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UTILIZATION OF ENZYME-CONTAINING PRODUCTS OBTAINED FROM FISH WASTE IN LEATHER PRODUCTION PROCESSES

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Abstract: *The properties of enzyme-containing product obtained from fish-processing waste were studied. High enzymatic activity and structural features e.g. compatibility with collagen, as well as the results of organoleptic evaluation and analysis of biogenic fibrous material in the form of semi-finished leather products (pelt) after modification by this product indicate its possible use in biotechnological processes of leather production.*

Keywords: *fish waste, enzyme-containing product, leather production, biogenic fibrous material, modification, structure, properties.*

1 INTRODUCTION

Nowadays the main trends in the development of food, light and other various branches of industry are the production of competitive products along with the rational use of material and energy resources, as well as the reduction of the harmful pressure on the environment. The ways of implementation of these trends are the improvement of technological processes through the development and introduction of more sustainable processing methods of effective, hazardous chemical materials. Such materials include enzymes - specific compounds which due to their protein nature and catalytic action are becoming more popular due to their positive effects on the course of technological processes, the quality of products, and their ability to ensure the respect of the resource conservation principles and environmental protection. Enzymes have already been used for genuine leather production in various forms: from saliva, manure, pancreas, in ancient and outdated methods of past years to the new generation drugs synthesized in modern chemical and bio-industries [1-5]. Despite the undeniable advantages of these materials, the lacking of Ukrainian supplies along with the presence of expensive foreign-made products is considered to be the biggest challenge today. Therefore, the search for new promising sources and forward ways to produce affordable and effective domestic product is as relevant as essential as ever.

A number of studies conducted in the past several decades [6-11] revealed that during the fish processing a significant amount of waste is formed, which contains many valuable components *e.g.* minerals, proteins, fats, enzymes. As a result, fish waste has found its applications in animal feed, food for healthy nutrition, biodiesel/biogas, natural pigments, food packaging, cosmetics, enzymes purification, Cr immobilization, soil fertilization and food moisture maintenance [8].

While studying the mass composition of pond fish that dwell the Volga-Caspian basin (grass carps, carps, silver carps), it was found that the bulk of the fish wastes consist of bone tissue (about 62%) and internal organs (about 21%). Taking into consideration the high collagen content in the bone waste (up to 40% of the total amount of protein), it is advisable to obtain from them first structure-forming compounds, and later, due to the higher minerals content (25% of the total chemical composition), mineral supplements. The main part of the internal organs of pond fish is represented by the liver, intestines, swimbladder and roe (milt). The analysis of the pond fish entrails chemical composition revealed a high protein content in the intestines and swimbladder (11-28%), therefore it is desirable to use them to obtain protein products. However, the proteins of the intestine and liver, which include the tissues of the pancreas and characterized by the presence of various enzymes, is advisable to use for enzyme manufacturing. Due to the fact that the intestine of silver carp is characterized by a high fat content (20%), it is proposed to use it for biofuels and lipolytic enzymes manufacturing [9].

Enzymatic methods became an integral part of the modern food and feed industry processes namely the production of a wide range of products for human and animal consumption. Since a huge amount of diverse genetic material in the aquatic environment is present it is recognized as a huge enzymes resource. In recent years, the enzymes from fish and aquatic invertebrates have been isolated and their characteristics have been determined. Moreover, several interesting applications related to marine enzymes in the food industry have been obtained. In a review of Canadian scientists [10] the current information on the digestive and muscle enzymes of fish and aquatic invertebrates was summarized, as well as the important advances in the use of marine enzymes in food was reported.

According to Arvanitoyannis and Kassaveti [8], fish waste should be recycled to obtain valuable enzymes that are beneficial to the world, and not improperly disposed, causing environmental pollution. It is useful to use fish waste as fish silage (a liquid product made from waste with the addition of acid or, less frequently, alkali), fishmeal for livestock farming, pharmaceuticals etc.

To convert fish waste into useful products, a group of Nigerian scientists used three sources (*Pseudomonas fluorescens, Enterobacter cloacae, and Bacillus megaterium*) for protease production. The most effective activity was detected at a temperature of 45°C and pH 9, thus promoting the fish stock decomposition. The protease enzymes can be used in the industry of baby food production, laundry detergents manufacturing, as well as in medicine to control blood clotting and pathogenic proteins decomposition [11].

The review of Ukrainian fish market in 2019 [12] as an optimal scenario for 2020 has maintained the fish and seafood imports within 400 thousand tons while increasing the total value of goods that are imported by expanding the range of imported fish products; the production volumes of own aquatic bioresources will be remained within the limits of 90-100 thousand tons. At the same time, an increase in fish products exports by 10-15% was expected due to the opening of new markets and an increase in shipping through the already existing contracts. The data from the State Statistics Service [13] showed that in 2020 some 76.5 thousand tons of own aquatic bioresources was extracted, 48.2 tons of which is fish. Although this result is lower than expected, it reveals a real source of fish waste generator in the country which could be used to produce secondary material resources for different industries.

In view of these facts, the aim of the present research was to study the properties of enzyme-containing product from fish-processing industry waste to identify its possible use as a secondary material resource in the production of genuine leather. For this purpose, the following objectives have been pursued:

- to perform the analysis of the product from fish-processing industry waste;
- to determine the enzymatic activity of the product;
- to find out the structural features of this product;
- to determine the suitability of the enzyme-containing product for the modification of biogenic fibrous material in the form of semi-finished leather products (pelt).

2 MATERIALS AND METHODS

The enzyme-containing product (ECP) was obtained from the waste of *Oncorhynchus mykiss*. ECP is a brown fine-fibrous substance that is readily soluble in warm water. Both common and advanced techniques - chromatographic, spectrophotometric, electrophoretic, infrared spectroscopy - were used to study the obtained product. The frozen mass of fish processing waste was suspended in the extraction buffer (0.1 M Na-phosphate (pH 7.0), containing 0.15 M NaCl, 1.5 mM ethylenediamine tetraacetic acid (EDTA), PEG-6000, and 0.1% Triton X-100) at the ratio 1:3, w/v and stirred continuously at 4°C for 1 hour. After that, the sample was centrifuged (Allegra 64 R Centrifuge, Beckman Coulter, USA) at 10 000 g for 30 min at 4°C.

The supernatant was purified by size exclusion chromatography on a Sephadex G25 column (GE Healthcare). After that, the supernatant was lyophilized (Telstar LyoQuest) and stored at 4°C until use. The lyophilized samples (50 $mg.mL^{-1}$) were dissolved in 10 mM Tris-HCl (pH 8.0) containing 5 mM $CaCl_2$. After centrifugation at 10.000 g for 5 min, the supernatant was loaded to a benzamidine sepharose column (flow rate of 180 mL per hour), which was pre-equilibrated with the same buffer. The bound fraction was eluted with 50 mM glycine (pH 3.0) containing 5 mM $CaCl_2$, and 1 M NaCl at a flow rate of 180 mL per hour. The obtained fraction was immediately neutralized to pH 7.4 to prevent loss of enzyme activity. Then the fraction was subjected to size exclusion chromatography on Sephadex G25 column pre-equilibrated with distilled water buffered with NaOH to pH 8.0. The fraction was loaded and the peak was collected at a flow rate of 45 mL per hour. The obtained fraction was lyophilized and used as the enzyme-containing product for further research.

The ECP was analyzed by sodium dodecyl sulfate polyacrylamide gel electrophoresis (SDS-PAGE) as reported [14] using 4% (w/v) stacking gel and 15% (w/v) separating gel. SDS-PAGE was performed using Mini-Protean Tetra System (Bio-Rad, USA) at 19 mA for stacking and 36 mA for separating gels. The samples were prepared by mixing with sample buffer (0.005 M Tris-HCl, pH 8.8, 2% SDS, 5% sucrose, and 0.02% bromophenol blue) at the ratio

of 1:1 (v/v). The samples were heated at 95°C for 1 min before loading in the gel. The total amount of proteins applied per well of gel was 20 μg. The gels were stained with 2.5% Coomassie brilliant blue R-250 in 10% (v/v) ethanol, 10% (v/v) acetic acid, 15% (v/v) isopropanol and the background of the gel was destained with 7% (v/v) acetic acid for 30 min. Apparent molecular weights of proteins were estimated using a protein calibration mixture (Bio-Rad, USA).

Zymography was carried out according to the method [15]. The separating gel solution (15%) was polymerized in the presence of fibrinogen (1 mg·mL^{-1}). The samples were not heated before loading in the gel. After electrophoresis, the gels were soaked in 2.5% Triton X-100 solution with shaking (30 min at 25°C) to remove SDS and renaturation of proteins. The gels were washed with distilled water for 10 min to remove Triton X-100 and then were incubated in 50 mM Tris-HCl (pH 7.5) at 37°C for 12 h. The digested bands were visualized as the unstained areas on the dark background of the gel. TotalLab 2.04 program was used to analyze the obtained electropherograms and zymograms. The represented electropherogram and zymogram are typical for the series of the repeated experiments (at least three in each series).

The enzymatic activity of ECP was analyzed by two different methods - using a chromogenic substrate Phe-Pip-Arg-pNA and by determining the caseinolytic activity. The activity against Phe-Pip-Arg-pNA was measured as described [16]. The reaction mixture consisted of 50 mM Tris-HCl (pH 9.0) and a sample of ECP (20 μg of total protein). The reaction was initiated by the addition of Phe-Pip-Arg-pNA (Renam, RF) (0.3 mM). The production of p-nitroaniline was monitored at regular intervals at 405 nm. The amount of p-nitroaniline realized from the substrate was calculated using a molar extinction coefficient of 10.000 M-1 x cm^{-1} for free p-nitroaniline. The caseinolytic activity of ECP was measured using casein as a substrate according to the method [17]. Casein (2%) in 50 mM Tris-HCl buffer (pH 7.4) containing 0.13 M NaCl was incubated in the presence of the sample of ECP (50 μg of total protein) at 37°C for 30 min. The reaction was stopped by the addition of trichloroacetic acid (7%) and the sample was kept for 15 min at 4°C. Then the sample was centrifuged at 15.000 g for 30 min. The absorbance of the supernatant was measured spectrophotometrically (SmartSpecPlus, Bio-Rad, USA) at 280 nm against the blank in which the test sample was replaced with an equal volume of 50 mM Tris-HCl buffer (pH 7.4) containing 0.13 M NaCl. The concentration of proteins in the ECP was determined by the method of Bradford [18], using bovine serum albumin as a standard.

Infrared spectroscopy was exploited to study the structural features of the ECP. IR-spectra were recorded in transmission mode by FT-IR spectrometer Tensor-37 (Bruker, Germany). The lyophilized samples and spectrally pure potassium bromide were pressed into thin pellets at a ratio of 2:100. The obtained spectrogram was processed using the baseline and internal standard methods; the interpretation of individual groups and bonds was performed according to [19-21].

The technological properties of ECP were studied taking into account the results of the use of ECP in the amount of 0.20, 0.35, 0.50 and 0.65% during modifying (softening) biogenic fibrous material in the form of a pelt. To exclude the influence of topographic areas of the skins, the experimental groups were composed using the method of asymmetric fringe [22]. Each group consisted of three samples. The control group #1 contained an unmodified limed hide. The modifications of groups #2-6 were carried out according to the standard method of the production of natural leather. The samples of group #2 were treated with 0.35% industrial enzyme preparation Enzymas 1072 while the samples of groups #3-6 - 0.20-0.65% ECP.

The effect of ECP consumption on the properties of the pelt was determined based on organoleptic assessment, welding temperature, moisture content, mass fraction of nitrogen, and gelatin melting. Sample preparation was carried out in accordance with ISO 2418. The welding temperature was determined as the temperature at which the pelt shrinks when heated in water in accordance with the requirements of DSTU 3177: Determination of welding temperature. The moisture content in the pelt was determined gravimetrically at a temperature of 100-105°C in accordance with ISO 4684. The mass fraction of nitrogen was determined by the Kjeldahl method. The Kjeldahl method was used to assess the state of a skin semi-finished product before and after its treatment with enzymes. The analysis was carried out in accordance with the requirements of DSTU ISO 5397.

Gelatin melting was estimated according to the protocol [22, 23]. The concentration of gelatin was determined using a calibration curve built on the basis of the results of determining the optical density of gelatin solutions of various concentrations (from 0.10 to 0.75 mg/mL).

3 RESULTS AND DISCUSSION

A product in the form of a brown fine fibrous substance soluble in warm water and containing various proteolytic enzymes was obtained by the primary sorption and further lyophilization of fish processing wastes. To purify the fraction of trypsin-like enzymes, which constitute a significant part of the proteases of the feedstock, and to stabilize the obtained enzymes, the chromatography on a benzamidine-sepharose column followed by lyophilization was carried out (Figure 1).

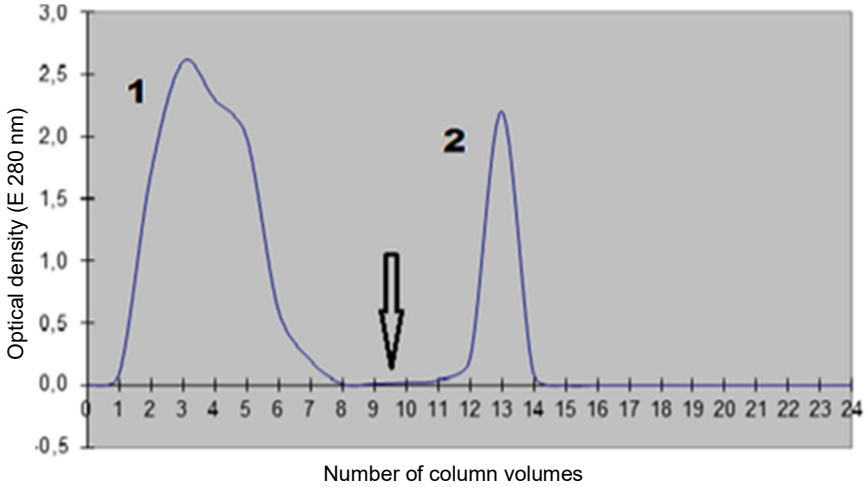

Figure 1 Chromatogram of the obtaining of ECP from the fish processing wastes a benzamidine-sepharose column: 1 - the fraction containing unbound proteins; 2 - the fraction of ECP the arrow indicates the point where the working buffer (10 mM Tris-HCl (pH 8.0) containing 5 mM $CaCl_2$) was replaced with the elution buffer (50 mM glycine (pH 3.0) containing 5 mM $CaCl_2$, and 1 M NaCl)

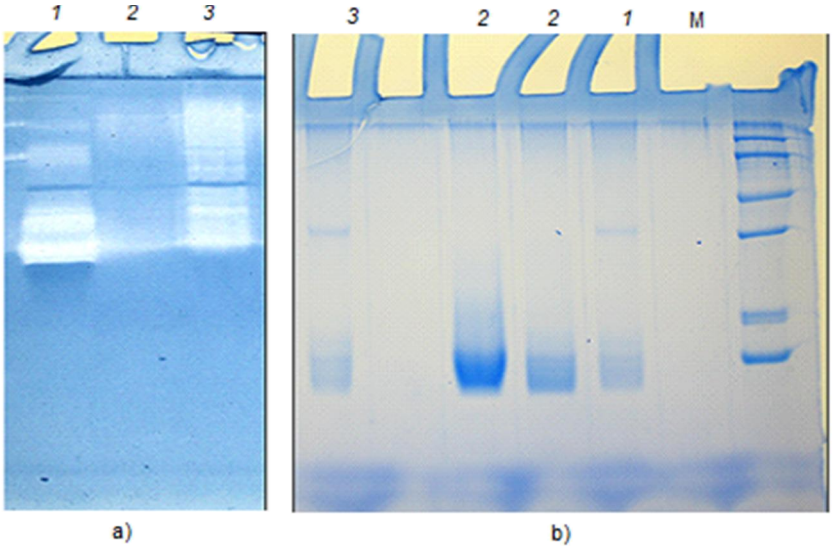

Figure 2 Substrate SDS-PAGE (a) and SDS-PAGE (b) analysis of ECP obtained by affinity chromatography on benzamidine-sepharose: 1 - starting material; 2 - the unbound fraction (10 and 20 μg of proteins per line); 3 - the fraction of ECP; M - molecular weight markers (97, 66, 45, 31, 21, 14 kDa)

The presence of active proteolytic enzymes in the fraction eluted from the benzamidine-sepharose column was confirmed by enzyme electrophoresis using fibrinogen as a substrate (Figure 2a). The result of the SDS-PAGE assay indicates the presence of several proteins in the fraction obtained after the affinity chromatography step: 60, 30, 21, 18 and 14 kDa (Figure 2b). Next, the enzymatic activity of ECP was evaluated using a specific chromogenic substrate Phe-Pip-Arg-pNA for serine proteases. According to the obtained data, the activity of ECP was 260 μmol $pNA.min^{-1}.mg^{-1}$ protein; the level of protein was 0.41 $mg.mg^{-1}$ extract. The caseinolytic activity of ECP was estimated to be 450 $IU \cdot g^{-1}$ product.

Since the treatment of biogenic fibrous leather materials during the production of natural leather is carried out in a wide range of temperature and pH, to which the enzymes are very sensitive, the influence of these factors on the enzymatic activity of ECP has been investigated. For this purpose, the enzymatic activity of ECP was estimated at temperatures of 25, 35, 45 and 55°C, and the range of pH from weakly acidic (pH 5.56) to basic (pH 12.45). As can be seen from Figure 3, the enzymatic activity of the ECP increased with increasing temperature. The maximum enzymatic activity was found at 55°C.

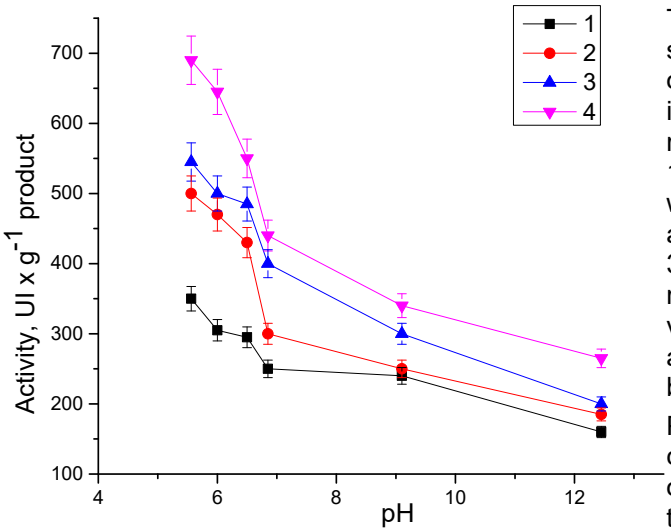

Figure 3 The influence of temperature and pH on the enzymatic activity of ECP: 1 - 25°C; 2 - 35°C; 3 - 45°C; 4 - 55°C

However, the increase in pH caused a decrease in the enzymatic activity of ECP. On the whole, it can be said that the enzymatic activity of ECP was more pronounced at pH of 6.0-7.0 and at a temperature not lower than 35°C. The obtained data indicate the possibility of using ECP in the leather industry, in particular in processes that occur at pH close to neutral and at increased temperatures, for example, during bating.

The compatibility of the materials used with the fibrous collagen, which is the major component of the skin, is important for the effective modification of biogenic objects such as animal skin. It is known from the theory and practice of leather production that not only the uniform diffusion of chemical material but also its fixation in the microstructure of the dermis, is important for achieving the necessary technological effect. This largely depends on the structure of the material used.

To identify the structural features of ECP, an IR spectroscopic study was carried out. The analysis of the spectrogram (Figure 4) revealed that the most intense absorption bands were in the frequency range from 3700 to 3100 cm^{-1} and from 1750 to 1000 cm^{-1}, while less intense bands of absorption were in the range from 900 to 400 cm^{-1}. The wide absorption band was observed in the range of 3700-3100 cm^{-1} with peaks at 3333 and 3505 cm^{-1}. These results indicate the presence of bonded and free valence NH-groups, bonded OH-groups, and amides, which involved in the formation of hydrogen bonds.

Peaks in the frequency range of 2857 and 2928 cm^{-1} correspond to symmetric and asymmetric valence oscillation of CH_2-groups. The oscillation in the range of 3100-3000 cm^{-1} with peak at 3068 cm^{-1} characterizes the valence and deformation vibrations of the N-H bond in the NH_3^+ group and valence vibrations of the CN-group of the Amide II overtone. The presence of peaks at 1750-1480, 1743, 1663, 1629 and 1527 cm^{-1} could be a result of the oscillation of NH-bonds (Amide I and Amide II). This suggests the valence oscillation of the bond (O=C-NH-), as well as β- and α-conformation of molecules. A significant band of absorption with a peak at 1234 cm^{-1} was observed in the frequency range of 1280-1000 cm^{-1}, which indicates the valence oscillation of CN-groups in Amide (Amide III) and deformation oscillation of the O-H bond. In the same region of the spectrum, there are bands at 1167-1064 cm^{-1}, which are typical for the valence oscillation of CN, C-O, and C=C groups and bonded NH groups. A wide band at the range of 850-450 cm^{-1} characterizes the valence oscillation of bounded NH-groups with rather intense peaks at 707 and 603 cm^{-1} (Amide V) and a less intense peak at 530 cm^{-1} (Amide VI). The obtained data show the multifunctional nature of ECP and its ability to interact with the functional groups of other chemicals, such as dermis collagen.

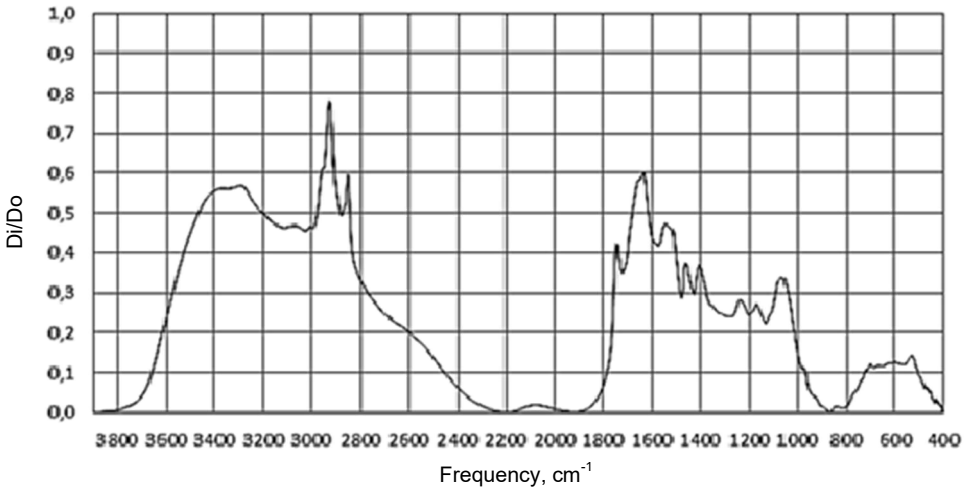

Figure 4 IR-spectrogram of ECP

Table 1 Effects of the modification conditions on the physicochemical properties of biogenic fibrous material (M±m; n=5)

Parameter	Group					
	*1**	*2***	*3*	*4*	*5*	*6*
	Product consumption [%]***					
	0	*0.35*	*0.20*	*0.35*	*0.50*	*0.65*
Welding temperature [°C]	60.00±3.0	61.00±3.0	56.00±2.8	58.00±2.8	59.00±3.0	59.00±3.0
Moisture content [%]	83.00±4.0	79.60±3.0	79.60±3.0	81.20±3.0	77.80±3.8	78.10±4.0
Mass fraction of nitrogen [%]	15.93±0.8	14.74±0.5	14.24±0.5	16.50±0.6	16.85±0.5	16.79±0.6
Gelatin melting [%]	10.95±0.5	14.40±0.6	15.40±0.5	23.60±1.0	26.20±1.2	25.20±1.2

*Note: *Starting material after liming, without modification (without bating); **Modification (bating) of the material with softening agent Enzymas 1072; ***by the weight of the sample.*

The next series of experiments was devoted to the study of the technological characteristics of the obtained enzyme-containing product. Given the results of our previous research, the modification of biogenic material in the form of a semi-finished leather product (pelt) from sheepskin during softening was investigated. According to the typical technique [24], softening is carried out at a pH close to the neutral value and a temperature of 36-38°C. The experiment was performed according to the conditions shown in Table 1. No difficulties were met in processing the samples of experimental groups #3-6; the treated samples were soft; they had a clean front surface and an imprint retained when pressed on the front surface. The samples of group #5 (ECP consumption was 0.50% of the sample weight) were the best in the terms of organoleptic evaluation. The face surfaces of the samples were more plastic and silkier.

As can be seen from Table 1, an increase in the ECP consumption in groups #3-6 from 0.20 to 0.65% does not significantly affect the hydrothermal stability of the samples. However, an increase in the mass fraction of nitrogen and melting of gelatin was observed. In determining the melting of gelatin, the color and the intensity of the solution in flasks differed significantly (Figure 5).

Figure 5 Flasks containing analytical reagents for spectrophotometric evaluation

The increase in the yield of gelatin could be explained by the removal of interfiber protein residues from the structure of biogenic material under the influence of ECP; the increase in the softness, the plasticity of the skin - due to the peptization of non-collagen components, and loosening of the dermis structure as the result of removal of residues of microfibrillar proteins; the increase in nitrogen levels - due to the formation of a certain number of nitrogen-containing groups as a result of enzymatic modification of proteins in the dermis.

4 CONCLUSIONS

The analysis of scientific and technical literature revealed the need for an affordable and effective domestic enzyme-containing remedy for biotechnological processes of leather production. The additional source for the manufacture of such preparations may be fish-processing waste that is rich in minerals, proteins, fats, enzymes, and other biologically active compounds. The enzyme-containing product ECP was hence obtained from the fish-processing waste in the form of a finely fibrous substance of brown color, which is readily soluble in warm water. Both common and advanced techniques - chromatographic, spectrophotometric, electrophoretic, infrared spectroscopy - were used to study the obtained product.

Electrophoretic fractionation of the product showed the presence of enzymes with a molecular weight in the range from 93 to 14 kDa. High proteolytic activity of enzymes was identified, which, in a recalculation on a specific chromogenic substrate, was 260 μmol pNA·min^{-1}.mg^{-1} protein. The high activity of the studied object was also confirmed by the estimation of caseinolytic activity (450 IU.g^{-1} product). According to the Bradford method, which is based on the ability of proteins to bind to Coomassie Brilliant Blue G-250, it was identified that the protein content in 1 mg of the extract is 0.41 mg.

The results of infrared spectroscopy showed the multifunctional nature of the product with the presence in Bradford the structure of various groups and bonds: bounded and free NH-groups, bounded OH-groups, and amides, which take part in the formation of hydrogen bonds; NH-bond in the NH_3^+ group, CN-group of the Amide II overtone, as well as NH-bonds of Amide I and Amide II, which provides an argument to speak about the valence fluctuations of the -CO-NH- bond, as well as the presence of β- and α- conformations of enzyme molecules.

While studying the effect of temperature and pH on the enzymatic activity of the product, it was found that the increase in the temperature for every 5°C results in the increase of enzymatic activity by 30-50 units; the pH factor acted in the opposite direction. Generally speaking, the highest enzymatic activity of the product was recorded in the pH range of 6-7 and at a temperature of at least 35°C. The data allowed to predict the possibility of using the product in the processes that will take place at a pH close to neutral and at elevated temperatures.

After modification of biogenic fibrous material in the form of semi-finished leather products (pelt) with an enzyme-containing product at the stage of bating, the semi-finished product became soft-touch, flexible, and had a clean front surface. The best result was achieved while using 0.50% ECP when a deeper gelatin melting (26.21%), a higher total nitrogen content (16.85%), and a better organoleptic evaluation were applied, compared to the results of modification with an industrial enzyme remedy at consumption of 0.35% by weight of samples, when the organoleptic assessment and the same indicators were worse (14.40 and 14.72%, respectively).

This effect can be explained by the ability of enzymes contained in the product obtained from fish-processing waste to loosen the structure of the dermis and remove interfiber proteins from it. The results indicate the potential technological capabilities of this product and the feasibility of further research in the chosen direction.

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INVESTIGATION OF THE TENSILE PROPERTIES OF BIFURCATED BRAIDS

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Abstract: *The modern 3D braiding machines with controllable switches can produce different braid structures like tubular, core and sheath, some flat and 3D braids. One of its interesting natures is its ability to produce bifurcated structures, which are demanded in different technological areas including medical applications. The aim of this paper is to study the physical properties (tensile strength, stiffness, elongation and mass per unit length) of bifurcated braids, produced with variable braiding angle combinations. Using cotton and polyester yarns, bifurcated braids with 30° and 45° braiding angle combinations, were braided using Herzog VF braiding machine and the properties of bifurcates and unified parts were tested and analysed.*

Keywords*: Bifurcation braiding, tensile strength, modulus of elasticity.*

1 INTRODUCTION

Braided structures have large and wide applications as candles, ropes, high performance fibre cover of mandrels for production of composites, reinforcements, medical applications, etc. The applications where the axial force transmission is important, such as marine and climbing ropes require loops, slices or special end fasteners in order to connect them to the remaining infrastructure. The modern braiding machines with controlled switches allow production of closed loops, produced almost continuously on the same machine placing bifurcation (splitting of the braid into two regions) and then back together into one. This possibility was reported already few times, but there is no available investigation about the mechanical properties (strength) of the bifurcated area. This work has the purpose to investigate the mechanical properties of such areas for one type of material.

In the braided products yarns are interlacing the product axis under angle *α*, named braiding angle. It can be between 1° and 89°, but usually is chosen in the range of 30°- 80° and represents the most important geometrical parameter of braided structures [1]. According to Chiu [2], braids with lower braiding angle have higher specific energy absorption. Tensile properties of braids are depending on the braiding angle as review papers and detailed investigations demonstrate [3-5]. For larger elongations braided structures exhibit nonlinear responses [6]. Additionally to the braiding angle, which is depending on the take up speed, the braid pattern influences the mechanical properties of the braids too [7]. The behavior of important characteristic points of the complete curve of the force - elongation diagram with prediction of breaking force is a complex task, discussed in [8]. As is shown in the figure below (Figure 1b), the behavior of stress elongation curve at different regions of the curve are explained. The region 0 to A corresponds to the load from relaxed state up to the first jamming state between the yarns in which the braiding angle starts to decrease and the yarns only change their orientation to each other. At point A, the yarns touch each other - and this position can be calculated based on geometry relations as the first jamming state [17]. At the region between A to B, there is minimal movement of the yarn axes which is only caused by the change of the yarn cross-section based on increased lateral compression and bending. The region B to C is characterized with predominant tensile loading of the material. Considering the orientation of the yarns (Figure 1a), the maximal force that the rope achieves will be less than the strength of all constituent yarns. In simplified way the strength can be calculated as:

$$P = F_{yarn} \cdot N \cdot cos\alpha \quad (1)$$

where: F_{yarn} is strength of the yarn, N is number of yarns in the braid and α is the braiding angle [8].

This equation does not consider the loss of strength based on the crimp of the yarns, but it provides evaluation of the maximal strength of one braided part at given angle. The real value will be always smaller than this evaluation because the loop strength of the yarns in crimped state is lower than the strength in straight position.

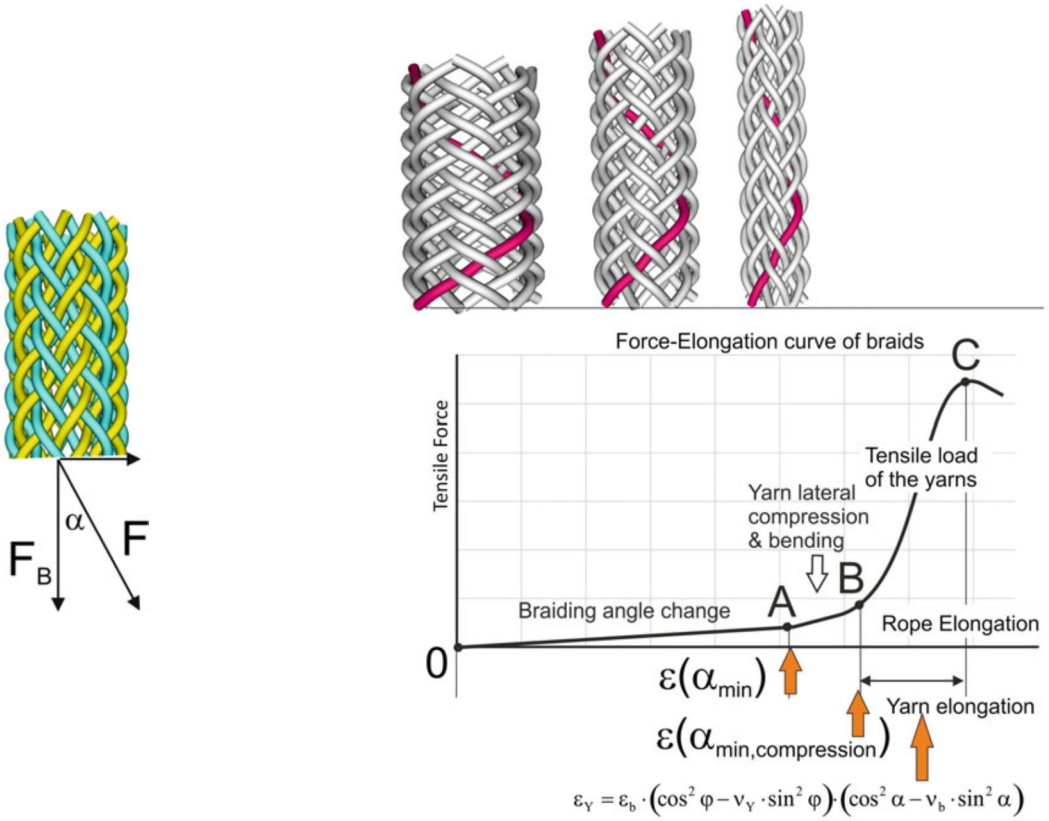


Figure 1 Contribution of the yarn to the braid strength (a) and characteristic points of a force elongation diagram of a biaxial braid with equation between the braid and yarn elongation (b) [8]

Based on these considerations and the equation (1) it can be expected that the braids with lower braiding angle will have higher strength. The yarns in a braid with lower braiding angle will have less freedom to move, so the region between 0 and A will be shorter and, in this way, the total elongation at break will be lower too, but still significantly higher than the elongation of the single yarns. For specific industrial braids, researchers prove that it can be up to at least four times that of the yarn [9]. As result, the elasticity modulus of braids with lower braiding angle is expected to be higher. More detailed analysis about influence of the braiding angle and the tensile and bending properties for different braid types and materials is presented in several works, as for example [10], or for composites [11]. The braids with bifurcations have different regions with different braids and their joint behavior is not enough investigated yet. There are only a few works related to bifurcated braids that are limited mainly to the specific applications specially in composites [12], or the medical area [13, 14] and do not cover investigation of the mechanical properties in the bifurcated areas. This work presents theoretical considerations and experimental investigation of the strength and elongation of braids with bifurcations for two materials. Considering the equation (1) can be seen that the strength of the braids is proportional to the number of single yarns but decreases with the increase of the braiding angle.

2 EXPERIMENTAL SETUP

The experiments are performed on the variation braiding machine VF of company Herzog GmbH, Oldenburg, Germany. It consists of a 4×4 arrangement of four slots horn-gears. Between the transfer points of the horn-gears, the travelling tracks for the bobbins are equipped with pneumatically triggered switches which allow the track to be changed multiple times during the production [1, 15].

This allows braiding of one larger tubular braid with all carriers and after that splitting them into two groups into separated areas of the plate. Using this technique, it is possible to produce basic braids, splitting up in to multi bifurcated braids and consolidating back into a basic braid, using only one braiding machine, just by changing the pattern of the track plate. The braiding machine can produce different structural braids like 3D structure, tubular, flat and core and sheath structural products [16, 17].

The materials used for this experiment are cotton and polyester yarns. The cotton yarn is a ply yarn of 4 single yarns having 37 Nm count.

The count of the ply one is 9.25 Nm. The polyester has 1100 dtex (9.091 Nm) count (Table 1). Both have almost the same count while the cotton has slightly higher count with 0.16 Nm. This shows that the cotton sample was to some extent finer.

Table 1 Used materials

Materials	Fineness
Cotton	27 tex x 4 = 108 tex (37 Nm) = 9.25 Nm (4 ply)
Polyester	1100 dtex (9.091 Nm)

Using the 4x4 VF braiding machine, bifurcated products with 30°and 45° braid angle combinations were produced. 16 carriers were used for the production. All the 16 carriers crossed each other to produce the unified part of the product and for the bifurcates, the carries are divided into two groups of 8 carriers and produce two braids separately which will be combined again after some steps. With 30° and 45° angle combinations, 4 different braids of both polyester and cotton products were produced (Table 2).

Table 2 Angle combination of the structure

Samples	Angle combination	
	bifurcate	unified
1	30°	30°
2	30°	45°
3	45°	45°
4	45°	30°

The actual braiding angles of the products were measured with ImageJ software. Each sample has both, unified and bifurcated parts one after the other (example in Figure 2).

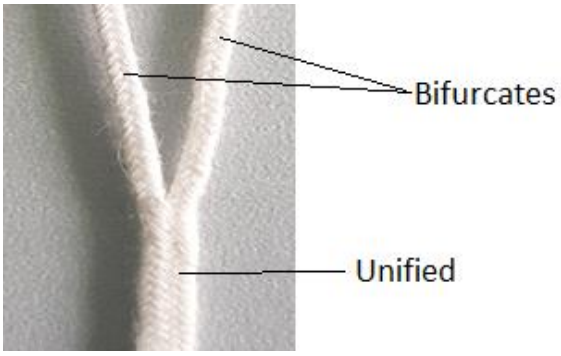


Figure 2 The braided structure of cotton sample

The input data for the machine to produce these specific angles with required part length is given in the Table 3 and images of the prepared braids are presented in Figure 4. The configuration steps of the machine for production of the bifurcated structures are presented in Figure 3 and consists of several steps: Pattern 1 - crossing loop for 1x16 to 2x8 to 1x16 geometry. Steps: 1 - start position; 2 - change crossings, 3 - move forward, 4 - change crossings: end position; 5 - start position, 6 - change crossings, 7 - move forward; 8 - change crossings [18].

Table 3 The input data given to the machine

Braiding angle	Cotton				Polyester			
	Lay length [mm]		Part length [mm]		Lay length [mm]		Part length [mm]	
bif/uni	bifurcated	unified	bifurcated	unified	bifurcated	unified	bifurcated	unified
30°30°	20	25	90	120	20	25	90	150
30°/45°	20	14	90	100	20	14	90	100
45°45°	8	14	50	100	8	14	50	100
45°30°	8	25	50	150	8	25	50	170

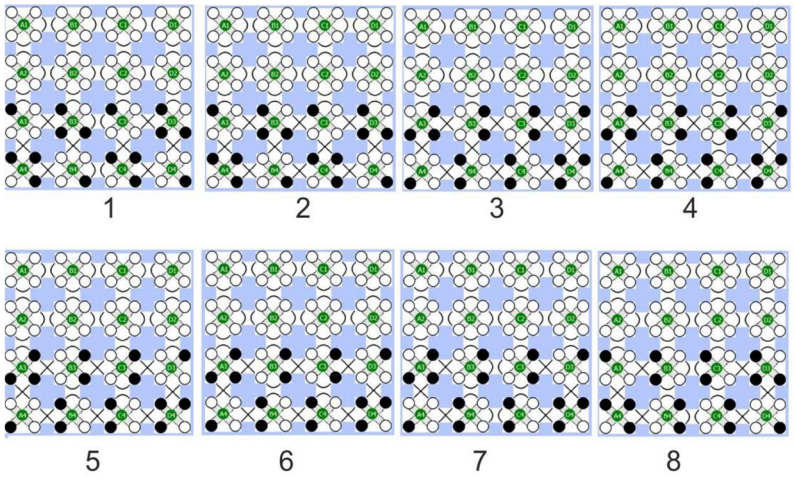


Figure 3 Configuration and crossing steps during braiding

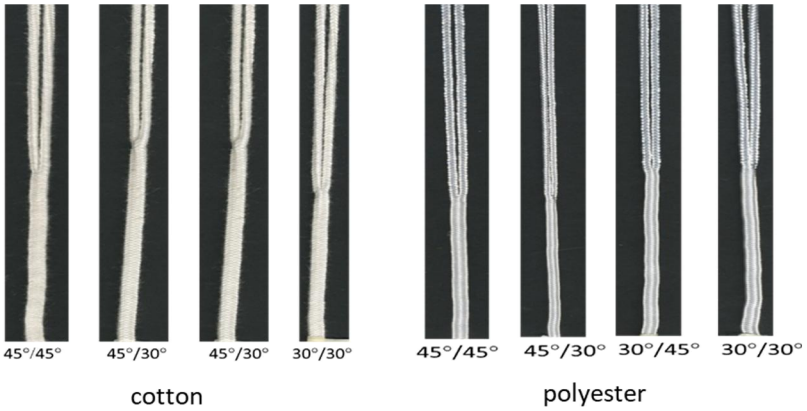


Figure 4 Bifurcated products of cotton and polyester yarns with different angle combinations

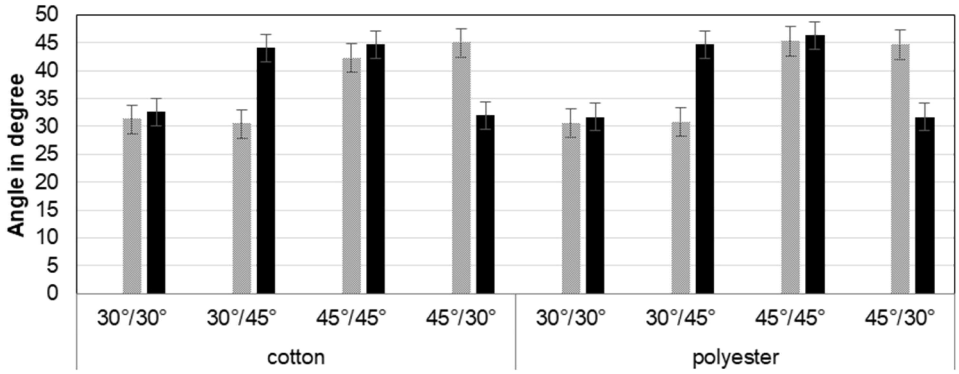


Figure 5 Braiding angles of the bifurcated products as measured by ImageJ software

In addition to these braiding steps, an additional step is included for the turning point from 45° - part length to the next part length. In this region a height correction of the braiding point is required. When 45° part is produced, the take-off velocity is lower and the braiding point moves at lower position, closer to the top of carriers. During the rearrangement of the carriers for the next pattern, some more yarn length is taken from the bobbins. In order to keep these yarns under similar tension for the next process, it is efficient to move the complete braids up taking some length off. For this process a separated programming step in the machine control is required.

The real braiding angle of the different areas of the samples was measured with ImageJ software as shown in the Figure 5. In the angle combination representation, a/b: a shows the angle of bifurcate part and b the angle of unified one. For instance, 30°/45° shows that the theoretical angle of bifurcates part is 30° and the angle of unified part is 45°.

After measuring the angles of the samples, the tensile strength, the modulus of elasticity and mass in one-meter length of each sample were measured with tensile strength and weight balance machines. From the tensile testing data and force versus strain graph the modulus of elasticity is calculated. By using the Hook's law, equations for parts which contain two parts were derived and modulus of elasticity was calculated using the formulae for comparison with the result from the load vs strain graph of the measured value.

Tests were made for the single bifurcated braid separately, two twin bifurcates together, the unified braid parts and combination of the twin bifurcates with the unified part which can represent the strength of the whole product (Figure 6).

The tensile testing was performed in test standard based on DIN EN ISO 2062 (with 20 kN measuring head) with Zwick 1455 tester. As shown in the figure below, samples with 10 cm length were taken for all four types of products to test their tensile strength.

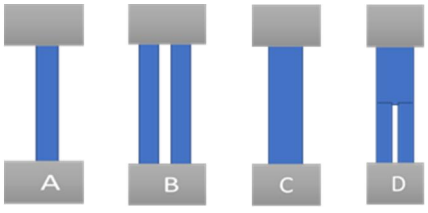


Figure 6 Tensile testing sample structures
A - bifurcated single sample, B - twin bifurcates together, C - unified, D - unified and bifurcates together

2.1 Elasticity of parallel braids in the bifurcated area

According to Carey [19] longitudinal elastic modulus of a braided tube is less than that of one composed of laminated layers. Considering Hook's law equation, the equivalent stiffness of the system can be determined [20-22].

$$F = k \cdot x \tag{2}$$

where: *F* is tensile or compressive force in N, *k* is spring stiffness (spring constant) (in N per m deflection) and *x* is the spring deflection in m.

For the bifurcated parts the principle of parallel and series springs can be used. The equivalent stiffness for n springs in parallel is obtained simply by adding together all the individual stiffnesses:

$$k_{eq} = k_1 + k_2 + k_3 \ldots \ldots + k_n \tag{3}$$

In case of series springs the reciprocal of the equivalent stiffness (spring constant) is equal to the sum of the reciprocals of stiffnesses of the springs:

$$\frac{\perp}{k_{eq}} = \frac{1}{k_1} + \frac{1}{k_2} \ldots + \frac{1}{k_n} \tag{4}$$

Therefore, the stiffness of the samples consisting of two bifurcates together and unified with bifurcates should be the equivalent values of the components.

As shown in Figure 5, sample B and sample D have two components.

Sample B has parallel arrangement and the equivalent stiffness value is double value of sample A.

$$k_b = k_a + k_a = 2k_a \tag{5}$$

where: k_b [N/m] is stiffness for sample B and k_a [N/m] is stiffness for sample A.

But sample D has both, parallel and series arrangements. The stiffness of sample D is the equivalent stiffness of sample A and sample B since it contains both parts together.

$$\frac{1}{k_d} = \frac{1}{k_c} + \frac{1}{k_b} \tag{6}$$

Substituting $2k_a$ for k_b:

$$\frac{1}{k_d} = \frac{1}{k_c} + \frac{1}{2k_a}$$

$$k_d = \frac{2k_a k_c}{2k_a + k_c} \tag{7}$$

where: k_a, k_b, k_c and k_d are in N/m.

The spring constants are valid for a spring with defined length L_0. If the spring deflection gets expressed as elongation, the equivalent to elasticity modulus will be obtained. Connection between the spring constant *k* at given length L_0 and the elasticity modulus *E* can be obtained after applying the expression of the stress σ and engineering stain ε trough the applied force *F* and cross section *A* of the investigated element (rope or rod) and elongation ΔL and initial length L_0.

$$E = \frac{\sigma}{\varepsilon} = \frac{\frac{F}{A}}{\frac{\Delta L}{L_0}} \tag{8}$$

$$k = \frac{F}{\Delta L} \tag{9}$$

$$E = \frac{\sigma}{\varepsilon} = \frac{\frac{k}{A}}{\frac{1}{L_0}} = \frac{k \cdot L_0}{A} \tag{10}$$

From the last equation can be concluded, that for a fixed initial length and cross section the elasticity modulus *E* and the spring constant *k* are proportional and the resulting elasticity modulus of single or bifurcated parts can be obtained using the same equations (4)-(7). From other point of view, the cross-section *A* cannot be determined efficiently for fibrous structures, for this reason instead of the engineering elasticity modulus, where the relation between the stress and strain is used, here the relation between the force and strain will be used:

$$E = \frac{\sigma}{\varepsilon} = \frac{\frac{k}{A}}{\frac{1}{L_0}} = \frac{k \cdot L_0}{A} \tag{11}$$

3 RESULTS AND DISCUSSION

3.1 Tensile strength

The load vs strain curves of the tensile strength tests are given in Figure 7 for cotton and in Figure 8 for polyester. Each diagram contains the load vs strain curves of 4 samples represented as A, B, C & D.

As shown in Figure 7, the single bifurcated samples (A) in cotton yarn have almost nearer values of tensile strength in both 30° and 45° angles. But the elongation increases in 45° cotton samples. In polyester single bifurcated samples, the tensile strength reduces and the elongation increases from 30° to 45°.

The result of the two bifurcates tested together (sample B), in cotton sample shows maximum strength and elongation at 45° of the 45°/30° combination. At the 45°/45° combination of cotton sample, the yarns of the 45° bifurcates started breakage in lower load than the same sample in 45°/30° bifurcations. But the last breakage takes higher load than the 45°/30° bifurcates. In polyester, the two bifurcates have maximum strength and elongation at 45°/30° combination and lower strength at 45°/45° combinations.

For the unified sample (C), maximum strength is recorded at 45°/45° combinations for cotton and at 45°/30° combinations for polyester. In both polyester and cotton samples, the unified samples have maximum elongation at 45°.

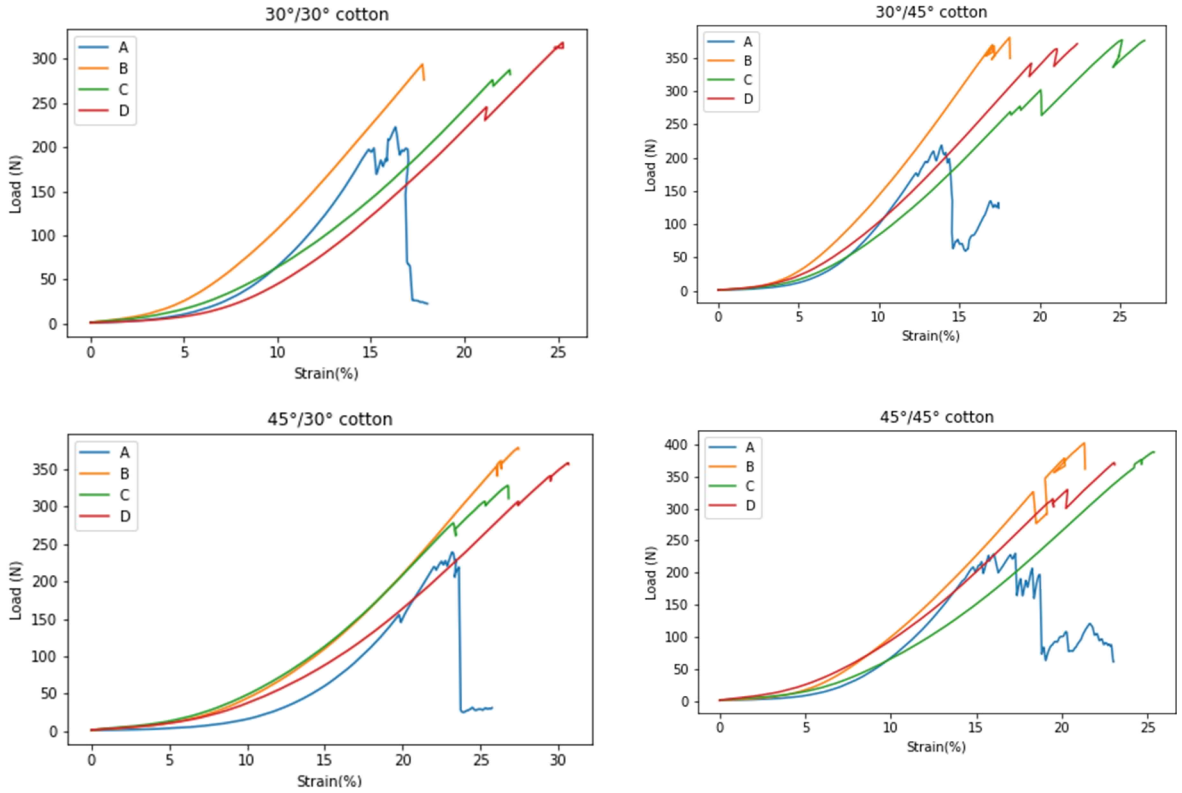


Figure 7 Load vs strain curves of cotton yarn samples with different angle combinations, A - bifurcate single, B - twin bifurcates (together), C - unified sample, D - unified and bifurcates together

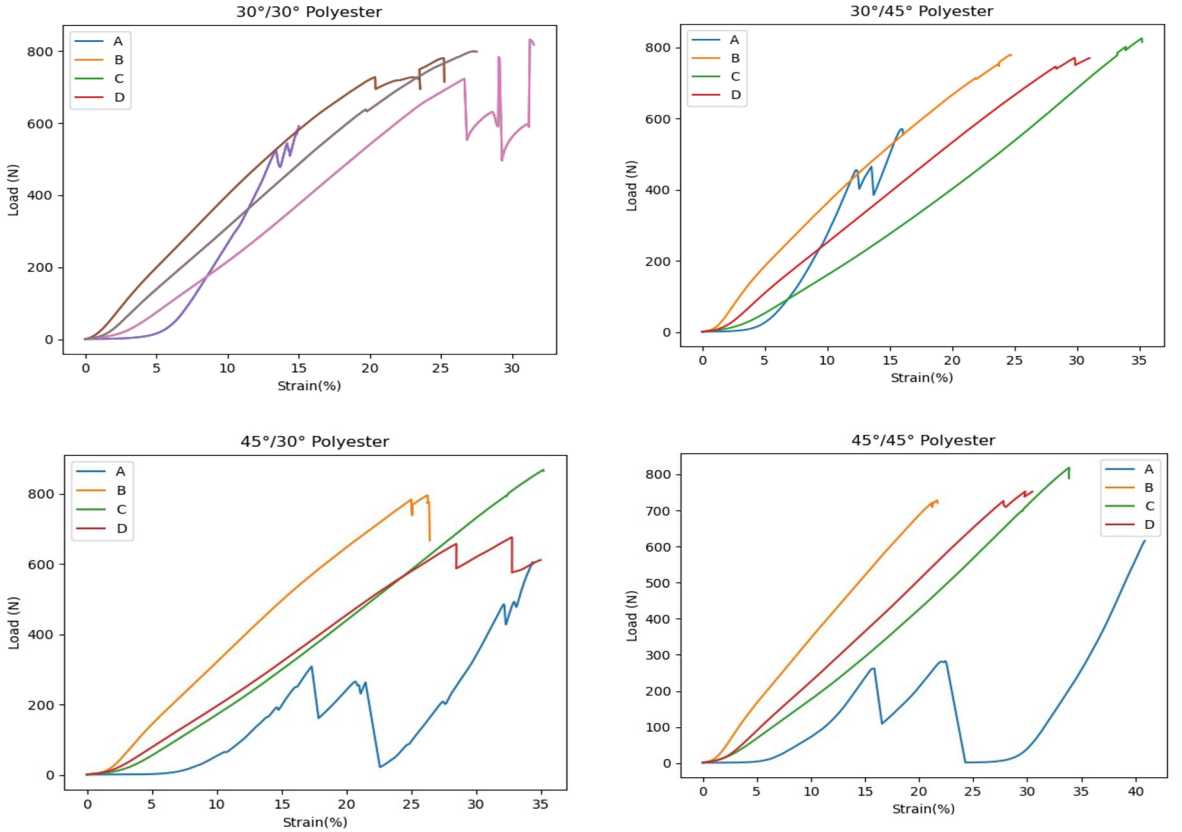


Figure 8 Load vs strain curves of polyester yarn samples with different angle combinations, A - bifurcate single, B - twin bifurcates (together), C - unified sample, D - unified and bifurcates together

The sample, which contains the unified and bifurcate parts together (D), represents the property of the material as a whole. In cotton samples, 45°/30° combination has the higher tensile strength and elongation. But in polyester the higher strength is recorded on 30°/30° combination.

In cotton sample, the strongest sample of the tests is sample B which is the strength of twin bifurcated parts. And the sample with least tensile strength is sample A, the single bifurcated part. But in case of polyester samples, the strongest part is sample C which is the unified part while sample A is the weakest like cotton sample.

Generally, the polyester sample meets the theoretical fact and results of previous works showing the reduction of strength and increment of elongation as the braiding angle increases from 30° to 45°. But in cotton samples, some contradicting results are seen. This is due to the influence of the angular combination and bifurcation braiding process. In addition to the braiding angle, the braiding mechanism has an influence on the tensile strength of the braids.

3.2 Modulus of elasticity

The modulus of elasticity can be calculated from the load vs strain diagram data [23].

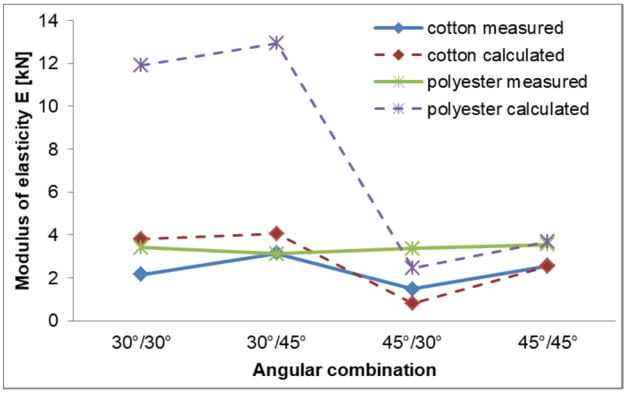
$$E = \frac{\Delta F}{\Delta \varepsilon} = \frac{F_2 - F_1}{\varepsilon_2 - \varepsilon_1} \tag{12}$$

$$k = \frac{E}{L_0} \tag{13}$$

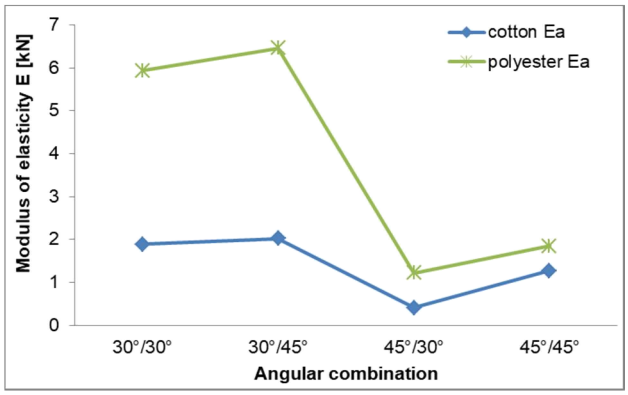
For samples B and D (Figure 5), the values of the modulus of elasticity (E_b, E_d) can also be calculated from the values of k_a and k_c using equations (5), (6) and (13): $k_b = k_a + k_a = 2k_a$

and for k_d: $k_d = \frac{2k_a k_c}{2k_a + k_c}$

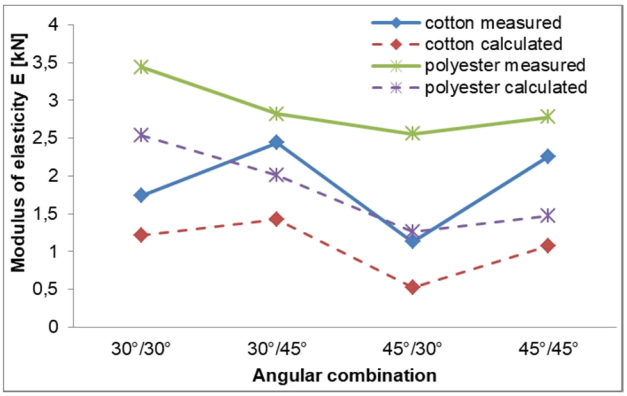
As shown in Figures 9b and 9d, the modulus of elasticity of the samples decreases in 45° than in 30°. In all the samples the 45°/30° combinations have lower modulus of elasticity. In this arrangement, the bifurcates are 45° braiding angle and the unified is 30°. On the other hand, the 30°/45° arrangement which contains 30° bifurcated part and 45° unified part, has the maximum amount of modulus of elasticity in both the samples. Comparing cotton and polyester (Figures 9b and 9d), the polyester sample has the higher modulus of elasticity than cotton sample. Almost all the values calculated by the principle of the spring constant in sample B (Figure 9a) are higher than the measured values (Table 4).



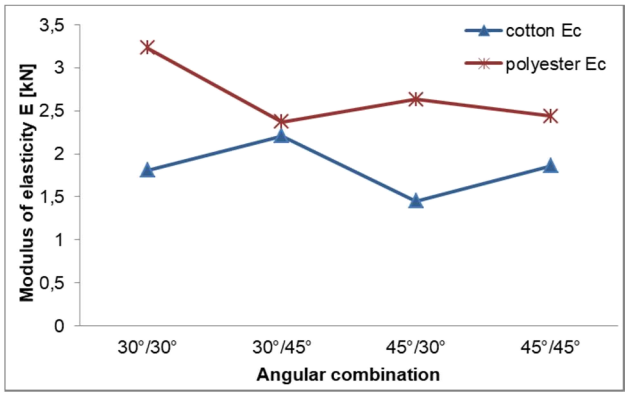
a) E_b measured and calculated for sample B



b) E_a for cotton and polyester



c) E_d measured and calculated for sample D



d) E_c for cotton and polyester

Figure 9 Modulus of elasticity of the samples

Table 4 Modulus of elasticity from the load vs strain graph and Hook's law calculations

Sample types		Modulus of elasticity E [kN]					
		E_a	E_b		E_c	E_d	
			measured	calculated		measured	calculated
Cotton	30°/30°	1.89	2.15	3.79	1.81	1.74	1.22
	30°/45°	2.02	3.13	4.04	2.21	2.44	1.43
	45°/30°	0.41	1.48	0.82	1.45	1.13	0.53
	45°/45°	1.27	2.57	2.55	1.86	2.26	1.07
Polyester	30°/30°	5.94	3.40	11.88	3.23	3.44	2.54
	30°/45°	6.46	3.12	12.93	2.37	2.82	2.01
	45°/30°	1.22	3.37	2.44	2.63	2.56	1.26
	45°/45°	1.85	3.54	3.69	2.44	2.78	1.47

On the other hand, the calculated values for sample D are less than the measured values. In other words, spring constant value for the parallel arrangement of samples, the calculated value is higher than the actual measured modulus of elasticity of the samples. On the contrary, in a sample which contains the combination of both parallel and series arrangement the calculated value for the samples is lower than the actual measured modulus of elasticity.

3.3 Mass in one-meter length of the samples

In each type of the sample, having similar braiding angle, the cotton sample has greater mass than the polyester one (Figure 10).

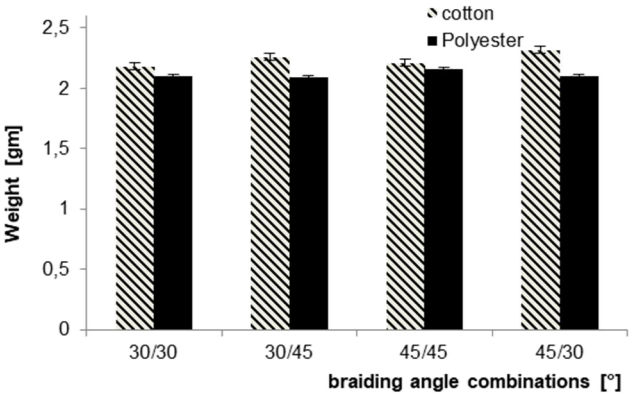


Figure 10 Weight of one-meter length for combined samples (bifurcated and unified) of cotton and polyester braids

The cotton yarn used was 0.16 Nm greater than the polyester yarn. That means cotton sample was slightly finer than the polyester yarn. But the mass of the braided samples shows that cotton braids have higher mass in unit length with the same braiding angle. This shows that because of the natural behavior of cotton, the yarns were compacted forming a crimp in the cotton samples. As it can be expected, the unified parts in both cases have higher mass in a unit length (Figure 11). 30° bifurcated cotton samples have shown higher mass than the 45° ones which shows the tension in 45° braiding reduced the length of crimp formed. As described before, the mass in gram of one-meter length in cotton is greater than in polyester due to the crimp formation and compactness property of cotton fibre.

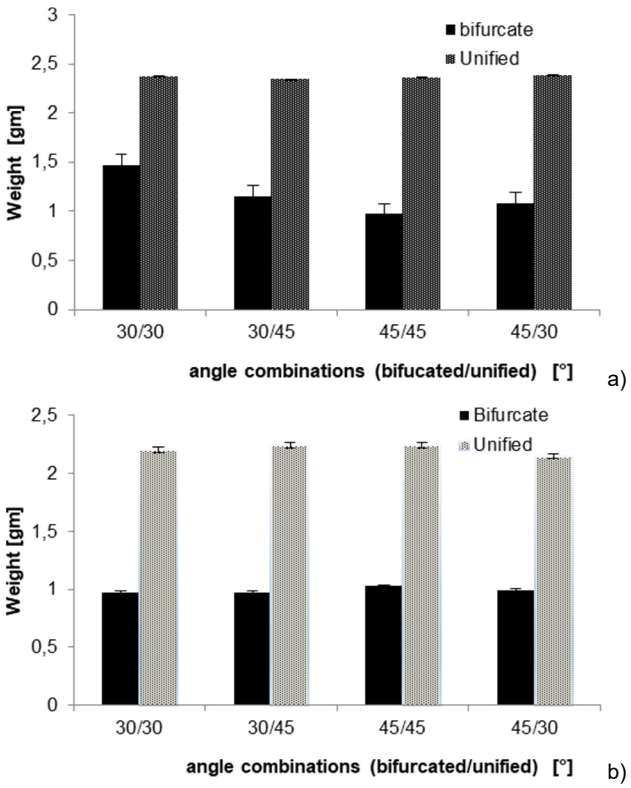


Figure 11 Weight in one-meter length of (a) bifurcated cotton and (b) polyester samples

4 CONCLUSION

In this experiment, bifurcated braid products were produced with different braiding angle combinations from two materials namely cotton and polyester. Tensile strength tests and measurement mass per unit length of the samples were made to identify the effect of braiding angles and materials on the bifurcated braids. From the tensile test, the modulus of elasticity of the samples were determined and the modulus of elasticity of samples consisting of branches in parallel or both parallel and series arrangement were compared with the values calculated by spring constant method.

Comparing the 30° and 45° braiding angle braids, in most tests, relatively higher tensile strength is measured in 30° braiding angles than the 45°, with some exceptions in cotton samples. In case of elongation, the higher the braiding angle, the higher is the elongation. The elongation at break is also higher for 45° than on the 30°. The tests in combination of angles show that samples with 30°/45° combination have higher modulus of elasticity and the 45°/30° angle combination has the lower modulus of elasticity.

Having similar braiding angle, polyester products have higher modulus of elasticity than the cotton samples. In addition to type of material and increment of braiding angle, the combination of the braiding in the bifurcation braiding, has an influence on the modulus of elasticity of the material. The mass in length value of cotton braid has been higher than that of polyester due to the compactness nature of cotton yarns. The weight in length of samples with 45° has in most tests higher value than the 30° samples.

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INFLUENCE OF MATERIAL COMPOSITION OF BLENDED YARNS CONTAINING PHOTOLUMINESCENT PP AND PA6 FIBRES ON THEIR COLOUR EFFICIENCY

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Abstract: *World`s leading companies in all industrial fields try to protect effectively their sophisticated products against counterfeiting. One of affordable and cost-effective access to originality protection of textile and clothing products is application of modified fibres containing photoluminescent pigments, which besides colour change emit also light under UV lamp. The contribution focuses on preparation of twisted yarns based on standard unmodified and modified polypropylene and polyamide filaments. The polypropylene and polyamide fibres incorporate organic photoluminescent pigment with a concentration of 0.01 wt.% and 0.10 wt.% in the fibre. Degree of efficiency of the photoluminescent pigment has been evaluated objectively on packages of blended yarns by change of colour expression defined by means of b* colour coordinate in the CIE LAB colour space and by optical expression of photoexcitation under UV lamp.*

Keywords: *Photoluminescency, photoluminescent PP fibres, photoluminescent PA6 fibres, blended yarns, change of colour expression.*

1 INTRODUCTION

Criminal activities in the field of intellectual property have significant economic consequences. It deprives legitimate companies of incomes and public administration of taxes. The counterfeit goods can have, besides the economic losses, also significant influence on health and safety of the consumers as well as adverse consequences for the environment. Slovakia loses millions of Eur due to falsification every year. The counterfeit and pirated goods are usually made by anonymous subjects which do not respect requirements for the protection of human health, safety and quality and do not provide any assistance, guarantee or instructions for use after purchase of the goods. Luxury products, clothing and accessories are traditionally the most popular categories of fake products (and belong among the cases disclosed the most frequently). Manufacture of fake clothing is growing in the frame of EU every year; the organized criminal groups affix false trademarks on imported, unbranded clothing. This way they reduce a risk of discovery of the clothing during transport [1].

An affordable and cost-effective access to protection of originality of products is application of photoluminescent dyestuffs and pigments, which besides colour change emit also light under UV lamp. Phenomenons involving absorption of energy and subsequent light emission are classified generally as luminescence. There are several kinds of luminescence depending on way of excitation. Photoluminescent dyestuffs and pigments, available in organic as well as inorganic form, are particularly interesting for the purpose of protecting product originality. Protection of originality of textile materials can be assured by luminous colour expression of the modified polymer fibres under UV light, whose intensity grows with increasing content of the photoluminescent dyestuff in the modified polymer fibres. Unmodified polymer fibres do not show any luminous colour expression under UV light [2].

The contribution focuses on solution how to protect originality of textile products using blended yarns with different content of modified polypropylene (PP) and polyamide (PA6) filaments containing 0.01 wt.% and 0.10 wt.% of special protective photoluminescent (FL) organic pigment in the fibre.

2 EXPERIMENTAL PART

2.1 Materials used

Standard unmodified PP (**PPš**) and PA6 (**PAš**) friction textured filaments and modified PP and PA6 friction textured filaments have been used to prepare the blended yarns:

- PP fibre containing blue organic photoluminescent pigment (PP FLV) with concentration of 0.01 wt.% (**PP FLV** $_{(0.01)}$) and 0.10 wt.% (**PP FLV** $_{(0.10)}$) in the fibre;
- PA6 fibre containing blue organic photoluminescent pigment (PA FLV) with concentration of 0.01 wt.% (**PA FLV** $_{(0.01)}$) and 0.10 wt.% (**PA FLV** $_{(0.10)}$) in the fibre.

PP and PA6 fibres were of the same linear density 180 dtex.

Table 1 Composition of the blended yarns containing PP FLV incorporating 0.01 wt.% of FL pigment in the fibre

Designation of the blended yarn	Composition of the blended yarn		Percentage of PP FLV in the blended yarn [%]
	Quantity of PP FLV in the yarn	Quantity of PPš fibres in the yarn	
A-$PP_{š}$	0	4	0
B-$PP_{(0.01)}$	1	3	25
C-$PP_{(0.01)}$	2	2	50
D-$PP_{(0.01)}$	3	1	75
E-$PP_{(0.01)}$	4	0	100

Table 2 Composition of the blended yarns containing PP FLV incorporating 0.10 wt.% of FL pigment in the fibre

Designation of the blended yarn	Composition of the blended yarn		Percentage of PP FLV in the blended yarn [%]
	Quantity of PP FLV in the yarn	Quantity of PPš fibres in the yarn	
A-$PP_{š}$	0	4	0
B-$PP_{(0.10)}$	1	3	25
C-$PP_{(0.10)}$	2	2	50
D-$PP_{(0.10)}$	3	1	75
E-$PP_{(0.10)}$	4	0	100

Table 3 Composition of the blended yarns containing PA FLV incorporating 0.01 wt.% of FL pigment in the fibre

Designation of the blended yarn	Composition of the blended yarn		Percentage of PA FLV in the blended yarn [%]
	Quantity of PA FLV in the yarn	Quantity of PAš fibres in the yarn	
A-$PA_{š}$	0	4	0
B-$PA_{(0.01)}$	1	3	25
C-$PA_{(0.01)}$	2	2	50
D-$PA_{(0.01)}$	3	1	75
E-$PA_{(0.01)}$	4	0	100

Table 4 Composition of the blended yarns containing PA FLV incorporating 0.10 wt.% of FL pigment in the fibre

Designation of the blended yarn	Composition of the blended yarn		Percentage of PA FLV in the blended yarn [%]
	Quantity of PA FLV in the yarn	Quantity of PAš fibres in the yarn	
A-$PA_{š}$	0	4	0
B-$PA_{(0.10)}$	1	3	25
C-$PA_{(0.10)}$	2	2	50
D-$PA_{(0.10)}$	3	1	75
E-$PA_{(0.10)}$	4	0	100

2.2 Technology of preparation of the blended yarns

The blended yarns have been prepared using twisting technology on the multipurpose twisting device DirecTwist2B. The twisting has been performed under the same technological conditions of manufacture, i.e. winding speed 6.66 m/min., drum revolutions 1 350 rev/min, quantity of twists 150 twists/m, twist direction Z.

Each blended yarn consisted of 4 filaments. Composition of the blended yarns is given in the Tables 1-4.

Each blended yarn has been wound on a dark mat in two layers. The color expression of the blue FL pigment has been evaluated on the prepared samples.

2.3 Evaluation of efficiency of the colour expression

Colour expression of the FL pigment has been evaluated in two ways:

1) objectively by a change of colour expression defined by means of b* colour coordinate in the CIE LAB colour space (Figure 1) using ULTRASCAN XE device according to the standard STN ISO 105-J03. The b* colour coordinate defines shades between yellow and blue colour. Shift of the b* coordinate to the positive values (+b*) causes colour transition to the field which comes up to the yellow colour spectrum. Shift of the b* coordinate to the negative values (-b*) causes colour transition to the field which comes up to the blue colour spectrum. The lower (more negative) b* value is measured the stronger intensity of emission is observed [3].

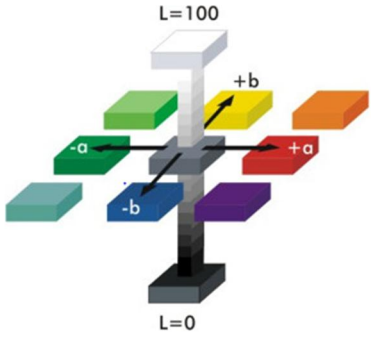

Figure 1 Colour space CIE LAB

2) by optical expression of photoexcitation using device Fluotest with UV lamp enabling to demonstrate luminescent effects of the materials in the range of short-wave and long-wave ultraviolet (UV) radiation. Ultraviolet radiation is inter alia an approved method to identify presence or absence of substances (e.g. photoluminescent pigments, optical brighteners) for the purpose of distinction of material differences which cannot be distinguished in the visible light. The blended yarns wound on a dark mat have been exposed to filtered UV radiation in the region of electromagnetic radiation (Figure 2) for long-wave radiation (UV-A region) and intensity of emission of FL blue organic pigments has been assessed visually.

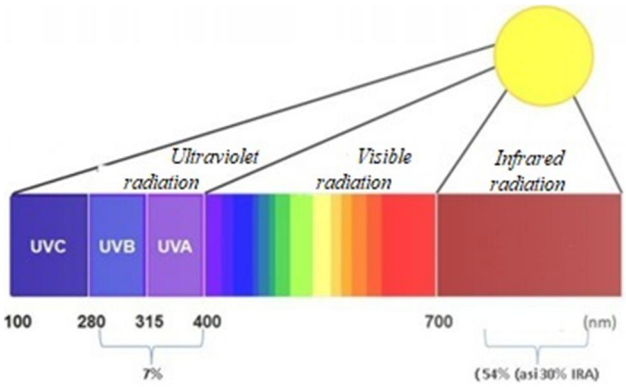

Figure 2 Electromagnetic spectrum

3 RESULTS AND DISCUSSION

PP blended yarns have white colour in the daylight and neither material composition of the yarn nor concentration of FL pigment in the fibre is distinguishable with naked eye of an observer (Figure 3).

Optical excitation in a form of blue light emission, enabling distinct identification of the blended yarns containing PP FLV has been observed on PP blended yarns with naked eye using UV lamp (Figures 4 and 5).

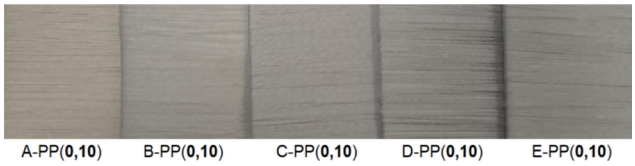

Figure 3 PP blended yarns with different content of PP FLV containing 0.10 wt.% of FL pigment in the fibre in the daylight

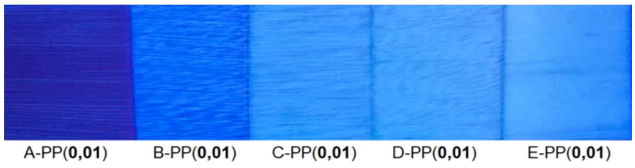

Figure 4 PP blended yarns incorporating PP FLV containing 0.01 wt.% of FL pigment in the fibre after exposition to long-wave UV radiation in the device Fluotest

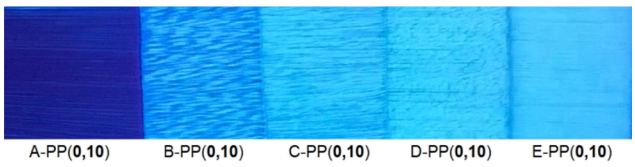

Figure 5 PP blended yarns incorporating PP FLV containing 0.10 wt.% of FL pigment in the fibre after exposition to long-wave UV radiation in the device Fluotest

All PP blended yarns emit bright pale blue colour whose intensity increases with increasing share of PP FLV in the yarn. Yarns prepared from standard PP fibres (designation of the yarns A) have deep dark blue colour. Blue light emitted by the yarn prepared from 100% PP FLV is brighter and its depth and brightness moves with increasing share of the standard unmodified PP fibre to the yarn prepared from 100% unmodified standard PP fibre. From a viewpoint of concentration of FL pigment in PP fibre we can state that the blended yarns prepared from PP FLV containing 0.10 wt.% of FL pigment in the fibre emit brighter blue light that the yarns prepared from PP FLV containing 0.01 wt.% of FL pigment in the fibre, what confirm also results of measurement of b* colour coordinate (Figure 6).The blended yarns incorporating PP FLV containing 0.10 wt.% of FL pigment in the fibre have by about 60% higher intensity of blue light emission than blended yarns containing 0.01 wt.% of FL pigment in the fibre. Efficiency of FL pigments can be expressed also by means of colour difference between a yarn prepared from standard PP fibre and/or yarn prepared from modified PP FLV and blended yarns with different content of PP FLV. Provided that a yarn prepared from 100 wt.% of standard PP fibre (designation of the yarn A) will show emission intensity 0% and a yarn prepared from 100 wt.% of PP FLV (designation of the yarn E)

will show emission intensity of blue light on a level of 100%, then decrease and/or increase of emission intensity of blue light in the blended yarns (designation of the blended yarns B – D) can be expressed also as percentage. Emission intensity of blue light of the blended yarns prepared from PP FLV containing 0.01 wt.% and/or. 0.10 wt.% of FL pigment in the fibre is, in comparison with a yarn prepared from 100 % modified PP FLV containing 0.01 wt.% and/or 0.10 wt.% of FL pigment in the fibre, on a level of:

- 51% and/or. 60% at 25% of PP FLV in the blended yarn (designation of the blended yarn B),
- 77% and/or. 83% at 50% of PP FLV in the blended yarn (designation of the blended yarn C),
- 95% and/or. 94% at 75% of PP FLV in the blended yarn (designation of the blended yarn D).

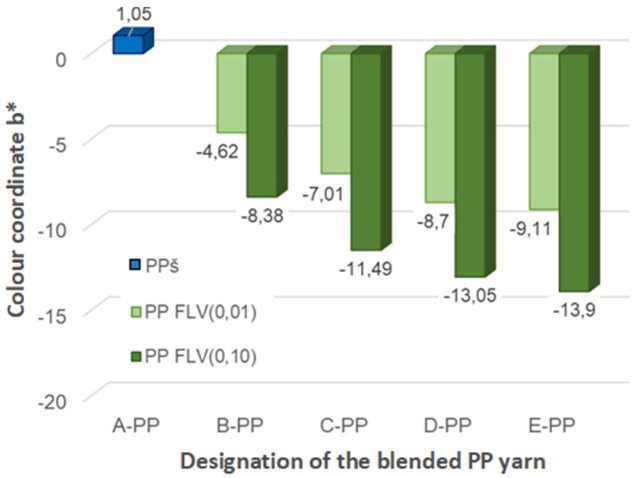

Figure 6 Influence of material composition of PP blended yarns on a change of b* colour coordinate

PA blended yarns, as well as PP blended yarns, have white colour in the daylight and neither the share of PA FLV in the blended yarn nor the concentration of FL pigment in PA FLV is distinguishable by the naked eye of an observer (Figure 7).

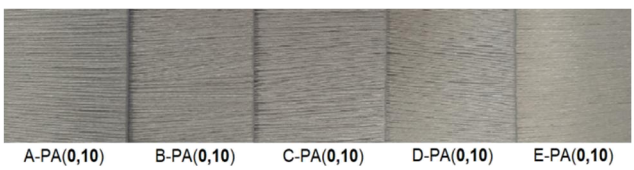

Figure 7 PA6 blended yarns with different content of PA FLV containing 0.10 wt.% of FL pigment in the fibre in the daylight

PA blended yarns emit in the region of long-wave UV radiation blue light of various depth and brightness, whose intensity depends on content of PA FLV in the blended yarn (Figures 8 and 9).

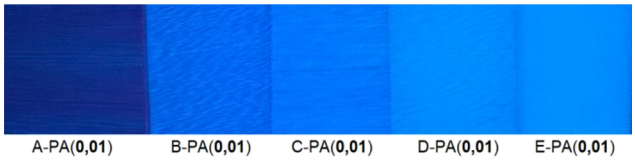

Figure 8 PA6 blended yarns incorporating PA FLV containing 0.01 wt.% of FL pigment in the fibre after exposition to long-wave UV radiation in the device Fluotest

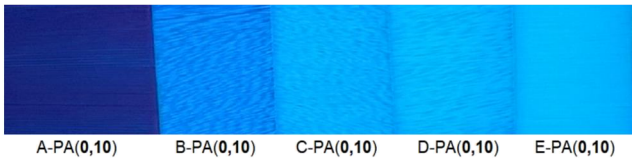

Figure 9 PA6 blended yarns incorporating PA FLV containing 0.10 wt.% of FL pigment in the fibre after exposition to long-wave UV radiation in the device Fluotest

From a viewpoint of concentration of FL pigment in PA fibre we can state that the blended yarns prepared from PA FLV containing 0.10 wt.% of FL pigment in the fibre emit brighter blue light than the yarns prepared from PA FLV containing 0.01 wt.% of FL pigment in the fibre, what confirm also results of measurement of b* colour coordinate (Figure 10).

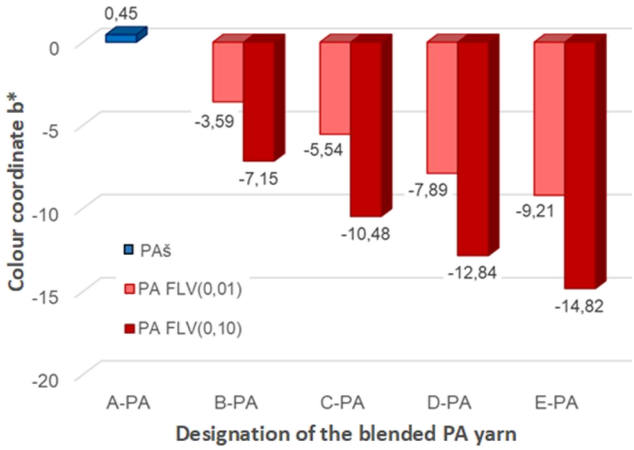

Figure 10 Influence of material composition of PA6 blended yarns on a change of b* colour coordinate

Emission intensity of blue light of the blended yarns prepared from PA FLV containing 0.01 wt.% and/or 0.10 wt.% of FL pigment in the fibre is, in comparison with a yarn prepared from 100% modified PA FLV containing 0.01 wt.% and/or. 0.10 wt.% of FL pigment in the fibre on a level of:

- 39% and/or 48% at 25% PA FLV in the blended yarn (designation of the blended yarn B),
- 60% and/or 71% at 50% PA FLV in the blended yarn (designation of the blended yarn C),
- 86% and/or 87% at 75% PA FLV in the blended yarn (designation of the blended yarn D).

Figures 6 and 10 demonstrated clearly that emission intensity of blue light with PP and PA6 blended yarns with the same concentration of FL pigment in the fibre is comparable and content of FLV in the blended yarn has influence on emission intensity of blue light.

4 CONCLUSION

Polypropylene and polyamide fibres are widely used in the assortment of textile and clothing products and their application depends on intended use of the final products. Photoluminescent fibres, whose added value is their simple identification at specific wavelength out of region of visible radiation, can be prepared by modification of PP and PA6 fibres using special additives, e.g. photoluminescent organic pigments applied to mass of the polymer system. The photoluminescent fibres incorporated in a form of protective elements in the assortment of textile and clothing products are able to ensure effectively authenticity and originality of products and reduce risk of falsification. Photoluminescent pigments belong among rather expensive materials, increasing price of the modified PP and PA6 fibres. One of the solutions in the field of textile workability is optimization of rate of the photoluminescent fibres in the yarn construction while ensuring sufficient excitation of colour light at a specific wavelength. On the base of achieved results it is possible to state that for identification of the photoluminescent PP and PA6 fibre in the blended yarns concentration of 0.01 wt.% of blue photoluminescent organic pigment in PP and/or PA6 fibre and 25% content of photoluminescent PP and/or PA6 fibre in the blended yarn is sufficient. In comparison with a yarn made of 100% PP FLV emission intensity of blue light of the blended yarn containing 25% of PP FLV is on a level of about 50% and emission intensity of blue light of the blended yarn containing 25% of PA FLV is on a level of about 40%. The achieved emission intensity of blue light of the above-mentioned blended yarns is sufficient for human perception to distinguish blue colour under long wavelength UV radiation.

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SCIENTIFIC DEVELOPMENT OF INNOVATIVE TECHNOLOGIES OF OBTAINING COMPOSITE MATERIALS FROM OF OILSEED FLAX FIBERS

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Abstract: *The article examines the ways to solve the problem of developing a scientific basis for obtaining composite materials of different functional purposes from oilseed flax fibers. Composite materials came to the forefront of production and demand for goods several decades ago, thanks to unsurpassed specific and mechanical properties as a result of growing consumer and industry demand for high-tech materials and structures. However, the combination of natural fibers with a polymeric material or matrix increases the difficulty of the process of forming composites and, as a rule, leads to problems in the physicochemical processes of interaction of the matrix and the filler. Composites with synthetic fillers have obvious advantages, but their disposal is difficult, requires the development of environmental processing technologies. The best way to save the environment when processing composite materials is to use non-toxic natural materials for their production, but this requires the development of innovative technologies for forming composite materials with natural fibrous fillers. The paper covers theoretical and experimental research in the area of processing flax raw materials. The purpose of the study is to provide scientific substantiation of developing the technologies for obtaining fillers to reinforce composite materials. In order to do it, we performed modification of oilseed flax fiber and developed technologies for processing oilseed flax straw with regulated technological and performance characteristics. The article also presents the results of the research on determining causes of low wettability of oilseed flax bast. In order to find out the causes of low bast wettability, we conducted research on examining chemical composition and anatomy of straw stems. The formulation for preparing the fiber aimed to be used as filler for reinforcement of composite materials is offered. The study suggests evaluation of the quality of composite materials produced on the basis of using modified oilseed flax fibers.*

Keywords: *oilseed flax, straw, bast, fiber, composite materials.*

1 INTRODUCTION

Oilseed flax is a valuable industrial crop of versatile use. Its botanical name Linum usitatissimum means “early maturing”. According to the FAO data, the sown areas of oilseed flax cover almost 3.5 million hectares over the globe [1, 2]. Flax is grown in many countries of the world (Figure 1). Oilseed flax takes more than 70% of sown areas in the world. Recently, oilseed flax production has been intensively developed in Canada and the USA.

Analysis of the global oilseed flax production shows that the leading producers of oilseed flax in the world are Canada, China, India, Argentina, USA and Russia. The total gross seed yield in these countries makes 1.2 million tons. In Ukraine this crop has not been paid attention over many past years due to social and political processes occurring in our country in the course of centuries. Nowadays oilseed flax returns to Ukraine. A wide range of varieties, their diversity and high profitability contribute to rapid spreading and annual growth of sown areas for this crop cultivation.

Figure 1 Flax-growing countries (hatched)

Recently, Western Europe and other countries of the world have shown great interest to the use of oilseed oil in manufacturing different products of industrial purposes. The global tendency for expanding sown areas to grow this crop can be explained by the fact that it is a main source for production of industrial oil and is characterized by excellent biological and technical properties: high drought-resistance; lodging-resistance; fast

maturation, it matures later than cereal crops. Moreover, oilseed flax is a good pre-crop for winter crops, high-protein feeds for animals, it has high seed yields (over 20 c/ha) and a high commodity price on the international market [1]. The article presents the results of theoretical and experimental research aimed at developing technology for the production of fillers for the reinforcement of composite materials.

In Ukraine flax seeds are processed to obtain oil in small volumes, and most of them are exported. The crop stems are rarely processed and largely burnt in the fields. The research conducted at Kherson National Technical University (Ukraine) showed that oilseed flax stems contain a sufficient amount of fiber similar by its structure to short fiber of linen flax.

Oilseed flax stems, like linen flax stems, contain cellulose fiber in their bast. However, oilseed flax fiber has not been used in the global industry so far. After separating seeds, straw residues have been mostly burnt and mixed into soil, i.e. they were applied as fertilizer. Recently the issues of using oilseed flax have been paid much attention over the globe, but these studies are mainly aimed at processing seeds. Only few of them investigate the processing of oilseed flax stems to obtain fiber.

The article introduces the results of theoretical and experimental studies on obtaining composite materials of different functional purposes from modified oilseed flax fibers. Examination and generalization of the results of the modern theoretical research in the area of production of composite materials allowed formulating the main hypothesis of the study: modification of oilseed flax fiber must result in the formation of its quality indexes, determined by physical-mechanical and chemical properties, parameters and modes of processing raw materials that can be considered as a complex criterion affecting final characteristics of composite materials of different functional purposes obtained from oilseed flax fiber. Confirmation of this hypothesis will support scientific development of innovative technologies for obtaining composite materials of different functional purposes by using oilseed flax fiber.

1.1 Literature review

The leading world scientists L.A. Chusina, H.A. Tikhosova (Ukraine) and V.V. Zhivetin (Russia) prove that oilseed flax fiber is suitable for manufacturing industrial textile of different purposes. The scientists of many countries of the world, namely Langer E. (Germany), Kathleen V.D.V. (Belgium) carried out research on using natural fibers, in particular, oilseed flax fiber to obtain internal panels for cars. There are studies of Ton-That MT, Denault J. (Canada) on applying fibers to manufacture products for technical purposes. The papers of Mieleniak B., Bagley C., d'Anselme T., Guyader J. (USA) evidence that oilseed flax is traditionally grown on 700-800 thous. ha in the west of Canada. Annual yield of oilseed flax straw is nearly 1 million tons, and only 15-20% of this straw is used in production, mainly, to manufacture cigarette paper. Factories producing pulp and paper are located in the states North Carolina and New Jersey (USA). The studies of Pallesen (Denmark) highlight that fiber obtained by the technology of Rome research center (IPZS), after enzymatic treatment, extracting, active warm ventilation and carding process are used to produce composite materials, and shives – to produce chip boards. Zelenetskyi S. (Russia) succeeds in conducting research on modification of natural fibers to produce polymeric composite materials with natural fibers as fillers [2-14].

Application of oilseed flax straw will allow using annually renewable, environmentally friendly and safe raw materials, in particular, oilseed flax fiber to manufacture fillers for reinforcement of composite materials. In order to determine suitability of the obtained bast for production of industrial textile, in particular, fillers for composite materials, production trials were carried out at the state enterprise “Plastmas” (Ukraine).

To conduct research and carry out experiments, we selected three varieties of oilseed flax – Evryka, Liryna and Aisberh, significantly different from each other by technological characteristics. These varieties were grown in the climatic conditions of the South of Ukraine. Flax oilseed straw was obtained at the state enterprise “Research Farm “Askaniiske” of the National Academy of Agrarian Sciences of Ukraine.

The variety Evryka has been in the List of Plant varieties of Ukraine since 2004; it was created by the Institute of Irrigated Agriculture of the NAAS of Ukraine by the method of hybridization with further individual-family selection. The variety is designed to obtain oil for food and industrial needs and protein meal to feed animals. The plant height is 57-62 cm. The stem is rounded, its thickness being 3-4 mm, with branches in lower and upper parts. The length of the growing season is 81 days. The inflorescence is umbellate, 25-32 cm long. The fruit is a rounded capsule with 7-10 seeds. The seeds are black. The weight of 1000 seeds is 7-8 g. It is resistant to lodging, capsule cracking and seed shedding. The variety is stable in terms of yields. It is medium-resistant to pests and diseases, suitable for all growing zones. The seed productivity is 28.8 c/ha. Seed oil content is 39.4%.

The variety Aisberh has been in the List of plant varieties of Ukraine since 2001; it was created by the Institute of oilseed crops of the UAAS by the method of induced mutagenesis of Tsian variety seeds through irradiation with gamma-rays. The plant height is 54-57 cm; the duration of the growing season is 86-88 days. The variety is

characterized by drought and lodging resistance. In the field experiments of the Institute of Agriculture of the Southern region of the UAAS (2004) its seed profitability was 20.8-21.8 c/ha.

The variety Liryna has been in the List of plant varieties of Ukraine since 2002; it was created by the German plant breeders of "Deutsche Saatveredelung AG". The variety is of an intensive type of usage. It generates high stable yields 25-29 c/ha. The growing season lasts 107-128 days. A large number of capsules with seeds ensure high yields even under low seeding density. The plant height is 58-78 cm. The weight of 1000 seeds is 5.6-7.2 g. Oil content is 44.3-46.1%. Plants are characterized by uniform maturation. It is recommended for growing in forest-steppe and steppe zones.

2 RESEARCH METHODOLOGY

The tasks set in the work were solved with the help of modern methods of theoretical and experimental research. Studies of chemical components were performed by traditional methods. Studies of anatomical structure were performed using microscopes. The quality of the obtained phenoplasts, reinforced with oilseed flax and cotton fibers was evaluated according to the requirements of TU U 25.2-32512498-001-2004 with amendments 1, 2, 3, 4 "Pressing phenolic mass".

In order to obtain fiber, the straw stems were mechanically processed using a modernized flax-scutching machine without a technological operation of flax straw preparation. The main reason for not preparing flax straw lies in the fact that agricultural enterprises are not interested in producing oilseed flax straw. It is related to additional financial, energy and labor costs, which agricultural producers cannot afford because of a lack of stable market for selling flax straw.

After processing straw stems of oilseed flax using a modernized flax-scutching machine, we selected samples of the obtained bast and determined its physical-mechanical properties: a mass portion of shives and impurities, breaking load, bast output [15, 16].

Analysis of the obtained results showed that bast, obtained from oilseed flax straw stems, has a high mass portion of shives 27.2-30.6% and low strength 3.9-5.0 daN. However, bast output after mechanical processing by a modernized flax-scutching machine is sufficiently high: with the average stem length of 32.9-36.0 cm, this index amounts to 32.4-39.4%. Thus, we can draw a conclusion that bast-fiber raw materials with such high indexes of a mass portion of shives and impurities do not meet the requirements of producers of industrial textile of different purposes. Therefore, in our further research bast was additionally purified from shives by means of the operation of machine hackling.

The experimental research resulted in obtaining bast with high quality indexes due to optimization of the modes and parameters of mechanical processing of oilseed flax straw stems. After the second hackling by the hackling machines with the rotational frequency of the main cylinder of 555 min^{-1} and the opening between it and the knife of 1.5 mm, we obtained bast with the following physical-mechanical characteristics: the staple length 18.10 mm; the mass portion of shives and impurities 0.01%; linear density 0.35 tex.

In order to determine suitability of the obtained bast for manufacturing fillers for reinforcement of composite materials, we carried out field trials at the state enterprise "Plastmas" (Ukraine). As it is known, the main index of filler adhesion to phenol formaldehyde resins is wettability. Currently at the enterprise "Plastmas" cotton lint with the wettability of 120 g is used as filler. Therefore, when carrying out the research, we determined the wettability of oilseed flax bast of the three varieties under study: Aisberh, Evryka and Liryna. The results of the experimental research showed that the average values of this index ranged from 5.0 to 6.3 g. Analysis of the obtained results indicate that bast fiber of oilseed flax has wettability that is 24 times less than cotton lint does. According to the requirements of the regulating documents, the filler for phenoplasts should have wettability of 116-120 g. Therefore, oilseed flax bast, obtained after mechanical processing of straw stems is unsuitable for producing fillers to reinforce composite materials on the basis of phenol formaldehyde resins because of its low wettability [17].

To find out the causes of such low wettability, the chemical composition of oilseed flax straw was studied and the anatomical structure of oilseed flax straw (Figure 2) was studied.

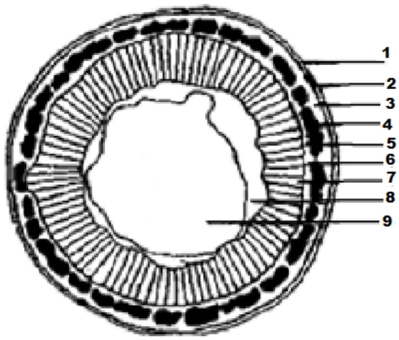

Figure 2 Anatomical structure of oilseed flax straw: 1 - cuticle; 2 - epidermis; 3 - parenchyma cow; 4 - fibrous bundles; 5 - floem; 6 - cambium; 7 - xylem; 8 - parenchyma; 9 - cavity

The study of anatomical structure of lounge oilseed flax showed that from the outside the stem is covered with a cuticle 1, under it there is

an epidermis 2, behind it - a cow parenchyma 3, which surrounds fibrous bundles 4. From the inside of the fibers located floem 5, closer to the center there are cambiums 6 and wood 7, 8 (xylem and parenchyma), and the center of the stem is cavity 9.

After reaching full maturity of oilseed flax straw stems and their mechanical processing by means of hackling, bast completely gets rid of its woody parts: phloem, xylem, parenchyma, and cuticle is left on the exterior of fibers, that adds hydrophobic properties to fibers. Cuticle is structureless transparent covering existing between fibers in the form of hairs. Cuticle consists of substances called cutins. As the study of Ordina N.A. proves, cutins are high molecular fatty acids, oxyacids, waxes and fats [18]. They are resistant to the effects of strong chemical reagents such as concentrated acids and alkali. Cutins do not dissolve in sulphuric and chromic acids and even in copper-ammonia solution in which cellulose dissolves. It is availability of cutins on oilseed flax bast that causes its low wettability and a lack of adhesion to polymeric matrix.

Thus, the results of the theoretical and experimental research allowed establishing that after mechanical processing of straw stems of oilseed flax by means of a modernized flax-scutching machine and double hackling of the obtained bast using a hackling machine to remove incrusts and cutins, it is necessary to perform chemical processing (boiling) of bust.

3 RESULTS

Therefore, the second stage of developing the technology for obtaining fillers to reinforce composite materials from oilseed flax is chemical modification, i.e. purifying fibers to remove cellulose and substances accompanying cutin – high molecular fatty acids, oxyacids, waxes and fats.

Having studied all the available methods for purifying bast to remove extraneous substances and waxes, the scientists of Kherson National Technical University developed a number of techniques to obtain cellulose from oilseed flax bast and fiber. The most effective technique is acidification that ensures high cellulose output. This technique was used in boiling oilseed flax bast of the varieties under study obtained after double hackling. The stages of chemical treatment of oilseed flax fiber are given in Table 1.

Table 2 presents the results of the research on wettability and chemical composition of oilseed flax fiber of the variety Aisberh after boiling for 1, 2 and 3 hours. The results of the chemical analysis of oilseed flax fiber composition of the three varieties under study allowed establishing that different varieties of oilseed flax have similar content of chemical components. Therefore, we used mediated values of the experimental research of the oilseed flax variety Aisberh. Analysis of the data in Table 2 shows that boiling oilseed flax bast of the variety Aisberh according to the mode given in Table 1 for 1, 2 and 3 hours allowed obtaining fiber which meets the requirements for wettability of fillers set by the enterprise “Plastmas”. In the course of the previous research we determined that this index for oilseed flax bast was only 5.0-6.3 g, and due to the technological operation of boiling the fiber wettability increased to the necessary indexes – 104.94-122.78 g, a mass proportion of α-cellulose rose from 76.88% in the control variant (without boiling) to 86.88-90.01%.

Table 1 Stage of chemical treatment of oilseed flax fiber

Stage of treatment	Composition [g/l]	Mode
1. Oxidation boiling	Hydrogen peroxide (100%) – 4.0	1. Boiling at 100°C, 60-180 min.
	Sodium hydroxide – 10.0	2. Rinsing with cold water, 10 min.
	Calcined soda – 2.0	3. Sulfuric acid H_2SO_4 concentration 96% (2 g/l), 10 min.
	Sodium silicate – 5.0	4. Rinsing with cold water, 20 min.
	Sodium tripolyphosphate – 1.0	
	Wetting agent – 0.3	
2. Fiber drying	-	Pressing out to moisture content of 60% and drying at 100°C

Table 2 Physical-chemical indexes of fiber quality of the oilseed flax variety Aisberh

Number of experiments	Physical-chemical indexes					
	Duration of boiling, hours					
	1		2		3	
	wettability [g]	mass portion of α-cellulose [%]	wettability [g]	mass portion of α-cellulose [%]	wettability [g]	mass portion of α-cellulose [%]
1	104.88	87.32	105.92	87.78	122.80	90.96
2	104.45	86.67	104.86	88.28	123.50	89.64
3	105.22	86.83	105.76	87.53	119.70	90.04
4	104.98	87.06	105.58	87.22	124.70	89.76
5	105.15	86.54	104.84	87.58	123.20	89.67
Average value	104.94	86.88	105.39	87.68	122.78	90.01

Table 3 Physical-mechanical quality indexes of the phenoplasts with oilseed flax and cotton fibers

№	Quality indexes	Type of filler					
		standard indexes	control variant	oilseed flax fibers of the variety Aisberh after boiling [hrs]			cotton
				1	2	3	
1.	Color	from light-brown to dark-brown	complies	complies	complies	complies	complies
2.	Fluidity [mm]	40-140	200	185-190	180-190	200	125
3.	Appearance of pressed samples	without cracks and blisters	does not comply	complies	complies	complies	complies
4.	Notch-toughness by Charpy on samples without a cut,[kJ/m2], not less	8.8 (9.0)	16.43	9.5	11.3	8.2	12.38
5.	Bending stress under damage [MPa], not less	58.8 (600)	632.55	571	562	706	692
6.	Specific volume electric resistivity [Ω.cm]	1.10^9	-	$2.89.10^{12}$	$1.96.10^{13}$	$6.9.10^{13}$	$5.0.10^9$
7.	Electric robustness [kW/mm]	6.0	-	10.8	10.6	12.0	-

The quality of the obtained phenoplasts, reinforced with oilseed flax and cotton fibers was evaluated according to the requirements of TU U 25.2-32512498-001-2004 with amendments 1, 2, 3, 4 “Pressing phenolic mass” [19]. The obtained composite materials were compared by their color, appearance, fluidity, notch-toughness by Charpy on the samples without a cut and electrical robustness. Physical-mechanical quality indexes of the phenoplasts U1-301-07, reinforced with cotton fiber and oilseed flax fiber are given in Table 3.

Analysis of the quality indexes of the obtained composite materials given in Table 3 evidences appropriateness of reinforcing phenoplasts with oilseed flax fibers on the basis of using thermosetting resin. Boiled for 1 and 2 hours, oilseed flax fiber of the variety Aisberh complies with the requirements of TU U 25.2- 32512498-001-2004 by all the indexes under study except bending stress under damage. It equals to 571 kgf/cm^2 after 1 hour of boiling and 562 kgf/cm^2 after 2 hours of boiling, i.e. a bit less than the standard index that must be not less than 600 kgf/cm^2 that is determined by low strength of oilseed flax fiber [20].

Oilseed flax fiber can be used as fillers to reinforce composite materials after mechanical processing and chemical modification. The results of the research on chemical composition of bast obtained after mechanical processing and analysis of the anatomy of oilseed flax stem structure allowed determining that high hydrophobic behavior and low wettability of bast are caused by the presence of cuticular layer on fiber. It consists of high molecular fatty acids, wax substances and fats, and their average content in bast equals to 3.85%. We established that in order to use oilseed flax bast for reinforcing composite materials, in addition to mechanical purifying to remove extraneous substances and shives, it is necessary perform chemical cleaning of cellulose to remove incrusts, i.e. lignin and pectin substances, and also high molecular fatty acids, wax substances and fats.

After performing theoretical analysis of advantages and disadvantages of different methods for obtaining cellulose, we chose a new acidification method for purifying bast to remove substances accompanying cellulose developed by the scientists of Kherson National Technical University. The research results showed that after boiling oilseed flax bast of the variety Aisberh for 3 hours we obtained samples of phenoplast that complied with the requirements of TU U 25.2-32512498-001-2004 by all the indexes except notch toughness being 8.2 kJ/m^2, whereas the standard index equals not less than 8.8 kJ/m^2. The samples of phenoplast obtained by means of oilseed flax bast without boiling, i.e. the control variant, complied with TU U 25.2-32512498-001-2004 by physical-mechanical properties, but the disks had numerous blisters and cracks, that did not make it possible to test these samples by electric indexes.

Thus, analyzing the results of experimental studies, we can conclude that the flaxseed fiber after boiling by the proposed method can be used as a reinforcing component for the manufacture of composite materials based on thermosetting resin alone or in combination with other types of cellulose fiber. Therefore, the scientific development of innovative technologies for obtaining composite materials from oil flax fibers for various functional purposes will allow the use of annually renewable, environmentally friendly and safe raw materials, which will promote resource-saving technologies and improve the environment and supply new products for various functional purposes.

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EFFECT OF PHYTIC ACID ADDITION ON STRUCTURAL CHARACTERISTICS OF ACRYLIC POLYMER FILM

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Abstract: *The influence of phytic and citric acids additives on the structure of polymer films formed from the Neoprint PNA/S acrylic polymer was investigated in order to develop finishing compositions for fire protection of textile materials. Standardized methods were used to study the structure formation of acrylic polymer films filled and unfilled with phytic and citric acids in various ratios. The degree of interaction between the components of the polymer system has been estimated and it was found that increasing the concentration of phytic acid enhances the interaction between the filler and the matrix. It was shown, that with an increase in the concentration of phytic and citric acids in the composition of the polymer film, the degree of crosslinking and the fraction of active chains of the acrylic polymer increase. The formation of a significant carbonized residue of the polymer film containing phytic and citric acids after exposure to an open flame is shown. The absence of splashing of the burning polymer melt, in contrast to the film formed from pure polymer, was noted, which excludes the potentially destructive effect in the form of melt dripping, which causes additional hot spots. The results obtained are of practical importance for the development of composite fire retardant finishing compositions for textile materials.*

Keywords: *acrylic polymer, phytic acid, citric acid, polymer film, structure formation, coke-forming ability, fire retardant finishing compounds.*

1 INTRODUCTION

The reason for the most active research areas and the introduction of non-halogenated flame retardants into the production cycle of finishing textile materials was the environmental restrictions imposed by many states on the use of organic substances that pose a threat to the environment. One of the most effective substitutions includes phosphorus-containing materials in inorganic or polymeric form, or in the form of low-molecular-weight additives to synthetic or natural polymers [1].

The advantages of phosphorus-containing fire retardants include the fact that phosphorus can act both in the vapor phase and in the condensed phase, depending on the specific phosphorus compound and the chemical composition of the polymer, in contrast to halogens, which act only in the vapor phase [2]. In the last decade, striving to further design and use products with a lower environmental impact, scientists have shown research on products of biological origin, such as proteins, nucleic acids, pomegranate peel extracts, banana pseudostems juice, etc., as effective fire retardants for natural (for example, cellulose) or synthetic (mainly polyester) textile materials [3-8]. These substances have three main advantages. First, their chemical structure and composition are very suitable for imparting fire resistance to fabrics, since biomacromolecules and products of biological origin contain key elements (phosphorus, nitrogen, sulfur) that are responsible for the activation of fire retardant mechanisms [9]. In addition, they tend to be readily dispersed or soluble in water, which is advantageous because the use of organic solvents, which have a strong environmental impact and are highly toxic, is prohibited in many countries. Also, the processing of fabrics can be carried out using existing finishing devices, for example, industrial impregnation / pressing / drying equipment.

Phosphorus-containing substances based on bioorganic compounds of phytic acid have attracted wide interest in the processing of textiles, especially as a fire retardant. Phytic acid (PA), known as inositol-hexakisphosphate acid or phytate in the form of a salt, is regarded as a “green” molecule and is found in abundance in plant tissues such as beans, grains and oilseeds [10, 11]. As a biocompatible, environmentally friendly, non-toxic and easily obtained organic acid, phytic acid is already widely used in antioxidant, antitumor, biosensor, cation exchange, nanomaterial and other fields due to its special structure of inositol hexaphosphate [12].

Phytic acid contains 28 wt.% phosphorus in terms of molecular weight and is promising as one of the effective fire retardants. Phytic acid was used as a doping acid to significantly improve the flame retardant characteristics of composite paper deposited with polyaniline [13]. PA/chitosan and

PA/nitrogen-modified silane hybrids were used by layer-by-layer assembly to make thin fire-resistant films on cotton fabric [14]. The potential fire protection effect of various metal phytates was assessed as a biosource of phosphorus additives for polylactic acid-based composites [15].

One of the significant disadvantages of processing textile materials with bioorganic molecules, limiting their use in the production of textiles, is the instability of the finish to water treatments.

2 THE GOAL OF THE STUDY

For many applications, the wash resistance of flame retardant fabrics is an important issue that can significantly limit their practical use. The high solubility of phytic acid in water causes an almost complete loss of the flame retardant coating after rinsing, resulting in significant weight loss for all flame retardant systems. To increase the resistance to washing, compositions are being developed using organosilicon substances, deposition by the sol-gel method, layer-by-layer assembly, etc. To increase adhesion to the fiber, textile materials are treated with polymer compositions. In this regard, it is relevant to study the effect of phytic acid on the spatial characteristics of the polymer in order to use it as a matrix for obtaining fire retardant coatings on textile materials.

3 MATERIALS AND METHODS

The 30 wt.% aqueous acrylic dispersion Neoprint PNA/S (LAMBERTI IBERIA S.A.U., Spain) was studied as a polymer matrix capable of providing a set of required properties. Phytic acid (Xi'an virgin Biological Technology Co.Ltd., China) was studied as a fire retardant. A tribasic carboxylic acid – citric acid (CA) (Ukraine) – was used as a carbon source.

An initial polymer film was formed on a glass substrate, as well as films with the addition of phytic and citric acids in a ratio of 1/1 and 3/2, respectively, followed by drying of the studied compositions at a temperature of 80°C.

To study the structural characteristics of acrylic polymer films filled and unfilled with phytic and citric acids in various ratios, we used methods based on the properties of crosslinked polymer systems to swell limitedly in various solvents.

4 RESULTS AND DISCUSSION

Most often in the finishing industry, aqueous dispersions of acrylic polymers are used, which is dictated by the requirements of environmental friendliness, as well as high adhesion properties, mechanical strength and availability.

Considering that most organic polymers are highly flammable objects, it was of interest to study the effect of phytic acid additives on the fire resistance of the polymer formation.

The selected ratios of phytic and citric acids are dictated by obtaining the most possible options for providing fire retardant properties to textile substrates, as well as by solubility in water. An increase in phytic acid in the composition leads to the formation of an insoluble precipitate, which during the processing of textile materials can adhere to the shafts of finishing machines and textile materials, leading to products defect.

Phosphorus-based biomacromolecules, as fire retardants, usually use the condensed phase mechanism, contributing to the formation of stable aromatic coke [3, 4]. This is possible due to the formation of phosphoric acid species during the activation of the biomacromolecule, which promotes dehydration reactions on the underlying textile substrate, significantly limiting the formation of organic combustible gases, which can additionally fuel the combustion process. This mechanism is further enhanced when phosphorus-containing biomacromolecules are combined with a carbon source [16].

The nature of intermolecular bonds (crosslinks) largely determines the properties of the polymer network. To obtain information about the crosslinked polymer matrix, the number of crosslinks in the volume of the polymer matrix is determined and the kinetics of swelling, as well as the relationship between the degree of crosslinking and the properties of the composition, are studied. The efficiency of the studied polymer films crosslinking was evaluated by the amount of acetone-insoluble fractions of the formed polymer films during the extraction of samples in a solvent. The polymer films were extracted with acetone in a Soxhlet apparatus for 24 hours. After removing and drying the films to constant weight, the degree of their curing was calculated using the formula:

$$C = \frac{W_1}{W_0} \times 100\% \quad (1)$$

where: W_0 - initial film weight [g]; W_1 - film weight after extraction [g].

The results of determining the degree of polymer films curing are shown in Figure 1. Analysis of the acetone-insoluble fraction of polymers shows that the individual polymer film Neoprint PNA/S is slightly soluble in acetone and is capable of providing a high-quality polymer coating. In Figure 1 shows a diagram characterizing the effect of different ratios of phytic and citric acids on the resistance of polymer films to the action of an organic solvent, which depends on the degree of their curing. As a result of the study, it was found that the film with the addition of phytic and citric acids in a ratio of 1/1 is characterized by the highest degree of curing of 90.38%. The cure rate of the individual polymer film is 83.48%. The degree of curing of the polymer film with the addition of phytic and citric acids, taken in a 3/2 ratio, is 75.02%.

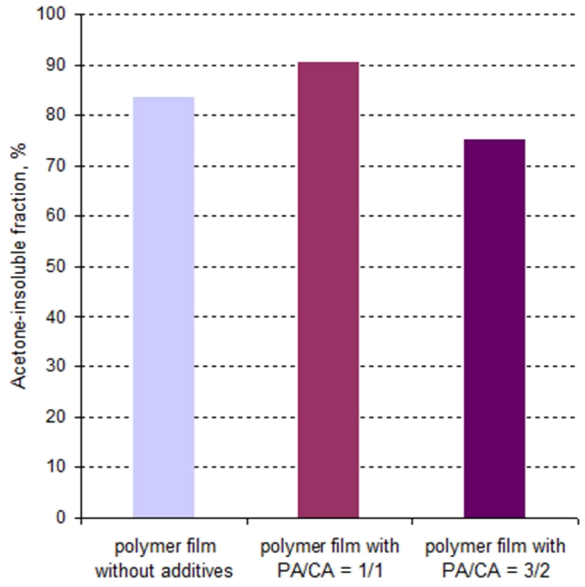

Figure 1 Degree of polymer films curing

Thus, it can be concluded that the presence of phytic acid in low concentrations contributes to an increase in the degree of polymer cure by 7.64%. However, an increase in the concentration of acids in the polymer film negatively affects the amount of the acetone-insoluble fraction, and the degree of cure decreases by 16.99%, which will contribute to the washout of the polymer during the operation of the textile product.

The efficiency of polymer-filler interaction was determined by the equilibrium swelling method and calculated using the Lorentz and Parks equation [17].

The ratio Q_f/Q_g characterizes the degree of interaction between the filler and the matrix, moreover, the higher the Q_f/Q_g values, the lower will be the extent of interaction between the filler and the matrix. The subscripts *f* and *g* refer to filled and unfilled polymer films, respectively.

Q is defined as grams of solvent per gram of polymer which is calculated by the formula:

$$Q = \frac{M_s - M_d}{M_d} \quad (2)$$

where: M_s - the swollen weight [g]; M_d - the dried weight [g].

The results of determining the polymer-filler interaction are shown in Table 1.

Analysis of the results of the Table 1 shows that polymer films with the addition of phytic and citric acids in a ratio of 3/2 have the highest interaction between the filler and the matrix.

To calculate changes in the structural parameters of the network of acrylic polymer, individual and with additives of phytic and citric acids, the sol content was determined by sol-gel analysis. A polymer sample weighing 1 g was first extracted with acetone to remove soluble products, then with benzene in an inert atmosphere. The weights of the samples were calculated before and after extraction with benzene. The degree of gel crosslinking was determined from the equilibrium degree of the studied polymer systems swelling. The data obtained are presented in Table 2.

The data obtained (Table 2) indicate that the structural characteristics of polymer films change depending on their filling with acids. When phytic and citric acids are added in equal amounts, the density of the polymer cross-link network is reduced by 37% compared to the unfilled film. In this case, the fraction of active polymer chains decreases to 0.59, in comparison with a film without additives, the fraction of active chains of which is 0.74. With an increase in the concentration of acids in the polymer film, the degree of crosslinking slightly increases by 5.79%, and the fraction of active chains increases to 0.76.

Table 1 The extent of interaction between the filler and the matrix

Index	Polymer film without additives	Polymer film with PA/CA=1/1	Polymer film with PA/CA=3/2
Degree of polymer-filler interaction Q_f/Q_g	6.74	5.04	1.26

Table 2 Structural characteristics of the formed polymer films

Crosslinking agent	Sol fraction *S* [%]	Equilibrium degree of polymer swelling *a* [%]	Degree of polymer crosslinking *j*	Fraction of active polymer chains V_c
Without additives	3.29	16.16	4.66	0.74
PA/CA=1/1	7.36	0.17	2.90	0.59
PA/CA=3/2	2.99	3.66	4.93	0.76

Figure 2 demonstrates the dependence of the formation of the fraction of active chains of unfilled and acid-filled acrylic polymer films on the change in the degree of polymer crosslinking.

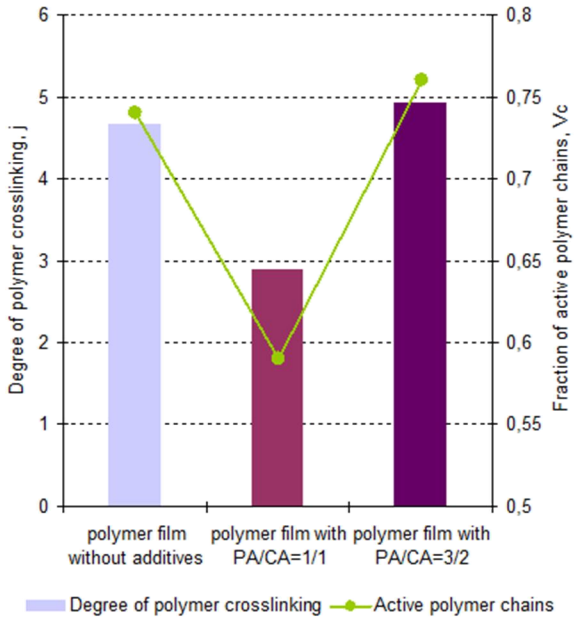

Figure 2 Influence of phytic and citric acids additives on the degree of crosslinking and the fraction of active polymer chains

Graphical dependencies in Figure 3 show that with an increase in the concentration of phytic and citric acids in the composition of the polymer film, the degree of crosslinking and the fraction of active chains of the acrylic polymer increase.

The supposed mechanism for the interaction of phytic acid with an acrylic polymer is via hydrogen bonds. To confirm this hypothesis, it is necessary to further study the nature of the chemical interaction using IR spectroscopy.

Based on the comprehensive studies carried out, it can be concluded that the use of phytic acid as a phosphorus-containing product that acts as a fire retardant, and citric acid as an additional source of carbon to increase coke formation, without deteriorating of the structural parameters of the acrylic polymer film is recommended in a ratio of 3/2, respectively.

At the next stage of work, the formed samples of unfilled and acid-filled acrylic polymer films were tested for resistance to open flame. The experimental results are shown in Figure 3.

Photo in Figure 3 demonstrates the results of exposure to flame on polymer films. The unfilled sample is characterized by fast ignition and high burning rate. In addition, the polymer film melted, burning droplets were formed, which could potentially cause the formation of new hot spots, as well as an increased risk of burns.

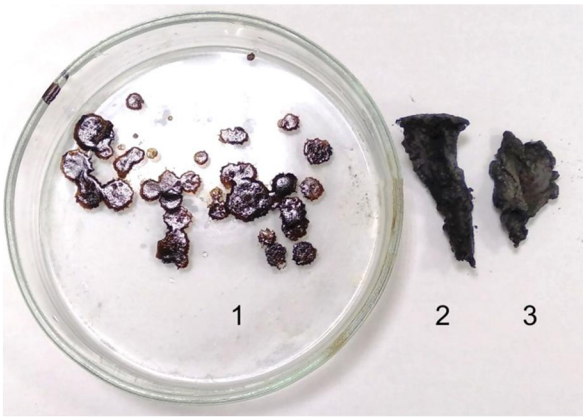

Figure 3 Resistance of polymer films to open flame: 1 - residue after splashing the polymer film melt without additives; 2 - carbonized residue of the polymer film with the addition of phytic and citric acids in a ratio of 1/1; 3 - carbonized residue of the polymer film with the addition of phytic and citric acids in a ratio of 3/2

5 CONCLUSIONS

Samples of polymer films with additives of phytic and citric acids showed the presence of a significant charred residue after combustion compared to unfilled film. It was also noted that there was no dripping of the polymer melt, which eliminated the potentially destructive effect that could cause additional hot spots.

Further research will be directed towards the development of composite environmentally friendly fire-protective finishing compositions for textile materials.

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DEVELOPMENT OF HYDRO-CENTRIFUGAL METHOD OF FORMING WOMENS HEADWEAR

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Abstract: *The article is devoted to the development of a new hydro-centrifugal method of forming the heads of women's headwear from fabrics of suit and coat assortment. As a working environment, it is proposed to use a liquid-active working environment (LAWE) for the formation of complex spatial forms. Theoretical substantiation of the formation method is performed by developing a physical model of the process. A method, technique and equipment for hydro-centrifugal formation of women's headwear have been developed. The research of the method by conducting one-factor and multifactor experiments was performed and the optimal parameters of formation were established. The influence of main input factors on the quality of formation of headwear parts was investigated. The adequate mathematical models of the second order of formation process were obtained. The rational parameters of the hydro-centrifugal forming process, in which the best quality of formation is achieved, were identified for each fabric.*

Keywords: *hydro-centrifugal forming method, LAWE, women's hats, formation, methods of formation, coefficient of form stability, women's headwear.*

1 INTRODUCTION

During the existence of mankind, fashionable costume has undergone many transformations. A large number of inspirations for the costume [1, 2] created a multifaceted palette of shapes of various items of clothing. This is especially true for a women's clothing. Modern fashion is characterized by variety forms of women's hats. The most difficult in the technology of forming garments is the way to obtain a three-dimensional shape that approaches the surface of hemisphere. The complexity is due primarily to the anisotropy of properties of the fabrics, which is manifested in different stresses of material at the base, weft and at an angle to them in the deformation process [3]. The final shape of the part, obtained in the forming process, can be different from the shape of the forming element. Recently, in the field of formation, scientists are working in the development of a new ways, which take into account the deformation properties of textile materials in order to ensure a given shape. Formation of clothes details can occur according to application of classical and non-traditional methods of humid-thermal treatment (HTT) [4]. The classic methods of formation are: with the use of irons, presses and steam-air mannequins (formation in the field of static loads). But they have a number of disadvantages [5]: different pressure of pillow surfaces on a different parts of textile materials; low mobility of the rough structure of textile materials; mismatch of the forms of a top and bottom pillow; impossibility of using a different pillow for the manufacture of products; small change of angles between systems of threads; deterioration of the quality of formation and increase in energy consumption due to the use of a small number of forming surfaces. Given these shortcomings of the classical methods of forming, it is justified to search for non-traditional, alternative, technologies for the formation of garments and hats (formation in the field of dynamic effort). The development of methods for forming parts of three-dimensional garments is accompanied by the search for ways to improve the forming process, from which we can identify four main areas [6-8]:

1. change of a number of forming surfaces and their location;
2. search for an alternative (rational) nature of the forming effort;
3. search the ways of force on the fabric without contact with the working bodies of the equipment;
4. search of an effect of firming force on the external and internal surface of the fabric.

In this case, the deformation of the textile material can be performed in different working environments and when using different in nature loads - forming methods.

In the works [4, 5, 8-13] the author's found that the dynamic methods of formation require attention in the study of the process of forming parts of garments and hats. They allow to a more efficient transformation of a flat textile material into a three-dimensional part by increasing the mobility of rough structure of the fabric. Also - changes occurring at the level of fine structure.

Among the known technical solutions should be noted the use of the centrifugal method of HTT garments [14-17]. It allows you to get rid of metal-cushions, reduce total costs compared to the press and increase productivity by 90%. It should be noted that in this method of forming the working environment is a pair in combination with a microwave, and also use one forming element.

The membrane method of a forming garment details is also original [18-21]. The peculiarity of this method is that the desired shape of the part is provided when using a heat-resistant elastic membrane. It is located under a rigid heating surface, which receives air of constant or pulsating pressure. In this case, the elastic shell, which is filled with air, provides a uniform force load on the material and increases contact with the forming surface. The greatest treatment effect is achieved in 25-30 cycles at loads of 0.02 MPa less than in static forming methods. The disadvantage of the membrane method of forming of garments should be considered the lack of interaction of the working environment, with the being processed fabric, as well as the unilateral action of the force load.

There is also a new technology of vibro-treatment using the energy of electromagnetic waves [22-25]. This forming method is based on the using a pulsating electromagnet field, created by the interaction of two spiral flexible coils: lower and upper. There are some disadvantages of such method: the presence of two forming elements; increased energy consumption of the method; humidification is performed using a spray, which effects on the forming quality. A method of forming, using dynamic load forces is also effective [23, 24, 26]. This technology is based on two ways of interaction of forming organs and parts of a clothing: vibrating and vibrating-impact.

According to the work data [27] dynamic methods of exposure on polymeric materials reduce the coefficient of friction between the fibers and threads of the fabric. This has a positive effect on the reconstruction of the rough structure of the material in the process of three-dimensional formation and provides the conditions for obtaining the spatial shape of the parts at low loads (p = 3…9·10^3 Pa).

Analysis [28] showed, that in order to successfully solve the problem of improving the quality of garments, reducing labor intensity and saving energy resources, it is necessary to develop a new special equipment and improve technological processes of HTT, based on the development of new ways of forming, finding a new working environments, etc.

Depending of the method of impact of forming load on material there are following types of forming: pneumatic, hydraulic, mechanical and combined. Modern technologies of the forming three-dimensional products from flat blanks provide a combination of different ways for creating the forming load [10]. The penetration of water into the fibers leads to decrease of the forces of internal interaction between the chains of molecules and, accordingly, to a decrease of the resistance of textile materials to forming forces [9]. In addition, it is advisable to use water as a plasticizer, because plasticization occurs without destroying the supramolecular structure of the material. There is a partial swelling of the material, which leads to a change in the diameter of the fibers [29].

Analysis of the formation methods [30-34], in which is used LAWE as a working environment, showed the features of solving a number of disadvantages of classical formation methods:

- there is no need for the second forming element – it's functions are performed by LAWE;
- lack of temperature action as the main factor of classical technology;
- the forming force is transmitted to the deformable fabric directly by the working environment, and not by the working elements of the equipment.

The existence of such methods of the formation of garment parts and three-dimensional parts of hats proves the feasibility of studying LAWE and finding ways to optimize its properties.

At the same time, scientists don't pay enough attention to the development of various methods of headwear forming. Existing works [35-40] describe only certain aspects of objective task that considered in the work. Therefore, there is a need for a study of aspects of hats formation more thoroughly.

Recently, scientists are interested by the method, which use centrifugal forces. This method in combination with the new LAWE working environment can be the basis for the development of energy-saving and low-operating technology, which will perform high-quality forming operations. Therefore, the development of a hydro-centrifugal method of forming the women's headwear is an actual question.

2 EXPERIMENTAL

2.1 Physical model of hydro-centrifugal method of headwear forming

The study of the proposed method of formation should be based on the actual reflection of the studied phenomena. For this a physical model was used, which allowed to study the influence of individual parameters on physical processes. The process of forming parts in LAWE is very complex, is continuous and fills the space in the working chamber and the structure of the material without voids and gaps. Due to its fluidity, it has forces evenly distributed over the volume [10]. Any volume of liquid can arbitrarily change its shape under the action of any small forces.

Peculiarities of the force field formation and the nature of its distribution on the forming element taking into account the fabric structure were investigated in [14, 41]. The obtained models illustrate the physical essence of the action on fabric of the phenomenon of centrifugal force in the process of formation. The development of a physical model of the process of centrifugal formation in LAWE is to analyze the action of forming forces applied to the fabric, taking into account the surface characteristics of the forming element. Therefore, it is necessary to determine the model of behavior of systems of fabric threads as well as the nature of the distribution of forming forces on the surface of the headgear during a centrifugal forming.

When rotating a cylindrical drum around the vertical axis of the OY with an angular velocity ω there is a centrifugal force F_B (1). It operates on each elementary section of the textile material δS of the part, which is located on the formation element and fixed to the wall. The centrifugal force *Fb(H)* is determined by the formula [42]:

$$F_B = m(1).\omega^2.R \quad (1)$$

where: *m(1)* - the mass of a single elementary section of textile material part, taking into account the moisture content [g/m^2]; ω^2 - angular velocity of the shaft, rotating the drum, on which the forming element and the part are fixed [rad/s]; *R* - smallest distance from the axis of rotation to a given elementary section of the material [m].

Since the working environment of the forming is LAWE, therefore it is necessary to determine the mass of a single elementary part of the material, taking into account the moisture content by the formula:

$$m(1) = M_s.w \quad (2)$$

where: M_s – surface density of textile material [g/m^2]; *w* - coefficient that takes into account the moisture content of the molded part.

The moisture content *w* of the textile material is determined by the formula:

$$w = \frac{m_B - m_C}{m_C}.100 \quad (3)$$

where: m_B - the mass of the part immediately after formation [g]; m_C - the mass of a completely dry part [g].

It is also necessary to take into account the angular velocity of the shaft with uniform rotation, which is as follows [42]:

$$\omega = 2\pi.n \quad (4)$$

where: *n* - rotation frequency (number of revolutions per 1 second) [s^{-1}].

Substituting in the formula (1) expression (4), where find:

$$F_B = 4m_{(1)}.\pi n^2.R \quad (5)$$

Consider the action of forming forces, namely the centrifugal force, as well as the interaction of the warp and weft threads of the textile material between themselves and the forming surface [14]. Based on the system of action of the deforming force F_V in the area of the warp thread, it is possible to determine the normal F_V^N and tangential F_V^T components:

$$F_V^N = F_V.\cos\varphi \quad (6)$$

$$F_V^T = F_V.\sin\varphi \quad (7)$$

The normal component of the centrifugal force F_V^N provides partial deformation of threads on thickness, without changing a network corner. In contrast to the normal tangential component F_V^T causes a shift of the main threads, resulting in a change in the magnitude of the network angle between the threads of the warp and weft. The magnitude of the shear deformation Δl largely depends on: the tangential component of the centrifugal force; the frictional properties; the physical and mechanical properties of the textile material. The change in the fabric thickness and especially its pile structure depends on the normal component, as well as the elastic properties of the elements of the textile material.

Due to the fact, that the surface of the forming element has a hemispherical shape, respectively, each elementary section δ_S of the textile material is at different distances *R* from the axis of rotation. Therefore, the magnitudes of the forming forces vary over the entire surface of the material. The smallest centrifugal force will be concentrated on the least convex part of the forming element, and the largest - at its base. Since the drum contains a liquid (LAWE), it is necessary to investigate the nature of its action on the forminf process. As the drum rotates at a certain frequency, the liquid (LAWE) in it will gradually gain the same angular velocity ω as the drum, and its free surface will change [43-45]. In the central part the level of RARS will decrease, and at walls - will increase and all free surface will become some surface of rotation (Figure 1).

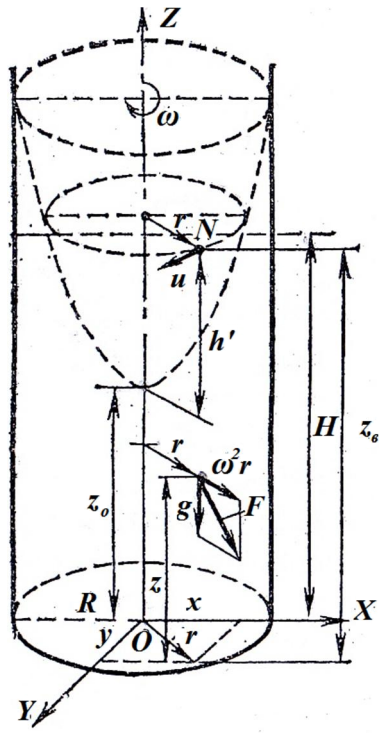

Figure 1 Determination of the free surface of a liquid rotating in an open cylindrical container: *OXYZ* -coordinate system in which the cylindrical container is placed; *R* – the radius of the cylindrical tank; *ω* - the angular velocity of rotation; *H* - fluid level at rest; z_0 - the vertex of a free surface of liquid; z_v - coordinate of a free surface of the liquid; *N* - an arbitrary point on a free surface

When considering the motion of a liquid, it is broken down as solids into individual small elements [46-48]. To describe the motion of a liquid, we select an arbitrary liquid volume *T*, limited by the surface *S*. Write for him an expressing equation - the law of movement quantity: the time derivative of amount of system motion is equal to the sum of external forces, acting on it. This equation is an integral form of the fluid motion equation. Using the formulas of vector analysis, we obtain the differential form of the fluid motion equation:

$$\rho\overline{F}+\frac{\partial\overline{p}_x}{\partial x}+\frac{\partial\overline{p}_y}{\partial y}+\frac{\partial\overline{p}_z}{\partial z}=\rho\frac{d\overline{u}}{dt} \tag{8}$$

This equation is a vector form of the equation of motion of a fluid in stresses, which is equivalent to three equations in the projections on the coordinate axes x, y, z, having the form:

$$\begin{aligned}
F_x+\frac{1}{\rho}\left(\frac{\partial p_{xx}}{\partial x}+\frac{\partial p_{yx}}{\partial y}+\frac{\partial p_{zx}}{\partial z}\right)&=\frac{du_x}{dt};\\
F_y+\frac{1}{\rho}\left(\frac{\partial p_{xy}}{\partial x}+\frac{\partial p_{yy}}{\partial y}+\frac{\partial p_{zy}}{\partial z}\right)&=\frac{du_y}{dt};\\
F_z+\frac{1}{\rho}\left(\frac{\partial p_{xz}}{\partial x}+\frac{\partial p_{yz}}{\partial y}+\frac{\partial p_{zz}}{\partial z}\right)&=\frac{du_z}{dt}.
\end{aligned} \tag{9}$$

The height at which the point of free surface of liquid (arbitrary point N) is raised above the vertex of paraboloid is equal to:

$$h'=z_B-z_0\frac{\omega^2r^2}{2g} \tag{10}$$

Ordinate z_0 the vertex of paraboloid of free surface at a given angular velocity depends on volume of liquid in the tank. If before the rotation of a tank the liquid level was horizontal and set at a height *H*, then the volume of liquid was equal to πR^2H. As the drum rotates, a free surface becomes parabolic, the shape of liquid volume changes, and its value at density ρ=const remains unchanged:

$$\int_0^R(z_0+\frac{\omega^2r^2}{2g})2\pi r dr=\pi R^2H \tag{11}$$

After integration, get:

$$H=z_0+\frac{\omega^2R^2}{4g} \tag{12}$$

Or

$$z_0=H-\frac{\omega^2R^2}{4g} \tag{13}$$

Assuming that z_0=0, then it is possible to find the angular velocity *ω*, in which a free surface of the liquid will touch the bottom of drum:

$$\omega=2\frac{\sqrt{gH}}{R} \tag{14}$$

Substitute the obtained data into the equation of equilibrium of incompressible fluid with density *ρ,* which is a consequence of transformations of the equations of motion of Euler fluid:

$$d_p=\rho(Xdx+Ydy+Zdz) \tag{15}$$

where *p* – hydrostatic pressure.

$$dp=\frac{\partial p}{\partial x}dx+\frac{\partial p}{\partial y}dy+\frac{\partial p}{\partial z}dz$$

Then get:

$$d_p=\rho(\omega^2xdx+\omega^2ydy-gdz) \tag{16}$$

After integrating equation (15) obtain:

$$p=\rho(\frac{1}{2}\omega^2r^2-gz)+C_1 \tag{17}$$

Performing mathematical transformations and substituting a found value C_1 in (16), obtain:

$$p=p_{am}+\rho g(z_0-z)+\frac{\omega^2r^2}{2g} \tag{18}$$

Knowing that $h'=\frac{\omega^2r^2}{2g}$ for any point it is possible to use the obtained results and write in the form:

$$p=p_{am}+\rho g\rho(z_0-z+h')$$

or

$$p=p_{am}+\rho gh_з \tag{19}$$

where $h_з$ – depth of immersion of a point under a free surface or a distance, measured vertically from a parabolic surface to the point under consideration.

Thus, in a liquid that is in a cylindrical container, the rotating pressure is distributed vertically by the hydrostatic law. According to Pascal's law, the value is the same for all points of fluid volume. So, given the property of hydrostatic pressure, it can be affirming, that the pressure applied to a free surface of the liquid is transmitted to all points of this liquid in all directions equally.

If limit the top of the open cylindrical container with a lid, with increasing angular velocity, the liquid near the side walls will begin to rest on its upper part, creating a hydrostatic pressure (Figure 2). In this case, the above law of pressure distribution is also valid.

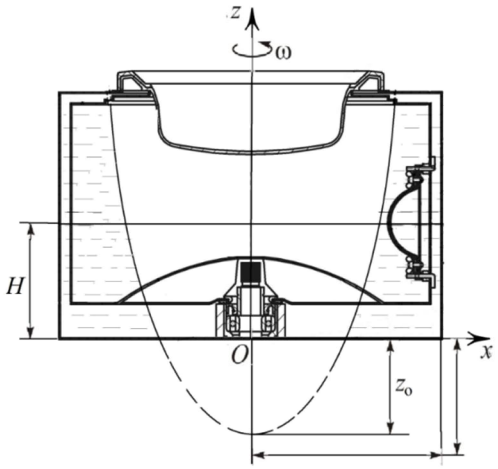

Figure 2 Determination of free surface of fluid rotating in a closed and cylindrical cavity: *OYZ* - coordinate system, in which the cylindrical capacity is placed; *R* - radius of cylindrical capacity; *ω* - angular velocity of rotation; *H* - fluid level at rest; z_0 - the top of free surface of the liquid

Therefore, the physical essence of the improved centrifugal forming method using LAWE as a working environment is in action on the textile material, which is fixed on the forming element by centrifugal forces and circular flows of LAWE, which moisten the material and create additional forming force in the form of hydrostatic pressure.

2.2 Materials

A quality of forming operations primarily depends on the properties of material from which the garment is made. For manufacture of hats use different textile materials that differ in structure, fibrous composition, physical, mechanical and operational properties. There are: coat, suit fabrics, knitted fabrics, artificial and natural leather, suede and fur, velour, felt, duplicated materials, cotton, linen and silk fabrics.

One of the main properties that effect on forming process is a forming ability of the textile material, scratchiness and stiffness. They must be taken into account when choosing materials for headwear manufacture. A material and its structure significantly influence on forming ability. The characteristics of the structure of textile materials on which the forming ability depends are:

- fibrous composition;
- thickness;
- density of material by warp and weft (number of main threads or weft threads located per 100 mm of length or width);
- linear density;
- surface density;
- linear filling (characterizes the density of fabric as a percentage of the maximum possible taking into account the thickness of threads and shows, what part of the fabric area is filled with parallel threads of the warp and weft);
- surface filling (indicates which part of the fabric is filled with threads of a two systems, taking into account the overlap of threads on each other when weaving);
- volumetric filling (shows what part of the volume of fabric is the volume of warp and weft threads);
- three-dimensional mass (shows the weight per unit volume of fabric);
- filling by weight;
- total porosity (characterized by the volume of fabric that is not filled with fiber);
- binding.

Structural characteristics in a complex determine the structure of fabric and effect on its physical and mechanical properties: strength, elongation, rigidity, drapeability, immutability, hygroscopicity, change of linear dimensions at HHT, ability to form, etc. The formation of textile materials is possible due to the fact, that they occupy a significant volume of air (the surface density of most fabrics doesn't exceed 500 g/m^2, porosity is close to 50-80%), and also the presence of mobile and stable connections in a structure of the textile material. With a decrease in the density of textile material, but with the same thickness of threads and weave, with a decrease in the number of threads by 10 cm, the fabric becomes more mobile.

For the formation of textile materials using LAWE as a working environment, it is proposed to use medium-density fabrics of linen, satin, satin or twill weaves with long overlaps, from hardware yarn, containing natural (preferably woollen) and chemical fibers. To study the centrifugal method of formation in LAWE, three fabrics of suit and coat range were selected, which are presented on the modern market and are in demand among consumers. These fabrics are used to make hats that can be combined with other garments (suits, coats), and thus create a complete ensemble. Selected textile materials are different in weave, fibrous composition, linear and surface density, thickness, etc. This will make it possible to investigate the proposed method of formatting for different fabrics, as well as to choose the optimal parameters of the forming process.

2.3 Experimental equipment

To implement an improved a centrifugal method of forming the textile materials, due to the use LAWE as a working environment, was developed experimental equipment, the scheme of which is presented on Figure 3.

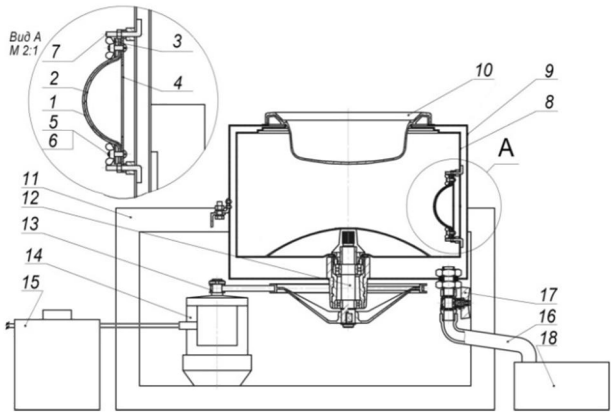

Figure 3 The scheme of experimental equipment for formation of the textile materials by hydro-centrifugal method: 1 - textile material; 2 - forming element; 3 - clamping ring; 4 - fastening platform; 5 - nut; 6 - screw; 7 - bolt; 8 - drum; 9 - forming chamber; 10 - camera cover; 11 - installation frame; 12 - shaft; 13 - belt drive; 14 - electric motor; 15 - laboratory transformer, 16 - valve; 17 - pipeline; 18 - tank

The main characteristics of developed experimental equipment are presented in the Table 1.

Table 1 Main characteristics of the experimental equipment

Characteristic	Specifications
Number of formation elements	3 psc
The height of the formation element	39 mm
The mass of one formation element	38.5 g
Drum speed	0…14 s^{-1}
LAWE volume	12…20 l
Laboratory autotransformer	RNO-250-2
Electric motor	INDESCO 1000 A
Overall dimensions of the drum diameter and height	470 mm 260 mm

Developed equipment provides a qualitative formation of headwear hats in LAWE. According to the active action of such environment provides effect on the fabric structure in the drum under pressure. This allows to form a stable shape of the hat in a relatively short period of the time, compared to the classic methods of hats forming. Developed equipment is presented on Figure 4 and shape fixation mechanism on Figure 5.

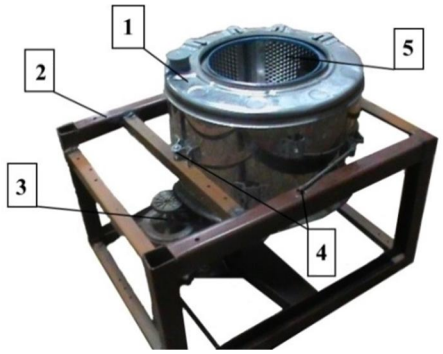

Figure 4 External view of developed experimental equipment of hydro-centrifugal method of headwear forming: 1 - forming chamber; 2 - installation frame; 3 - electric motor; 4 - drum fastening to the equipment; 5 - drum

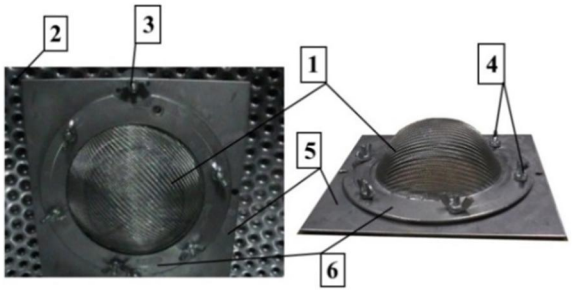

Figure 5 External view of form fixation element: 1 - forming element; 2 - drum; 3 - fixation of fastening platform; 4 - nut with screw; 5 - fastening platform; 6 - clamping ring

The fabrics used in the studies have their own characteristics (Table 2). The essence of the proposed method of formation on developed equipment is to action on the textile material by centrifugal forces and circular flows of LAWE, which moisten the material and create additional forming force.

Table 2 Physical and mechanical characteristics of fabrics

Characteristic	Fabrics type			
	Costume fabric №1	Costume fabric №2	Coat fabric №1	Coat fabric №2
Fibrous composition	100% wool	100% wool	78% wool 22% polyester	78% wool 22% polyester
Surface density M_s [g/m^2]	214	263	315	345
Number of threads per 10 cm, warp P_{wp}	158	229	138	154
Number of threads per 10 cm, weft P_{wf}	131	194	126	130
Binding type	twill 2/2	twill 2/2	twill 1/2	twill 2/2
Linear density of warp threads T_{wp} [tex]	75	25x2	100	120
Linear density of weft threads T_{wf} [tex]	75	54	102	124

2.4 Research method

Investigated method and equipment for forming parts of garments from textile materials under the action of a centrifugal force field and LAWE includes placing a sample of material on the forming element, fixing in the chamber for forming, wetting, and formation. Sampling of materials for forming and their cutting is performed from a roll or piece of fabric across its width with observance of parallelism of threads at cutting. Experimental investigations were performed using the method of small samples with a diameter of d = 170 mm. At least 3 tests are required to conduct experimental studies with an average guarantee error of 3% and a confidence level of 0.95.

The flat spot sample of textile material is fixed on a perforated forming element and a platform for fastening by means of a clamping ring. The platform with the forming element and the material is placed in the forming chamber on the bolts, fixed on the perforated wall of the drum. Then the chamber is filled with a certain volume of LAWE (t = 18-20°C) and closed with a lid. Through the laboratory autotransformer a voltage is applied to the AC motor, which provides rotational movements of the drum through the belt drive. The drum and forming elements rotate with a certain frequency, which is set by the regulator on the previously set scale of speeds, located on the transformer. The formation of the textile material takes place within the set time due to the action of centrifugal force and circular flows of LAWE, which moisten the material and create additional forming force.

3 RESULTS AND DISCUSSION

To verify the theoretical provisions set forth in the physical model of the process, a comprehensive experimental study was performed in terms of the choice of input parameters, their range, and their combined effect on the quality of the process.

In the work [14] to study the centrifugal method of forming a working environment which was a steam, the main input controlled parameters were the speed of drum rotation and steam pressure. A drum speed, vapor pressure and steaming time were used as input factors influencing on the centrifugal forming process.

For a preliminary study of the hydrocentric method of textile formation in LAWE on the experimental equipment, the input controlled factors were chosen: drum speed $n(x_1)$, LAWE volume $V(x_2)$ and forming time $t(x_3)$. The initial function and the criterion for optimizing this forming process is the coefficient of form stability K. Determining the influence of each of the factors separately on the process of centrifugal formation and their quantitative indicators was carried out in the framework of one-factor experiments. Levels and intervals of parameters variation that effect on the hydrocentric method of formation in LAWE are selected on the basis of literature sources [14], as well as based on the physical capabilities of this experimental setup are shown in Table 3.

Table 3 Levels and intervals of factors variation of influencing the process of centrifugal formation

Levels of variation	Parameters of the forming process		
	Frequency rotation n [s^{-1}] x_1	***LAWE volume*** V [l] x_2	***Formation time*** t [s] x_3
+2	14	20	180
+1	12	18	150
0	10	16	120
–1	8	14	90
–2	6	12	60
Interval of variation	2	2	30

Similar studies of the hydrocentric forming method were conducted for the three fabrics of suit and a three coat assortments, selected in the previous section. In the experimental study of coat fabrics, the regression equations of the dependences between selected input parameters and a coefficient of form stability K were obtained. This is done as a result of analysis of the influence of each factors separately on the process of centrifugal method of forming the parts from textile materials. They adequately characterize the studied process, which is confirmed by the reliability of the approximation. Regression dependencies $K = f(x_i)$ between the rotational speed, the volume of LAWE, the time of formation and a coefficient of shape stability, are described by polynomials of the third degree and are presented in the Table 4.

Table 4 Regression dependencies $K = f(n)$, $K = f(V)$ and $K = f(t)$ in the study of coat fabrics

Process factors	Regression equation	Reliability of approximation
Rotation speed [s^{-1}]	$K = 0.89 \cdot 10^{-3} n^3 - 0.02774 n^2 + 0.26508 n - 0.6974$	$R^2 = 0.99$
LAWE volume [l]	$K = 0.13 \cdot 10^{-3} V^3 - 0.00618 V^2 + 0.08509 V - 0.25989$	$R^2 = 0.98$
Formation time [s]	$K = -0.004.10^{6} t^3 + 0.003 \cdot 10^{-3} t^2 - 0.29 \cdot 10^{3} t + 0.0726$	$R^2 = 0.98$

Graphical dependences of the coefficient of form stability on main parameters of the process in the study of centrifugal forming method parts of hats from coat fabrics are presented in Figures 6-8.

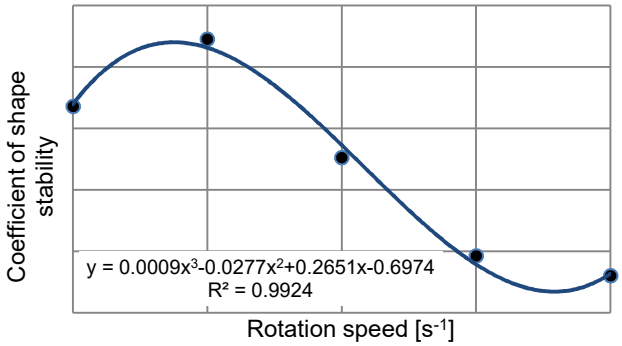

Figure 6 Dependence of the coefficient of stability on the rotation speed in the study of coat fabrics

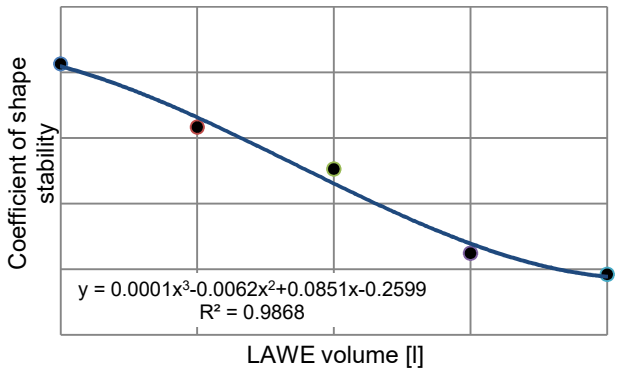

Figure 7 Dependence of the coefficient of stability on the volume of LAWE in the study of coat fabrics

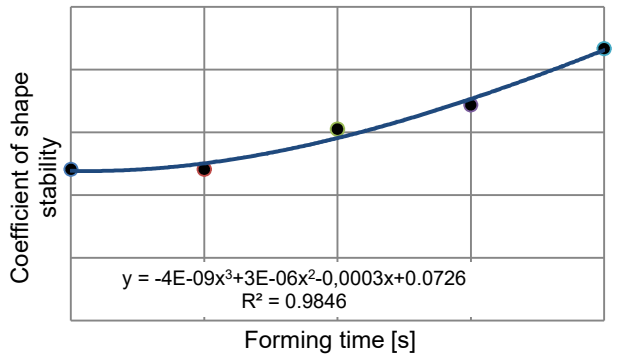

Figure 8 Dependence of coefficient of form stability on the formation time for a coat fabric in the study of coat fabrics

Analysis of the influence of each factors on the process of forming textile materials, which were studied, showed that the curves of dependence of shape stability coefficient on the main factors have the same character. Considering influence of a speed on the parts formation from textile materials by centrifugal method (Figure 4), there is a decrease in the coefficient of form stability with increasing factor. That is, the shape of the part is improved. Increasing the values of rotational speed leads to an increase in the forming forces acting on external and internal connections of textile material. This improves deformation properties of the studied fabrics, and confirms theoretical assumptions.

The graphical dependences (Figure 5) showed that increase in the volume of LAWE is accompanied by decreasing in the coefficient of shape stability. Thus, with increase of this factor the formation process is more intensive, due to increase of moisture content of textile material, which improves its deformation properties. It is determined that with increasing a forming time there is an increase in the coefficient of form stability and deterioration of the quality of formation operation (Figure 6). The results of experimental studies allow to conclude that the factors of centrifugal forming process - the speed, volume of LAWE and forming time significantly effect on the quality of molded part.

As can be seen from the graphs (Figures 4-6) and regression equations (Table 3), the relationships between input factors and a coefficient of shape stability are nonlinear and do not clearly indicate the importance of influence of parameters on this process. Therefore, a multifactorial experiment is required in the future. The analysis of one-factor dependences indicated that it is possible to narrow the ranges of the studied factors for a multifactor experiment. Narrowing of ranges is caused by inexpediency of use of process outside certain limits. This is due to the fact that the lower indicators of the selected parameters do not provide the appropriate quality, and higher indicators are not rational to use due to increased energy consumption with a slight increase in quality.

According to the graph (Figure 4) by changing the rotational speed of 6 to 14 s^{-1} it is possible to reject a range of values up to 8 s^{-1}, because the experimental equipment has not yet gone into operation and LAWE is moving chaotically. This is not a managed process because after 13 s^{-1} increased energy consumption. As the graphical dependence shows (Figure 5), analyzing the influence of LAWE volume, the range of values up to 14 l was rejected and after 19 l. The area of studied factor was narrowed from 13 to 18 l. The studied range of formation time (Figure 6) indicates that it is possible to reject the range of values up to 90 s and from 150 s, because with increasing duration of formation the quality does not improve. To perform a multifactorial experiment, were used the calculation matrices of the second-order rotatable plan, five levels of variation of parameters that effect on the forming process. The range of values of input parameters was adjusted on the basis of previous studies and formed the basis of a multifactorial experiment (Table 5).

Table 5 Levels and intervals of factors variation for study of coat fabrics

Levels of variation	Parameters of forming process		
	Frequency speed n [s^{-1}] *(x_1)*	*LAWE volume V* [l] *(x_2)*	*Forming time t* [s] *(x_3)*
+1.68	13	19	150
+1	12	18	138
0	10.5	16.5	120
–1	9	15	102
–1.68	8	14	90
Interval of variation	1.5	1.5	18

According to the results of experimental data of studied fabrics, a special program "Planning experiment" was used to determine the coefficients of regression equation, as well as to derive the regression equation of mathematical model of the centrifugal forming process according to the specified parameters. According to the selected plan of the experiment, the number of experiments was obtained $N = 15$, number of input factors $n = 3$, the number of repetitions of each experiment $k = 3$. On the basis of experimental researches of centrifugal formation of coat fabrics received the regression equation of mathematical model of process in the coded form:

$$y = 0.0601 - 0.0096x_1 - 0.0035x_2 + 0.0014x_3 + 0.0017x_1x_2 - 0.0094x_1x_3 + 0.0071x_2x_3 + 0.0053x_1^2 - 0.0030x_2^2 + 0.0188x_3^2 \quad (20)$$

To determine the weight of parameters of process a centrifugal formation of coat fabrics was substituted a values of factors x_1, x_2, x_3 (+1.68; 0; -1.68) in the regression equation (20) with the stabilization of two factors at zero level. Based on this, one-factor graphical dependences are built $K = f(x_1)$ and defined the influence of each of the factors on original function, that is, the coefficient of form stability.

The analysis of the obtained results shows that an indicator of form stability coefficient K is greatest influence by the duration of formation ($K = 0.0538$). The value of duration x_3 is at a minimum level -1.68. The next most important parameter is frequency speed, at maximum value +1.68, a coefficient of stability of which is $K = 0.0566$. Indicator value $K = 0.0575$ is achieved when the volume of LAWE is at the maximum level +1.68.

After conducting, an experimental research in one-factor and multifactor experiments were rationalized parameters of the process of hydro-centrifugal forming method. As a criterion for optimizing a process of centrifugal formation of textile materials, the coefficient of form stability was chosen. It is given by a function that goes to the minimum value. To determine the extreme (minimum) value of the original function of mathematical model, the equations are differentiated for each independent variable. The construction of response surfaces of the coefficient of shape stability from the input parameters (speed, volume of RARS and time of formation) gives the opportunity to get a clear idea of patterns of change of this optimization criterion [39].

For this purpose, the analysis of response functions was performed and was carried out rationalization of formation process. A task of rationalization was to find the values of factors that belong to the range of permissible values $x_i \in$ [-1.68; 1.68], at which output parameter has minimum value and condition is fulfilled $y \leq 0.2$.

Analyzing the nature of response surface of stability coefficient of from parameters of frequency of rotation and volume of water with the stabilization of the factor of formation time at the level $x_3 = 0$ ($t = 120$ s) in the study of coat fabrics, following conclusion can be drawn: the best quality of formation occurs at values of parameters of rotation frequency $n = 13$ s^{-1} and LAWE volume $V = 19$ l, as evidenced by the minimum value of optimization parameter (coefficient of stability) $K = 0.055$ (Figure 9).

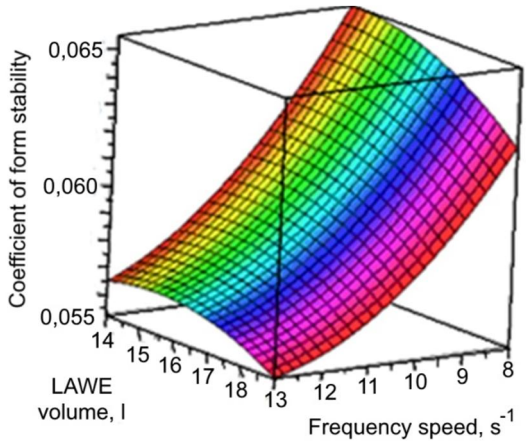

Figure 9 Dependence of response function of frequency speed and LAWE volume at $x_3 = 0$ ($t = 120$ s) in the study of coat fabrics

When analyzing the response surface of LAWE volume and formation time when stabilizing a speed at the level $x_1 = 0$ ($n = 10.5$ s^{-1}) for coat fabrics, it is determined that the most stable form is obtained at the following values of parameters – forming time $t = 90$ s and LAWE volume $V = 19$ l, as indicated by the minimum value of stability coefficient $K = 0.050$ (Figure 10).

Considering the nature of response surface of frequency speed and formation time at $x_2 = 0$ ($V = 16.5$ l) for coat fabrics, it is possible to draw a conclusion about optimal modes of formation to achieve the best quality of forming part, which constitute $t = 90$ s and $n = 12$ s^{-1} when the value of the optimization criterion $K = 0.053$ (Figure 11).

The graphical dependences of response function on Figures 9-11 reflect the complex influence of input parameters of centrifugal forming method on the coefficient of form stability. The optimal values of the response functions are represented by minimum that is declines of surfaces. Analysis of these graphic illustrations makes it possible to state that the speed, volume of LAWE and formation time significantly effect on the value of quality indicator of forming process of a coat fabric by centrifugal method.

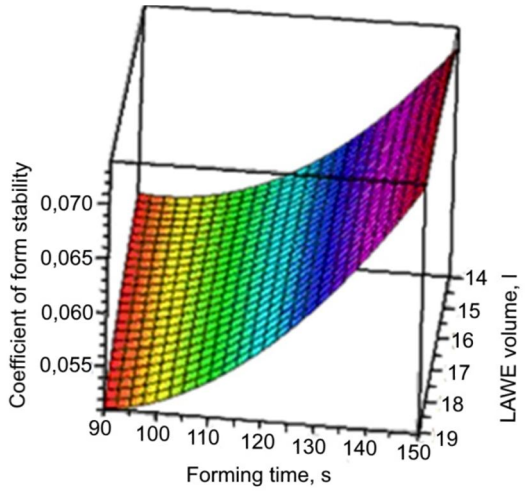

Figure 10 Dependence of response function of frequency speed and LAWE volume at $x_1 = 0$ ($n = 10.5$ s^{-1}) in the study of coat fabrics

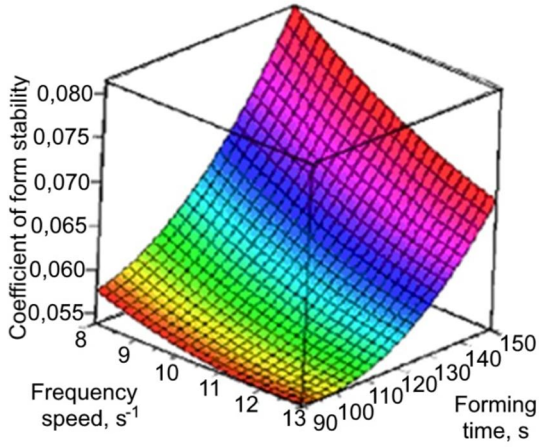

Figure 11 Dependence of response function of forming time and of frequency speed at $x_2 = 0$ ($n = 16.5$ s^{-1}) in the study of coat fabrics

Therefore, the rational parameters of the process of centrifugal forming in LAWE for this fabric are: frequency speed $n = 12.5$ s^{-1}; LAWE volume $V = 19$ l and forming time $t = 90$ s, as evidenced by the minimum value of the coefficient of stability K.

4 CONCLUSIONS

A hydro-centrifugal method of forming three-dimensional parts of women headwear has been developed by replacing the working environment of steam with LAWE. To implement this method the experimental equipment was developed for forming the details of three-dimensional shape of hats and the method of experiment conducting.

A physical model of the hydro-centrifugal forming method has been developed. The choice of factors for the formation process and materials for research is substantiated. The choice of the method of assessing quality of forming process using the value of coefficient of shape stability is substantiated.

Investigated the influence of main input factors on the quality of formation of headwear parts: for fabrics frequency speed $n = 8$-13 s^{-1}; LAWE volume $V = 14$-19 l and forming time $t = 90$-150 s. As a result of statistical processing data of multifactorial experiments the adequate mathematical models of the second order of formation process were obtained. For each fabric were identified rational parameters of the centrifugal forming process, in which the best quality of formation is achieved and they are: for coat fabric frequency speed $n = 12.5$ s^{-1}; LAWE volume $V = 19$ l and forming time $t = 90$ s with $K = 0.053$.

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THE HISTORICAL TIMELINE OF NIAS WAR ARMOR MATERIALS DEVELOPMENT AND TECHNOLOGY

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Abstract: *This study aims to unravel the relationship between technological developments, material aspects, and historical events in the form of clothing material's function in Nias war armor. The warlike culture of the Nias people has historically emphasized the importance of war costume and weaponry. The raw material, external influences, and technology is evident in Nias war armor reflect the development of civilization on the island. Data collection was carried out by literature review, interview, field observation, and descriptive analysis. This study observes that Nias war armor was made from natural materials such as Oholu tree bark, palm fibers, pandanus leaves, animal skin, iron, and tin. The use of material in Nias armor is highly adaptive, addressing the soldiers' needs for protection, resistance, comfort, and self-expression. This study also provides new insights into the adaptability of Nias war armor throughout history.*

Keywords: *historical timeline; material; Nias war costume.*

1 INTRODUCTION

This research addresses the question of how the material of Nias war armor developed over time through analysis of raw material, external influences, and technology. Nias war armor was used as a protective gear, however, today, the Nias war armor is used as a costume for welcoming tourist. The diversity of material used in Nias war armor lends particular interest to its historical timeline that has yet been discovered. Through the examination of Nias war armor, this study aims to unravel the relationship between technological developments, material aspects, and historical events in the form of clothing material's function.

Clothing reflects the culture and characteristics of a civilization. The emergence of new ways of dressing can mark a turning point in a culture's history by introducing new values to the social structure [1]. Clothing is a response to political, economic, artistic, and war conditions. Analyzing the historical development of clothing can reveal the circumstances that a society experienced. Clothing is a product of user-oriented development. The objective of a garment can be divided into practical and symbolic values. In the case of war clothing, the material serves a pragmatic purpose: to provide optimal protection and agility in combat [2]. Based on its material, war clothing can also show a function of symbolic value based on the traditional beliefs of a region.

Indonesia expressed its regional cultural values through traditional clothing. The significance of traditional clothing is tied to the cultural values embedded within it. These values are related to economic, social, and political aspects of life which have been translated into visual structures, materials, colors, and patterns.

Nias, a remote island located west of Sumatra, Indonesia (see Figure 1), is often forgotten; therefore, its civilization has developed in relative isolation from other parts of Indonesia. Over time, Nias became known as a warlike society that prioritized innovation in armor and weaponry [3]. The evolution of Nias war costume is evident in its varied material and technology. It is crucial to understand the history, purpose, and technology of traditional war clothing to recreate its design. Due to its isolation, Nias offers an interesting contribution to the discussion of cultural development through clothing. During the colonial period of Indonesia, Nias was not a prime area of interest for the Dutch colonists because it lacked spices. Historically, Nias has never enjoyed a high profile in the cultural, political, or economic realms of Indonesia. Moreover, Nias did not play a role in the history of the great kingdoms that once ruled the archipelago, namely the Sriwijaya Kingdom (in power from the seventh to the eleventh century) and the Majapahit Kingdom (in power from the thirteenth to the fourteenth century) [4]. At that time, Nias was described as an independent island by Arabic, Chinese, and Indian merchants [5]. The people of Nias have been isolated and often forgotten by the Indonesian central government due to their remote location [6]. However, this environment is ideal for performing a case study of Indonesian culture.

Figure 1 Map showing the location of Nias Island within Indonesia. Courtesy of Google Maps

Today, Nias remains an independent society that still practices megalithic traditions and creates stone crafts, wood carvings, and earthquake-resistant architecture. This expertise is a valuable asset to Indonesian culture. Therefore, documentation and analysis are necessary to highlight the importance of addressing social changes in Nias in order to preserve its culture and facilitate its continued progression.

Nias culture was built on a unique community system of war society and social hierarchy. Clans and families in Nias were separated based on distinct social classes: (1) *si'ulu*, meaning "those who are on high", namely the highest customary leaders or aristocrats who descended from the village founders; (2) *si la* ('he who knows'), the chief's advisors; (3) *ere*, or religious leaders; (4) *sato*, which means village children, or ordinary people; (5) *sawuyu*, the slaves who previously belonged to the aristocracy and lived outside the village [6-8].

War is a tactic typically employed to expand territory, property, and dignity. War in Nias was motivated not only by political or group factors, but also by the personal conflicts of the villagers [6]. In the Nias language, the concept of *ëfavanusa ndröra banuas* states that patriotic feelings of the village are more important than kinship relations. As a result, the villagers would fight to defend their village's honor. War was a constant presence in the daily life of the people of Nias. Therefore, the creation and innovation of weaponry and armor took priority over that of agricultural equipment. The abundant evidence of armor and weapons of war in Nias confirms that the development of war equipment was always prioritized. The war clothes demonstrated the pride of the soldiers, which is evident from the quality of the materials and the detailed decoration in the clothes. The society adapts and develops material and technology in making cloths for armor throughout history. It is evident that developments material and technology of Nias war armor through historical approach is suitable but the relevant data on this topic is limited.

Through further analysis of Nias war armor, the response of the Nias people to their environment can be revealed.

2 RESEARCH METHOD

This qualitative research aims to construct a timeline of material usage in Nias war armor through analysis of raw materials, external influences, and technology. The primary data have been collected from the online archives of the Nationaal Museum van Wereldculturen, a comprehensive museum organization for the management of several ethnographic museums in the Netherlands, founded in 2014 that consists of the Tropenmuseum in Amsterdam, the Museum of Africa in Berg en Dal, and the Volkenkunde Museum in Leiden (https://collectie.wereldculturen.nl/); the database was searched using the keyword "Nias" and filtered by column (objects) and category (battle and war). Data was also gathered from the online archives of the Musée du Quai Branly – Jacques Chirac, Paris (https://www.quaibranly.fr/en/explore-collections/) using the keyword "Nias." The collected data regarding Nias armor material, history, and artifacts are primarily based on field research at Nias in 22 to 24th of August 2019 in the Nias Heritage Museum and Bawomataluo Village's. Data collected in the Nias Heritage Museum are capturing object images related to Nias war equipment and in-depth interview with Nata'aluhi Duha, director of the Nias Heritage Museum. This paper also interviews Dalizi̇̈hi Manaö, grandson of the Bawomataluo Village's warlord regarding Nias warlike culture and oral history of warriors and war implements. The secondary data were obtained through books, journals, and proceedings related to the present topic. Data collected from primary sources are compared also cross-checked with the secondary sources and discussed in this article.

3 RESULTS

3.1 Nias war armor: basic structure and function

The villages in the Nias area were considered one clan. Members of one large village could separate and construct a new village due to differences in principles or opinions with the village leader. Wars between the newly separated villages were a common occurrence. When fighting at night, the similar appearance of the opponent's war clothing would confuse both parties. To differentiate between groups, a village war unit would create a special sign recognizable only to soldiers from that village [9].

Traditional Nias war clothing was composed of five pieces of protection: the helmet or head protector; *kalabubu*, the protective necklace; armor; loincloth; and *baluse,* the shield. Weapons of the Nias warriors included the saber (with *ragö,* a rattan ball used to store amulets) and the rifle, which the Nias stole from the Dutch army (see Figure 2). Psychologically, this structure made the soldier's body appear more substantial and intimidating to his opponent [10]. This basic structure was worn by soldiers and nobles alike; the warrior's status was differentiated by a circular detail on the back of the neck, which offered extra protection (see Figure 3).

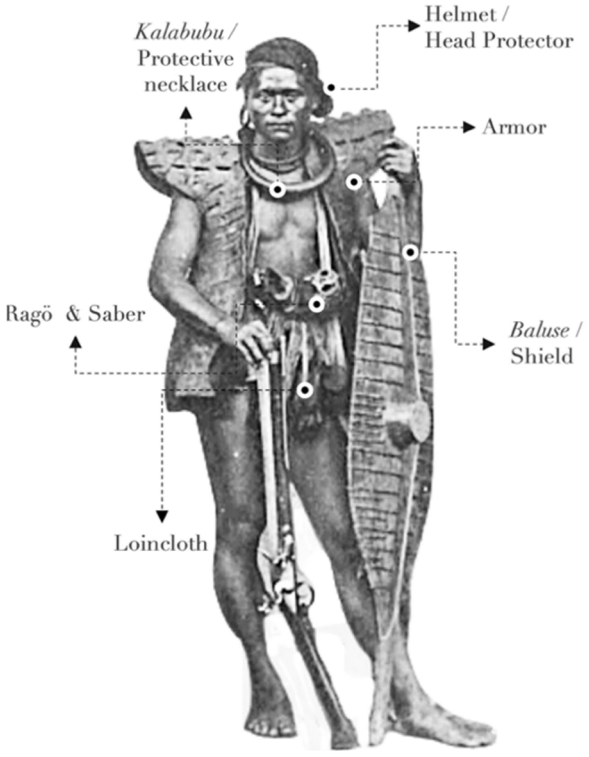

Figure 2 Basic structure of Nias armor. The basic structure consists of defensive tools: *kalabubu*, the protective necklace; armor; loincloth to protect vital body part; and *baluse*, the shield.; and attacking tools: sabres, and gun. This structure is seen on all Nias war armors (Collection Nationaal Museum van Wereldculturen. Coll.no. RV-A72-133, adapted by researcher)

Figure 3 The basic structure of the Nias war armor (flat sketch). It has a front opening, an A-line silhouette, a large shoulder section, and a circle detail on the neck. To differentiate the war armor and noble's vest is the circle detail on the neck. This detail gives a double protection for the warrior's neck alongside the *kalabubu* necklace

The Nias people recognized two types of war: *sifalau* and *sifatele.* In the case of *sifalau,* the enemy was attacked by surprise, while in *sifatele*, the war was prearranged. The significance of Nias war armor was evident during negotiations, encouragement before fighting, and battle. Before officially declaring war, the opposing parties would conduct negotiations to determine whether either side would concede defeat [8 - p. 457, 9]. In these meetings, the soldier, in all the splendor of his armor, would be seen by his opponent. Due to psychological influence, even a group with a tactical advantage could feel intimidated by its opponent (psywar) [10]. If an agreement could not be reached, and war was declared (*sifatele)*, the soldiers would gather to engage in morale-boosting rituals. Warlords and soldiers would shout, perform *hoho* (an oral tradition in the form of verse), and stomp their feet. These pre-battle rituals stimulated the soldiers' adrenaline and fostered a sense of group solidarity. Today, the Nias people reenact this tradition in the form of war dance to commemorate the greatness of their ancestral soldiers [11]. Before battle, war armor would be stripped of its decoration. The material of the armor provided the warrior with a form of defense, while its decoration offered him a means of self-expression. Nias war armor conveyed the identity of the wearer as an individual as well as his belonging to a group.

It can be concluded that Nias war armor served both external and internal functions. Externally, war armor could intimidate the soldier's opponent and offer protection for members of his village. Internally, war armor could bolster a soldier's self-confidence, foster a sense of belonging in a group, and display the wearer's creativity and craftsmanship.

3.2 Developments in materials and technology used in Nias war armor

Nias war armor has undergone several stages of material and technological development. The timeline of Nias war armor can be divided into

three stages: traditional materials and technology, foreign materials and technology. and a combination of traditional and foreign materials and technology. The technology for making war armor also developed alongside the materials used. In Nias language, armor that resembled a shirt was called *baru* (shirt), while more substantial armor was called *öroba* (armor). The Nias named war armor based on its material. This research mentions *baru oholu*, created from the Oholu tree; *baru leama*, braided from *leama* (palm fiber); *baru sinali*, weaved with *sinali* fiber (pandanus fiber), *öröba uli mbuaya*, created from *buaya* skin (crocodile skin); and *baru öröba*, known as the armor shirt (can be seen in Table 1).

Table 1 Analysis of the development of Nias war armors by year, name, material, phenomena and influence, ttechnology, material characteristics, image, and function

Year	Name	Phenomena & influence	Material	Technology	Material characteristics	Image	Function
12.000 BC	*Baru Oholu*	Simple techniques to protect the body, Hòa Bình and Toala cultural influences	Oholu tree bark	beating with a rock	thin, light		protection
5.000-4.000 BC	*Baru Leama*	The Austronesian race from Taiwan via the Philippines	palm fibres	braiding	thick, coarse, heavy		resistance
500-100 BC	*Baru Sinali*	Dong Son culture	pandanus fiber	weaving	soft, light, breathable		comfort
100 BC - 851 AD	*Öroba uli mbuaya*	Develops the pattern of weaving techniques on animal skin materials	animal skin	cutting, smoking, combining with *ösumö* technique	thick, strong		resistance
	Baru Öröba	Lack of crocodile skin material. Introduced new materials from Chinese, Arab, and Indian traders Ability to form materials into specific shapes	iron	forging, smoking	strong		resistance, self-expression
1894	*Baru*	Using a combination of local materials	bark, rattan, palm fiber	beating with a rock, weaving, and combining	thick, breathable		resistance, comfort, self-expression
1930	*Baru Öröba*	Incorporating several materials and decorations	crocodile skin, iron, rope, and yellow paint	forging, smoking, painting	thick, strong		resistance, self-expression

Year	Name	Phenomena & influence	Material	Technology	Material characteristics	Image	Function
1960	*Baru Öröba*	Using local materials with a combination of other materials.	tin	forging, smoking, sewing	strong, thin		protection, self-expression
2019	*Baru*	Using foreign materials with decorative patterns	cotton, yarn	sewing, embroidery	light, soft, breathable		resistance, self-expression

3.2.1 Traditional materials and technologies

The first excavations undertaken by the Nias Heritage Museum and Airlangga University found that the inhabitants of Nias Island first occupied the Tögi Ndrawa cave around 12,000 years ago. This conclusion was reached through research on the skeletal remains of vertebrates, as well as Paleolithic tools, such as stone flakes and arrowheads. The most compelling evidence of civilization included the discovery of an oval andesite stone with a character resembling a bat, a hammer, and a tool made from horns and mollusk shells (see Figure 4). Cultural similarities exist between the early Nias and the people of the Hòa Bình culture from Southern China and Vietnam, as well as the Toala culture from Sulawesi [12-14], in [15] argued that "bark cloth technology originated in the Huang and Huai Plains of North China, passed through the valleys of the Yangzi and Han rivers to south China, and then on to the islands of Taiwan and Hainan before moving onto Indonesia, via Vietnam." This finding aligns with the emergence of the Nias bark shirt.

Figure 4 Paleolithic tools found at Tögi Ndrawa cave: oval andesite stone with a character as a bat, a hammer, and a tool or spatula made from horns and mollusk shells (Courtesy of Nias Heritage Museum)

The Nias tribe used the bark of the Oholu tree to create barkcloth. Barkcloth was made by carefully peeling the Oholu bark to obtain a piece approximately 130-150 cm long and 40-50 cm wide. The peeled bark was then soaked in water for a few hours to make it malleable. The process of beating *(lalabago)* the bark peel with stone would make it wider, thinner, and more pliable [16, 17] (see Figure 5). Once dried, the bark peel would be sewn together into garments called *baru oholu* (see Figure 6). The manufacturing and maintenance process of barkcloth clothing was a time-consuming endeavor. This barkcloth was used as a body covering to protect the warrior from the island's climate. This is also in line with first and superior function of clothing as a protection of the human body injury of the skin [18]. In conclusion, the barkcloth armor's primary function was protection.

In [19] is presented DNA evidence that the first Nias people originated from Taiwan and accessed Nias Island via the Philippines. According to oral tradition, all knowledge and expertise were handed down by *Hia Walani Adu*, the most prominent figure in early Nias history. He passed on the rules of life in Nias culture, including customs, religion, agriculture, carpentry, and crafts. These settlers are estimated to have entered Nias around 5.000-4.000 BC, bringing progress in various fields, including weaving, carpentry, and metalwork [12, 20]. Like *baru Oholu*, *baru leama* used local materials sourced from nature. Palm fiber from the palm tree (*leama*) is a hard and coarse material. Therefore, it is rigid, uncomfortable, and heavy in its natural state [17]. However, the braiding technique employed by the Nias made the material easier to fabricate into clothing than barkcloth. Moreover, it added volume and strength to the soldier's body, protecting him from injury. According to [18], the first category of function of clothing after protection is the utility performance (strength, etc.). This article concludes that the primary function of palm fiber armor was resistance.

Figure 5 The process of recreating the barkcloth production. Peeling process of the bark of Oholu tree. This process is crucial since there should be no tear on while peeling. After soaking in the water, the pealed bark is hammered with a stone (Hòa Bình culture influence) (Documented by Nias Heritage Museum and Dina Waoma)

Figure 6 The process of recreating the barkclot. After cutting the bark according to the pattern, it is combined by sewing (modern technology) (Documented by Dina Waoma)

Heine-Geldern [21] stated that the late Bronze-Age Dong-Son in circa 500 BC and 100 BC, distributed weaving to Indonesian archipelago [22]. As the Niasan learned the weaving techniques of Dong-Son culture, their war armor pattern pieces became more refined and better structured. The double braiding technique was performed by hand to unite the patterns, improving the basic shape of the Nias armor. The woven material was made of pandanus leaves formed into a small and fine rope *(sinali)*. Based on its material, this armor is called *baru sinali.* The transition from palm fiber material to lightweight pandanus leaf material facilitated soldiers' movements during battle, allowing them to jump and run effortlessly. Although *baru sinali* was thinner than *baru leama*, this new armor improved the soldier's agility and confidence in battle. The second category of function of clothing after protection and utility performance is comfort performance (fitting to the human body) [18]. The primary function of this material can thus be considered comfort.

Previously, the Nias used *baru* (shirt), as the term for the armor. Upon the replacement of plant materials with animal skins, Nias war armor was officially declared an "armor," or *öröba*. During this period, Nias war armor gained popularity. Animal skins were sourced from the tiger, the buffalo, the pangolin, and, most famously, the crocodile.

Crocodiles occupy the rivers of Nias and are traditionally associated with the underworld (hell), or with punishment from ancestors [20]. Crocodiles are seen as elegant killers – they do not rush during a hunt, but rather monitor their prey in silence. Crafting *öroba uli mbuaya,* or crocodile skin armor, is a unique traditional Nias technique. The dorsal surface of the crocodile is protected by a natural armor of osteoderms, which are individual segments made of a porous bony core surrounded by dense bone. As a result, crocodile skin is flexible yet puncture-resistant [23]. The people of Nias modified the crocodile skin, fortifying its natural hardness to withstand enemy attack. To create this armor, the Nias cut the crocodile skin into eleven pieces, much like a clothing pattern, which were then joined using the *ösumö* technique [24]. The distinctive feature of this armor was the prominent display of the crocodile's dorsal skin on the armor's back. This skin piece was the largest of the eleven pieces of leather. Two large pieces of crocodile skin were

positioned to cover the chest to the waist, while two curved pieces provided shoulder protection. Pairs of crocodile hands and feet covered the armor, starting from the sides of the body and ascending to the armpits and the back of the neck [24]. Based on the material characteristics of the crocodile skin, the function of this armor was resistance.

Analysis of armor made with local materials and technology reveals the use of materials native to Nias nature and techniques passed down by Nias ancestors, as well as those adapted from foreign traders. Recorded materials and techniques used for making clothes during this period include barkcloth, made from hitting the bark of the Oholu tree with a stone; palm fiber, which was joined into a rope and then braided; woven pandanus fibers; and crocodile skin cut into patterns and combined with the *ösumö* technique. Nias war armor was initially manufactured for protection, comfort, and resistance.

3.2.2 Foreign materials and technology

The Nias soldiers' affinity for wearing crocodile skin caused the crocodile population to decline. As their source of armor material grew scarce, the Nias soldiers chose to move on to iron. Iron ore is estimated to have entered the island before the 851 AD. Iron ore is a material imported by traders and sailors from Arabia, China, and Indian, according to early notes from Sulayman and the *"Kitab adhaib al-Hind"* from India written in 950 [12]. Nias also bought brass and gold plates in exchange for skulls acquired from headhunting [5]. malleability allowed it to be shaped and hammered according to the desires of the soldiers [20]. The texture, shape, and detail of this novel metal armor were typically designed to imitate the texture of crocodile skin. However, the level of customization and detail possible with this armor offered the soldier the chance to reach self-expression, or the validation of self-esteem and craftsmanship in his creation. In Nias culture, the color black symbolizes the anger and ferocity of soldiers during battle. Therefore, before use, the armor would be hung from the second floor of the soldier's house and smoked until it reached a black color like crocodile skin armor [20]. The primary functions of this armor were resistance and self-expression.

Iron war armor was created due to the scarcity of crocodile skin, as well as the entry of new technology from trading with Arabia, China, and Indian merchants. The purpose of producing war armor with this material was to facilitate resistance and encourage the self-expression of soldiers.

3.2.3 Combination of local and foreign materials and technologies

In later years, Nias war armor was characterized by the combination of various techniques and decorations by the soldiers. Generations of experience taught Nias warriors to extract the best attributes from a variety of materials and techniques. Examples of late Nias war armor are documented in the archives of the Nationaal Museum van Wereldculturen in the Netherlands and the Musée du Quai Branly – Jacques Chirac in France.

In 1894, the Nationaal Museum van Wereldculturen (Coll. No. RV-985-1) acquired war armor made from a combination of bark, rattan, and palm fiber [25]. Nias warriors combined the best characteristics of each local material to create a sturdy, yet comfortable armor. The combination of materials emphasizes the basic structure while showing the creativity of the warrior. The barkcloth serves as the base of the armor, while the outer layer is constructed of woven fibers joined together with rattan stitches. This makes the war armor stronger and more comfortable, with a fierce appearance. The functions of this armor included resistance, comfort, and self-expression.

Circa 1900, Musée du Quai Branly – Jacques Chirac (Coll. No. 71.1912.3.237) documented war armor made from coconut fiber. The armor was constructed with a rattan frame built over a base of barkcloth. Palm fiber was tied to the rattan frame layer by layer. Trims of rattan rings adorned the armholes, hem, front, and neckline. At the back of the armor, the addition of four palm fiber "ponytails" was added. This armor displays the functions of resistance, comfort, and self-expression.

In 1930, the Nationaal Museum van Wereldculturen (Coll. No. TM-6402-1a) received a Nias war armor made from a mixture of crocodile skin, pangolin skin, and forged iron decorated with yellow paint to imitate gold. Yellow in Nias culture signified nobility and wealth [20]. The armor conveyed strength and displayed the warrior's creative design. The combination of animal skin and iron created a shield for the warrior's body, while the yellow accent color signified the victory of the warrior. This armor's functions included resistance and self-expression.

In 1960, the Nationaal Museum van Wereldculturen (Coll. No. TM-2918-25a) received another piece of Nias armor made from tin with decorative application mimicking the style of *baru oholu*. Like iron, tin was imported to Nias by traders and sailors. The tin plates were combined with traditional sewing methods to create a unique asymmetrical design. The armor's main functions were protection and self-expression.

It should be noted that the years referred to above are not the years in which the decoration, techniques, or materials were first formed. However, these findings indicate that one village could apply different materials to the same basic structure.

Today, Nias war armor is used as a performance costume intended to drive away malevolent spirits. These costumes are worn to escort respectable guests to meet the village chief, to perform dances

celebrating significant life events, and to attend major ceremonies [20]. This modern form of “war” armor is constructed of cotton with embroidered decorations. It no longer serves the purpose of protection, but now facilitates comfort and self-expression.

The conclusion gathered through analysis of combined materials and technology in Nias war armor reveals the ability of the Nias people to see the advantages of each material’s properties. Increased knowledge, expertise, and technology allowed Nias war armor to reflect the individuality of the Nias soldier. The Nias’ experiences in making armor taught them that in order to achieve their goals of increasing resistance, comfort, and self-expression, a combination of local and foreign materials was the best method. In this period, decorations were emphasized to show the character of the soldier (see Figures 7 and 8). The techniques of weaving, painting, embroidering, forging textures, and even decorating with gold were employed to achieve this goal.

Figure 7 A group of soldiers and nobles of Nias in their armor in 1915. Seen in the picture that the group has similar armor structure. The differentiation of the group's status is seen through the armor’s material and decoration (Photograph and courtesy of Feldman et al. 1990:111)

Figure 8 Three Nias soldiers with different war armor materials during the same period. Although there is a timeline on Nias war armor’s material, the warrior has their own decision on choosing the material and decoration (Collection Nationaal Museum van Wereldculturen. Coll. No. TM-10001506)

4 DISCUSSION

The main finding of this study is that the material development of Nias war armor exhibits the adaptive nature of the Nias people. Close observation reveals the connection between Nias historical events, artifacts, and the development of Nias war armor. According to [26], material has consolidated humanity’s need either to be protected from the environment (protective function), or desire to outwardly convey a message about themselves (self-expression). It is evident that over time, Nias war armor’s material progressed from local materials, such as the Oholu tree bark, palm fibers, pandanus leaves, and animal skin; foreign materials, such as iron and tin; and a combination of local and foreign materials. The chief functions of materials in Nias war armor were protection, resistance, comfort, and self-expression. These functions are in line with the concept of fabric function as clothing material according to [18], protection, utility performance, and comfort performance. The study of clothing can provide insight into the evolution of civilization. Clothing provides vital evidence to help us understand what occurred in the past and how this has affected the present. Similar study has been done by [27] with the development of Chinese clothing function that lies between protective function, beauty, and religion. Each of these changes are the result of interactive influence between the outside world and China's own dynastic tradition [27]. It is evident that the development of Nias war clothing was closely tied to historical phenomena in Nias; the most obvious proof is the correlation between Hòa Bình and Toala cultural influences and material developments in Nias armor in 12.000 BC with evidence of stone hammer founded in Tögi Ndrawa cave. This can also be seen in the arrival of settlers from Taiwan via the Philippines in 5.000-4.000 BC with evidence of the research of Nias DNA that was highly related with the Philippines and Taiwan descendants with braiding fabrication. The Dong-son culture migrating to Indonesia during 500-100 BC influenced the weaving technique with local material. Local technique of creating crocodile skin into armor with *ösumö* technique is one of the innovations of Nias culture. Evidence of iron trading was mentioned in 851 AD with Arabia, China, and Indian merchants. These historical events impacted the development of material and functionality in Nias war armor.

This research supports the idea that Nias warriors emphasized the importance of manufacturing war equipment by [3]. Therefore, the progression of material and technology was essential to create innovative war costumes. From armor artifacts, it is apparent that Nias warriors combined the best characteristics of both local and foreign materials to create armor that provided protection and facilitated self-expression.

This analysis provides new insight into the rich history of Indonesian traditional clothing, much of which has not yet been researched. Culture naturally evolves over time. As the creative work of the Indonesian people flourishes, Indonesian culture will continue to develop new values. This study of material and technology in Nias war armor can be applied to various other disciplines. Today, the people of Nias no longer use war armor for defensive purposes, but rather as a dance costume. This creates an opportunity for costume designers to adapt the Nias war armor into a decorative and theatrical structure.

This study encourages further research and development using contemporary technology and materials. For example, 3D printing techniques using plastic material could allow designers to create armor digitally and quickly. “Dry-fit” sportswear technology is another modern material that can be applied to improve the comfort of traditional Nias clothing. A hands-on research approach might involve creating a modern fashion collection based on the traditional structures, materials, and technology of Nias war armor, which could then be appreciated by the wider community.

5 CONCLUSION

This study investigated the relationship between Nias war armor and historical events. Detailed analysis established the chronological timeline of Nias war armor and revealed its relationship with historical events, materials, and technologies.

Traditional clothing offers valuable insight into the development of a civilization. Analyzing the timeline of traditional clothing can link historical events and technological developments with changes in local traditions. Clothing is a basic human need, but its function can shift from a purely utilitarian role to become a means of self-expression. Analysis suggests that the timeline of Nias war armor was defined by an early period of local materials (circa 12.000 BC until 851 AD), an interim period of foreign materials (circa 100-851 AD), and finally a combination of both local and foreign materials (circa 1894 AD). Each material has its own distinct function. These conclusions were reached by examining local myths and history. Through this analysis, it is clear that the adaptable nature of the Nias people will allow them to continue to adapt to the modern world.

The many forms of Nias war clothing are a testament to the Nias culture’s ability to adapt to changing science and technology. The development of materials in Nias armor was marked by the shift from local materials to foreign materials, and finally the mixing of the two materials to create a more advanced form of armor. This article shows the Nias’ usage of barkcloth, palm fiber, pandanus leaves, animal skin, and iron. The development of materials was accompanied by technological discoveries of how to process these materials: the technology of beating bark into cloth with stones, braiding, weaving, smoking, *ösumö*, forging, sewing, and embroidery. This research concludes that Nias war costumes, particularly those based on materials and technology, were adaptive and demonstrated functions of protection, resistance, comfort, and self-expression. Current study can contribute to uncovering the relationship between history, material, and technology in Indonesian traditional clothing as well as further research and development using contemporary technology and materials.

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THERMAL AND UTILITY PROPERTIES OF SOCKS

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Abstract: *The aim of this paper was to evaluate the thermal and utility properties of sock goods. The sock assortment had a different material composition and knitted structure. Material analyses were performed on samples of socks before of measuring properties. The basis of the research was the determination of thermal insulation properties by using of a thermal imaging camera. To do this, it was necessary to know the changes in length and weight before and after wearing the socks. It has been proven that the temperature of the foot is different in the monitored parts and the composition of the textile material and change dimensions and weight does not significantly influence the thermal insulation properties of the evaluated assortment.*

Keywords*: knitting structures, socks, thermal insulation, thermogram.*

1 INTRODUCTION

Socks are an important part of the wearer's feet protection. The quality of the socks assortment currently influenced by its thermal and aesthetic-fashionable properties, antibacterial and antimycotic effects, structure, surface, and color. The socks are composed of a toe, a foot, a heel, and a hem [1]. They usually have a doubled hem in the upper part, into which a rubber strip is inserted, or elastic yarns. Some types of socks are folded into a cuff at the top. In the shape of a sock, they are produced by knitting technology, for example on single - cylinder three - system automatic knitting machines (Ange 15 - universal Lonaty GK 616, etc.) [2]. The following structures knitted fabrics are used: plain, ribbed, terry and velour. The preparation of the sock pattern itself consists of the following operations:

- design of a graphic pattern,
- design of a program for knitting socks,
- transfer of the pattern to a specific program and type of sock knitting machine,
- knitting of model socks,
- verification of sock dimensions, stretch and adjustment,
- modification of the graphic pattern resp. adjust the number of chains and lines.

Then the socks are adjusted to the final shape by operations such as: forming, ironing, sorting, sewing labels, strapping, and packaging. The following types of socks are known (Figure 1):

a) invisible socks - they reveal the whole instep, covering only the heel,
b) sneaker socks - leave the ankle exposed, the hem ends just below the ankle,
c) ankle socks - cover the ankle,
d) high socks - reaching up to the calf muscle,
e) socks with a hem - these are high socks, the upper part folds and creates a cuff in the ankle part,
f) knee socks - ending below the knee,
g) socks above the knees - reaching above the knees,
h) loose socks - are wide, worn lowered and gathered to knee height,
i) tabi socks - characterized by their separate big toe,
j) toe socks - have a place for each toe separately [1].

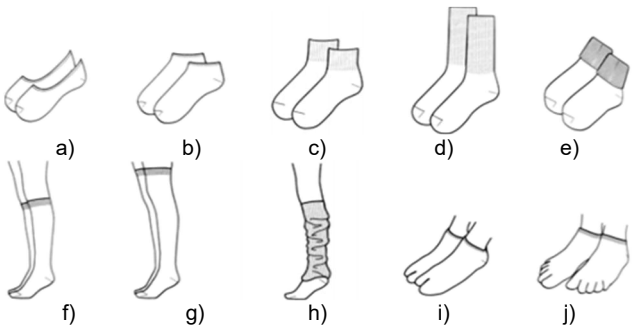

Figure1 Types of socks

Socks of different shapes made of different materials can be inappropriately shaped and prevent the transfer of residual heat and moisture during wearing. From the point of view of thermal comfort, it is therefore most important to maintain the optimal temperature between the foot, the sock and the shoes during the transfer of heat and moisture, especially by convection and conduction [3]. Human temperature is typically around 37°C, a known fact. However, the skin surface has a lower temperature, in the range of approximately 33.5 to 36.9°C.

Warmer spots are in the muscle areas, but the tendons and bones, as well as the feet, are colder [4]. It is important to ensure optimal temperature and humidity in this area of the body to achieve comfort when wearing socks. Moving and pressing the foot on the sock and shoes increases or decreases the generation of heat and moisture in this area. The most important is the dynamics of the spread of moisture and heat in this part of the body, which affects the ability to wick sweat from the body surface through the sock into the environment (or through shoes into the environment) [5]. In case of insufficient moisture wicking, the body overheats, and moisture liquefies and accumulates between the skin and the socks [6]. When the feet meet with the sock and the shoe, this process slows down significantly compared to other parts of the body, even though the surroundings can absorb sweat. Based on the above information, the basis of the paper was:

- temperature measurement using a thermal imaging camera at selected points of the foot (measurement was performed at certain time intervals before and during the wearing of the sock),
- changes in the distance of the socks before and after wearing, since changes in temperature can also be caused by the shape instability of the socks,
- -the observed properties may also be related to the weight of the sock, therefore the change in the weight of the sock due to wear was also evaluated.

Based on the measurements, the most suitable material composition and knitted structure for thermal properties of the sock were chosen. The differences in temperature due to the wearing of socks were also evaluated. The presented measurement of temperature differences represents only a qualitative evaluation of a certain estimate of the degree of thermal insulation of different socks. The work is informative rather than research. These measurements were preceded by a detailed analysis of the material composition of the examined range of socks.

2 EXPERIMENTAL PART

2.1 Materials

Natural materials such as cotton and its combinations with polyester, polyamide and Lycra are mainly used to produce socks. Table 1 shows the base materials (yarns used) in a wide range of combinations. Figure 2 shows electron microscope images. These are longitudinal and transverse views of the fibers and longitudinal views of the yarns that have been used in the range of socks. Twisted combed mercerized cotton yarns with a fineness of 14.5 tex are used to improve strength, affinity for dyes and smoothness. The yarn made of cellulose fibers provides good antibacterial properties. Instead of 100% cotton yarn, a single blended yarn of 29.5 tex with 60% cotton and 40% polyester is used. Polyester fiber gives the yarn greater strength and longer life. They retain their shape well even when repeatedly washed and worn. Polyester is less absorbent than cotton, but also forms less stains. One of the special materials is the profile polyester fiber Coolmax®, the main feature of which is the increased specific surface. The fiber wicks sweat away from the body faster and thus speeds up its drying. Polypropylene fibers Prolen® with Siltex treatment prevent the multiplication of bacteria, fungi and molds. The reason is that additives based on biogenic silver ions were used in the production of the material. Resistex® Carbon yarn has similar effects. It contains 34% of polyamide 6, which has been cast on the surface with conductive carbon and coated with 66% of structured polyamide 6.6. The fibers ensure that no electrostatic charge is generated.

Table 1 Material composition of yarns

Sample no.	Material composition of yarns
1.	33% wool, 19% polyamide,46% polyester, 2% elastane
2.	98% cotton, 2% Lycra
3.	93% cotton, 5% polyamide, 2% elastane
4.	47% cotton, 47% viscose, 4% polyamide, 2% elastane
5.	65% cotton, 23% polypropylene, 10% polyamide, 2% elastane
6.	98% cotton, 2% elastane
7.	98% cotton, 2% elastane
8.	83% wool, 15% cotton, 2% elastane
9.	100% cotton
10.	33% cotton, 19% polyamide, 48% polyester

2.2 Methods

The research process mainly involved temperature measurements. Related are measurements of the change in weight and length of a sock. These properties were measured before and during the wearing of socks.

2.2.1 Weight of socks

The socks absorbed moisture, sweat and dust while wearing them. This support bacterial growth and weight gain. It is a sign of reduced comfort when wearing socks. For these reasons, it was necessary to detect a change in the weight of the socks during wearing. Samples were weighed before and after use on the proband. The time interval for wearing socks was 12 hours.

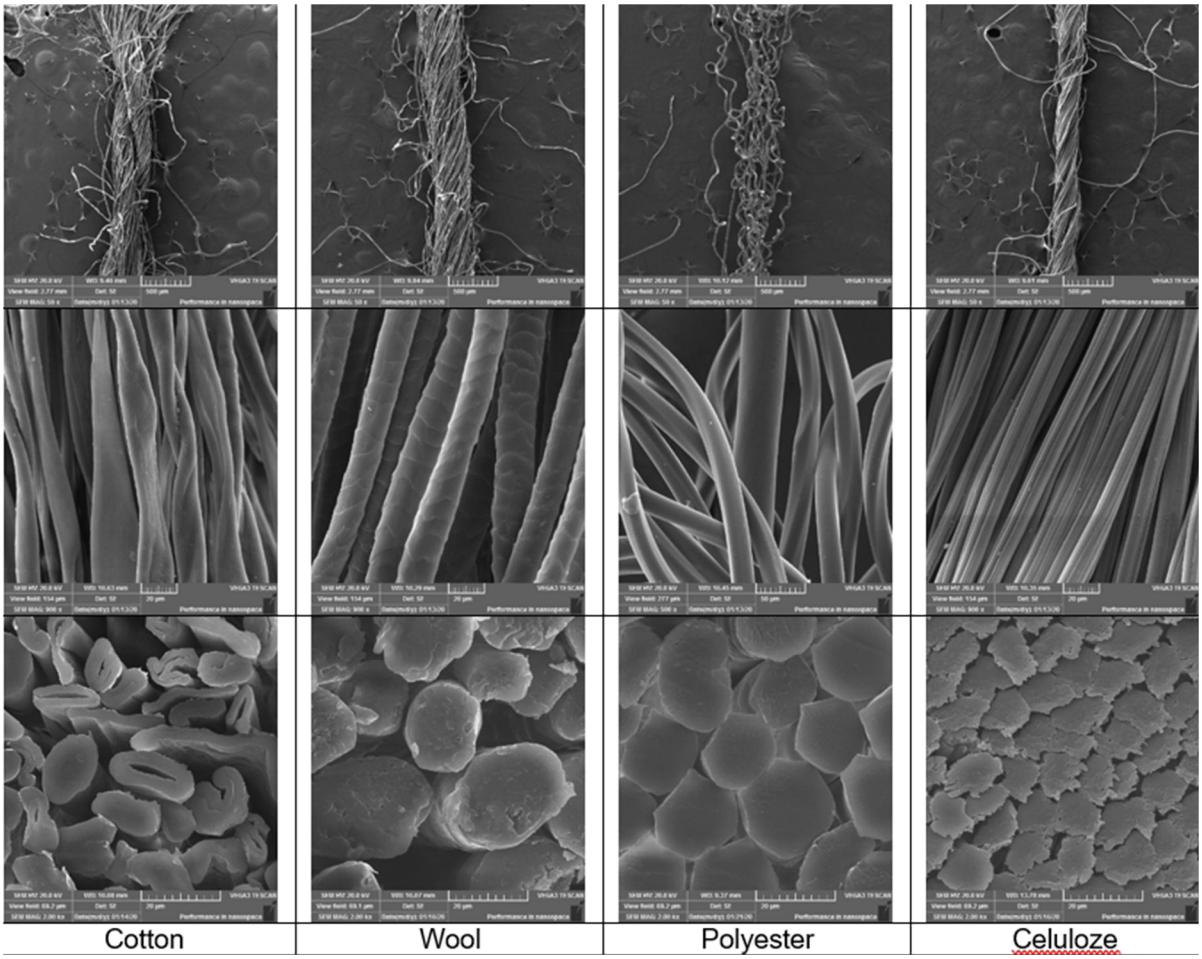

Figure 2 Types and shape of yarns and fibers

2.2.2 Length of socks

The shape deformation of the sock mainly leads to a change in its distance. This fact causes problems during wearing the sock and can influence the measured temperature of the sock. Changes in the shape of socks are mainly influenced by the material composition and the knitting structure used. Therefore, it was the sock length was measured before and after wearing. The distance in the part of the foot was measured from of the heel to the toe (Figure 3). The sock was fixed in position on the pad during the experiment.

2.2.3 The temperature measurement using a thermal imaging camera

Thermo Vision can be called a technology that detects temperature differences between the environment and objects in the foreground of the image. It uses infrared imaging techniques for this purpose. It deals with the capture, processing, analysis, display of a thermal radiation [7]. The result is infrared images called thermograms. It is a graphical representation of the heat [8]. The temperature of the head and foot in real conditions in a dressed person is clearly visible on the thermogram.

The clothing area usually radiates less heat than the naked skin. The more layers or thicker the clothing the body have, the lower the temperature measured from the outside.

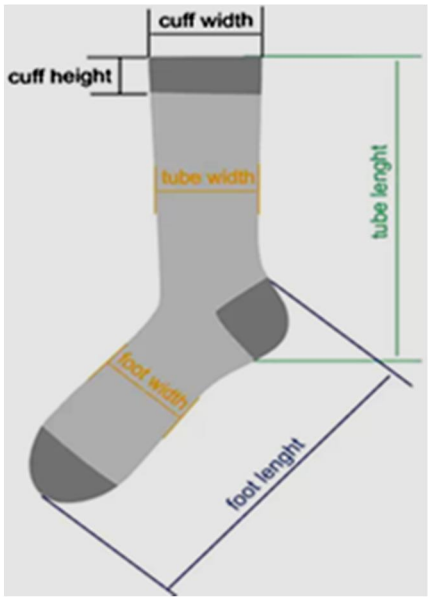

Figure 3 Measurement of the length of the sock

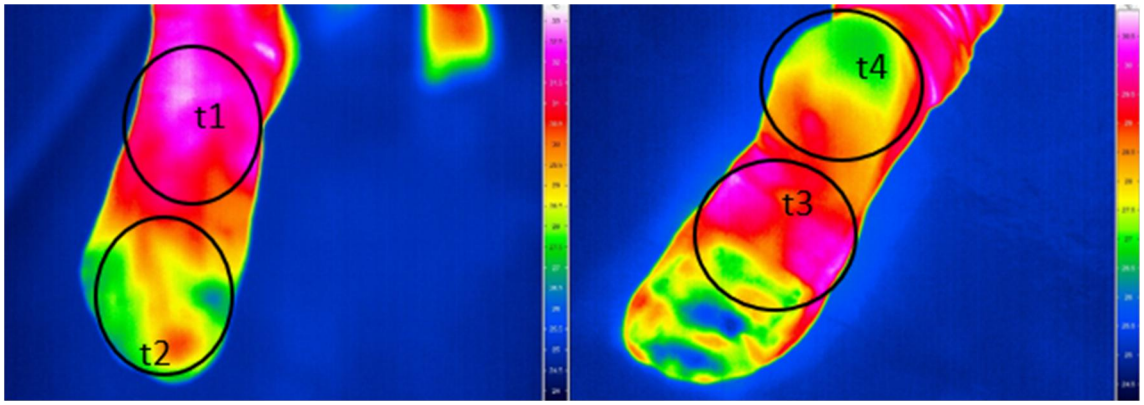

Figure 4 Thermal images of the upper and lower part of the bare foot

The infrared thermal imaging camera measured the surface temperature of the end of the wearer foot, which was at first without a sock and then covered by a sock. The thermal energy that a body receives, or transfers was measured. In this experiment a thermal imaging camera Jenoptik Vario CAM (serial no. 10381) was used with following settings:

- resolution 2048x1536 pixels,
- detector resolution 1024x768 pixels,
- temperature range -40°C to +1200°C,
- detector: uncooled FPA microbolometer, 17 μm, 7.5 μm to 14 μm [9].

Measurements performed by a thermal imaging camera and subsequent assessment of thermal properties are performed according to the standard STN EN 13187 [10]. The thermal imaging camera enabled the setting of the emissivity coefficient in the range of values 0.1 - 1 and the value was set to 0.85. The captured data was recorded in the form of digital images (thermograms) on a storage medium and further evaluation.

In the study, the bottom of the foot was monitored under light load. The experiment procedure consisted of the following steps:

- the foot was placed stationary on the stand,
- the distance of the foot from the thermal imaging camera was length 1 m and height 0.4 m,
- measurement of the temperature of the foot without the sock (before the measurement, the foot was bare for 15 minutes to acclimatize in the laboratory),
- the measurement of the temperature of the foot with the sock after 30 minutes of wearing the sock with loose bedroom slippers.

Environment conditions in the laboratory were set at a temperature of 20°C and relative humidity 60%. It was also important to prevent heat radiation and air flow on the measured probant. In selected areas of temperature fields, minimum, maximum as well as mean values of surface temperatures were identified on all analyzed thermograms. After the actual sensing of the temperature field, it was necessary to sort these fields. The knowledge of the anatomy of the foot was used here. The points of temperature changes were selected from the record (Figure 4), where is the temperature t_1 on the instep, t_2 on the toe, t_3 in the middle of the foot and t_4 on the heel.

3 RESULTS AND DISCUSSION

3.1 Change in the weight of socks

Measurements of the weight were taken before and after wearing the socks. The weight was measured using an analytical balance. It was assumed that due to the material composition of the socks, there would be larger weight gains for those samples that contained a higher percentage of cotton and wool. This assumption was not confirmed by measurements and weight changes due to wear were minimal (Figure 5).

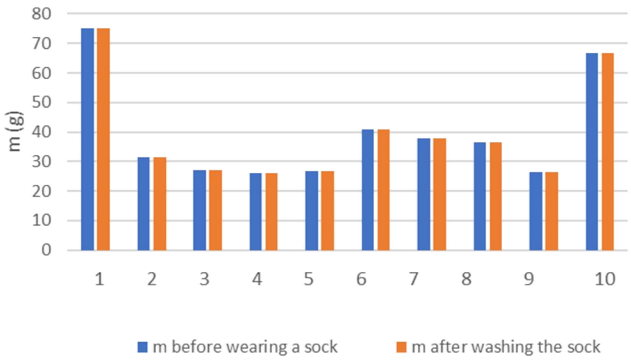

Figure 5 Weight *m* [g] of the socks before and after washing

3.2 Change in the length of socks

The measurement of the distance of the socks was focused on the length before and after wearing the proband. The results in Figure 6 show that the length of the socks was reduced by 3 to 16% of the original length. Samples without elastane no. 9 and 10 can be deformed the most.

This assumption was not confirmed by measurement. However, socks containing a wool component were the most deformed (samples no. 8 and 1). It is also not possible to determine from the measurement results which material composition of the socks will have the lowest shape change due to wear.

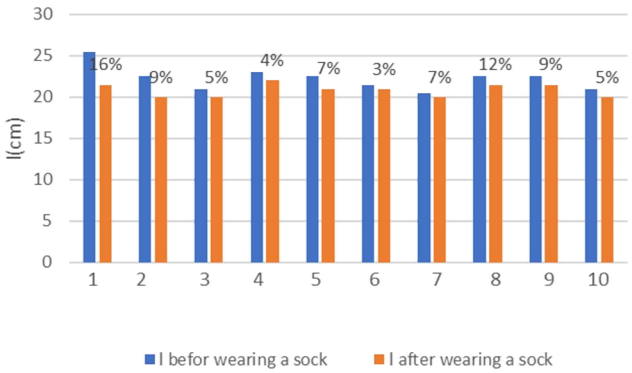

Figure 6 Length *l* [cm] of the sock before and after wearing

3.3 Temperature sensing using a thermal imaging camera

The surface temperature distribution on the sample was measured using a thermal imaging camera. Five images were taken from the upper (instep area) and the lower (heel area) of the foot. Measurements were performed at short time intervals. Figure 7 shows selected images of a foot with a sock. The temperature of the foot with the sock was variable, which results from a visual analysis of all images.

However, it is not possible to determine exactly which material composition will be the best heat insulator from the results of thermograms.

When evaluating the thermal images, the detection was performed with different data, which in our case represent the temperature in the parts of the foot. Minimum, maximum, as well as mean values of surface temperatures were identified on all analyzed thermograms [11]. The average results of their analysis and elementary statistical processing in this system are given in the Table 2, where t_{min} is the minimum temperature in the heel area (t_4 on the heel), t_{max} is the maximum temperature in the instep area (t_1 is on the instep), $t_{average}$ mean temperature in the monitored areas and s is standard deviation of temperatures. The high temperature variability of the foot with the sock was confirmed from all measurement results.

Table 2 Results of thermograms analysis

sample no.	t_{max} [°C]	t_{min} [°C]	$t_{average}$ [°C]	s [°C]
1	33.0	24.0	28.5	2.50
2	32.5	27.0	29.75	1.49
3	32.5	27.5	30.0	1.32
4	32.0	25.0	28.5	2.14
5	32.0	25.0	28.5	1.65
6	32.5	25.0	28.75	2.35
7	31.5	27.0	29.25	0.95
8	32.5	25.0	28.75	2.46
9	32.0	27.5	29.75	1.38
10	33.0	25.0	29.0	2.27

Figure 8 shows a mean temperature of specific foot parts. In all measurements, it was found uniformly that the highest temperature is reached by the foot in the instep (t_1) and the lowest temperature in the heel (t_2). The foot temperature is similar in the area from the center of the foot to the toe (t_2, t_3). Next experiment containing outputs from thermograms was a comparison of the average temperature of the bare foot and the foot with the sock. From the results in Figure 9 it follows that within 30 min wearing the foot did not retain its temperature. In all cases, there was a slight decrease in foot temperature with a sock ranging from 0.25 to 1.5°C.

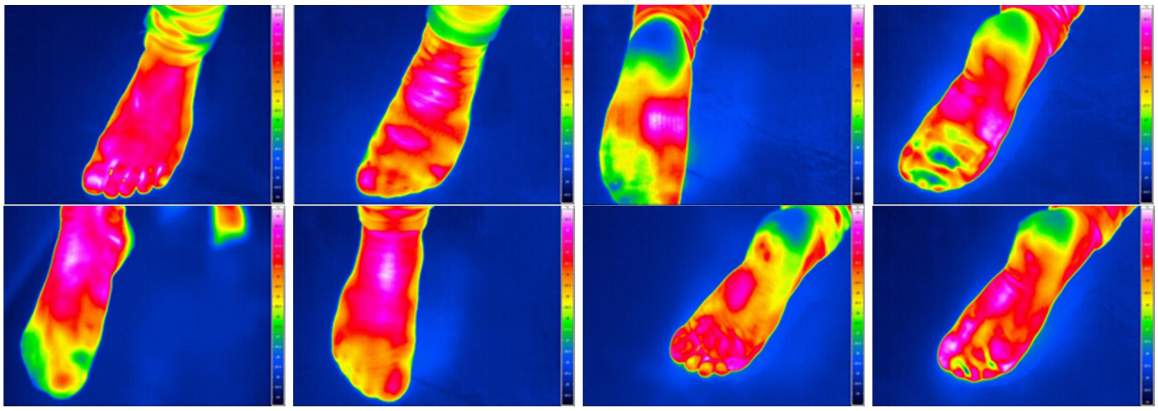

Figure 7 Selected images of thermograms front and back of the foot

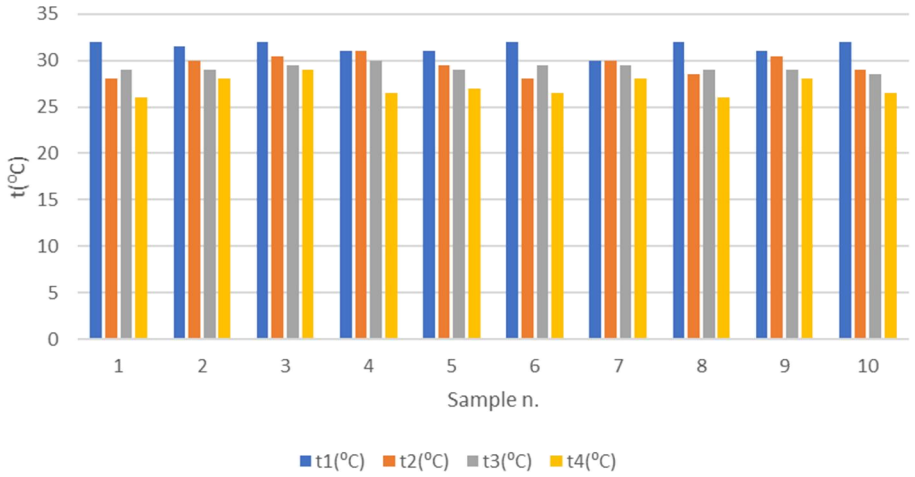

Figure 8 Foot temperature where t_1 is on the instep, t_2 on the toe, t_3 in the middle of the foot and t_4 on the heel

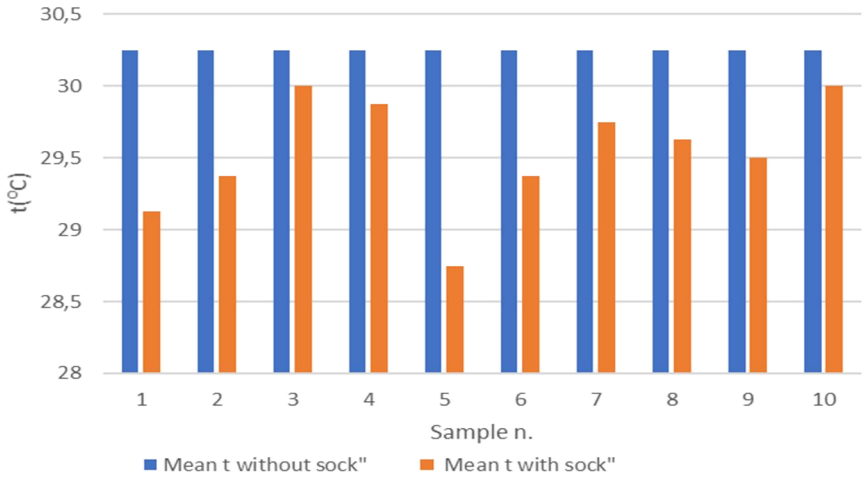

Figure 9 Mean temperature without and with the sock

4 CONCLUSIONS

The main aim of the work was to measure and evaluate temperature changes in parts of the foot and sock and to determine the change in length and weight of socks due to wearing.

From the measured results, it was found that changes in the weight of the socks due to wearing and maintenance are minimal. This means that the change in the weight of the socks will not affect the measured temperature fields.

The length of the socks was reduced by 5% to 16%. The content of elastane in the mixture did not significantly affect this distance. Its main role is to maintain the shape of the sock on the foot during wearing. Samples no. 1 and 8 had the worst dimensional stability. The reason may be the content of the wool component or the knitted structure.

The composition of the material shown in Figure 2 was for information only. A detailed analysis of the influence of the structure of linear textiles on the thermal insulation properties of socks will be the subject of further articles.

Temperature measurements were performed using a thermal imaging camera on a bare foot and on a foot with a sock at a proband during a time interval of 30 minutes of wearing. The result was thermographic images in the instep and foot part. Specific temperatures t_1, t_2, t_3, t_4 in these parts of the foot were selected. Based on them, it was found that the temperature of the foot with the sock is different in parts of the foot. In places where the skin is coarsest and the blood supply is lowest (the heel t_4), the temperature was lowest. The temperature was highest in the upper instep (t_1). It has also been shown that the temperature of the bare foot is higher than the temperature of the foot with the sock in the range from 0.25 to 1.5°C (Figure 9). However, from the results of the thermal imaging camera, it is not possible to conclusively determine which material composition of the sock is best heat insulation.

In the part of the sock where a denser knitted structure or terry and filler weave structures was used, it is possible to improve the insulating and mechanical properties [13]. The used method of temperature measurements on foot skin and a sock based on infrared camera may suffer for very low measurement precision. The temperature detected by infrared thermometers depend strongly on the surface emissivity of the measured object, and on the radiation energy penetrating from the background of the measured object (here human skin).

For knitted fabric structures, the penetration coefficient may reach 10%. Regretfully, the emissivity of a skin differs from emissivity of textiles. Moreover, emissivity of a skin depends on the skin moisture level. Therefore, the use of this method in the textile area is limited. Strictly scientifically, the measured knitted fabric surface temperatures will also depend on boundary condition on free surface, namely on the free convection heat transfer coefficient.

Moreover, temperature measurements on bare and dressed foot do not bring the information about thermal resistance of socks which also involves the effect of thermal resistance of the air gaps. Thus, the temperature difference data should be accompanied by thermal resistance data of the socks plus the knowledge of the average level of the air gaps.

These inaccuracies in thermal imaging camera measurements can be specified using thermal resistance and porosity measuring instruments. The main benefit of the paper is the evaluation of temperature variability in different parts of the foot, while its inaccuracies are also justified.

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PATTERN 210 FOR DESIGNING LONG-SLEEVED SHIRTS WITH *SANGGIT* BATIK MOTIF

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Abstract. *The purpose of this research is to formulate a pattern of batik motifs on cloth measuring 115x210 cm which can be made into long-sleeved shirts of various sizes whose motifs are still sharp. The research was conducted using qualitative descriptive methods and participatory action studies. The descriptive method is to identify and develop a draft pattern, while the participatory follow-up method is to test the draft pattern by actively involving batik entrepreneurs, motif designers and tailors. The research was conducted at the Jalidin batik business, Sragen, and data were collected through observation, interview, FGD and document analysis techniques. The results of the research are a pattern of 210 batik motifs for long-sleeved shirts in the shape of a rectangle, the length of the pattern is 210 cm and the width of the pattern is 115 cm. This pattern is relevant for developing batik motifs with an efficiency of 16%, and the resulting batik fabric can be made into long-sleeved batik shirts in sizes S, M, L, XL and short-sleeved shirts in size XXL with strong motifs. The complexity (kesanggitan) of the motif is located on the front of the shirt, the left side, the right side, the pocket, as well as certain motifs on the cuffs and collar. The shirt motif pattern is very effective and efficient for developing random or a-symmetrical batik motifs.*

Keywords: *pattern 210, motif, long-sleeved shirt, sanggit batik.*

1 INTRODUCTION

Indonesian batik motifs pattern can be broadly divided into three categories based on their feature, namely the long or *jarit* cloth batik pattern, the shirt batik pattern, and the jarit-shirt batik pattern. First, the long cloth batik pattern is a rectangular pattern for making a batik motif, and the batik cloth it produces, is used as a whole without cutting or stitching. For instance, batik cloth is used to produce *jarit* for women, sarongs for men, scarves, headbands, kemben, sheets, tablecloths, and other products. Dependent on the design of the position of the motifs, long cloth batik patterns in Indonesia can be divided into at least seven patterns, namely the morning-evening pattern, the one-headed pattern, and the two-headed pattern [1], *parang* pattern, ruffled pattern, upright symmetrical pattern, and random pattern.

Batik cloth is used as a *jarit* for women, a sarong for men, a scarf, headband, kemben, mats, tablecloth, and other objects. Based on the form of the position of the motifs, the types of long cloth batik patterns in Indonesia can be divided into at least seven patterns, namely the morning-evening pattern, the one-headed pattern, and the two-headed pattern. The batik cloth with the long cloth pattern described above is expected to be used as a *jarit* or sarong for women. If the cloth is used to produce a shirt or blouse, the resulting clothing will have a motif that cannot be *sanggit* in some parts of the *jarit*.

Second, the jarit-shirt batik pattern is a pattern for creating batik motifs; the resulting batik can be used for *jarit* as well as made into shirts of different sizes with stick motifs. This jarit-shirt has four types of batik patterns: 1) long-sleeved *jarit* shirt pattern, 2) short-sleeved *jarit* shirt pattern, 3) asymmetric long-sleeved *jarit* shirt pattern, and 4) asymmetric long-sleeved *jarit* shirt pattern, asymmetrical short sleeve shirt pattern.

Third, the shirt batik pattern is 1) a rectangular picture that is used as a guide for producing batik motifs; the batik cloth created by the pattern can then be made into shirts or blouses in a variety of sizes (M, L, XL, XXL) while the motive remains *sanggit*. [2], 2) a short-sleeved shirt batik pattern [3], and 3) a jumbo-size shirt batik pattern. The drawback of the three batik shirt patterns is that they can only be used for shirts and not for sewing or sarongs. A shirt is an essential piece of clothing, especially for men. Since the shirt's incremental success in the nineteenth century until today, global market demand for the shirt has been increasing [4].

Specifically for making long-sleeved shirts of various sizes whose motifs remain *sanggit*, based on the above pattern, it can be used 1) batik pattern for long-sleeved shirts with cloth size 250 cm, 2) jumbo shirt batik pattern measuring 270 cm, and 3) batik jarit pattern, long-sleeved shirt 260 cm cloth size. The three patterns listed above, however, have drawbacks. The downside of the long-sleeved shirt batik pattern is that it needs a 250 cm long cloth, the jumbo shirt batik pattern requires a 270 cm long cloth, and the long-sleeved-long-sleeved shirt batik pattern requires a 260 cm long cloth and cannot produce asymmetrical motifs.

This research identified 210 shirt patterns based on the flaws of the three types of patterns. The 210 shirt pattern is a rectangular pattern that uses a fabric 210 cm long x 115 cm wide as a guideline for creating batik motifs, and the resulting batik cloth can be made into a long-sleeved shirt of different sizes with motifs that remain *sanggit*. As compared to existing patterns, this 210 shirt pattern has the potential to increase the cost efficiency of batik production by 16% (250-210/250).

The batik industry, batik motif designers, batik makers, batik dyes, batik patterned clothing fashion designers, the convection industry, and batik consumers will all benefit from this 210 long-sleeved shirt pattern. The advantages for the batik industry include: a) the potential to produce a variety of batik motifs, both symmetrical and asymmetrical, for long-sleeved shirts with sanggit motifs; and b) the potential to save up to 16 % of batik production.

Benefits for motif designers include the fact that this pattern makes it easier to work on designing batik motifs for long-sleeved shirts, despite the fact that the motifs produced are of a high level of difficulty. Benefits for batik makers and dyers: this pattern makes it simpler to do batik and color the fabric based on the pair of motifs, even though the position of the motifs spreads out.

The advantage for tailors is that it will be easier for them to determine the broken sections of the long-sleeved shirt design that is produced so that the tailor will find it easier to cut the batik cloth to make it into long-sleeved clothing. The advantage for the consumer community is that by looking at the motive on the back, they would be able to better grasp the batik motifs as a whole if the batik cloth has been sewn into a shirt.

2 REVIEW OF RELATED LITERATURE

There has been a lot of researches conducted on clothing patterns, both men's and women's clothing patterns, however specific research on patterns for producing motifs on clothes, where the patterned cloth produced can be made into clothes with *sanggit* motifs has been uncommon.

One related clothing pattern research, for example [5], examines the making of party dresses for students of Syafi'i Akrom Pekalongan Vocational High School, and he uses combination patterns more effectively than construction patterns. When using hybrid patterns, the resulting clothing is better and the time required is shortened. The disadvantage is that since the *draping* pattern uses the main material directly, certain errors in cutting the fabric would result in an additional cost, it is best to be cautious when creating a combination pattern.

The research results of [6] on the Dotted-Board Model (DBM) and Extended Local Search (ELS) for the optimization of the layout of fashion patterns on patterned materials using pattern harmony rules, the overall average result of the combination of ELS and DBM contributed positively in increasing the computation time, namely the computation time of ELS with DBM. The optimum resolution and Nmo that can be obtained by combining ELS and DBM are 5 and 3, respectively, with an average efficiency of 56% and a computation time of 381 seconds. In this research, the patterned materials used are those with geometric and repetitive motives, making it easier to match the motifs. If the motif is not geometric or even abstract, the situation would be different. According to that research, there are three major items that have been studied in relation to motif patterns, namely fashion patterns, the creation of motifs without paying attention to patterns, and the application of fashion patterns. Combining the three items listed above, namely compiling a pattern on a piece of cloth, a strategy for placing a clothing pattern on the cloth so that the motif can be organized, and a strategy for placing a pattern on a clothing pattern. Thus, the patterned cloth produced by this pattern model is used to make shirts with *sanggit* motifs. To produce a shirt pattern, several factors must be considered, such as the various types of broken shirt patterns, the standard size of the adult body, various standard sizes of fabrics as shirt material, various types of motifs, factors that influence the quality of shirt items, etc. There has been a great deal of research on the development of clothing motifs. For instance, research on coffee and cocoa as a source of inspiration for Jember batik motif creations [7]. Local arts and culture can also be explored and developed to inspire the design of Balinese batik motif designs [8]. Regional icons have also become a source of inspiration for Indonesian batik motif development [9]. Archaeological site elements include elephants, elephant heads, elephant legs, and bones, which were used to create batik motifs as in Sragen region [10].

There are three types of adult shirt patterns used for the manufacturing of long-sleeved shirts: body broken pattern, sleeve broken pattern, and collar broken pattern.

Upper chest circumference, shirt length, long sleeve length, arm circumference, wrist circumference (cuff), neck circumference (collar), and pocket size are all adult body measurements that must be taken. The actual sizes used in the small, medium, big, X-large, and XX-large labels vary depending on the producer's region and target market. Manufacturers whose products are marketed to adult men offer a larger range of clothing sizes than producers whose products are marketed to young men [11]. Examples of the two standard indicators for upper chest circumference proposed by British Standards and widely used by manufacturers in the UK are shown in Figure 1.

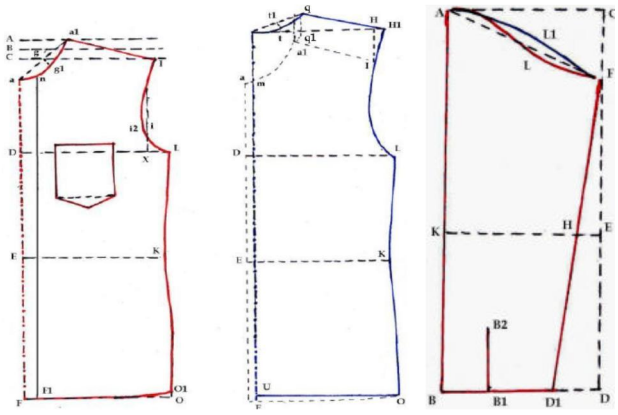

Figure 1 Men's fashion body and long sleeve designs

Table 1 Standard upper chest circumference [cm] according to British Standards

Target consumers	S	M	L	XL	XXL
Young man	<94	97-102	103-109	112-117	119-125
Adult male	94-99	99-107	107-114	114-122	122-130

Table 2 The size [cm] for broken pattern of men's shirts [11]

Target consumers	S	M	L	XL
Upper chest circumference	92	100	108	116
Neck circumference	38	40	42	44
Sleeve length	80	82	84	86
Wrist	17	17.8	18.6	19.4
Half the width of the back	19	20	21	22

Table 3 Size [cm] for broken pattern of the shirt [22]

Descrption	S	M	L
Low chest	18	22	24
Shirt length	57	66	70
Shoulder width	33	40	46
Half the width of the face	22	25	28
Shoulder width	11	15	16
Short sleeve length	19	21	23
Arm circumference	28	31	34
Neck circumference	30+2	35+2	37+2

Various types of cloths that are commonly used for batik shirts include primissima cotton cloth, prime cotton cloth, and 115 cm wide silk. Batik motifs that are considered beautiful on a piece of cloth may not be as appealing when made into a shirt or blouse. This is induced by the process of making clothes by cutting and stitching the cloth. For example, a piece of well-patterned batik cloth is then made into a shirt garment by cutting it to the size of the pattern and stitching it. The motifs on the joint of the clothes will no longer be able to connect, so the motif does not appear intact, does not meet, or does not *sanggit*. Thus, the batik cloth, that will serve as quality clothing for the sanggit motif, must be designed in such a way that the motif's aesthetic value can be obtained. The motif pattern layout must be designed based on the type of motif, clothing pattern, production techniques and adult body standard size.

There are seven aspects that need to be considered in designing product designs, namely functional, technical, ergonomic, economical, environmental, socio-cultural, and visual aesthetics [12]. In terms of motif pattern, the most important factor to remember is the aesthetic aspect, namely that the motif can still be pitted in various sizes of shirts. Functional aspect is precisely the suitability of motives and their implementations. The economic aspect is specifically the match between the pattern size and the cloth size required for the shirt. In terms of the environment, Fanina and Suaedi [13] claimed that Solo batik products have a significant correlation between consumer willingness to consider environmental conditions and batik sustainability. In term of clothing in general, Algahni and Al-Dabbagh [14] stated that consumers prefer sustainable clothing that is attractively designed, of excellent quality, and reasonably priced. Besides, consumers have emphasized the importance of having sufficient information about clothing trends on social media.

Furthermore, several important factors in the design process of textile products (batik) include products that can be produced, products that can be marketed, products that can be used, and products that are attractive [15]. Shirt pattern design, including design in general, is a creative industry business which is unique amongst cultural industries. Design innovation requires the incorporation of a wide range of expertise, such as the one shared by designers, consumers, and company performance [16].

To create a batik patterned long sleeve shirt pattern, one must first understand the batik manufacturing process that will be used on the long sleeve shirt pattern. The written batik technique, the resin printing technique, the wax printing technique, and the full print technique are the batik textile production techniques that are essential for working on long-sleeved shirt patterns. There are several different types of batik motifs that can be used in long-sleeved shirt patterns, including geometric motifs, symmetrical motifs, asymmetrical motifs, abstract motifs, italic motifs, and random motifs.

3 METHODS

Using a qualitative descriptive approach and participatory action research, this research was conducted in the Jalidin batik industry in Sragen and the tailors of Royal Sukoharjo, Central Java Province, Indonesia. The qualitative descriptive method is used to establish a draft long-sleeved shirt pattern which is standard for adult sizes, and the participatory action testing method is used to evaluate the pattern draft before it becomes a standard shirt pattern. The data sources in this research include participants, documents, and events. Participants consisted of 1 businessman as well as motif designer, 2 batik makers, 1 batik dye, 1 tailor, and 3 batik consumers. The documents used are mainly the 210 batik patterns tested, screen motifs, symmetrical batik motifs, broken long-sleeved shirt patterns, and batik shirts produced from the patterns studied.

All of the processes related to the making of the 210 long sleeve motif pattern, including the pattern design process, motifs design process, batik production process, pattern production, cloth cutting process and shirt sewing process, were observed. The results were presently documented in the research. The data sources were based on purposive techniques, snowball and time sampling.

Then the data were collected through observation techniques [17] on the events and the resulting documents, interviews [18] with participants and informants, focus group discussions [19] with all participants, and literature studies, and further data analysis using a flow model [20].

4 RESULTS AND DISCUSSION

To discuss the pattern of long sleeves with the Sanggit pattern, the standard shirt pattern size for adults (Indonesia) must be determined. Based on several references, such as [21-23], a split component size of the long sleeves shirt pattern for Indonesian adults has been determined as described in Table 4.

Table 4 Size [cm] of broken long sleeve shirt pattern for Indonesian adult

Broken shirt pattern	M	L	XL	XXL
Half rear body circumference	54	56	58	60
A quarter of the circumference of the front	30	32	34	36
Shirt length	76	78	80	82
The circumference of the arm base	48	50	52	54
Long sleeve length	57	58	59	60
Short sleeve length	21	23	24,5	26
Neck circumference/collar	38	40	42	44
Wrist circumference	17	17,8	18,6	19,4
Cuff	23	23,3	23,6	24
Pocket	12x14	12x14	12x14	12x14

The pattern of the 210 long sleeved sanggit shirt (Figure 2a) is used as a reference for developing a batik motif in a sleeve of 115x210 cm where it is possible to produce batik clothes in long-sleeved shirts in size S, M, L & XL, and short-sleeved XXL and the motifs can remain sanggit .The produced sanggit pattern motif lies in the front buttoned shirt, the right side seam, the left side seam, and the upper left chest pocket with the motifs around it.

This 210 long-sleeved shirt pattern is divided into 7 parts, namely the back or back area of A, measuring 73 cm wide and 90 cm long; fields B and C, right chest and left chest area measuring 42 cm wide and 90 cm long, respectively; plane D and E, right and left arm, measuring 60 cm at arm's circumference and 61 cm in arm length, respectively; F area, cuff and collar, measuring 12 cm wide and 120 cm long; G area, the pocket section measures 42 cm by 30 cm.

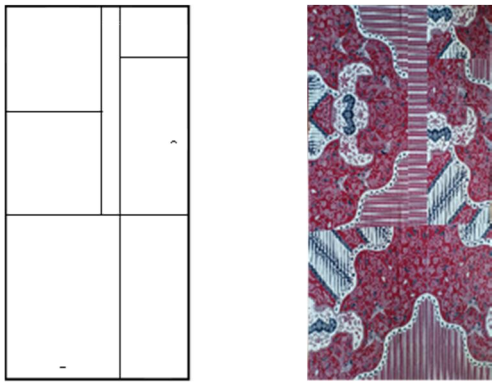

Figure 2 The 210 pattern motif (a) and the batik of 210 pattern (b)

Figure 3 Asymmetrical long-sleeved front *sanggit* shirt, sides, pockets

Asymmetrical batik motif design (master motif shown in plane A) extended to long sleeve shirt pattern 210. Furthermore, the 210 pattern is batik on cloth which measures 115x210 cm (Figure 2b). Furthermore, the patterned batik cloth is made into a long-sleeved shirt in size M (Figure 3), such that the shirt is visible from the front, behind, left side, and right side. The motif on the front is reversed with the motif on the back. The sanggit is depicted on the front shirt motif, as well as the pocket motif and the surrounding motifs (Figure 3a). The left and right sections of the shirt motif (Figure 3c) are both sanggit (Figure 3d). The motifs on the cuffs and collar are striped in shape, as compared to the main motif, which is in the shape of a machete and an *ukel* flower.

Pattern 210 analysis for a long-sleeved shirt

A trial analysis of 210 long-sleeved shirt-patterned batik cloth is produced into a long-sleeved shirt in sizes XXL (Table 5), XL (Table 6) and M (Table 7).

Table 5 shows that:

1. The broken portion of the shirt pattern shown is only the broken shirt pattern linked to the shirt pattern's formulation. Half back body circumference, front body quarter circumference, shirt length, sleeve circumference, long sleeve length, cuffs, collar, and pockets are the pattern's split components.
2. The pattern size and code are the codes for A to H on the pattern, and the size is that which is specified on the pattern.
3. Kampuh is the edge of the cloth which binds one cloth to another. The circumference of the base of the arm, for instance, is given an excess of 3cm for *jarit*, the right and left halves of back are given an excess of 2x2 cm = 4 cm, and the length of the shirt is given an excess of 6 cm for seams and *jarit*.
4. Body, the amount of cloth required to make a shirt in sizes ranging from M to XXL.
5. The total means that the required pattern size plus the size of the camp or seams.
6. Difference, namely the size of the cloth pattern minus the total amount of clothing required to produce the shirt.

According to Table 5, the relationship between the size of the pattern (Table 5) and the required XXL size shirt is as follows. Code A, the size of the back half of the body circumference in the pattern is 73 cm, although it takes 64 cm to make a shirt so that the cloth is only 9 cm wide. The length of the shirt in the pattern is 90 cm, however the length of the shirt required is 88 cm, so there is still 2 cm of cloth remaining. Code B or C, measure a quarter of the circumference of the right or left front of the body in a pattern measuring 42 cm, while a shirt takes 38 cm, so there is still 4 cm of cloth left. Thus, for the entire body circumference there is still cloth remaining as wide as 15 cm (9+2+4 cm).

Code D or E, the sleeve circumference pattern size is 60 cm, although it takes 57 cm to produce a shirt, remaining 3 cm of cloth. The length of the pattern's long sleeves is 61 cm, but it takes 63 cm to produce long sleeves, so "the length of the fabric is less than 2 cm" (-2 cm). As a result, this 210 long sleeve shirt patterned batik cloth cannot be used to produce XXL long-sleeved shirts. Meanwhile, when it is used to produce a short-sleeved shirt, it takes 33 cm of the sleeve length, leaving the cloth 28 cm with full body circumference of 149 cm (73+42+42-8 cm).

Code F, cuffs and collars in a pattern measuring 12x120 cm, enough to render two cuffs measuring 12x24 cm = 48 cm long and a shirt collar measuring 12x24 cm = 48 cm long (12x44 cm). As a result, the F cloth is 12x20 cm. G code, the upper left chest pocket in the pattern is 42 cm wide x 30 cm high, whereas making the pocket requires a cloth measuring 17x21 cm, so there is a width and height excess of 25 and 9 cm. The excess of the pocket pattern size of 25 cm wide and 9 cm high is used to provide flexibility in positioning the motif on the pocket with the motif on the chest between the sizes M, XL and XXL so that the motif on the pocket size (13x15 cm) can be *sanggit* with motifs around it.

The analysis in Table 5 shows that this 210 pattern cannot be made into a “long-sleeved” XXL size shirt with a *sanggit* motif, but only a “short-sleeved” XXL size shirt with a *sanggit* motif.

Table 5 Comparison of pattern size [cm] for long-sleeved shirt 210 and fabric requirement for long- sleeved shirt size XXL

Part of the broken pattern	Pattern codes	*Kampuh* (Hem)	Body	Total	Difference
1	2	3	4	5=4+3	6=2-5
Half rear body circumference	A-73	4	60	64	9
Shirt length	A-90	6	82	88	2
A quarter of the front body circumference	BC-42	2	36	38	4
The base arm circumference	DE-60	3	54	57	3
Long-sleeved length	DE-61	3	60	63	(-2)
Short-sleeved length	DE-61	7	26	33	28
Cuffs and collars	F-120	4; 4	2x24; 44	52; 48	20
Pocket	G-42x30	4+6	13x15	17x21	25-9

Table 6 Size comparison [cm] of 210 long sleeve shirt patterns with a specification for an XL long-sleeved shirt

Parts of the broken pattern	Pattern codes	*Kampuh*	Body	Total	Difference
1	2	3	4	5=3+4	6=2-5
Half rear body circumference	A-73	4	58	62	11
Shirt length	A-90	6	80	86	4
A quarter of the front body circumference	BC-42	2	34	36	6
The base arm circumference	DE-60	3	52	55	5
Long-sleeved length	DE-61	3	59	62	(-1)
Cuffs and collars	F-120	4; 4	47.2; 41	51.2; 45	23.8
Pocket	G-42x30	4-6	12x14	16x20	26x10

According to Table 6, the relationship between the size of pattern 210 (Table 6 codes) and the required size of the shirt XL size is as follows. Code A, the size of the back half of the body circumference in the pattern is 73 cm, while it takes 62 cm to make a shirt so that the cloth remains 11 cm wide. The length of the shirt in the pattern is 90 cm, however the length needed is 86 cm, leaving 4 cm of cloth. Code B or C, measure a quarter of the circumference of the right or left front of the body in a pattern measuring 42 cm, while a shirt takes 36 cm, leaving 6 cm of cloth.

Code D or E, the sleeve circumference pattern size is 60 cm, but it takes 55 cm to make a shirt, so there is still 5 cm of cloth remaining. The pattern's sleeves are 61 cm in length, but it takes about 62 cm to produce a shirt, so "the length of the fabric is less than 1 cm." The required seam length is 3 cm, but only 2 cm of cloth is available; this 1 cm deficiency can be made up using the cloth from the F (collar) part. As a result, this 210 long sleeve shirt patterned batik cloth can be used to make XL size of long-sleeved shirts.

Code F, cuffs and collars in a pattern measuring 12x120 cm, enough to make 2 cuffs measuring 12x23.6 cm = 47.2 cm long and a shirt collar measuring 12x23.6 cm = 47.2 cm long (12x41 cm). As a result, the fabric element F is 12x23.8 cm. The upper left chest pocket in the pattern is 42 cm wide x 30 cm high, however the pocket includes a cloth measuring 16 20 cm, so there is a width and height excess of 26 and 10 cm.

The conclusion of the analysis in Table 6 is that this 210 batik pattern can be made into an XL size long-sleeved shirt with sanggit motifs.

In addition, Table 7 shows the relationship between the size of the motif pattern 210 and the cloth required to make a long-sleeved shirt size M. Since the pattern's half-body circumference is 73 cm and the required shirt size is 58 cm, there is still 15 cm of cloth remaining. The pattern's shirt is 90 cm in length; however the required shirt length is 83 cm, so there's still 7 cm of cloth remaining. The remaining 7 cm cloth will be discarded later so that the tailor may select which batik motif to use or discard, the top or bottom motif. It is also possible that the motif chosen is the middle part, in which case the 3.5 cm upper and 3.5 cm lower motifs are discarded.

Codes B and C, measure a quarter of the front body circumference to the left or right in the pattern measuring 42 cm, while a shirt is needed 31 cm, leaving a cloth 11 cm wide. In codes D and E, the size of the cloth at the base sleeve is 60 cm, while the required shirt sleeve circumference is 51 cm, leaving 11 cm on the cloth. The pattern's arm length is 61 cm, thus it takes approximately 60 cm to produce a shirt so that the cloth remains 1 cm.

The pattern's cuffs and collars are 12x120 cm, while the fabric required to produce two cuffs is 12 cm wide, 4 cm long + (2x23 cm) = 50 cm, and the cloth required to produce a shirt collar size M is 12 cm wide and 4 cm + 38 cm long = 42 cm. It takes 92 cm of cloth to produce cufflinks and collars, leaving 28 cm of cloth. The pattern pocket is 42 cm wide x 30 cm high, although making a pocket requires a size of 16x20 cm, then there is still an excess of 26 cm in width and 10 cm height. The pocket pattern's excess size is used to provide flexibility in locating the pocket motif on the pocket with the motifs around it. Eventually, this 210 motif pattern can be modified into M size long-sleeved shirt.

Table 7 Comparison between cloth criteria for long sleeve shirt size M and pattern size [cm] for long-sleeved shirt size 210

Parts of the broken pattern	Pattern codes	*Kampuh*	Body	Total	Difference
1	2	3	4	5=3+4	6=2-5
Half rear body circumference	A-73	4	54	58	15
Shirt length	A-90	6	76	83	7
A quarter of the front body circumference	BC-42	2	30	31	11
The base arm circumference	DE-60	3	48	51	9
Long-sleeved length	DE-61	3	57	60	1
Cuffs and collars	F-120	4; 4	46 38	50; 42	28
Pocket	G-42x30	4-6	12x14	16x20	26x10

The Efficiency of 210 Pattern

There are currently at least three patterns of batik motifs which can be used to produce long-sleeved shirts in a range of sizes with *sanggit* motifs. These patterns are (1) long-sleeved shirt pattern, (2) long-sleeved shirt pattern and (3) motif 210 long-sleeved shirt pattern. The following is a comparison of the three patterns:

The pattern of a long-sleeved shirt-long rectangular size 115x260 cm, can produce batik cloth with motifs that are used for *jarit* and can be made into long-sleeved shirts of various sizes with *sanggit* motifs (Figure 4).

a) b) c)

Figure 4 The results of the pattern can be made into (a) *jarit* and (b, c) long-sleeved shirts with symmetrical sanggit motifs

a) b)

Figure 5 Symmetrical motif (a) and asymmetrical motif (b) of long-sleeved *sanggit* shirt

Long-sleeved shirt pattern in the form of a rectangle measuring 115x250 cm, can produce batik motifs into long-sleeved shirts of various sizes with *sanggit* motifs. This pattern can be used to produce both symmetrical and asymmetrical motifs for shirts, but not *jarit* motifs (Figure 5).

The pattern of 210 long sleeved shirts is rectangular size 115x210n cm, can produce batik motifs to be made into long-sleeved shirts of sizes M, L, and XL, which motifs are *sanggit*, and the XXL size of the short sleeve shirts.

This pattern can only be used to produce symmetrical or asymmetrical motifs for long-sleeved shirts; it cannot be used to produce motifs for *jarit*.

The 210 pattern has an advantage over the previous pattern in that it only needs a 210 cm cloth length. As compared to the long-sleeved shirt pattern, which is 250 cm in length, the 210 pattern has a 16%.

Efficiency rate = (250-210)/250 = 40/250 = 16%

That is, if producing one piece of batik cloth using the previous pattern can cost Rp. 500.000, but using this 210 pattern it will cost only Rp. 420.000, then the efficiency value of the 210 pattern is Rp. 80.000. (16% x Rp. 500.000). The limitation of this 210 motif pattern is that it cannot be used to make long-sleeved XXL shirts with sanggit motifs, but it can be used to make short-sleeved XXL shirts.

5 CONCLUSION

Based on the discussion above, it can be concluded that if batik producers will produce batik cloth which is projected to only be used as long-sleeved shirts with a sanggit pattern, then the right solution is to produce batik using the "sanggit pattern 210 for long-sleeved shirt". The sanggit pattern 210 for long sleeve shirt measures 115x210 cm, it can be made into long-sleeved shirts in sizes S, M, L, and XL with sanggit, as well as a short-sleeved XXL shirt with sanggit motifs.

The advantages of this sanggit 210 pattern are as follows:

1) It has a batik production cost efficiency of 16%. For instance, if one piece of batik cloth costs Rp. 500.000 to produce, then using this pattern of 210 motifs can result in an efficiency of Rp. 80.000.
2) The *sanggit* 210 batik cloth can still be made into long-sleeved shirts in sizes S, M, L, and XL with the sanggit motif, as well as short-sleeved shirts in size XXL.
3) The 210 motif pattern can be used to produce highly difficult batik sanggit motifs, such as asymmetrical motifs, oblique motifs, and abstract motifs. Moreover, shirt motifs on the collar and cuffs may be made with motifs other than the main motif.

To manufacturers of shirt-patterned batik fabrics, it is recommended that when marketing the 210-patterned batik cloth to include guidelines for the broken pattern of 210 design. The pattern image can be used as a reference for consumers and tailors when reading batik motifs while the batik cloth is still in the form of a sheet (raw material), as well as reading parts of the motif based on the broken pattern of long-sleeved shirts. Before purchasing a batik cloth with a long-sleeved shirt pattern, customers should first read and understand the motif so that the batik purchased is in accordance with their desires and needs.

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THE SOLUTIONS OF TEXTILE BRANDS TO THE INVENTORY PROBLEMS CAUSED BY THE COVID-19 PANDEMIC IN SWITZERLAND

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Abstract: *This study focuses on the solutions of textile brands to the inventory problems caused by the Covid-19 pandemic in Switzerland. The aim of this research is to present a wide range of solutions to the inventory problems of the fashion industry and to analyse which solutions are most commonly used by apparel companies. An online survey is conducted for the research. 15.79% of the companies participating in the survey do not try any approach to reduce their inventory; however, all of these companies express that they do not even have inventory problems. On the other hand, 84.21% of the respondents select at least one approach from the list, which they use for inventory reduction. Companies find the approach to improve the service levels the most helpful. This approach is followed by improving forecast accuracy. Besides, the approaches: setting more focus on the quality, values, and sustainability and the three-season-strategy are popular among the companies.*

Keywords: *fashion industry, Covid-19, coronavirus, textile, supply chain, inventory.*

1 INTRODUCTION

For a considerable time, textile industry has been criticized for its unsustainable and polluting nature. Although there have been attempts to put more focus on sustainability and to slow down fast fashion, most brands produce several collections per year. The products of fast fashion collections are often manufactured in low-wage countries, where employees work under desperately miserable conditions to keep the costs and prices low. As a result, the quality of the products is poor leading to an increase in the number of products purchased per year per customer.

The lockdowns lead to a severe increase in inventories as the goods cannot be sold. The crisis awakens the companies of the sector, and they try to focus on a radical reinvention. This research paper focuses on the solutions that textile brands developed to reduce their high stocks.

2 AIM, HYPOTHESES, AND RESEARCH QUESTIONS

Current paper possesses two aims. One of them is to present a wide range of solutions with detailed descriptions and examples for the inventory problems of the fashion industry thus supporting textile companies to overcome their stock problems. Secondary sources are collected and analyzed to provide a compiled list of actions that textile companies might take to solve their stock issues. The other goal is to examine which measurements have already been introduced or are planned to be taken in the future by fashion brands to overcome the crisis in Switzerland. The research focuses on the behavior of apparel companies as they try to optimize their stocks. The hypotheses and research questions are the followings:

H1: 75% of Swiss textile companies face increased inventories due to the Covid-19 pandemic.
Q1: What is the percentage of textile companies facing excess inventories?

H2: There is no difference between the bridge brands and all other brands in terms of their inventory situations.
Q2: Are there any significant differences between the bridge brands and all other brands in terms of their inventory situations?

H3: The average inventory level of Swiss textile companies does not increase due to the Covid-19 crisis and lockdowns.
Q3: Does the average inventory level of Swiss textile companies increase due to the Covid-19 crisis?

H4: Textile companies take several measures to reduce their inventories.
Q4: Which approaches do textile companies use to reduce their inventories?

3 LITEREATURE REVIEW

3.1 Related terms of warehouse management

There are several terms related to warehouse management. This part of the research paper sums the most crucial terms up to give an overall understanding of them.

Warehouse management is a field of activity efficiently operating the warehouse and distribution system [1]. The purpose of warehousing is to store goods, to preserve their condition, and to balance the material flows of the supply chain as needed. Warehouses preserve the quality and quantity of goods without loss, and through their material and goods handling system, they allow goods to be removed and stored as necessary. Warehouses are complex facilities that have their own specific network of relationships and internal processes. Successive phases in the logistics chain are connected through warehouses, which provide the material requirements for production or delivery. Warehouses and storage systems are primarily needed to compensate for the differences in the economic capacity of various work processes [2].

Warehouse management and inventory management have overlapping areas, but these terms are not synonyms. Inventory management is a *«system designed for the management of quantities and locations (storage locations) and especially their interrelations»* [1]. The inventory management focuses on those products and goods that are hold in the inventory as well as on the management of the quantities and storage locations [1]. Two strategies, namely constant inventory and variable inventory are available in case of the inventory management. On the one hand, the constant inventory strategy should be used when the demand is constant over time. On the other hand, the variable inventory strategy is more appropriate if the demand fluctuates, and there are very high peaks in the demand from time to time [3].

Warehouse management analyzes the warehouse and distribution system. It concentrates on the journey of goods within the warehouse between their arrival and dispatch [1].

Demand management matches the supply with the demand proactively to decrease the probability of disruptions [4].

The target of logistics is to have:
- the right goods
- at the right time
- in the right quantity
- in the right quality
- at the right location
- at the right costs. [1]

3.2 The Covid-19 pandemic and its impact on the fashion inventory management

The first infection with the virus of Covid-19 was registered on December 8, 2019 in China [5] and the infection series was classified as pandemic on March 12, 2020 [6].

The rapid spread of the virus quickly led to various closures at different places all over the world. Although the Covid-19 pandemic is a health crisis, it has a major impact on the economy and ultimately leads to an economic crisis. As the fight against the virus requires social distancing, lockdowns and postponements of events become necessary. Additionally, the spread of the virus generates the feeling of insecurity in citizens; thus, they reduce their consumptions to save financial resources up [7].

The Covid-19 pandemic has a negative effect on many sectors including the textile industry. Various players of this sector have already experienced the consequences of the pandemic. First of all, retailers struggle as they have to look for new distribution channels due to the lockdowns. Furthermore, customers stay away due to uncertainty of the future. Additionally, due to the production cancellations, sweatshop workers of the Far East lose their jobs leading them to deep poverty [8]. Moreover, lockdowns cause job losses, layoffs, supply problems, declining in sales and net profits across most fashion brands. Both small and large companies and companies of all price levels are affected by the decline in sales. Usually, customers adapt to crises by preferring products from the lower price range instead of the more expensive ones bought earlier. However, during this crisis, they behave differently and refrain from buying products [9].

Although the crisis brings many challenges, Buheji and Ahmed [10] emphasize that the situation creates many opportunities, which can have a positive effect, too. The crisis directs the attention to the areas not working currently. In these areas, the crisis leads to problems that need to be addressed thus triggering a positive outcome and a better solution for the future. One of the problems is the increase in the stock of the retailers and brands [11]. During the pandemic and lockdowns, brands and retailers cannot sell as many products as they used to before the crisis. This circumstance leads to the brands having higher stocks [12]. Additionally, huge amounts of their products follow short-term trends; therefore, these are more complicated to get sold later. These reasons lead to discounts after the lockdowns and to lower turnover.

The fashion industry works based on seasons; most of the brands have at least two seasons each year: spring/summer and autumn/winter. A lot of companies have additional capsule collections to follow the trends and constantly attract customers with new products [13]. Due to fast fashion, the number of micro seasons can reach up to 50-100 for a brand each year. Nowadays, customers are trend-focused, and due to the constantly changing trends, they buy 60% more clothing items than in 2000. At the same time, the time garments are kept in just half as long as before. As these examples highlight, the length of fashion cycles is steadily decreasing [14].

Calculating the demand for a collection is challenging as brands would like to benefit from the economies of scale and produce the highest possible quantity to satisfy the demand but at the same time, avoiding overproduction and sitting on the stocks for a longer time [15].

The problem of high inventories due to the Covid-19 pandemic was hinted at in February 2020. Back then, it was estimated that 10 to 15 million products were likely to be involved [16]. Currently, it is estimated that the industry groans under an overstock of around 40% [17]. As Freutel [18] wrote in January 2021, the inventory level of clothing retailers has never been as high as it is currently, even the value was lower in April 2020. Normally, 12% of the inventory remains unsold. Currently, it is more than the double of this industry's standard level [19]. Some companies stockpile products to protect themselves from sales through high discounts. However, the high stock levels mean space problems for the retailers [20].

3.3 Fashion supply chain model

The fashion supply chain is complex, and several stages of it are affected by inventory management. Thus, raw-material suppliers, textile producers, and garment manufacturers need all ingredients in their inventory to be able to start producing. Moreover, the distribution channels depend on the right inventory to fulfill the orders. On the other hand, if companies have overstock, they lock down capital and cash flow that makes them less adaptable to changes. Therefore, inventory management plays an important role in the success of companies operating in the textile industry.

There are several models available that visualize the supply chain process of the fashion industry. Based on the models of Shen and Mikschovsky [14], EURATEX [21], Martin [22], Gries et al. [23], Brito et al. [24], and the Swiss Federal Office for the Environment [25], a model is developed as part of current research. It models the actors and stages of the textile supply chain.

Similarly to Shen and Mikschovsky's [14] and Brito et al.'s [24] models, the developed model is based on a closed loop due to the fact that fashion industry pays more and more attention on sustainability and recycling. Even if today's recycling practices are not fully integrated in the supply chain of the textile industry, the possibility for this kind of action is marked with dashed lines.

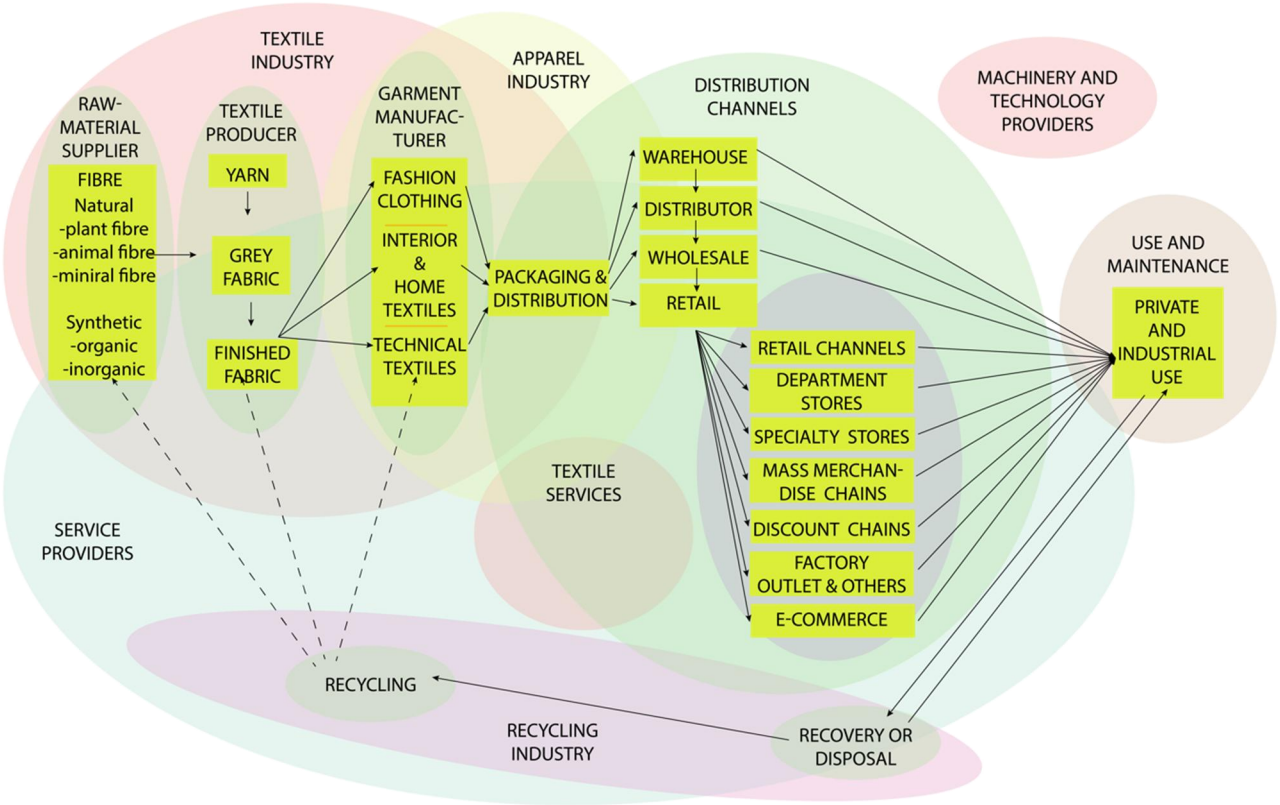

Figure 1 Fashion supply chain model

3.4 Approaches to solve the inventory problems in the fashion industry

The Fashion Inventory Management is especially important because it avoids stockouts, increases customer satisfaction, keeps carrying the costs low, helps in optimizing storage places, prevents tying up capital, and helps in maintaining the company's health [26].

As described above, nowadays, the fashion industry faces the problem of high inventory. There are approaches available to solve the problem [27]. Due to the Covid-19 crisis, several companies have already recognized that the reorientation of the fashion industry is necessary. Especially the concept of fast fashion and its environmental, social, and economic impact is questioned. According to designers and textile industry professionals, the adjustment of the seasonal cycles has been due for years; therefore, the return to the traditional two collections per year should take place. Additionally, the focus should go toward the direction of sustainable fibers and fabrics [13]. There are a lot of possibilities for the fashion industry to become more sustainable than it is currently; a high amount of their supply chain consumes natural resources [14]. According to Emig [28], the demand for high-quality products purchased for a lifetime is increasing likewise the aim of customers to support their local retailers. In order the fashion industry to be able to return to slow fashion, the customers need to make conscious apparel purchases [15].

Several brands have already committed to slow down, among others Giorgio Armani. He plans to reduce the number of their collections and to set more focus on the quality, values, and sustainability [29]. Besides Giorgio Armani, Gucci has committed to create non-seasonal collections and to reduce their shows, as well [30].

A different approach toward slower fashion has been taken by Ralph Lauren. The brand expands its on-demand manufacturing and personalized garment offerings. This approach not only allows the company to be more responsive to customer needs but to reduce inventory and waste as well as to focus more on the full-price business [31].

Moreover, sales are supported by comprehensive data analyses and tools. Dynamic pricing can be used to sell surplus stock faster and with higher margins [19].

The blending of retail and online warehouses can be helpful, as well. Retailers can use their numerous shops as warehouses for the delivery of online purchases. This method can shorten delivery times and reduce inventory and delivery costs [31].

Furthermore, some brands postpone the launch of part of their collections [32]. Callersten et al. [11] recommend a three-season strategy for fashion brands. This solution could increase both stock monetization and profitability over at least three selling seasons. In the long term, this method could replace the short-term solution of discounting. Besides reorientation, fashion brands have several possibilities to improve their inventories. Blanchard [33] defines 12 ways to reduce inventory:

- reduce demand variability
- improve forecast accuracy
- re-examine the service levels
- address capacity issues
- reduce order sizes
- reduce manufacturing lot sizes
- reduce supplier lead times
- reduce manufacturing lead times
- improve supply reliability
- reconfigure the supply chain
- reduce the number of items
- eliminate the questionable practices.

4 METHODOLOGY

In the first part of the paper, the theoretical foundations are uncovered with secondary sources. The related terms of warehouse management, the impact of the Covid-19 pandemic on the fashion inventory management, the approaches to solve the inventory issues in the fashion industry, and the fashion supply chain model are explained.

The next section, the primary research of the study, focuses on the practical behavior and handling of textile companies. The aim of this research is to collect and analyze the data provided by textile companies to identify their behavior. For this, a quantitative research is conducted. The answers of companies are measured with a Likert Scale. The respondents are able to agree or disagree with the statements on a predetermined multi-level answer scale. Additionally, yes-or-no questions are asked.

The quantitative survey is sent to 216 Swiss textile companies that are members of the "Swiss Textiles", the association uniting the actors of the Swiss textile industry [34].

Of the emails sent, 17 are not delivered; thus, 199 email requests are successfully received. The survey in the form of another email is sent again as a reminder after 10 days. Due to the anonymity of the survey, it is not possible to contact exclusively those companies that do not fill out the survey. That is why multiple reminders are not sent. During the two-week period when respondents could complete the survey, the survey is completed 19 times. This results in a response rate of 9.55%.

$$Response\ rate = \frac{19}{199} = 0.09547739 = 9.55\%$$

5 FINDINGS

The findings of the quantitative study are summarized in this section. For a better understanding of the situation of the companies, their demographical characteristics including their field, location, and positioning are examined. Of all companies, their headquarters are located in Switzerland. 21.05% of them have offices in other countries as well but 78.95% are based exclusively in Switzerland.

When comparing the companies' sizes, it can be seen (Figure 2) that most respondents to the survey are small and medium-sized companies and large corporations do not participate in the survey to a great extent. This factor is a limitation of the study and leads to the fact that the findings apply to the small and medium-sized companies and not to the large corporations.

Furthermore, the survey records the market levels where the companies operate. Companies could choose from:

- Haute Couture - meets the four main requirements of the French Ministry of Industry, offering made to measure products
- Luxury Fashion - high quality designer brands
- Bridge Brands - great quality clothing at a more adequate price point
- Diffusion Lines - created by luxury labels as more budget friendly, secondary lines
- High Street Fashion - accessible, quality fashion with a longer life span than economy items at affordable prices
- Fast Fashion - well-known, affordable brands, extremely quick turnover in fashion and trends
- Economy Fashion - mass production, low prices, low quality, and short lifespans of products

52.63% of the brands operates just at one market level and 47.37% operate at two market levels.

The luxury fashion and bridge brands market levels are the most common with 25%-25% of the respondents operating in both segments. It is followed by haute couture. High street fashion is indicated by 14.29% of the surveyed companies. 10.71% of the textile companies sell diffusion lines. Fast and economy fashion are very rarely marked by the respondents.

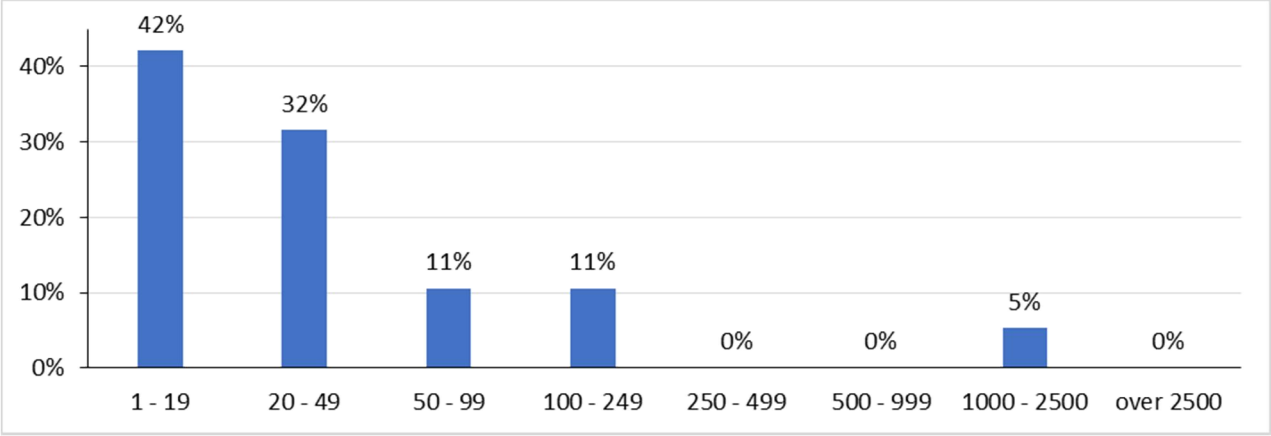

Figure 2 The total number of employees at all locations of the company

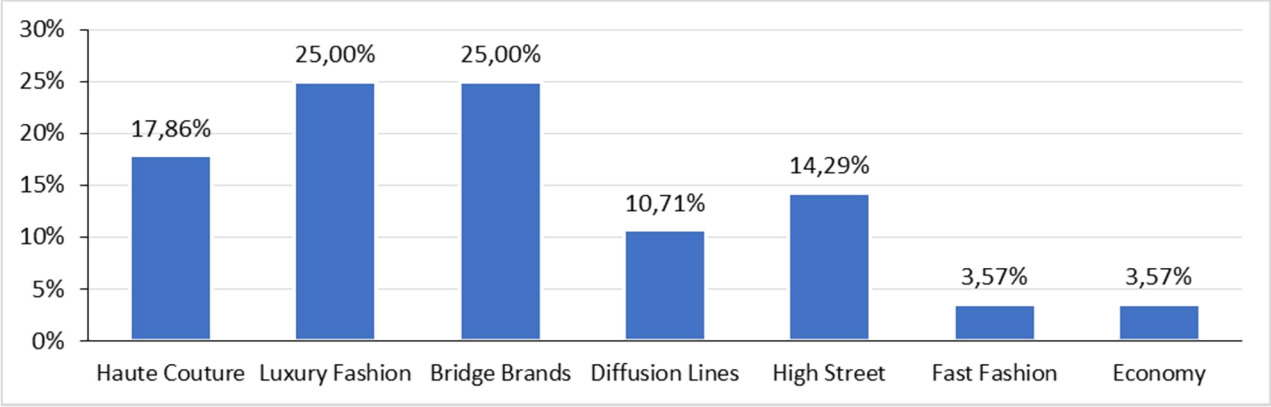

Figure 3 The breakdown of the companies' presences at different market levels

5.1 The investigation of the first hypothesis

The first examined hypothesis and research question are the following:

H1: 75% of Swiss textile companies face increased inventories due to the Covid-19 pandemic.
Q1: What is the percentage of textile companies facing excess inventories?

H_0: x=75%
H_a: x≠75%

To answer the research question, it has to be analyzed how many companies have to deal with increased inventories. Overall, 47.37% of the respondents face higher stock levels due to the Covid-19 pandemic. 52.63% of the companies do not feel any negative impact on their stock levels due to the virus and lockdowns.

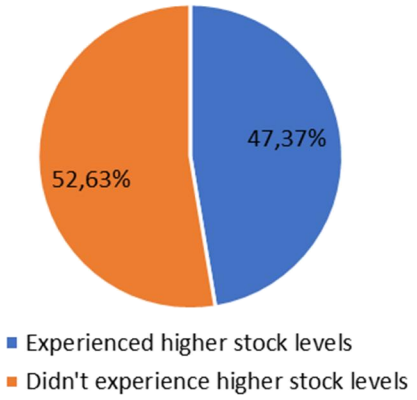

Figure 4 Experiencing higher stock levels

As 47.37% experience higher stock levels, p=0.4737. P_0 is 0.75 as it is attempted to find out whether it is 75%.

$$z = \frac{\mathrm{p} - \mathrm{P0}}{\sqrt{(\frac{P0(1-P0)}{n})}} \sim N(0,1) = \frac{0.4737 - 0.75}{\sqrt{(\frac{0.75(1-0.75)}{19})}} \sim N(0,1) =$$

$$= \frac{-0.2763}{0.0993} \sim N(0,1) = -2.7825$$

As this is a double-sided test, the absolute value of -2.7825 is used for further calculations, which is 2.7825.

z_{calc}=2.7825

The level of significance is 95%, which means α=0.05. However, as the test is double-sided, for the calculation $\alpha/2$ has to be used, which is 0.025. The critical value is 1.96.

z_{crit}=1.96

If $z_{calc} > z_{crit}$, then H_0 can be rejected. In this case, the requirement is fulfilled; therefore, the hypothesis has to be rejected; there is a significant difference. Not 75% of Swiss textile companies face increased inventories due to the Covid-19 pandemic.

5.2 The investigation of the second hypothesis

The second examined hypothesis and research question are the following:

H2: There is no difference between the bridge brands and all other brands in terms of their inventory situations.
Q2: Are there any significant differences between the bridge brands and all other brands in terms of their inventory situations?

In order to detect the factors that lead to differences between the companies and have an impact on the inventory levels, deeper analyses have to be carried out. Thus, it is investigated whether the activity of the different market levels has an impact on the increased inventory. It is studied at which market level operating companies have the most problems. Based on Figure 5, it is apparent that most of the distributors of bridge brands have higher stock levels due to the Covid-19 crisis. 54.55% of the companies that distribute these fashion lines have inventory problems. The chart shows the distribution of stock problems at all the market levels.

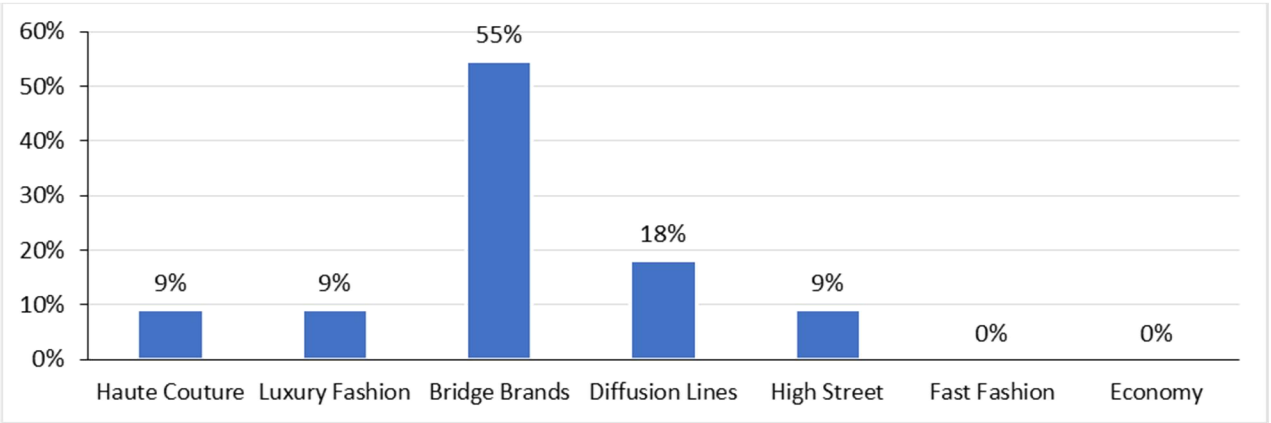

Figure 5 The breakdown of stock problems by market levels

To examine the problem further, a two-sample t test, which is based on the relation of all survey attendees and those that experienced stock problems, is conducted.

H_0: P1-P2=0
H_1: P1-P2≠0

$$z = \frac{p1 - p2}{\sqrt{\bar{p}\bar{q}(\frac{1}{n1} + \frac{1}{n2})}} = \frac{0.8571 - 0.2632}{\sqrt{0.41 * 0.59(\frac{1}{7} + \frac{1}{21})}} =$$

$$= \frac{0.8571 - 0.2632}{\sqrt{0.2419(\frac{4}{21})}} = \frac{0.5939}{\sqrt{0.04608}} = \frac{0.5939}{0.2147} = 2.7661$$

Where:

- *p1*=0.8571, which represents the percentage of those bridge brands that suffered by the Covid-19 pandemic caused inventory problems
- *n1*=7
- *p2*=0.2632, which represents the weighted average of all other price level brands that suffer by the Covid-19 crisis caused inventory problems
- *n2*=21
- As there are companies that sell goods in more than one category, the companies are examined as subsidiaries, raising the total number of items to 28.

$$\bar{p} = \frac{n1p1 + n2p2}{n1 + n2} = \frac{7 * 0.8571 + 21 * 0.2632}{7 + 21} =$$

$$= \frac{5.9997 + 5.5272}{7 + 21} = \frac{11.5269}{28} = 0.411675 \approx 0.41$$

$$\bar{q} = 1 - \bar{p} = 1 - 0.41 = 0.59$$

The level of significance is 95%, which means α=0.05. However, as it is a double-sided test, for the calculation $\alpha/2$ has to be applied, which is 0.025. The critical value is 1.96.

z_{crit}=1.96
z_{calc}=2.7661

If $z_{calc} > z_{crit}$, then H_0 can be rejected. In this case, this requirement is fulfilled; therefore, the hypothesis has to be rejected because there is a significant difference. In terms of their inventory situations, there is a difference between the bridge brands and all other brands. One particularly interesting finding is that companies struggling with higher inventory levels are those operating in only one market segment. Over three quarters of the companies having stock problems serve exclusively one market segment with their products. Of all the companies that are active in several market segments solely 22.22% have stock problems.

This result may indicate that companies representing multiple market segments are able to act more agilely and to find solutions quickly to align their actions with the needs of the market.

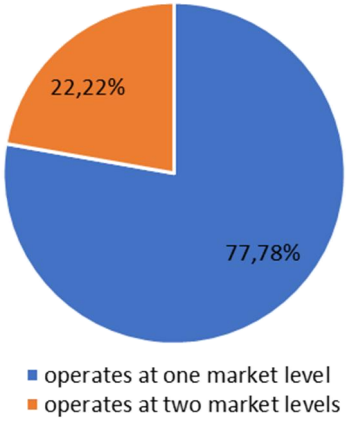

Figure 6 The breakdown of stock problems based on the operation at different market levels

5.3 The investigation of the third hypothesis

The third hypothesis and research question state:

H3: The average inventory level of Swiss textile companies does not increase due to the Covid-19 crisis and lockdowns.
Q3: Does the average inventory level of Swiss textile companies increase due to the Covid-19 crisis?

H_0: *μ1 = μ2 = μ3*
H_a: Not all population means are equal

47.37% of the companies do not have available information regarding their stock levels. These companies are excluded from the research on the development of the stock levels over the last seasons. To analyze the means of the groups, the statistics analysis of Anova is conducted. The level of significance is 95%, which means α=0.05.

Table 1 The Anova Table

SUMMARY						
Groups	***Count***	***Sum***	***Average***	***Variance***		
Before the Covid-19 crisis	10	1.13	0.113	0.00713444		
End of the SS20	10	1.5	0.15	0.00944444		
End of the FW20/21	10	1.37	0.137	0.00880111		
ANOVA						
Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
Between Groups	0.0070467	2	0.0035233	0.41646966	0.66353423	3.354130829
Within Groups	0.22842	27	0.00846			
Total	0.2354667	29				

Because the p-value is not <=α, the H_0 cannot be rejected. Thus, it is indicated that there is not enough significant difference between the changes in the stock levels during the examined seasons. As it is visible in the Table 1, the average percentage of the remaining stocks at the end of the seasons before the Covid-19 crisis is 11.3%. The average stocks of the textile companies have increased to 15% by the end of the season spring/summer 2020. After this peak, the average started to decrease again, and it is solely 13.7% at the end of the season fall/winter 2020/2021.

5.4 The investigation of the fourth hypothesis

The fourth hypothesis and research question examine the several approaches taken by the companies to reduce their inventories.

H4: Textile companies take several measures to reduce their inventories.

Q4: Which approaches do textile companies use to reduce their inventories?

Solely 15.79% of the companies participating in the survey have not tried any approaches to reduce their inventories, and all of these companies indicate that they do not have inventory problems at all. On the other hand, 84.21% of the respondents have selected at least one approach from the list, which they have already used for inventory reduction.

Figure 7 presents according to the companies, which approaches have contributed the most to them for the reduction of their inventories. It can be seen that the most helpful is "improving service levels". This approach is followed by "improving forecast accuracy". Besides, the approaches "setting more focus on the quality, values, and sustainability" and "three-season-strategy" are popular among the companies, as well.

Figure 7 The contribution of various approaches to the reduction of stock level

6 CONCLUSION

This research addresses the inventory problems of apparel companies. Four hypotheses are formulated, which are investigated with the help of research questions.

The research provides the following evidence: overall, 47.37% of the respondents face higher stock levels due to the Covid-19 pandemic. 52.63% of the companies do not feel any negative impacts on their stock levels due to the Covid-19 crisis and lockdowns.

One particularly interesting finding is that companies struggling with higher inventory levels are those operating in only one market segment. Over three quarters of the companies that have stock problems serve exclusively one market segment with their products. Of all the companies that are active in several market segments, solely 22.22% have stock problems.

15.79% of the companies participating in the survey have not tried any approaches to reduce their inventories, and all of these companies indicate during the survey that they do not have inventory problems at all. On the other hand, 84.21% of the respondents select at least one approach from the list, which they have already used for inventory reduction. Companies find the most helpful to "improve service levels". This approach is followed by "improving forecast accuracy". Besides, the approaches "setting more focus on the quality, values, and sustainability" and the "three-season-strategy" are very popular among the companies, too.

A limitation of the study is the low number of participants in the survey. Only 19 companies participate in the questionnaire. In addition, most of the participating companies are small and medium-sized enterprises. Therefore, the results might rather apply to the small and medium-sized companies alone and not to the large corporations.

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TECHNOLOGY OF MAKING THERMAL TRANSFERS

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Abstract: *Nowadays, there are several ways to print images on textiles used by modern manufacturers. The choice of technology for applying the image depends on many factors: the number of products, area of the image, the number of colors in the picture, the raw material composition of the fabric, fabric colors. This work is devoted to studying the quality and stability of thermal transfer on fabrics made by screen printing. This method of applying images to textile materials in many ways is similar to screen printing. The difference lies in the fact that the idea is applied directly to the cut details or garment with direct screen printing. In contrast, transfer printing might be used to an intermediate carrier that is transferred. A study of the technological parameters of applying thermal transfer, which we made by screen printing on materials of various compositions, was carried out. The technology of applying the thermal transfer performed by the method of screen printing was developed. Recommendations for the use of equipment were developed.*

Keywords: *images, textile materials, printing methods, screen printing, digital printing, sublimation printing, thermal transfer printing, printing cost, performance.*

1 INTRODUCTION

The light industry of Ukraine and some European Union countries is developing rapidly. Unlike metallurgy, mechanical engineering, and other branches of the national economy, fair industry products are aimed at the end consumer, who constantly follows fashion trends. With this in mind, every clothing company tries to compete with other industries in different ways. One way to increase product competitiveness is to improve product quality while maintaining the final retail price or slightly to increase it.

To improve the quality of the product, manufacturers use various finishing methods, namely: decorating products with prints [1], applications of artificial stone [2], finishing with fake pearls, the use of complicated elements, and so on. Finishing products with different patterns focused on a particular brand, different age categories, gender, and youth movements significantly improves the appearance of products. Methods of drawing on garments or finished products include direct digital printing, screen printing, sublimation, thermal transfer of films, flock coating, as well as the application of thermal transfer [1]. Drawing images by direct digital printing using DTG technology or direct screen printing requires costly specialized equipment, so companies with low production capacity turn to contractors specializing in decorating clothing. The disadvantage of such cooperation is the high cost of transporting large volumes of cut or finished products. The use of thermal transfers, which contractors can also manufacture, significantly reduces logistics costs, and the task of the manufacturer is only to apply a finished pattern on the product in its production.

The described general technological process of screen printing [3] is printed on knitted, textile materials, artificial and natural leather, and other complex and elastic surfaces. In the light industry, the so-called "indirect printing" technology is used - transfer, which is partially described in [1], but this technology requires independent research. The essence of the technological process is to apply to the surface of the intermediate carrier – a particular paper or film of a single or multicolor image, followed by transfer to the fabric. The working surface of the middle page - transfer has low adhesive properties with printing inks and undergoes antistatic treatment.

1.2 Discussing ideas

The research aims to identify differences in drawing images by thermal transfer printing in contrast to direct screen printing on textile materials, natural and artificial leathers, rolled materials.

The main tasks to be solved:

- analyze the main methods of making transfers;
- identify the technological features of different methods of making transfers.

2 METHODS

Today we can identify several excellent technological processes of making transfers:

- screen printing;
- a combination of offset and screen printing;
- DTF (direct to films) printing;
- laser printing.

Each of these methods of making transfers involves applying the image on an intermediate medium - transfer (paper or film) and its subsequent transfer to the material of the workpiece. Not only knitted and textile materials can is used as blanks. Application is possible on artificial and natural leather, plastic and metal surfaces, wood, etc. The transfer is a specially treated paper or film. Typically, the print surface is coated with low adhesion to the transfer, for example, silicone, which significantly facilitates the extraction of the media after image transfer. Some manufacturers achieve common adhesion properties between the paint and the transfer surface by machining the latter (polishing) or combining machining and coating. Also, for carriers, antistatic treatment is performed by applying a special coating that prevents the accumulation of static charge or simply removing static stress from the surface of the page during its machining. Consider in more detail each process.

3 EXPERIMENTAL

The technological process of applying the image to the transfer surface by screen printing contains some differences from the direct screen printing method [1, 4]:

- design preparation - mirror image;
- drying transfer (used for paper media, to remove excess moisture and prevent further deposition, the film, unlike paper, is non-hygroscopic, so it does not require this operation);
- applying colors of multicolor images in the opposite direction;
- application of a layer of paint - an adhesive base that covers the entire image to create a unique adhesive surface (usually performed with white paint or anti-migration paint, which then acts as a barrier when applied to colored and black fabrics);
- application on the prepared film of paint special powder glue (as a rule, powder glue was made of polyester (Diethylene glycol phthalic anhydride polymer CAS # 32472-85-8) or polyurethane crushed to the sizes from 1 to 600 microns);
- cleaning the surface of the transfer from the powder glue on the surface free of paint, because despite the special antistatic treatment of the transfer surface, the powder glue particles, during the application, receive a significant static charge, which leads to their adhesion to the surface of the carrier free of paint;
- drying of the transfer workpiece (this technological operation takes place at the melting temperature of the polymer adhesive; the manufacturers recommend a range of 105 to 115°C for these materials [5]);
- image transfer from the transfer surface to the workpiece surface for which press equipment is used (transfer temperature modes depend on the type of media, paint, and material on which the drawing is applied, and range from 170 to 190°C, exposure from 10 to 15 s, under pressure in the range from 35 to 40 psi (from 0.24 to 0.31 N/mm^2) [6]. In cases of low-temperature resistance, the workpiece material is allowed to reduce the temperature, but not less than the melting point of the polymer adhesive, while increasing the processing time.

To apply the image to the transfer surface by screen printing, special liquid adhesive mixtures of transparent or white color are used. White is used for colored and black fabrics and fine only for white.

In the case of liquid adhesives, applying a layer of paint and applying powder glue is not present in the process, but taking into account surveys of manufacturers and users of transfers, the use of powder glue has advantages due to better adhesion abrasion resistance, washing.

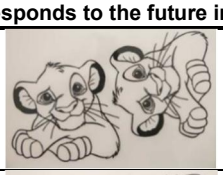
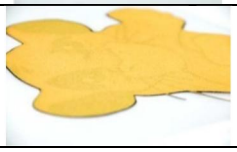
4 RESULTS

For screen printing, the preparation of vector and raster images also has specific differences and features.

Table 1 (as an example) shows photos of the stages of the technological process of applying a vector five-color image. When using a multicolor image, it is necessary to dry the intermediate colors to prevent the pre-applied color from sticking to the next printing plate.

At the moment of paint drying, its insignificant deposition - deformation owing to fast evaporation of moisture is observed. To avoid shrinkage of the carrier, its pre-drying or deposition in tunnel-type dryers or drying chambers are used. To prevent the residue of the ink layer, manufacturers use grid numbers on printing plates in the range from T61 to T100 (Glamor, TM Gektor, ltd Ornament Print-F). As the grid number increases, the thickness of the paint layer decreases and, accordingly, the probability of significant deposition. Another way to combat the undesirable phenomenon of deposition of media and paint is a unique design preparation. Table 1 shows an example of applying an image containing five colors (black contour, brown, pink, yellow, and orange), which are used with a slight overlap.

Table 1 Technological stages of making a vector image

The name of the stage	Photo of the print side	Photo from the side of the film (corresponds to the future image)
Application of the first color of the image		
Applying a second color image		
Applying the third color		
Applying the fourth color (pink nose)		
Apply the fifth color, which is the basis of the image		
Applying an adhesive base (plastisol or powder)		
Drying of the adhesive base		

This design and application scheme prevents the formation of "gaps" or substantial overlaps of colors when performing multicolor images. Such overlaps are permissible from the printing side (photo of the printing side in Table 1) but invisible in the finished painting (picture from the film side, Table 1). As a rule, the last turn put the color occupying the extensive drawing area, which further put powder glue. If the future transfer is planned to be applied to black or dark fabrics, then on top of the whole image, use an anti-immigration base or white paint and then powder glue.

In the case of raster image application, it is necessary to divide the image into a certain number of channels following the color scheme (RGB, LABColor, CMYK, or according to the pontoon of the available colors [7-9]). The division into one or scheme and the verification of the conformity of the received transfer to the original deserve more attention and are not considered in this work.

The CMYK color scheme was chosen as the image's color scheme, as it is the most common in the printing industry and full-color direct screen printing.

The technological process of preparing a raster image contains the following stages (Figure 1):

- translation of the image into the appropriate color scheme (Figure 1b);
- division into channels according to the color scheme and creation of separate images corresponding to each channel (for the CMYK color scheme, it is four channels, respectively and four photo templates) (Figures 1c-1f);
- translate each image into raster. When converting to a raster, separate raster angles are used for each channel, and the ruler, according to the screen number of the printing plate, is taken into account. The use of different angles during rastering prevents the appearance of moire (Figures 1c-1f);

- production of the control image in the CMYK color scheme (Figures 1c-1f). The control image is performed by creating a new file in the CMYK color scheme and then placing the corresponding raster image in the channel of the same name. This image makes it possible to control the appearance of moire, as well as the correspondence of color reproduction to the reference image;
- making a photo template of the base of the drawing, which acts as an adhesive base. As in the previous process, it can be made with an outline of white or black. As a rule, white paint is used as a basis, which allows you to transfer to light and dark fabrics. The peculiarity of making the basis for raster images is the possibility of its manufacture in two ways. The first method is to convert the image to grayscale with its subsequent rasterization or to a black and white photo of the control image in the color scheme CMYK (Figure 1i). This method is more suitable for light colors of fabric. The second method is to select the entire area of the figure and fill it in black (Figure 1j). This method is better for black and dark fabrics.

The following CMYK channels are accepted in polygraphy: Cayan - 150, Magenta - 750, Yellow - 00, Black - 450. Since textile and knitted fabrics have a specific weave pattern, and more, as a parasitic pattern, occurs due to interference of raster grids of patterns division into colors and alignment during printing [7-11], the probability of the moire itself on textile materials increases significantly. Therefore, when translating split images into raster, they also experiment with the shape of the raster dot and the ruler, followed by applying the test image to the fabric.

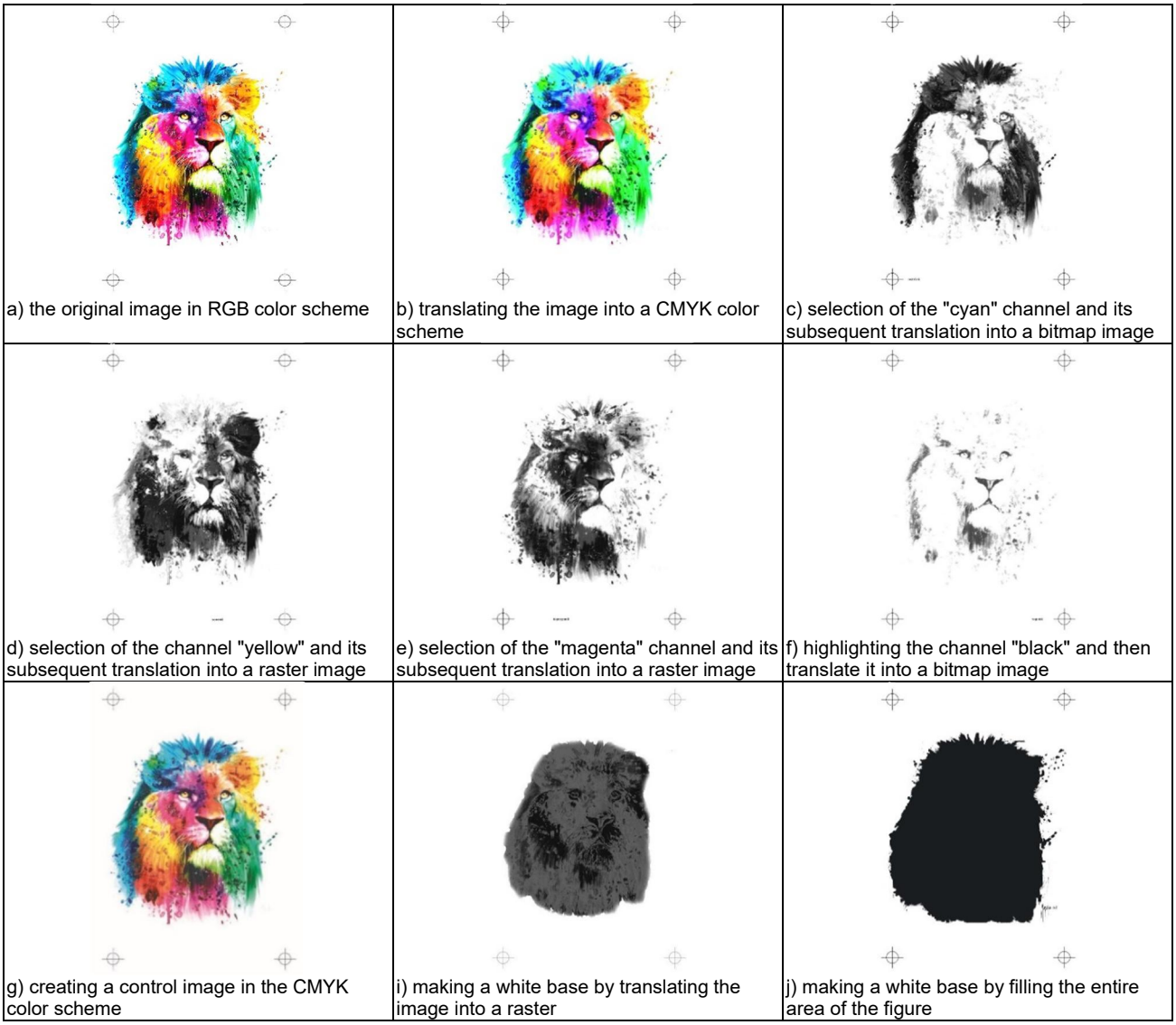


Figure 1 Technological process of making photo templates for raster image

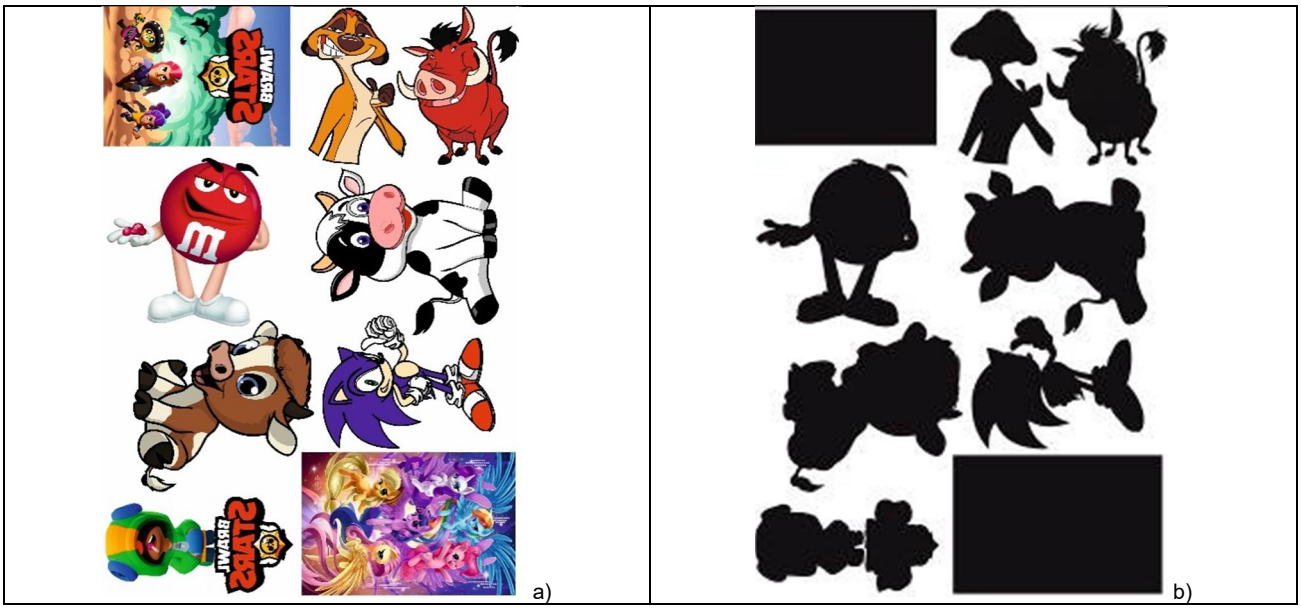


Figure 2 Production of transfer by offset printing a) offset printing b) a photo template for making a transfer

The next type of transfer, which, in our opinion, deserves attention, is obtained from a combination of two methods - offset image printing and screen printing. The technological process of manufacturing the transfer contains the following steps (Figure 2):

- drawing the image on the transfer medium on special offset machines used in the printing industry (Figure 2a). The most similar paints used in printing to plastisol are oil-based paints. After their transfer to the fabric surface, they retain elasticity, unlike other types of stains. Because offset machines are highly productive and require special prepress, which takes a long time, so printing small batches of images becomes impractical. Service firms work with minimum orders of 1000 sheets. The most common print formats are A2 and A1, while A3 and A3 + machines are less popular, so to reduce the cost of making the transfer on the print format, place an array of the same type or several different images (Figure 2a);
- preparation of a photo template and production of a printing form. Depending on the type of drawings, the desired effect, and the color of the fabric of the workpiece or product, the photo template (Figure 2b) is made with a black or white outline. In the case of a white design, the area of the photo template will be smaller than the area of the image, otherwise – large;
- overlap the picture with paint. As a rule, overlappings provide white color that will give a qualitative light basis. Using a photo template with a black outline, a white outline is obtained, respectively. Such a transfer is better to apply to white fabrics if the manufacturer does not intend to intentionally get the effect of a white outline on black and colored fabrics. If you use a photo template with a white design, then the paint that overlaps the pattern does not protrude beyond its contour, and such a transfer can be applied to white and black fabrics;
- application of powder glue on the surface of the paint;
- removal of adhesive residues from the free surface of the transfer;
- drying of the glue.

Production of DTF transfer by printing (direct to films) involves the application of special ink on the surface of the transfer on an inkjet printer. Paints have the same name DTF, which is resistant to high temperatures (up to 200°C), essential when transferring the image from the transfer surface to the material. The main feature of these printers is the ability to print with CMYK inks and overlap the entire image with white ink, which acts as a basis for the glue. In the case of applying white paint on top of a color pattern, the transfer can be used to black fabrics without losing image quality.

Other technological stages of transfer manufacturing are similar to the previous technology: application of glue and its drying. The operation of sprinkling the bond is done manually and drying in special dryers.

Today, there are automated transfer systems, including a DTF printer, a roll holder, an adhesive application system (Figure 3a), a dryer (Figure 3b), as well as a mechanism for winding the finished transfer into a roll (Figure 3c).

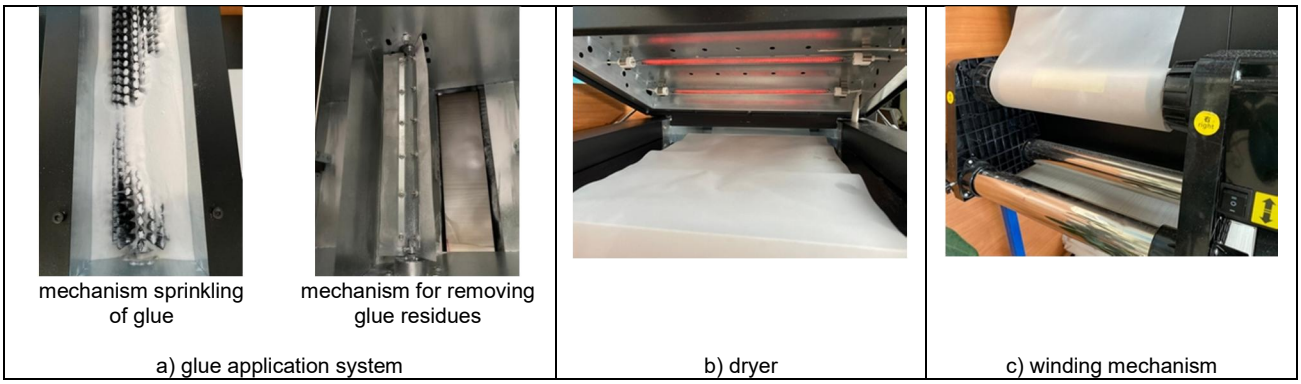


Figure 3 Automated transfer systems

Making transfers by laser printing has two options:

Option 1 Print on particular media on a laser printer, which allows you to print both CMYK and white toner. Different media are used for dark and white fabrics. After printing, the patterned media is transferred to the transfer. The image transfer from the transfer is similar to the previous cases on the press equipment or with an iron. The disadvantage of this technology is the need to manually cut the pattern along the contour of the transfer.

Option 2 The image is printed on a laser printer, which allows you to print CMYK colors on the transfer, used in screen or offset printing. After that, a photo template is made, and operations similar to offset transfer making are performed.

5 CONCLUSIONS

As a result of the carried-out work, technological features of processes of drawing by thermo transfer methods of the press are considered: by a screening method of the media; a combination of offset and screen printing; DTF (direct to films) printing; laser printing.

The differences between these methods from direct screen printing on textile materials are established.

The main methods of making transfers are analyzed, their technological parameters are revealed, and the peculiarities of transferring images made in different ways to textile materials are established.

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INFLUENCE OF WASHING OF WOVEN LABELS PREPARED FROM POLYPROPYLENE AND POLYAMIDE PHOTOLUMINESCENT FIBRES ON INTENSITY OF LIGHT EMISSION

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Abstract: *The contribution focuses on examination of photoluminescent effect of woven labels prepared from polypropylene and polyamide photoluminescent fibres using concentrate containing 0.10 wt.% of photoluminescent blue organic pigment. The photoluminescent polypropylene and polyamide fibres are incorporated into construction of the label directly in the technological process of weaving. Photoluminescent effect of blue pigment has been evaluated before and after 20 washing cycles. Influence of washing on change of intensity of blue light emission in the woven labels has been assessed by two methods, using FLUOTEST device in the region of electromagnetic radiation in short-wavelength and long-wavelength ultraviolet radiation and by evaluation of b* colour coordinate in the CIE LAB colour space using ULTRASCAN XE device. Intensity of light emission of the photoluminescent polypropylene and polyamide fibres allows distinct identification of photoluminescent fibres in the labels aimed at protection of products against counterfeiting.*

Keywords: *photoluminescent fibres, photoluminescent pigment, originality protection, change of colour expression, electromagnetic radiation.*

1 INTRODUCTION

World`s leading companies in all industrial fields try to protect their sophisticated products against counterfeiting. Clothing and fashion accessories represent a significant share of counterfeiting and it is estimated that counterfeiting constitutes more than 10% of the world fashion-related trade [1].

One of affordable and cost-effective access to originality protection of products is application of photoluminescent dyestuffs and pigments, which besides colour change emit also light under ultraviolet (UV) lamp. Phenomenons involving absorption of energy and subsequent light emission are classified generally as luminescence. Photoluminescent (FL) dyestuffs and pigments, available in organic as well as inorganic form are particularly interesting for the purpose of protecting product originality [2, 3].

Originality protection of textile products made of unmodified polymer fibres can be ensured using various safety protective patterns from photoluminescent polymer fibres in the products, e.g. logo of the manufacturer, safety protective label, extra courses in the clothing products etc. This way authentication of textile products can be ensured in the cases where it is necessary to prove their originality. Basic way of designation is a structured code with an identification mark. The mark is made of a thread/yarn incorporating photoluminescent fibre reflecting light backwards when exposed to UV light [4].

The aim of this contribution is to examine permanency of photoluminescent (FL) effect of a protective identification component in the labels containing 0.10 wt.% of blue organic pigment in polypropylene (PP) and polyamide (PA6) photoluminescent fibre (FLV).

2 EXPERIMENTAL PART

2.1 Materials used

Following fibre types with linear density of about 90 dtex have been used to prepare the woven labels:

- PP fibre containing FL blue pigment with concentration of 0.10 wt.% in the fibre (**PP/FLV**);
- PA6 fibre containing FL blue pigment with concentration of 0.10 wt.% in the fibre (**PA/FLV**);
- textured standard (unmodified) PP fibre without content of FL pigment (**PP**);
- textured standard (unmodified) PA6 fibre without content of FL pigment (**PA**).

Workability of the above-mentioned fibres has been evaluated in an assortment of woven labels. Base of the label was made from black polyester fibre on which a pattern in a shape of full circle with 32 mm diameter has been woven using PP and PA FLV (Figures 3, 6 and 7). Weaving of the labels was performed on a width of 1.2 m and length of 0.5 m.

Edges of the labels remained smooth, undamaged when cutting the labels to a final width of 40 mm with a content of PP FLV and PA FLV.

2.2 *Methods used to evaluate efficiency of blue light emission*

Photoluminous expression and intensity of blue light emission has been evaluated on the prepared labels using methods as follows:

- objectively by a change of colour expression by means of b* colour coordinate in the CIE LAB colour space using ULTRASCAN XE device according to the standard STN ISO 105-J03 [6]. The more negative b* value is measured the stronger intensity of emission is observed [5].
- optically by a form of photoexcitation after light exposure in the device FLUOTEST with UV lamp in short-wavelength (UV-C region) and long-wavelength ultraviolet radiation (UV-A region). Region of the electromagnetic radiation in shown in Figure 1.

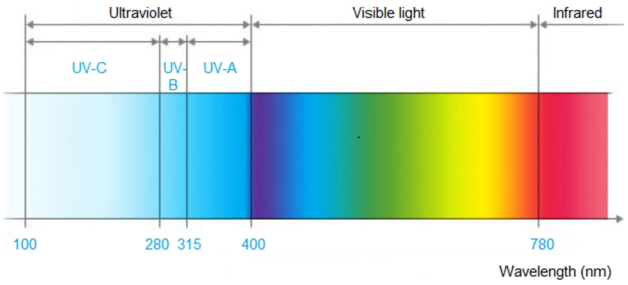

Figure 1 Electromagnetic radiation fields

3 RESULTS AND DISCUSSION

Intensity of blue light emission on the prepared woven labels made from PP FLV and PA FLV containing 0.10 wt.% of FL pigment has been evaluated by an objective instrumental method using b* colour coordinate in the CIE LAB colour space on ULTRASCAN XE device. This device enables to evaluate influence of blue FL pigment and its concentration on change of photoluminescent expression of the FLV. Degree of efficiency of FL pigments in the woven labels before washing without content (standard) and with 0.10 wt.% content of FL pigment in PP FLV and PA FLV is shown in Figure 2.

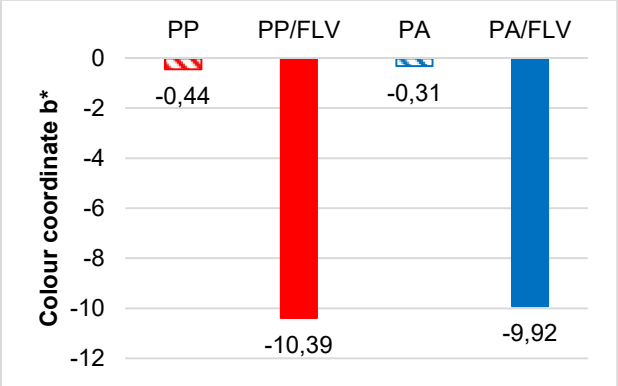

Figure 2 Intensity of light emission on the labels made of standard PP/PA fibre and PP FLV, PA FLV containing 0.10 wt.% of blue FL pigment in the fibre before washing

More remarkable intensity of blue light emission has been achieved with PP fibre containing 0.10 wt.% of FL pigment. The b* colour coordinate of the used PP FLV is at the level of -10.39. PP FLV containing 0.10 wt.% of FL pigment used in the label has higher intensity of blue light emission by 4.52 % than PA FLV (-9.92) with the same content of FL pigment.

Intensity of blue light emission (Figure 3) has been demonstrated optically on the prepared labels using FLUOTEST device to confirm photoluminescent effect of the used FL pigments.

Figure 3a) indicates that the labels with FLV as well labels without FLV have white colour in the daylight and they are not distinguishable with naked eye of an observer. The labels made of PP FLV and PA FLV shined brighter in UV region with long-wave radiation than in UV region with short-wave radiation. Optical excitation emitting brighter blue light is observed by naked eye allowing clear identification of labels containing FLV in comparison with the labels without content of FLV (Figures 3b, 3c).

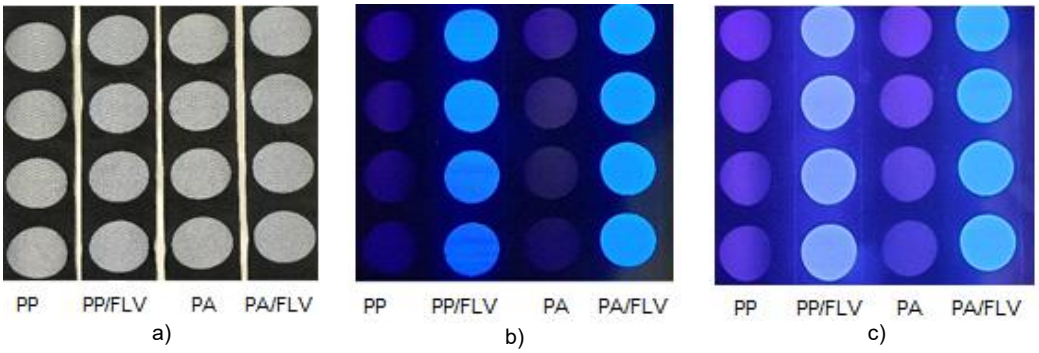

Figure 3 Woven labels: a) in visible spectrum of solar radiation, b) in UV-C region with short-wave radiation, c) in UV-A region with long-wave radiation

Permanency of blue photoluminescent pigment in PP FLV and PA FLV used in construction of the woven labels has been evaluated after the 1st, 2nd, 3rd, 5th, 10th, 15th, and 20th cycle of washing and drying with subsequent assessment of b* colour coordinate and optical expression of photoexcitation under UV lamp. Washing and drying of the labels has been performed according to the standard STN EN ISO 6330: 2012 [7] under 4N procedure at water temperature of (40±3°C) using a reference detergent (phosphate-free powder detergent without optical brightening agent and without enzymes). Drying has been performed under C procedure, i.e. flat drying in horizontal position. Influence of washing on change of b* colour coordinate of the woven labels is shown in Figures 4 and 5.

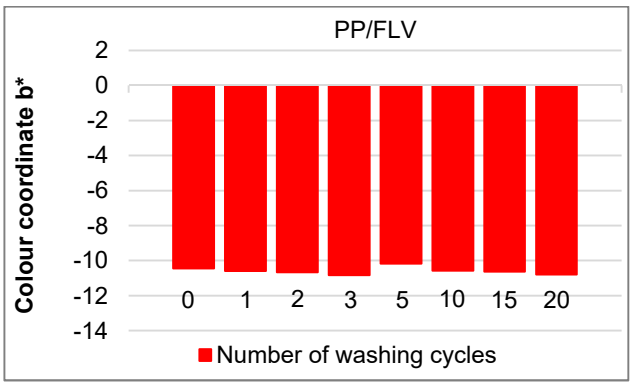

Figure 4 Influence of washing and drying of the woven labels from PP FLV containing 0.10 wt. % of pigment in the fibre on change of b* colour coordinate

The photoluminescent effect of any woven label has not been changed with increasing number of washing and drying cycles. Washing and drying of the labels has no influence on reduction of intensity of blue light emission. There is no reduction of intensity of blue light emission of the labels from FLV, which has been confirmed by the measured values of b* colour coordinate (Figures 4 and 5).

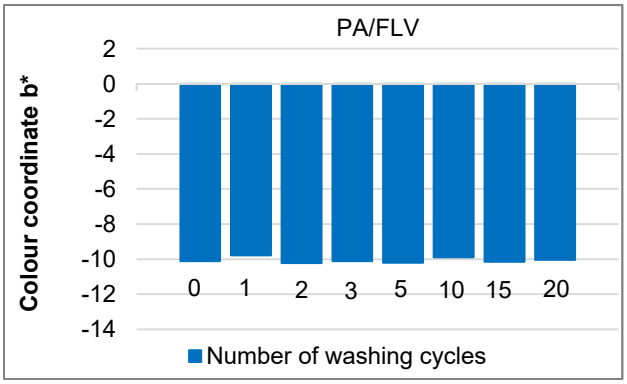

Figure 5 Influence of washing and drying of the woven labels from PA FLV containing 0.10 wt. % of pigment in the fibre on change of b* colour coordinate

Continuous decrease/increase of intensity of blue light emission before and after 20 washing and drying cycles is comparable. We consider resulting difference in intensity of light emission on a level of ±3% to be negligible as it can be caused by measurement error, device, influence of knitted construction – its density after washing etc.

Intensity of blue light emission on the prepared labels from PP FLV before and after washing and drying (1 – 20 cycles) is shown in Figure 6. Intensity of blue light emission on the prepared labels from PA FLV before and after washing is shown for comparison in Figure 7.

As established with labels prepared from PP FLV containing blue organic pigment, more pronounced intensity of blue light in UV region with long-wave radiation has been noted in the case of the labels from PA FLV as well.

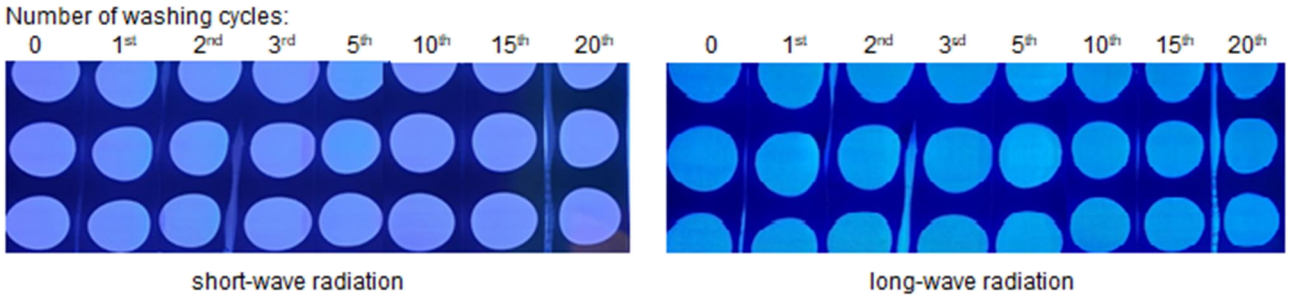

Figure 6 Photoluminescent effect of the woven labels from PP fibre containing 0.10 wt.% of pigment before washing up to the 20th cycle

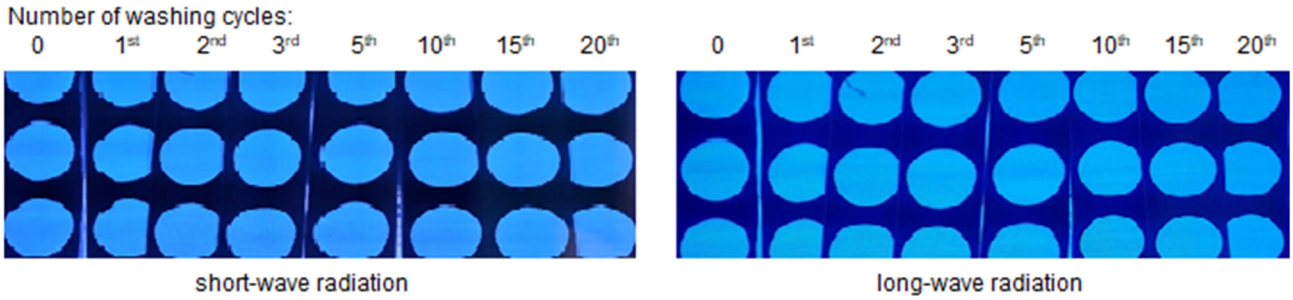

Figure 7 Photoluminescent effect of the woven labels from PA fibre containing 0.10 wt.% of pigment before washing up to the 20th cycle

4 CONCLUSION

The woven labels prepared from PP fibres containing 0.10 wt.% of photoluminescent pigment have retained the same intensity of blue light emission after 20 washing cycles as before washing. As far as intensity of blue light emission is concerned, intensity of emission of the labels from PA FLV is comparable with intensity of emission of those made of PP FLV. The woven labels prepared from PP FLV are characterized by visibly lower intensity of blue light emission in short-wave radiation than labels from PP FLV of the same quality in long-wave radiation. Equally, woven pattern on the label from PA FLV has visibly brighter intensity of blue light emission in long-wave radiation. Continuous decrease/increase of intensity of photoluminescent blue pigment emission in PP and PA fibre before and after 20 washing cycles is comparable. Intensity of blue light emission of the woven labels containing blue photoluminescent pigment has not decreased visibly from the 1st washing cycle. Intensity of blue light emission on the labels with incorporated PP FLV and PA FLV containing 0.10 wt.% of FL pigment is as significant before washing as after the 20th washing cycle. With increasing number of washing cycles of the labels washing out the photoluminescent pigment from PP FLV and PA FLV did not occurs, the proof is optical expression of photoexcitation of these FLV after lighting in the optical device FLUOTEST with UV lamp with short-wave and long-wave radiation.

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FATIGUE STUDY OF SPIRO[INDOLINE-NAPTHOOXAZINES] PIGMENT USING COLORIMETRIC DATA IN A CONTINUOUS MODE OF UV IRRADIANCE

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Abstract: *The fatigue behavior of photochromic pigment applied on fabric is studied under different asymmetric UV irradiance times in seconds of one photochromic cycle and developed a rapid testing method for studying such chromic compounds in the future. The fatigue property of chromic compounds plays a significant role in designing possible applications such as UV sensors for outdoor applications, optical rewritable devices, and smart wearable textile-based sensors. The photochromic woven fabric was prepared using the screen-printing technique using chromic pigment. Previous studies have demonstrated the sensitivity of photochromic pigments to UV radiation's intensity during the experimentation. This work provides a different approach in testing the fatigue resistance of such photo-induced transform chromic pigment of the spiro-indolines-oxazine family. The pigment was applied on the surface of the fabric to harness as a UV sensor for outdoor applications. We found that color intensity performance and fatigue resistance can be evaluated using a relatively small amount of necessary photochromic cycles and an advanced time/intensity UV radiation exposure setup.*

Keywords: *Photochromism, fatigue behavior, chromic pigment, UV exposure time, color intensity values.*

1 INTRODUCTION

Photochromism is a part of photochemistry; the chromic pigments or colorant undergo reversible or irreversible color change under the fluence of UV light (Figure 1), whereas the reverse reaction can occur upon the removal of the UV source. The difference in absorption spectrum is measured as a reflectance using a specifically designed spectrophotometer, which runs in a continuous irradiance mode. A well-known example is photochromic sunlight glasses which darken the tint of optical wearable glass lenses under daylight, which contains the UV range wavelength. The commercially available photochromic pigments forms are spiropyarns (SPs), spirooxazines (SOs), diarylethenes, stilbenes, naphthopyrans, and fulgides. These compounds commercial application is optical storage, rewritable data storage, ophthalmic industry, sensors, surface coatings, microencapsulation, chemotherapy, phototherapy, wearable textile sensors, and many others.

$$\boldsymbol{A}(\lambda_1) \underset{h\nu_2,\ \Delta T}{\overset{h\nu_1}{\rightleftharpoons}} \boldsymbol{B}(\lambda_2)$$

Figure 1 General scheme of photochromism from A to B [1, 2]

The spirooxazines (SOs) show high fatigue resistance, as its open-colored form extends the coloration in visible absorption range from 520 to 650 nm [3]. The colorability of chromic compounds, which are spirooxazine based pigments, has been studied. There is a need to design the fatigue experiment for the chromic compounds in a continuous irradiance mode. The spirooxazine compounds undergo photoinduced irreversible change under a UV light source at its dominant wavelength. The colored photo merocyanine (PMC) form occurs after opening the heterocyclic cleavage of the spiro C-O bond of the colorless, forming the purple color. These photochromic compounds react with molecular oxygen, which is responsible for the photodegradation rate. The fatigue resistance property of photochromic compounds restricts such dyes and pigments [4].

The fatigue behavior of photochromic compounds which undergoes reversible change under the influence of UV light source can be studied under flash and continuous mode of irradiance. Depending upon the chromophore in the compound structure and the elements at a particular position, the opening of such compounds shows a bathochromic (red shift) or hypsochromic (blue shift) through a change in wavelength after irradiation in the visible range of the electromagnetic spectrum. The photochromic compounds might undergo the fatigue products and have their isomeric forms, but it won't provide us with colorimetric data. The amount of intensity used to activate the compound or the opening of the cyclic ring

of compounds is dependent on the time used for radiance and the amount of dose, which are mutually reliant on each other.

Photochromic properties, including photo coloration, photobleaching, and fatigue resistance of 1,3,3-trimethylspiro[indoline-2,3′-[3H]naphtho[2,1-b][1,4] oxazine] doped in poly(methyl methacrylate) (PMMA) and epoxy resin, have been investigated. The photo coloration was followed spectrophotometrically by observing the adsorption curve. The open form of the pigment showed a purple color at its dominant wavelength (555 nm) [5]. The photo coloration process follows the first-order exponential model in solid form, but it might follow the second order in the polymer matrix. The photoinduced reaction was carried out using a UV light source (366 nm), resulting in the photo merocyanine (PMC) form. The reverse reaction was carried out by using visible white light. The results show a bathochromic shift attributed to the polarity of epoxy resin more than PMMA. Spirooxazine doped in epoxy resin offers better fatigue resistance than that doped in PMMA [5].

The photochromic properties of 7′,8′-dichloro-1,3,3-trimethylspiro[indoline-2,3′-[3H]benzo[b][1,4]oxazine] doped in PMMA and epoxy resin has been investigated [6]. The open form shows a more purple-redder intense color. A bathochromic shift has been observed due to the polarity of epoxy resin and PMMA. The photobleaching rate of spirooxazine is two times slower in PMMA than in epoxy resin. The fatigue resistance of SO doped in epoxy resin is better than that of doped in PMMA [6].

The commercialization of the photochromic compounds requires essential criteria such as follows [7, 8]:

1. a photo coloration and a thermal bleaching rate fast enough over an extensive temperature range
2. a suitable colorability in the optimal concentration range
3. significant resistance to photodegradation (a fatigue resistance).

The chromic compound's fatigue behavior follows the photochromic cycle – the coloration and decoloration mode of conduct under the presence and absence of UV light source, respectively. The photochromic process generally includes thermal stabilization, growth phase (UV+), decay phase (UV-). The dynamics of photochromic material when exposed to external stimuli, i.e., UV source, the different stages of material - 1,2,3 - as shown in Figure 2. The various stages of a photochromic cycle have been illustrated by the Figure 2 [9-11]:

1. with a UV(-) - the thermal stabilization phase of the instrument starts where the sample is colorless at the initial step. Due to the device, which is an essential step, it is required to stabilize the instrument at a specific or used temperature to reach that state from the ambient temperature.

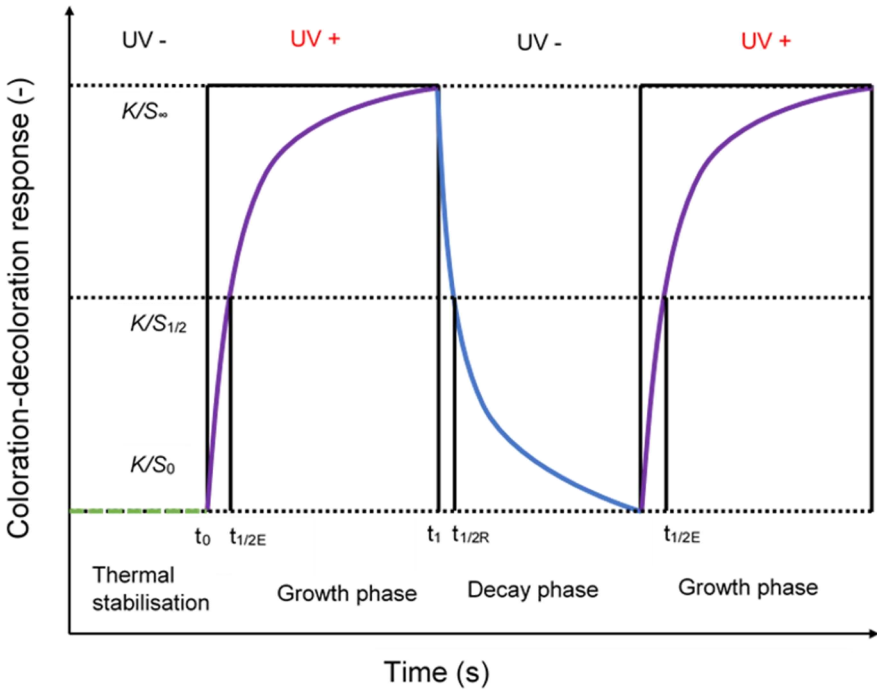


Figure 2 Kinetic phases of the photochromic cycle, where: t_0 = UV+; t_1 = UV-; $t_{1/2E}$ = half-life measure during growth phase; $t_{1/2R}$ = half-life measure during decay phase; $(K/S)_0$ = coloration value at beginning; $(K/S)_{1/2}$ = half of the total coloration value before reaching the equilibrium state, $(K/S)_\infty$ = maximum coloration value after reaching the equilibrium state

2. with UV(+) - the growth phase (coloration) starts at the time t_0, the change in reaction gradually starts between the light source and the photochromic sample. The optical density is visualized at its dominant wavelength in spectra as a measure of reflectance values over the range of the electromagnetic spectrum. It achieves its maximum-colored intensity value and remains in equilibrium under light source till the time of exposure.
3. with UV(-) - the decay phase (decoloration) starts where the color intensity of the photochromic sample fades away with time and comes back to its original color. The color change can be observed in the decay phase from a colored to a colorless state. Decoloration rate or bleaching rate is measured in the fading phase of the sample.

The investigation of such chromic compounds can be carried out using colorimetric measurements. In photochromism, the photoinduced color change is measured using a specifically designed spectrophotometer only, which provides us to measure the change in these colorations and decoloration modes of used pigment as a change in reflectance value in a visible range of the electromagnetic spectrum. However, the photochromic cycle consists of thermal stabilization, coloration mode utilizing the UV light source, and decoloration mode using a visible light source. For a certain amount of UV exposure time in seconds, the pigment behaves accordingly in its growth and decay phases. The color change is measure as a change in the reflectance, absorbance, or scattering of the light can be visualized as chromogenic phenomena. The coloration of the pigment is a measurable property using the Kubelka-Munk function. As the Kubelka-Munk function, as well as Beer's law, is monochromatic. The color values *(K/S)* were calculated from the reflectance spectra using the Kubelka-Munk function [12-14].

$$f(R)_\lambda = \frac{(1 - R_\lambda)^2}{2R_\lambda} \quad (1)$$

where: $f(R)_\lambda$ is equals to $(K/S)_\lambda$, K_λ is the absorption coefficient, and S_λ is the scattering coefficient of the paint or pigment at its dominant wavelength.

The first-order kinetic association model describes the photo-induced transformation reaction process when the photochromic sample is exposed to UV irradiance. The color intensity at a time t_0 is $f(R)_0$ without exposure, the color intensity changes when it is exposed to UV irradiance is $f(R)_\infty$ at time t_∞ (Figure 3) [15].

The rate of color intensity *df(R)/dt* is directly proportional to the difference to a color intensity at time *t* and in equilibrium given by Equation 2:

$$\frac{df(R)_t}{dt} = -k\,(f(R) - f(R)_\infty) \quad (2)$$

For *t=0*, $f(R) = f(R)_0$, integrating the Equation 2 between the limits from (0, t), $(f(R)_0, f(R))$ leads to the exponential equation for exposure phase is given by Equation 3:

$$f(R)_t = f(R)_\infty + (f(R)_0 - f(R)_\infty)e^{-kt} \quad (3)$$

where: *k* is rate constant at given time *t and f(R);* $f(R)_\infty$ are the color intensity at the time (0, t).

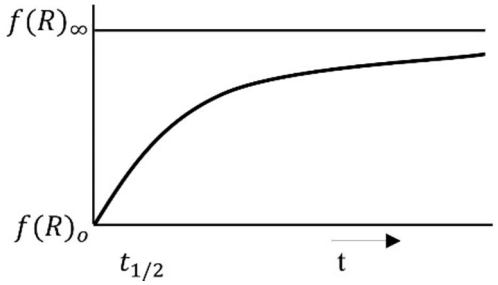


Figure 3 Photokinetic trace – exposure phase

The first-order kinetic dissociation model describes photodegradation of color intensity upon the removal of UV light source. The change in color intensity reduces from $f(R)_\infty$ to $f(R)_o$ during the decay phase (Figure 4). The basic presumption is again of first-order kinetics as for exposure. The rate of color intensity *df(R)/dt* is directly proportional to the difference to a color intensity at time *t* and equilibrium (Equation 4) [15].

$$\frac{f(R)}{dt} = -k\,(f(R) - f(R)_\infty) \quad (4)$$

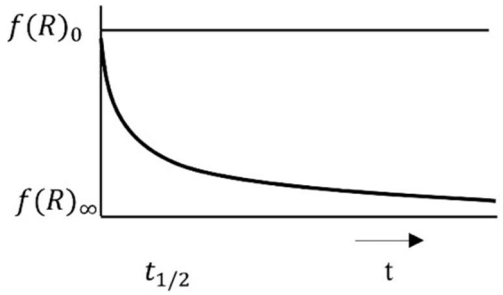


Figure 4 Photokinetic trace - decay phase

For *t*=0, $f(R) = f(R)_0$, integrating the Equation 4 between the limits from (0, t), $(f(R)_\infty, f(R))$ leads to the exponential equation for decay phase (Equation 5):

$$f(R)_t = f(R)_0 + (f(R)_\infty - f(R)_0)\,e^{-kt} \quad (5)$$

The half-life $t_{1/2}$ was measured using Equation 6:

$$t_{1/2} = \frac{\ln 2}{k} \quad (6)$$

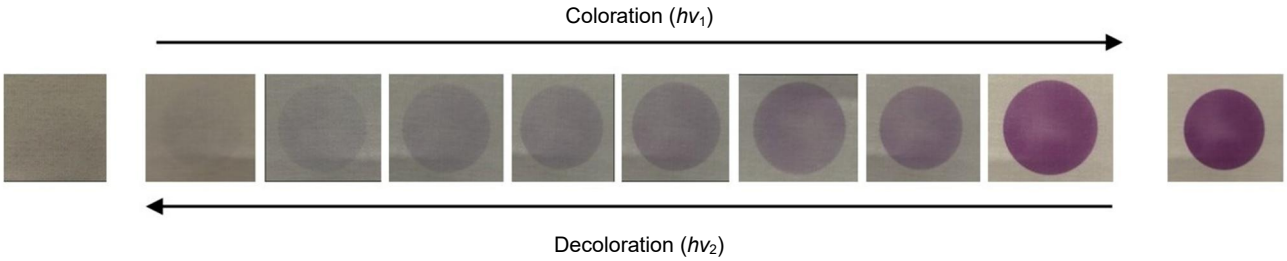


Figure 5 Coloration and decoloration of photochromic system

The half-life time of exposition represents the time required to develop pigment coloration or dye at excitement to the half value of maximum absorbance at a specific wavelength during the exposure phase. The half-life time of reversion estimates the color fading rate during the decay phase to reach its original color state. The half-life time during the decay phase represents the time required for the decrease of the color from the absorbance at a specific wavelength in one cycle to the half value. Fatigue resistance describes the efficiency of chromic compounds through its photodegradation of such pigment or dyes.

The coloration and fading of the photochromic sample can be visualized in Figure 5. The numbers represented in the respective image show us how the coloration of the sample occurs stepwise, in the forward direction. In contrast, in the reverse direction, it shows us the decoloration of pigment occurs gradually to reach its original state from intense colored state. It allows us to give an idea visually, how the fatigue behavior of such photochromic system changes under the influence of UV light source and upon removal of the same.

The change in coloration and decoloration of the photochromic fabric sample was observed during the exposure and the decay phase, with and without the UV irradiance respectively. In this study, five irradiation cycles are used to study the fatigue resistance of used chromic pigment in continuous mode. The obtained monochromatic spectroscopic data were fitted using the first-order exponential decaying function at its dominant wavelength at which the equilibrium is attained. The first-order kinetic association and dissociation model were used to compute the exposure and decay phase kinetic parameters from $f(R)_\lambda$ values at its dominant wavelength.

The photodegradation of the chromic compounds follows exponential decay in regards to the amount of dosage of the used UV light source for activation. In a continuous mode of irradiance, the absorbance values vary with the radiance time used for studying the fatigue resistance of such chromic species.

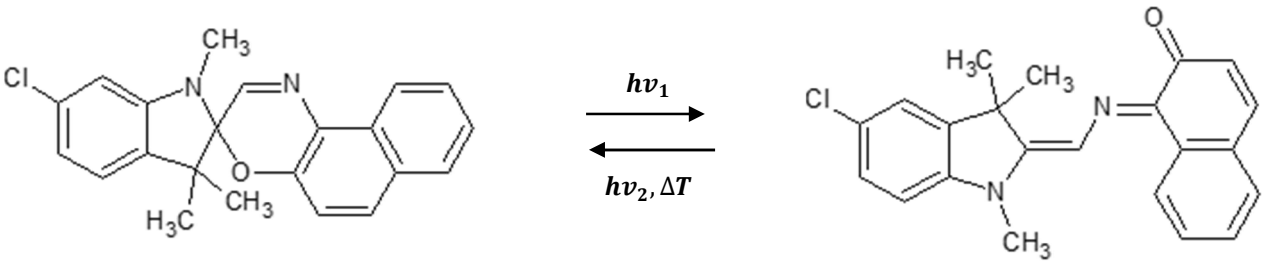
The integrated form of the first-order rate law for the photodegradation process can be rewritten as follows (Equation 7):

$$ln\frac{I}{I_0} = -kt \quad (7)$$

where: I/I_0 is the relative intensity ratio measured from the reflectance data as a colored intensity values, k is the rate constant and t is the time used to observe the photochromic response.

The UV irradiance dose can be measured: UV dose [mW.s.cm^{-2}] = UV energy received [mW.cm^{-2}] of the used UV light source of specific wavelength * UV irradiance time [s], which is used for excitation prolong to the exposure time used to measure the fatigue resistance of the chromic compound. This will give us the logarithmic relationship of the energy used to excite the photochromic species to the exposure time used. However, one can even measure for large UV irradiance cycles; the long-term fatigue behavior of the chromic compounds can also be analyzed.

The open forms are constituted during continuous irradiation mode, but the photo isomers having a long half-life accumulates, and the peak is observed at its dominant wavelength (λ_{max}). The colorability of the isomeric forms might be the same; only the opening of the heterocyclic ring of the compound structure is at a different planar angle. The most stable photo isomeric form will be visualized and the measured reflectance values can be used further to obtain the spectrokinetic parameters. The spectrokinetic analysis is carried out under UV and Visible light sources in a continuous irradiance mode. The kinetic parameters were computed by fitting the data using the one-phase association followed by the plateau function. The photochromic properties of its compounds include the color intensity, the thermal bleaching rate, half-life. The obtained parameters characterize the photochromic behavior of the used pigment under different UV irradiation times of one cycle. The dominant wavelength (λ_{max}), the thermal bleaching rate (k), the colored intensity values – which are measured as a function of the Kubelka-Munk function.



Closed-form (Colorless) Photomerocynaine (PMC) form (Purple)

Figure 6 The photochromic transition of used pigment

Table 1 Measured physical properties of used fabric to study photochromic response

Bending length [cm] Warp	Bending length [cm] Weft	Fabric thickness [mm]	Fabric areal density [g.m^{-2}]	Flexural rigidity [mg.cm] Warp	Flexural rigidity [mg.cm] Weft	Bending modulus [kg.m^{-2}] Warp	Bending modulus [kg.m^{-2}] Weft
4.7	3.2	0.326	170.7	949	543	330	184

2 MATERIALS AND METHODS

2.1 Photochromic pigment and its photo isomeric forms

Woven fabric, a blend of polyester:cotton 65:35, was printed with 5-chloro-1,3,3 trimethylspiro [indoline-2,3'-(3H)naphtho(2,1-b)(1,4)-oxazine] pigment with concentration 100 g.kg^{-1} using a screen printing method. The chemical structure of this pigment and its photo merocyanine form (PMC) is as shown in the scheme in Figure 6. Then these prints were dried 5 min at 120°C and cured 3 min at 150°C.

The chromic pigment undergoes fatigue mode depending on the intensity of UV light used and the exposure time. It might result in the possible photo by-products or the isomeric photo forms. In previous studies, the following isomers have been reported of the spirooxazine based pigment. The purple color is observed after being irradiated with UV light. The photo merocyanine (PMC) form may undergo isomerism in the back reaction if it is illuminated in a continuous mode for a particular number of used UV irradiance cycles. The measured physical properties of the used fabric are as follows in Table 1:

2.2 Spectro kinetic measurements using FOTOCHROM 3

FOTOCHROM 3 device allows us to measure in continuous mode: UV+ and UV- mode for five irradiance cycles, as per the kinetics of one photochromic cycle. It consists of two light sources: excitation light source (360 nm UV light source) and visible light source, allowing to measure the used photochromic woven sample's reflectance data. The spectral power distribution (SPD) of used light sources was measured as shown in Figure 7. The obtained reflectance data were converted to color intensity values at its dominant wavelength using the Kubelka-Munk (*K/S*) function and treated further for evaluation. The dominant wavelength is 570 nm for the used pigment, which changes from colorless to purple. The color intensity values were fitted to the first-order kinetic exponential model at its dominant wavelength to compute the kinetic parameters: rate constant *k*, half-life $t_{1/2}$.

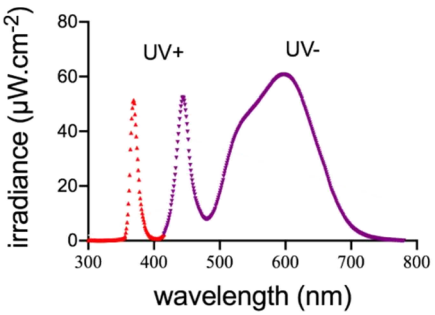


Figure 7 Spectral power distribution (SPD) of used light sources

Fatigue resistance of photochromic fabric samples was measured at 20±2°C using FOTOCHROM 3 device (Figure 8) for five irrespective photochromic cycles with a symmetrical arrangement of UV+(E) and UV-(D) timing of two different decay times 150E:600D, 300E:600D, and 600E:600D. Here, 000E:000D corresponds to the time used for the E=exposure phase and D=decay phase, the time used is in seconds. During testing, the UVA region's used constant spectral irradiance was 780 μW.cm$^{-2,}$ and the resulting dose (fluence) was calculated as a linear combination of spectral irradiance and used UV + time. The UV irradiance dose was measured as: UV dose [mW.s.cm^{-2}] = UV energy received [mW.cm^{-2}] * UV irradiance time [s] used for UV exposure under continuous UV irradiance using the FOTOCHROM 3 device.

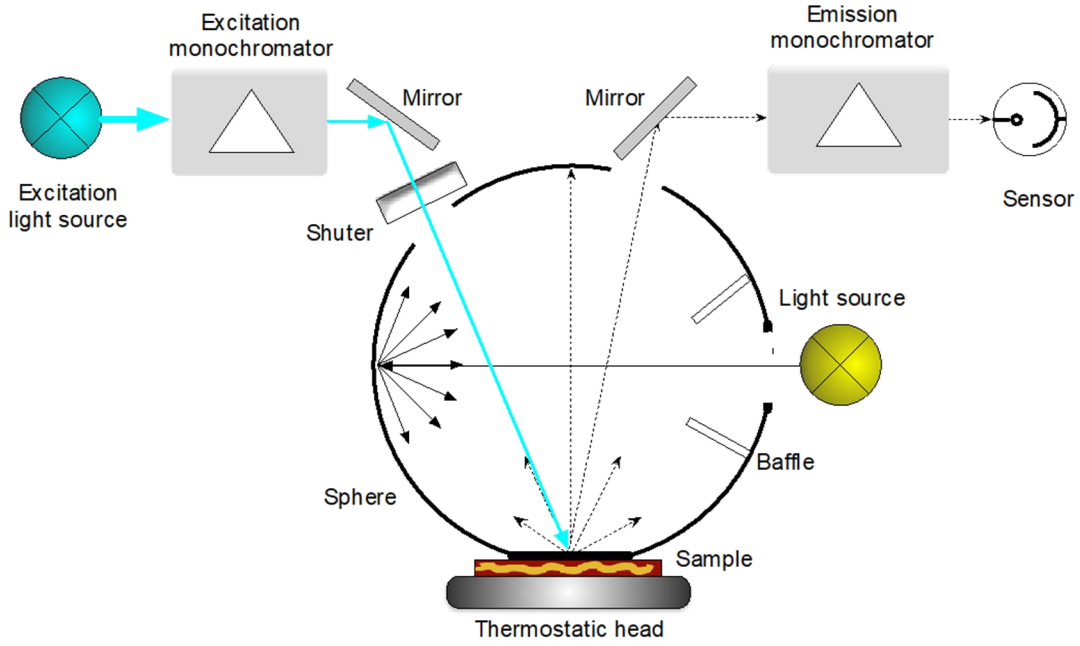


Figure 8 General scheme of FOTOCHROM 3 instrument [16]

3 RESULTS AND DISCUSSION

A new approach for measuring the fatigue resistance of the photochromic system has been described. The photodegradation of used chromic pigment is related to the specific amount of UV light dose of UV illumination (the number of photons absorbed by the molecule to open the ring form). The colorless state is absorbed to attain the colored state. The UV illumination dose is measured with the amount of UV irradiance time used during the exposure phase to open and activate the used pigment structure's cyclic ring. Figure 8 shows the used experimental set-up of used exposure time with constant relaxation time to study the fatigue behavior and the photodegradation of the chromic molecules under the UV influence. The experimental set-up was kept for five irradiance cycles in a continuous mode (Figure 9).

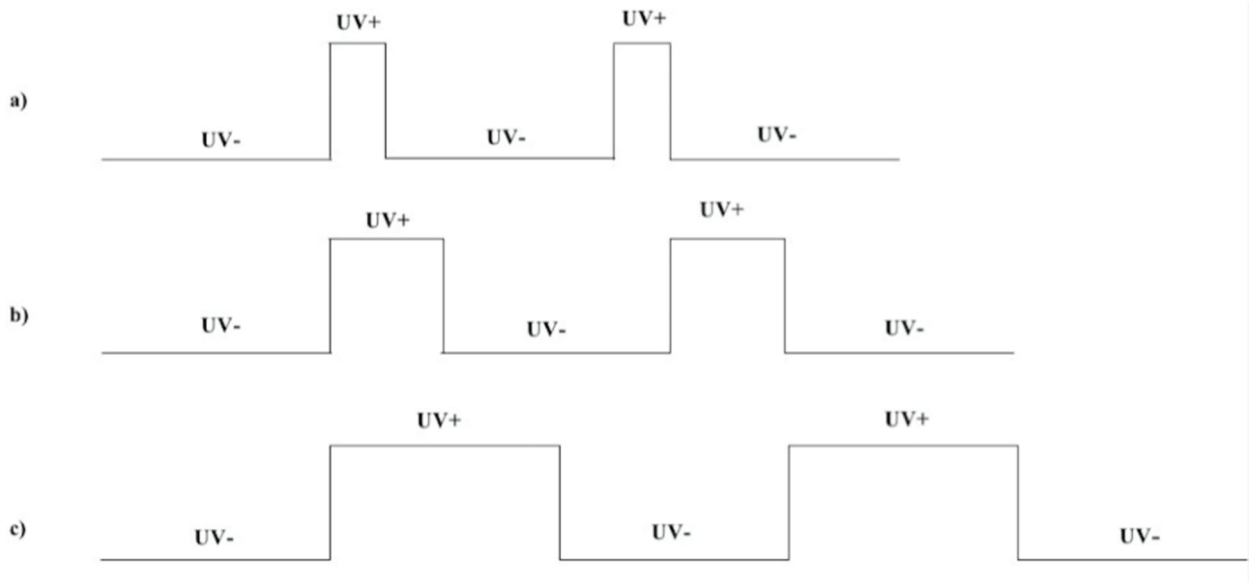


Figure 9 Schematic of the used experimental set-up

Figure 10 illustrates the relative intensity ratio irrespective of the first irradiance cycle to five exposure cycles as per the schematic experimental set-up as in the Figures 9a-c. It is computed from the obtained parameters - the span length value (coloration intensity *K/S* value) after fitting the *K/S* data using a one-phase association followed by plateau function. However, with an increase in the number of UV irradiance cycles, the color intensity (*K/S* values) decreases with a gradual rise in the number of cycles.

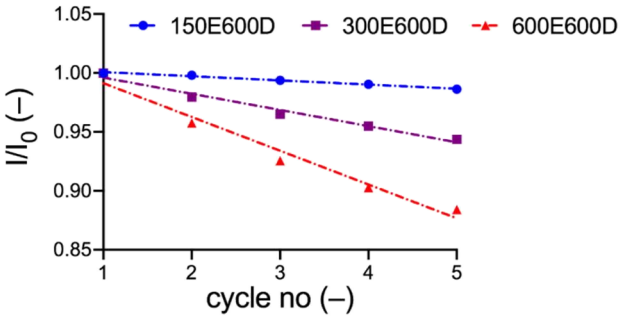


Figure 10 Dependence of I/I_0 and exposure time *t* of the individual cycle

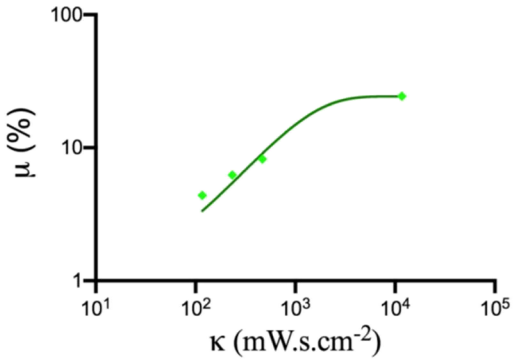


Figure 11 Three exposure times with 600 s decoloration time

We can observe the shift of intensity decay of the pigment for double decoloration time used for the same exposure time used.

The exposure time, decoloration time, and the amount of UV dose used for the exposure time are interrelated. The UV dose [mW.s.cm^{-2}] required to form photo merocyanine form to the decay rate [%] has been reported for the asymmetrical time used to study fatigue behavior.

The intensity decay [%] of the pigment molecules depends on the used exposure time and the amount of UV dose used for the activation, until the used exposure time. Figure 11 illustrates the UV dose behavior in corresponding to the photodegradation of the used chromic pigment with the relaxation time of 600 s. The data fit with exponential function with the coefficient of determination value 0.99. The photodegradation and used dose relationship can be put in general from as follows:

$$\mu = a + b\, e^{-c\kappa}$$

where: μ = photodegradation rate of the chromic compound undergo in regards to the applied UV dosage; κ = the corresponding used UV dosage for studying the fatigue resistance of the chromic compounds (the UV light source of specific wavelength).

Table 2 illustrates the computed kinetic parameters using the one-phase association followed by plateau function, the average values of five replicas of each measurement and the fitted data. It has been calculated irrespective of the five consecutive photochromic cycles during the growth phase. The pigment's half-life decreases over the consecutive number of irradiance cycles, as its degradation occurs with the same exposure time over the number of irradiance cycles used. The amount of UV energy at its dominant wavelength to activate chromic compounds is related to the photodegradation rate as a fatigue behavior in continuous mode.

The asymmetrical exposure time was used with constant decoloration time to see the effect of coloration and the amount of dose required for prolonged decoloration time.

With the photodegradation of the pigment with the amount of dose used, we observe the shift of intensity decay equally with 600 s of decoloration time. With more decoloration time with the same exposure time, the photodegradation rate increases with the higher number of irradiance cycles.

Table 2 Kinetic parameters with 600 s decoloration time ($t_{1/2}$ - half-life; *k* - rate constant)

Time [s] used for one cycle	Computed kinetic parameters										κ [mW.s.cm^2]	μ [%]
	1th cycle		2nd cycle		3rd cycle		4th cycle		5th cycle			
	$t_{1/2}$ [s]	k [s^{-1}]	$t_{1/2}$ [s]	k [s^{-1}]	$t_{1/2}$ [s]	k [s^{-1}]	$t_{1/2}$ [s]	k [s^{-1}]	$t_{1/2}$ [s]	k [s^{-1}]		
150E:600D	8.95	0.0775	8.96	0.07754	8.77	0.07898	8.69	0.07988	8.57	0.08098	117	4.38
300E:600D	8.72	0.07962	8.62	0.08054	8.68	0.07998	8.44	0.08216	8.47	0.08194	234	6.22
600E:600D	8.37	0.08296	8.25	0.084	8.16	0.08494	8.18	0.08484	8.14	0.0853	468	8.21

4 CONCLUSIONS

Durability and spectral sensitivity are the essential criteria allowing the comparison of various photochromic products, including textiles. The fatigue resistance of spirooxazine based chromic pigment that opens its heterocyclic ring under the influence of UV irradiance has been studied. The photostability of such chromophore species, which is responsible for the color change in their chemical structure of pigment which is dependent property to the amount of UV exposure time and the amount of UV dose used. Fatigue resistance of photochromic dyes is measured by evaluating time or cycles caused 50% of optical density decrease based on different method testing scenarios such as flash, continuous and cyclic degradation. An increase in the UV irradiance time used for the exposure phase shows a decline in the relative intensity decay during photo coloration and discoloration process of the used pigment. When the amount of spectral irradiance of the used light source is known, the system is more reliable to the relative decay intensity of photochromic color change to the dose of light used during the experiment. We conclude that the number of irradiance cycles used for the investigation should be considered for studying the relative intensity ratio curves, which also plays a vital role in studying the fatigue behavior of chromic compounds in a continuous UV irradiance mode. However, the time for relaxation of the pigment with a certain amount of exposure time used for the experiment plays a significant role in studying the photodegradation of the pigment molecules and the amount of UV dose used for the activation at the molecular level. The intensity decay of the pigment with an increase in relaxation time, the photodegradation is shifted a little with the same amount of exposure time. Moreover, the applied approach and the stated method can be used for other photochromic compounds to study their photodegradation rate and their kinetic parameters, which can be analyzed using colorimetric data. Such compounds can be used as a UV sensor applied on fabric and paper for outdoor and anti-counterfeiting applications respectively.

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MEASURING SELECTED PROPERTIES OF MATERIALS OF MILITARY CLOTHING FOR THEIR POSSIBLE INNOVATION

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Abstract: *The article deals with materials of service military clothing in the Czech Armed Forces (CAF) and their possible innovations. Useful properties were identified based on the results of a questionnaire survey in the field of satisfaction of soldiers with current military uniform. Properties as colourfastness, abrasion and pilling of materials became the subject of measurement experiment. The measurement took place in the Laboratory of Physiological Comfort of the Technical University of Liberec. The aim of the work was to create design possibilities for improving the application properties of materials. The results are a source of important information for the stage of design and innovation in the introduction of new service military uniform, leading to improved military equipment.*

Keywords: *abrasion, colourfastness, maintenance system, Martindale, military uniform, pilling, property measurement.*

1 INTRODUCTION

Military clothing has a long tradition and is influenced by a number of aspects, including gender, rank, type of activity performed, climatic conditions, level of protection, but also, for example, tradition. In order for textile materials to be used in the production of military uniforms, they must meet the requirements imposed on them during use, i.e. the purpose of the garment. Pre-specified useful and processing properties are placed on clothing materials, which are intended for their application on the market.

The Technical Specifications for Material for Personal Use of soldiers (TS MPU), which include military uniforms, set out the requirements associated with the requirements for the properties of the material used, design, dimensions and maintenance, including instructions for storage, marking and disposal of clothing.

The useful properties of the equipment material include those that are applied from the point of view of the consumer (soldier) in demanding conditions during the use of clothing. The characteristics of the equipment materials must be such that military clothing made from them performs all the required functions. They also have a psychological effect on its users. Useful properties are divided into four basic groups. It is durability and the possibility of maintenance, representative, aesthetic properties and clothing comfort. Equally important is the group of special properties, which are used mainly for certain garments requiring special treatment [1]. Durability is given by the resistance of the fabric to stress. It is the time for which the fabric is functional before it wears out and loses its properties. During use, the garment is exposed to a large number of external influences, both during wearing and maintenance. The fabric is gradually worn by light, sweat, abrasion, bending and stretching until it deteriorates. It can also lose its properties by improper washing and ironing [2]. Durability, maintenance, original appearance and properties are complicated by combinations of different clothing materials of different material composition, surface treatment such as coating. Adherence to the recommended product maintenance procedures is essential. The reason for the shorter service life is the use of atypical components or surface treatments of the material, which are prone to mechanical failure (abrasion). Garment products are made up of different types of fibers, so it is important to follow the most sensitive component of the whole system [3]. Aesthetic and representative characteristics are addressed in connection with the national tradition of uniforms and rank. The appearance of the fabric is influenced by the material used, the threads, the weave and its finishing. Tested properties include colourfastness, creasability, flowability, pilling resistance, and more. Abrasion and pilling resistance are key properties for textiles. These are the basic properties that determine the durability of the product and are the most common reasons for complaints about textiles, and therefore it is important to monitor them. Abrasion can characterized as a disturbance of the fabric surface. Abrasion occurs upon contact the fabric into contact with another fabric or rough surface. In this way,

the individual fibers are abrade, which then fall off, the bonding points are pierce and the fabric disintegrates. Abrasion resistance tests are simulation tests that imitate how long a fabric can withstand stress, i.e. abrasion, in practical use. Methods of abrasion testing are in the area, edge and random direction of the fabric [4]. The relation between abrasion resistance and fabric constructional parameters was found in the study [5]. The lint on the garment is manifest by lumps, which are the result of excessive wear or improper maintenance. The main factor here is the mechanical friction of textiles. It is a process in which the fibers are released from the fabric and their subsequent agglomeration and winding. The cream lumps are then tripe in the structure of the fabric, creating an unsightly impression. This phenomenon depends on the construction of the garment and the properties of the fibers and threads. This is a relatively common phenomenon in products made of synthetic fibers, most often polyester. Synthetic fibers are smooth and therefore easier to release from the base of the fabric. Thanks to its strength, it also holds better on the fabric. On the other hand, strength is one of the properties of textile fibers, which makes synthetic fibers more resistant to abrasion [1]. Three test methods for evaluating the abrasion or wear resistance of textile materials were compare in the study [6].

The article is focus on the need to improve the useful properties and construction of the existing uniform, which in the opinion of soldiers is obsolete and has a number of poor material properties, which affect in particular the representative properties of clothing and durability. It was necessary to verify the functionality and suitability of the maintenance of existing military uniforms by measuring selected properties of materials. Based on the evaluation of the survey of soldiers' satisfaction with wearing the uniform, tests of colourfastness when dripping with water, resistance to pilling and resistance to abrasion before washing and after thirty washes performed on the Martindale device. The output is a proposal of possibilities to improve the useful properties of equipment components.

2 EXPERIMENTAL

The questionnaire of satisfaction of soldiers with using current military uniform was conduct in experimental part. Based on the results of the survey, the properties for measurement were identified. The aim of the experiment was to measure selected properties of samples of materials together with other suitable materials for the production of military uniforms. Determining the functionality of existing materials was the purpose of the measurement. Tests for colourfastness to water dripping, resistance to pilling, abrasion resistance before and after thirty washes were performed. These tests were performed under normal operating climatic conditions specified in the standards: air temperature 20±2°C and relative humidity 65±2%.

2.1 Materials

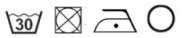
Two types of fabric samples used for the experiment, which used for the production of existing uniforms 97 (A) and 2005 (B). In addition, two types of fabric samples used (C and D), which are suitable for the production of suits and were selected as an alternative replacement for existing materials. Thirty-two test samples of the fabric were tested. Table 1 provides an overview of used materials of military uniforms and other used materials, including maintenance symbols, manufacturer and other information. Figure 1 shows examples of two types of military uniforms, whose material designation in Table 1 is marking A and B.

Figure 1 Examples of two types of military uniforms

2.2 Methods

The questionnaire of satisfaction of soldiers with the use of the current military uniform was conducted in February 2020. In the questionnaire was the sample of material A from the Table 1 indicated by the exact designation of the uniform 97. The examined sample of the Czech Armed Forces soldiers was 200 respondents.

Table 1 Overview of used materials of military uniforms and other used materials

Marking	Material composition	Thickness [mm]	Square mass [g/m^2]	Weave fabric	Maintenance system	Producer
A	45% wool, 55% polyester	0.59	315	Amplified atlas		VESTIMENTUM s.r.o. Czech republic
B	50% wool, 47% polyester, 3% elastane	0.24	210	Gabardine Multistage twill		Koutný spol. s.r.o. Czech republic
C	62% polyester, 32% viscose, 6% elastane	0.30	264	Gabardine Multistage twill		fabfab GmbH Gernamy
D	53% polyester, 43% wool, 4% elastane	0.15	205	Amplified atlas		fabfab GmbH Gernamy

Research question No. 1: Are the correct procedures followed when maintaining the equipment components of the uniform 97? Questions 5, 7 and 16 of the questionnaire survey assessed this question:
5. How do you maintain trousers 97?
7. How do you maintain the blouse 97?
16. How do you maintain the skirt 97?

Research question No. 2: Does the number of equipment components of the uniform 97 affect the frequency of maintenance? Questions 2, 3, 4, 6, 8, 15 and 17 from the questionnaire survey evaluated this question:
2. How often do you wear a uniform 97 at work?
3. How many pieces of blouse 97 (jacket) do you own?
4. How many pieces of trousers 97 do you own?
6. How often do you maintain your trousers 97?
8. How often do you perform maintenance on the blouse 97?
15. How many skirts 97 do you own?
17. How often do you maintain skirt 97?

Research question No. 3: Do soldiers perceive the comfort of wearing uniform 97 positively? This question evaluated by questions 9, 10 and 11 from the questionnaire survey:
9. Are you satisfied with the uniform 97 in terms of clothing design?
10. Are you satisfied with the uniform 97 in terms of the useful properties of the materials (i.e. resistance to pilling and abrasion, colourfastness...)?
11. Do you feel comfortable in the 97 uniform?

Research question 4: What would soldiers want to change to using and purchasing a uniform 97?

This question evaluated by questions 12 and 13 from the questionnaire survey:
12. What would you like to change on the uniform 97?
13. What would you like to change about purchasing a uniform 97?

The colourfastness test when dripping with water was performed according to the standard ČSN EN ISO 105-E07 (80 0145) [7]. Two samples of each type of material measuring 40 x 100 mm used. The first sample unloaded with water for comparison with the drip sample and the second sample for the test. The essence of the test was the incorporation of drops of distilled water by pipette into the test sample. After incorporating the water into the sample, a stain with a diameter of 20 mm formed. After 2 min, the change in hue at the edge of the stain evaluated according to a grey scale. The test sample air-dried at room temperature and the shade change re-evaluated according to the grey scale.

The determination of the inclination of fabrics for pilling by the Martindale method was performed according to the standard ČSN EN ISO 12945-2 (80 0837) [8]. Two samples from each type of material used before and after thirty washes, one for comparison and the other for testing. All prepared samples were washed in an automatic washing machine. A program for coloured laundry without prewash was chosen. The washing time was 120 minutes at 40° C. Wool and delicate washing gel was used for washing and no softeners were used. The squeeze was set at 800 rpm. The samples were knocked out and hung on a dryer so to the warp was in the vertical direction according to the ČSN EN ISO 6330 standard [9]. After drying, the samples were ironed according to the recommended maintenance symbols. The sample had a circular shape with a diameter of 140 mm. The essence of the test was the movement of a circular test specimen under a specified load on a friction surface formed by the same fabric, while the Lissajous pattern was observed. The test sample was also rotatable about its central axis perpendicular to the surface of the test specimen. The test specimens were mounted face up in the holders of the Martindale instrument. Category 2 was selected in the pilling test for fabrics. The abrasive was a face-to-face test fabric and a load weight of 415 ± 2 g used. The test run until evaluation stage 6 for 7000 revolutions was reached. At the indicated load, the test specimens moved along a friction surface formed by the same abrasive fabric material. The pilling was evaluated visually after the defined stages of the abrasion test. The determination of abrasion resistance of fabrics

by the Martindale method was performed according to the standard ČSN EN ISO 12947-2 (80 0846) [10]. One sample from each type of material was used before and after thirty washes. The method of washing the samples was described in the previous paragraph. The essence of the test was to abrade the circular sample clamped in the sample holder against the abrasive by a gradual movement that follows the Lissajous pattern. The abrasion resistance of the fabric was determined using a test speed interval until the samples damaged. For the abrasion load, a weight value of 595 g was determined for clothing and home textiles with a pressure of 9 kPa. On the sample-clamping holder, auxiliary materials placed under the sample in the order of foam material, felt and sample, which abraded by a defined abrasive fabric placed on the table. The dimension of the test specimen had a circular shape with a diameter of 35 mm. The test procedure was similar to the previous test, except that a wool abrasive fabric was used, which was attached to the holder of the Martindale instrument. The test specimen attached to the upper holder and abraded under the defined conditions specified until the first binding point ruptured or the fabric damaged. For each test specimen, a test interval was found in which the test specimen damaged or the first binding point of the fabric ruptured.

3 RESULTS AND DISCUSSION

A questionnaire was conducted to survey the satisfaction of soldiers using the current military uniform, and based on the results of the survey, the properties for measurement were identified. Tests for colorfastness to water dripping, pilling and abrasion resistance using the modified Martindale method were performed in the Laboratory of Physiological Comfort of the Technical University of Liberec.

3.1 Evaluation of the questionnaire of satisfaction survey

Although the maintenance symbols for trousers and the skirt of uniform 97 indicate a ban on washing in the washing machine, more than 60% of respondents said that they use this type of maintenance for these equipment components. The method of maintenance of the blouse 97 in the treatment plant is performed correctly by 129 respondents, which is 64.5%. As another method of maintenance, 6 respondents mentioned brushing. Based on the evaluation of the answers to questions No. 5, 7 and 16, it is possible to answer the 1st research question. In the case of trousers and skirts 97, the correct maintenance procedure is not followed. The components are washed in the washing machine. The blouse 97 is usually properly maintained in dry cleaners or the part is not maintained. Regarding the influence of the number of skirts on their maintenance, it can be said that the more pieces of equipment the user owns, the lower the frequency of its maintenance. Three respondents out of 40 women interviewed stated that they do not perform skirt maintenance. This is affected not only by the higher number of components they own, but also by the frequency of wearing. At least once every 14 days, 15 respondents perform maintenance. She wears the clothing component almost daily and each has two skirts available. Based on the evaluation of the answers to questions 2, 3, 4, 6, 8, 15 and 17, the 2nd research question can be answered. The frequency of maintenance of equipment components is affected not only by their number, which the user owns, but also by the frequency of wearing them.

When evaluating the questions, the statistical significance set to 5% confidence level between the frequencies of answers was divided into two parts, namely the group certainly yes, rather yes versus rather no and certainly no. In the case of satisfaction with the design of uniform 97, the result of 113 respondents satisfied (56.5%) versus 87 dissatisfied (43.5%) is not statistically significant and we cannot say with certainty that respondents are satisfied with the design of uniform 97. Satisfaction with the above-mentioned uniform in terms of the useful properties of the materials used expressed by 66 respondents (33%) and dissatisfaction by 134 respondents (67%). The above result is statistically significant and we can confirm that the respondents are not satisfied with the uniform in terms of useful properties of the materials used. The last question asked if the soldier felt comfortable in his/her uniform. The obtained result of 85 satisfied respondents (42.5%) versus 115 dissatisfied (57.5%) is statistically significant and it is possible to deduce that the respondents do not feel comfortable wearing a uniform 97. Based on the evaluation of the answers to questions 9, 10 and 11 answered to the third research question. Soldiers perceive comfort, which includes satisfaction with the design of the garment, useful properties and overall comfort when wearing the uniform 97, negatively. 82% of respondents would change the properties of the material used and almost half of those surveyed would change the design of the garment. Respondents would welcome the possibility of tailoring adjustments by the Dispensing Centres of Natural Clothing (VSNO) or sewing uniforms with a measuring system. Other preferred changes of respondents mentioned in the area of uniform 97 acquisition. 69.5% of respondents would increase the number of components to acquired, which are few when used almost daily. Soldiers would welcome the reintroduction of jacket shirts, perforated low shoes, winter boots and winter jackets. The soldiers emphasized their satisfaction

with the summer uniform of 2005, which unfortunately can no longer be obtained and is not suitable for winter weather. One respondent mentioned the unavailability of women's trousers 97 at a height of 189 cm. Based on the evaluation of the answers to questions 12 and 13; the fourth research question can be answered. Soldiers would like to change the use and acquisition of the uniform 97, in particular the properties of materials and increase the number of equipment components to purchase the uniform 97.

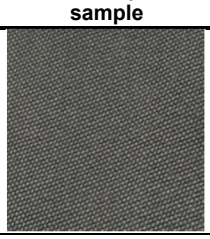
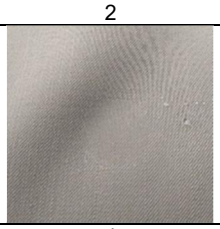
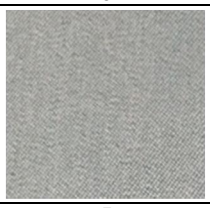
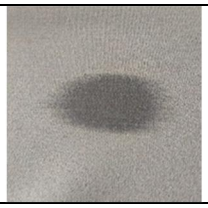
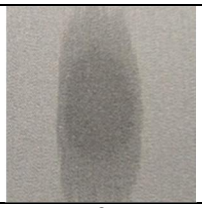
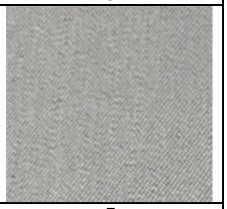
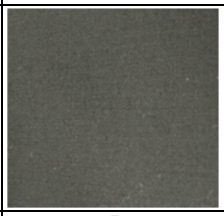
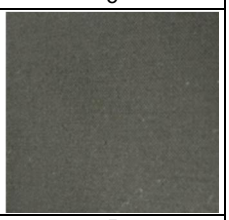
The results of the soldiers' satisfaction survey with the uniform 97 survey showed that most respondents are not well informed about the proper maintenance of service trousers and skirts. It turned out that the correct maintenance procedure not followed in this case. The components are normally machine washed, which can cause a change in the dimensions, colourfastness and useful properties of the materials. The soldiers also drew attention to the formation of permanent stains when dripping the uniform with water, such as rain. Improperly selected iron temperature during maintenance causes the material to flare up. According to the respondents, the material is biting, prone to dust and is very discomfort able in summer.

It has been show that the number of equipment components of the uniform 97 has an effect on the frequency of maintenance and on the frequency of wearing them. Soldiers are not satisfied in terms of comfort when using the uniform with useful features and overall wearing comfort. They perceive clothing comfort negatively. Only in the case of clothing design, 113 of respondents were satisfied, which is more than half. Regarding the opinions and attitudes of soldiers in what they would like to change to the use and purchase of uniforms, 134 respondents agree that they would like to change the useful properties of the materials used and increase the number of equipment components for their acquisition.

3.2 Evaluation of the colourfastness test when dripping with water

The evaluation was of the test performed based on a subjective evaluation of three experts in the laboratory. Subjects evaluated changes in colourfastness of samples by dripping with water after 2 minutes and after drying. All three persons agreed on the same test results, which shown in Table 2, together with photographic images of the test specimens. The images are illustrative.

Table 2 Evaluation of the colourfastness test when dripping with water for all samples

Marking	Untried comparative sample	Tried sample at the beginning of the test	Tried sample after 2 min and after shaking the drop	Tried sample after drying
A				
Degree of grey scale	5	-	2	4
B				
Degree of grey scale	5	-	4	5
C				
Degree of grey scale	5	-	2	5
D				
Degree of grey scale	5	-	4	5

The Table 2 shows that according to the grey scale for evaluating the change of shade according to ISO 105-A02, samples of materials marked B and D have the best colourfastness when dripped with water. Drying of both materials had the same grade 5 as the untested comparative material. In the case of material samples A and C, the results are slightly different. For material C, immediately after dripping the liquid with a pipette, the drop soaked into the fabric and formed a gradually enlarging map, first with a circular diameter of 2 cm up to an oval shape of 4 x 5 cm. However, after drying, the colour change was at grade 5, which is a satisfactory result. The material obviously did not have a water-repellent or oleo phobic treatment that would prevent such rapid absorption of the droplet, and therefore would not be suitable for the production of a uniform in its current state. In the sample of material A, a part of the contents of the drop was soaked into the fabric more slowly than in the case of gabardine. Nevertheless, the change in hue to level 2 from the original colour was significant after 2 minutes. After the material has dried, the drop left a significant change in the shade of grade 4, which is the value that stated in the criteria of required values for colourfastness in the TS MPU uniform made of material A.

3.3 Evaluation of the determination of the inclination of fabrics for pilling by the Martindale method

The evaluation was of the test performed based on a subjective assessment of 3 experts in the laboratory with the help of standards and a visual appraisal, which shown in Table 3.

Table 3 Visual evaluation of the test [11]

Degree	Description
5	Without changes.
4	Slight pulping of the surface and / or onset of lint formation.
3	Slight pulping of the surface and / or slight pilling. The lumps of various sizes and densities partially cover the surface of the sample.
2	Significant surface pulping and / or significant pilling. The lumps of various sizes and densities cover a large part of the sample surface.
1	Dense surface pulping and / or heavy pilling. The lumps of different sizes and densities cover the entire surface of the sample.

Pulping and pilling were evaluated visually after defined stages of the test. Each test sample assigned the degree of pilling according to Table 3. If the evaluation fell between two degrees, an intermediate degree was marked, e.g. 3-4. The standards used to evaluate the pilling test are shown in Figures 2 and 3. The test result for each individual evaluator was the average of the pilling degrees given to the test sample. The result of the test for the laboratory sample was the average value of the degrees of pilling assigned by all evaluators.

Figures 4 and 5 show a comparison of the degrees of pilling according to the standard during the evaluation at speeds of 2000, 5000 and 7000 for the samples marked A and B, which showed differences in the evaluation in the individual stages. Arithmetic mean was 4.125 before and 4.5 after 30 washes. Reliability limit of the average value was 1.640 before and 0.919 after 30 washes.

Figure 2 Standards for the evaluation of the degree of pilling 3 to 5

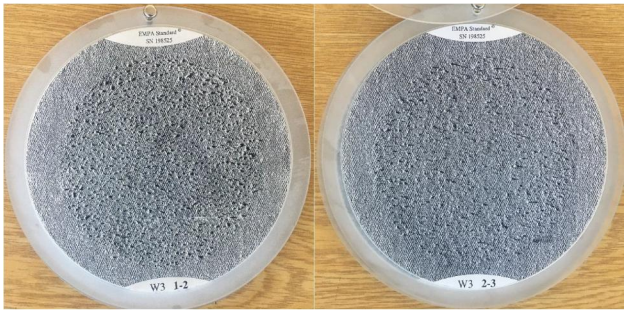


Figure 3 Standards for the evaluation of the degree of pilling 1 to 3

It can be seen from the graphs in Figures 4 and 5 that before washing, the degree of pilling is lower for both materials, so they have a greater tendency to pill than after 30 washes.

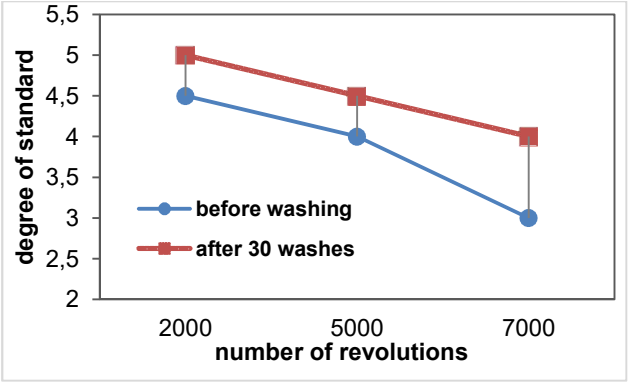


Figure 4 Comparison of average values of pilling for material A

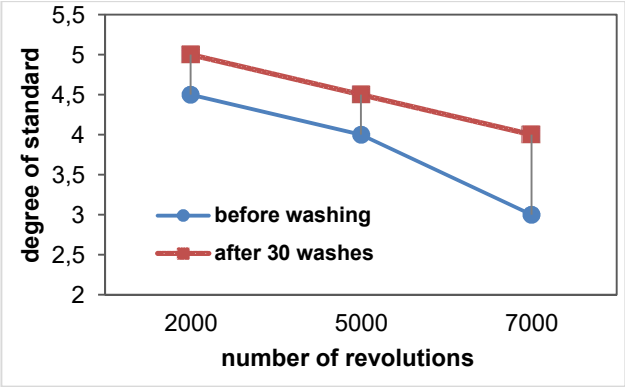


Figure 5 Comparison of average values of pilling for material C

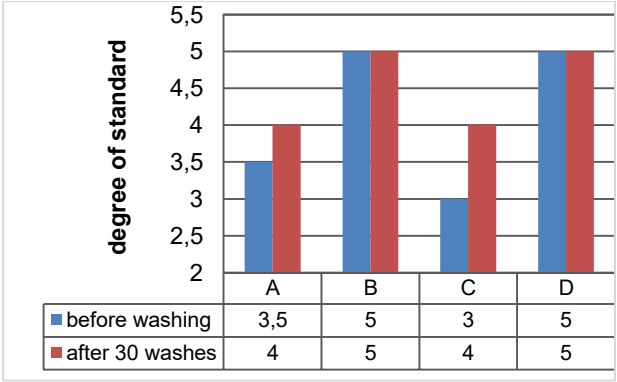


Figure 6 Comparison of average values of pilling for all samples

It follows from the above that, in the case of samples A and C, washing has a good effect on the pilling, since the degree of pilling after washing increases and thus the tendency to pill decreases. Washing releases the fibres, which probably worsened the pilling. Other test samples of other types of materials marked B and D showed no changes in surface pulping or the formation of lumps both before and after 30 washes. These samples evaluated at all times in grade 5, the evaluation of which is no change. Comparison of average values of pilling for all samples all samples is in Figure 6.

3.4 Evaluation of the determination of abrasion resistance of fabrics by the Martindale method

The evaluation was of the test performed based on the determined values of the number of revolutions at which the sample damaged or the first binding point of the fabric was torn. The graph in Figure 7 shows the resulting abrasion resistance values for damage to all samples.

Arithmetic mean was 35 000 number of revolutions before and 37125 after 30 washes. Reliability limit of the average value was 28642.01 before and 27471.65 after 30 washes. From the results of the abrasion resistance test, it can be stated that for the types of materials marked A and C, washing does not affect the abrasion resistance. Before and after 30 washes, the samples damaged at 50,000 revolutions per minute (rpm). For samples of material B, the first binding point ruptured at 26,000 rpm before washing and 9,000 rpm later for samples that washed 30 times. This means that in the case of B washing, it does not affect the abrasion resistance. For test specimens of material D, abrasion occurred after 30 washes 500 revolutions earlier. We can talk about a small, but still effect of washing, because washing can slightly loosen the bond, thin the material and thus reduce its strength. Comparison of abrasion resistance values for all samples can see in the graph in Figure 7.

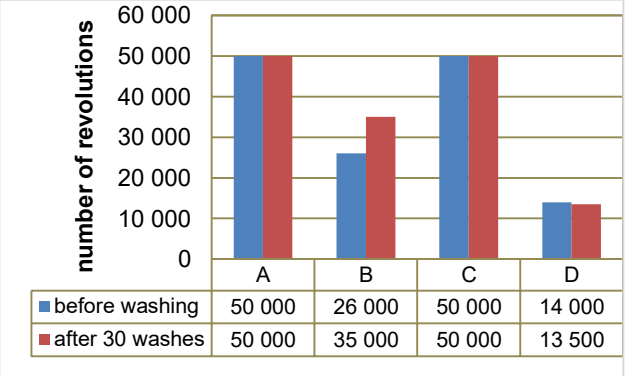


Figure 7 Comparison of abrasion resistance values for all samples

3.5 Proposal of possibilities of improvement properties of materials of military uniforms

In the evaluation of the survey, it considered that due to non-compliance with the proper maintenance of equipment components by washing, the useful properties of materials might deteriorate. The facts found above in the previous subchapter prove that the washing of materials B has no effect on the pilling and in the case of A material even has a favourable effect on the pilling resistance. This is an interesting finding, given that no type of material is allowed to be washed on the maintenance symbols and cleaning in the dry cleaner's is recommended, which will be significantly more expensive for soldiers. Washing these samples also does not affect the abrasion resistance. It follows from the above that in the case of skirts and trousers of materials A and B it is not necessary to maintain them in the dry cleaner's. For the sample of material A, which contains 45% wool and 55% polyester, it is recommended by the suit manufacturer to wash the clothes in a washing machine at 30°C, to gently spin them once, to dry without a dryer and to iron them up to 150°C. For trousers and skirts of material B, which contain 50% wool, 47% polyester and 3% elastane, it is sufficient to carry out maintenance by washing in a washing machine at 30°C, perform gentle single squeezing, dry without a dryer and iron to 110°C.

For both uniforms, the maintenance of the blouse would be left to the dry cleaner's, as the blouse is not maintained as often and it is sufficient and affordable to have the blouse treated professionally once a year.

The soldiers also drew attention to the formation of permanent stains when dripping the uniform with water, for example during the rain on a sample of material A. Yes, this happens, for example, during the ceremonial parades of professional soldiers in uniforms, when it starts to rain and an umbrella can used. The results of the experiment proved that the degree of colourfastness resistance when dripping with water agrees with the value required in TS MPU and is sufficient [12]. Here it is appropriate to think about whether the value determined in the TS MPU is correct and whether further mechanical action on the surface of the dripped fabric does not cause permanent stains. In a test carried out in accordance with the standard by dripping on a fabric that is no longer mechanically subject, there may be no permanent staining. The drop dries eventually. It is also important to consider the choice of determining the appropriate mechanism by the commander for the occasion. A field uniform can also be used for the winter. For the summer period, it would be more appropriate to use a uniform of material B, which shows excellent colourfastness values. However, the problem is insufficient equipment and little or no garments at present. It not known whether it will still be possible to purchase the said summer uniform 2005, which is in all respects a quality and functional equipment component.

To achieve the comfort of the soldier, it would be appropriate to ensure that each soldier has a service and walking uniform available for both the cold season and the summer season. Soldiers who wear uniforms daily in the office would especially welcome the restauration of the availability of uniforms made of B material for summer. The existing uniform made of material A calls for a change, which means both the properties and the design. Especially for women's blouses, there is no inner chest pocket and for trousers, the lining in the front part is missing. For possible innovation of military uniforms, two materials C and D selected, which could replace the materials of current uniforms.

The C type of material selected as a replacement for the material for the service uniform 97. A different variant of the material composition chosen than that of A, with a composition of 62% polyester, 32% viscose and 6% elastane. It is a woven, softly flowing, bi-elastic fabric suitable for business clothes, comfortable suits, trousers, skirts and blazers. Despite the fact that it does not contain wool, the material is pleasantly soft to the touch, as if woolly. The basis weight of the material is 264 g/m^2 at a width of 145 cm. The price of the footage is 279 CZK for 1 m incl. VAT.

From the results of the utility measurement experiment, the following was found. The tested material would be suitable in terms of research results only for abrasion resistance, where, as with material A, the final value was 50,000 revolutions before and after 30 washes. In terms of the pilling resistance, the substitute material ended up as the worst of all. It has a great tendency to pill. In the water drip test, the material reached grade 4 as well as A. The time of drop infiltration was different, when C immediately absorbed the drop and formed visible maps. This fact would eliminated by oleo phobic or waterproof treatment of the fabric. To replace the material of the uniform, I would recommend performing tests on three other suitable materials and increasing the number of tested properties by tests of dimensional changes during washing and ironing.

As an innovative material suitable for the summer variant of the military uniform, the D type of material selected. It has a composition of 53% polyester, 43% wool and 4% elastane. It is a woven, softly flowing, light, elastic fabric suitable for casual clothes, blazers, jackets, trousers and skirts. The material is soft to the touch. The basis weight of the material is 273 g/m^2 at a width of 150 cm. The price of the footage is 449 CZK for 1 m incl. VAT. Based on the results of the utility measurement experiment, it was found that, compared to material B, the substitute material has the same values of colourfastness to dripping water and resistance to pilling. The abrasion resistance of the innovative material is 22,000 revolutions before washing and 21,500 revolutions after 30 washes. For this reason, the material is not suitable for summer uniform innovation. According to the results of experimental measurements of colourfastness, pilling and abrasion resistance, the B material of the summer uniform ended up as the best of all. It meets all the requirements for sufficient comfort of the soldier when worn, and therefore there is no need to innovate it.

4 CONCLUSIONS

The paper dealt with the materials of the CAF military uniforms and its possible innovations. Based on the need to improve the performance and design of the existing uniform, it was necessary to verify the functionality and suitability of the maintenance of current military uniforms by measuring selected properties of materials.

A survey of soldiers' satisfaction with the use of uniforms 97 conducted. The aim of the survey was to find out the experience, opinions and attitudes of the CAF soldiers to use and quality of uniforms 97. The survey showed that most respondents not well informed about proper maintenance of service trousers and skirts. The correct maintenance procedure was not followed.

The components are normally machine washed, which can cause a change in the dimensions, colourfastness and useful properties of the materials. The soldiers also drew attention to the formation of permanent stains when dripping the uniform with water, such as rain. Based on the evaluation of the survey, the properties of colourfastness to water dripping, resistance to pilling and abrasion selected for the measurement experiment so that the above could objectively confirmed or refuted. Based on the results of the survey and experimental measurements, a proposal made to improve the performance and innovation of equipment material. The proposal evaluated the suitability or unsuitability of selected innovative materials C and D for Uniform 97 (A) and Summer Uniform 2005 (B) and recommended other possible procedures to improve the performance of the equipment. It has been proved that washing the equipment parts of the uniform 97 and the uniform 2005 does not affect the colourfastness and resistance to pilling and abrasion. According to the results of the experiment, the material of the summer uniform 2005 ended up as the best of all, it meets all the requirements for sufficient comfort of the soldier when wearing it, and therefore there is no need to innovate the material. The question remains whether it will be possible to restore the option of its acquisition and replenishment of the equipment by soldiers in VSNO, which currently stopped due to the termination of the contract with the manufacturer. All selected utility properties greatly affect the function of equipment components, as they are crucial for the durability of the product and its aesthetic properties, which are key in the case of military uniforms. The appropriate design of the garment is also important. It affects not only the function of the garment, but also the clothing comfort of the soldier who wears the uniform. Selected utility properties of equipment components affect their function in terms of durability of clothing, aesthetic properties, clothing design and the soldier's clothing comfort. The obtained research results and the proposal of possibilities to improve the useful properties of equipment components are a source of important information not only for the design and innovation in the introduction of new equipment components, but also lead to the improvement of military equipment.

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EFFECT OF SPINNING AND DRAWING CONDITIONS ON STRUCTURE PARAMETERS AND MECHANICAL PROPERTIES OF PLA FIBRES

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Abstract: *The biodegradable polymer, polylactic acid (PLA), is becoming more and more popular with manufacturers and traders in an effort to save our planet from plastic contamination. Pure or modified PLA is used in a variety of industrial areas, including fibres. Depending on the stereochemistry of the main chain, PLA can be partially crystalline or completely amorphous, from which its processing properties and method of use also depend. In this work the PLA of Luminy LX175 type was used. This PLA is a high viscosity, low flow, amorphous and transparent PLA resin suitable for film extrusion, thermoforming and also for fibres spinning. The influence of spinning temperature, PLA melt dosing and drawing on the basic parameters of the supramolecular structure (birefringence, sound speed and crystallinity), fineness and basic mechanical properties of fibres (Young's modulus, tenacity and elongation at break) were studied. It was found that the above-studied parameters have a significant effect on the evaluated properties of fibres.*

Keywords: *PLA fibres, supermolecular structure, mechanical properties.*

1 INTRODUCTION

The development of the bioplastics industry has changed dramatically since the early 1990s. The latest generation is moving towards durable bioplastics with a high content of biological materials. The main goal is to replace "fossil carbon" with "renewable carbon", a holistic strategy to mitigate climate change by minimizing the product's impact on the environment during its life cycle. Durable bioplastics are in demand for multiple long-term uses in the automotive, food biomedical but also textile industries. The preference for "renewable carbon" over "fossil carbon" stems from the very awareness of our need to reduce the consumption of non-renewable resources and greenhouse gas emissions [1, 2]. The current pandemic situation also supports the growing global demand for personal protective equipment such as masks, gloves, gowns and bottled hand sanitiser, leading to the accumulation of solid polymer waste [3-5]. The search for materials with similar technical plastics that come from renewable sources is becoming a reality in the 21st century. Although there are already several bio-based technical plastics available on the market, the aim is to take advantage of the price competitiveness and unique properties of polylactic acid (PLA). PLA offers unique properties of biodegradability, biocompatibility, thermoplastic processability and ecological safety [6-8].

PLA was discovered in the 1920s by Wallace Carothers the scientist who invented nylon, but at this time never had been successfully commercialized on a large scale. PLA is aliphatic polyester, due to the ester bonds that link the monomeric units generally producing a lactic acid synthesis that can be produced from renewable sources such as corn, starch, sugar or other biomass [9, 10]. It is high-potential biodegradable thermoplastic polyester due to its unique physical properties, making it useful in a variety of applications, including surgical and medical applications, fibres, films and packaging. PLA is naturally degraded by an in situ hydrolysis mechanism: water molecules break the ester bonds that form the polymer backbone. PLA serves as an alternative to certain petroleum-based plastics in commercial applications. At present, there is a comparable price on the market to commonly available plastics such as polypropylene [2, 6].

PLA fibres are produced using lactic acid as a starting material, which comes from the fermentation of various sources of natural sugars. PLA fibres are used to provide low moisture absorption and high rise by capillary for sports and performance clothing and products. They have a high resistance to ultraviolet light, which is beneficial for outdoor use of furniture and furnishings. In addition to coming from renewable sources every year, PLA fibres are easily melted and offer production benefits that lead to greater consumer choice [11, 5].

The paper presents the results of a study of the PLA, type Luminy LX175, specifically the influence of spinning temperature, PLA melt dosing and drawing on the supermolecular structure parameters and basic mechanical properties of prepared fibres.

2 EXPERIMENTAL AND METHODS

2.1 Materials

Polylactic acid LX175 (PLA) produced by Total Corbion PLA B.V with MFI = 12.8 g/10 min (210°C/2.16 kg) was used.

2.2 Fibre preparation

The samples of PLA fibres were prepared using the classical discontinuous process of spinning and drawing. The laboratory discontinuous line had an extruder with a diameter of 32 mm, with a discontinuous one-step drawing process. PLA biopolymer has been dried before spinning for 4 hours at 85°C. The fibres were prepared at two spinning temperatures of 210°C and 220°C with a final spinning process speed of 1500 m/min. Subsequently, the fibres were drawn to a drawing ratio of λ=1.4, 1.6 and maximum drawing ratio $\lambda_{max,}$, at a drawing temperature of 100°C and a final drawing process speed of 100 m/min. Two 25 holes spinning nozzles were used for spinning, with a diameter of nozzle hole 0.26 mm. The two different dosage amounts of polymer melt 34.7 g/min and 22.5 g/min during spinning were used. Under the above conditions, samples of PLA fibres 1-16, which are listed in Table 1, were prepared.

Table 1 The samples of PLA fibres prepared from biopolymer Luminy LX175

Sample No.	Dosage of polymer melt [g/min/nozzle]	Temperature [°C]	Drawing ratio λ
1	34.7	210	undrawn
2	34.7	210	1.4
3	34.7	210	1.6
4	34.7	210	1.68
5	34.7	220	undrawn
6	34.7	220	1.4
7	34.7	220	1.6
8	34.7	220	1.88
9	22.5	210	undrawn
10	22.5	210	1.4
11	22.5	210	1.6
12	22.5	210	1.66
13	22.5	220	undrawn
14	22.5	220	1.4
15	22.5	220	1.6
16	22.5	220	1.88

2.3 Methods used

Melt Mass-Flow Rate (MFR) of PLA was evaluated using a capillary rheoviscosimeter Dynisco Kayness according to EN ISO 1133-1 under conditions: a temperature: 210°C, a load of 2.16 kg, a detention time of 5 min, nozzle diameter of 2.095 mm, a nozzle length of 8.00 mm, shear stress of 19.5 kPa. The sample has been dried before measurement 4 hours at 85°C.

Birefringence

The orientation of macromolecular chains in fibre expresses the level of anisotropy of the oriented polymer system (fibre). The total orientation of prepared modified PLA fibres was evaluated using polarization microscope DNP 714BI. The refractive indexes of light in the fibre axis ($n_{//}$) and in the perpendicular direction of fibre ($n_{\perp}$) were determined. From the difference of refractive indexes of light, the fibre birefringence (Δn) was calculated.

The sound speed in fibres is given as the ratio of fibre length and time needed for the transfer of acoustic nodes across this length (expressed in km.s^{-1}). It is dependent on the internal structure of fibre arrangement and is served as a measure of fibre anisotropy. The sound speed in fibres was measured by Dynamic Modulus Tester PPMSR.

Crystallinity β represents the crystalline portion of fibre which may be evaluated using various methods. In this work the DSC-Q20 apparatus, TA Instruments was used for the evaluation of the thermal properties of PLA fibres. The non-isothermal process of analysis was performed. All samples of PLA fibres were heated by rate of 10°C.min^{-1} from 60 to 200°C under nitrogen flow. From melting endotherm of 1st heating of PLA fibres the cold crystallization enthalpy (ΔH_{cc}) and the melting enthalpy (ΔH_m) were determined. The crystallinity β of PLA was calculated according to the following equation 1:

$$\beta = \frac{\Delta H_m - \Delta H_{cc}}{\Delta H_{m,0}} \cdot 100\ \% \qquad (1)$$

where: $\Delta H_{m,0}$ is the melting enthalpy of a 100% crystalline PLA (93.6 kJ.kg^{-1}) [12].

Mechanical properties were measured using Instron 3345 equipment (USA) with a gauge length of 250 mm and clamping rate of 250 mm.min^{-1}. An average of at least 10 individual measurements was used for each fibre. The mechanical characteristics (tenacity at the break, elongation at break and Young's modulus) were determined according to EN ISO 2062 and fineness according to the STN EN ISO 2060.

3 RESULTS AND DISCUSSION

The spinning of the studied type PLA biopolymer Luminy LX175 on two 25 holes spinning nozzles at a dosage of PLA melts 34.7 g/min per nozzle was at both spinning temperatures 210°C and 220°C at the standard level. Also, the spinning processes with dosing PLA melt 22.5 g/min per nozzle were satisfactory at both temperatures without interrupting the flow of the polymer stream under the nozzle.

Table 2 Supermolecular structure parameters of PLA fibres

Sample No.	Dosing and spinning temperature	Birefringence $\Delta n.10^3$	$Vk_{\Delta n}$ [%]	Sound speed *c* [km/s]	Vk_c [%]	Crystallinity β
1	34.7 g/min 210°C	8.19	2.66	1.66	2.41	0.143
2		17.91	2.98	1.81	2.34	0.265
3		19.83	2.11	1.95	2.31	0.298
4		24.41	1.94	2.05	2.53	0.314
5	34.7 g/min 220°C	6.52	2.08	1.62	1.93	0.129
6		13.78	3.17	1.75	2.04	0.249
7		19.02	3.00	1,84	1.91	0.264
8		23.57	1.34	1.94	2.61	0.304
9	22.5 g/min 210°C	7.78	2.74	1.70	1.92	0.160
10		16.41	2.70	1.94	2.71	0.301
11		22.08	1.99	2.00	2.66	0.314
12		22.35	2.04	2.08	1.97	0.347
13	22.5 g/min 220°C	5.59	2.91	1.59	1.97	0.131
14		13.14	3.09	1.79	2.08	0.231
15		17.22	3.45	1.86	2.28	0.250
16		22.18	3.18	1.97	2.15	0.264

The drawing of the fibres to a drawing ratio of 1.4 and 1.6 was reliable, without break, at both higher and lower dosing of the melt during spinning. A drawing to the maximum drawing ratio, only samples spun at 220°C showed a standard level. The samples spun at 210°C showed deterioration, with occasional fibre break during unidirectional deformation (drawing).

First, the structure of PLA fibres prepared from biopolymer Luminy LX 175 was studied. The results of supermolecular structure parameters are listed in Table 2. The change of spinning temperature, i.e. increasing the temperature from 210°C to 220°C, affects the supermolecular structure parameters. The effect of the spinning temperature of the PLA biopolymer on the supermolecular structure parameters was compared under the same dosing of PLA melt per nozzle. It was found that increasing the temperature from 210°C to 220°C reduces all structure parameters of undrawn fibres (Table 2).

For fibres 1-8 with a melt dosage of 34.7 g/min/nozzle, the total average orientation of macromolecular chains (birefringence) decreases by 20%, while for fibres 9-16 with a melt dosage of 22.5 g/min/nozzle there is a decrease of 28%. The decrease in the orientation of macromolecular chains in surface areas (sound speed) in the spinning field at the spinning speed of 1500 m/min did not exceed 10% in both cases of PLA melt dosing during spinning. At the same time, with increasing spinning temperature, a decrease of crystallinity at 10% in fibres 1-8 with a melt dosage of 34.7 g/min/nozzle and at 18% in fibres 9-16 with a melt dosage of 22.5 g/min/nozzle was observed. As the drawing ratio increases, the parameters of the supermolecular structure increase proportionally, as we can see in Table 2. The effect of different PLA melt dosing on the parameters of the supermolecular structure at the same temperatures was not clearly evident in the fibres. Slight deviations were noted, but in most cases they did not exceed 10%.

The spinning temperature also affects the process of uniaxial deformation of the fibres - drawing. A higher maximum drawing ratio (λ_{max}=1.88) was achieved for fibres prepared at a spinning temperature of 220°C, independent of the PLA melt dosing, which is due to the higher mobility of macromolecular chains and their segments at higher temperatures.

The reduction of the PLA melt dosing from 34.7 g/min per nozzle to 22.5 g/min per nozzle was most significantly reflected in the change in overall fibre fineness, which was reduced by 33% (Figure 1). The defined parameters of super-molecular structure undrawn and drawn PLA fibres affected their mechanical properties (Figures 1b, 2a and 2b).

The decrease in crystallinity due to the higher spinning temperature (220°C, Table 2) results in an increase in the elongation of the PLA fibres compared to the fibres obtained at the spinning temperature of 210°C, compared at the same drawing ratios (Figure 1b). At the same time as the drawing ratio increases, the elongation of the fibres decreases. With a lower melt dosage of 22.5 g/min per nozzle and a higher spinning temperature of 220°C, the fibres with the highest elongation were obtained.

The tenacity of the fibres depends on several factors. The first significant effect on increasing fibre tenacity at break has a drawing ratio, as seen in Figure 1. The highest tenacity of 2.7 cN/dtex was achieved at fibre prepared at a lower spinning temperature, with lower dosing, at the maximum drawing ratio.

The second significant effect on fibre tenacity has the spinning temperature. It can be seen in Figure 2a that the tenacities at the maximum drawing ratio and 210°C are comparable and higher

compared to the tenacities at 220°C and λ_{max}, even though the higher drawing ratio was obtained with fibres prepared at higher spinning temperatures (Table 2). The different of the PLA melt dosing on the fibre tenacity at the spinning temperature of 210°C did not manifest itself. A reduction in the tenacity of more than 16% was found for fibres prepared at 220°C with a melt dosing of 22.5 g/min per nozzle, except for the fibre at a drawing ratio of 1.4.

Young's modulus increases as the drawing ratio accretion. It is related to the increment in crystallinity in the fibres with a rising drawing ratio (Table 2).

The effect of the different spinning temperatures was manifested especially at drawing ratios 1.4 and 1.6, as can be seen in Figure 2b. By increasing the spinning temperature, the Young's modulus decreased by 16% at a dosing of 34.7 g/min per nozzle and by 26% at a dosing of 22.5 g/min per nozzle.

It follows from the above that, as in the parameters of the supermolecular structure, the most significant changes occur in fibres with a dosage of 22.5 g/min per nozzle due to the spinning temperature.

The obtained values of the basic mechanical properties are in good correlation with determined values of their supermolecular structure parameters.

4 CONCLUSION

From the spinning processes (spinning speed of 1500 m/min) and drawing (drawing ratios λ=1.4, 1.6 and λ_{max}) it follows, that processes are stable, only at maximum drawing ratios some occasional fibre breaks were occurred.

The dependencies of influence of spinning temperature, dosing of PLA melt per nozzle and uniaxial deformation (drawing) to the supermolecular structure parameters and basic mechanical properties were evaluated.

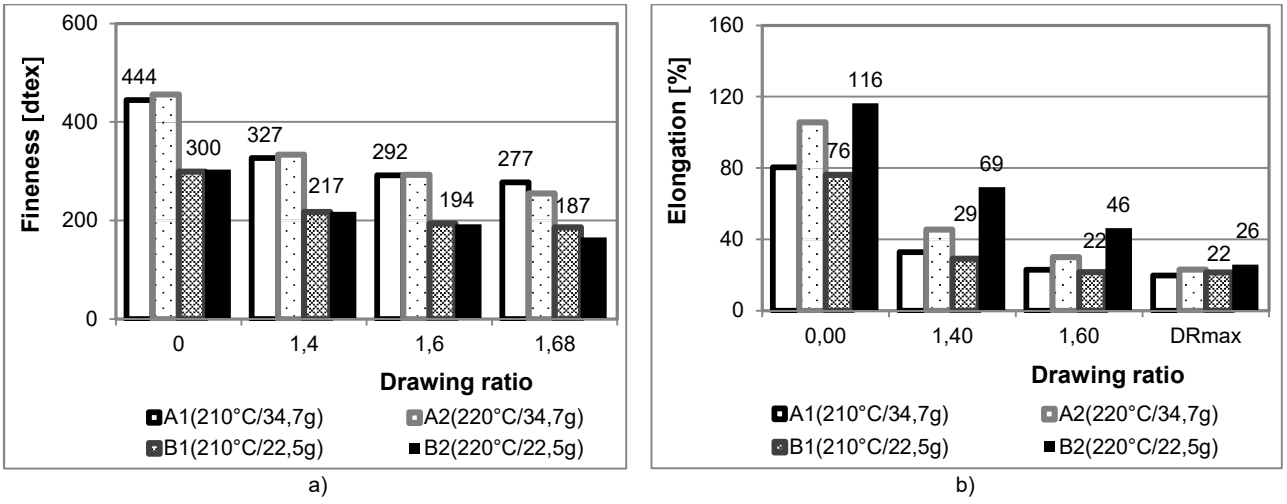

Figure 1 Dependencies of fineness and elongation at the break on drawing ratio of PLA fibres

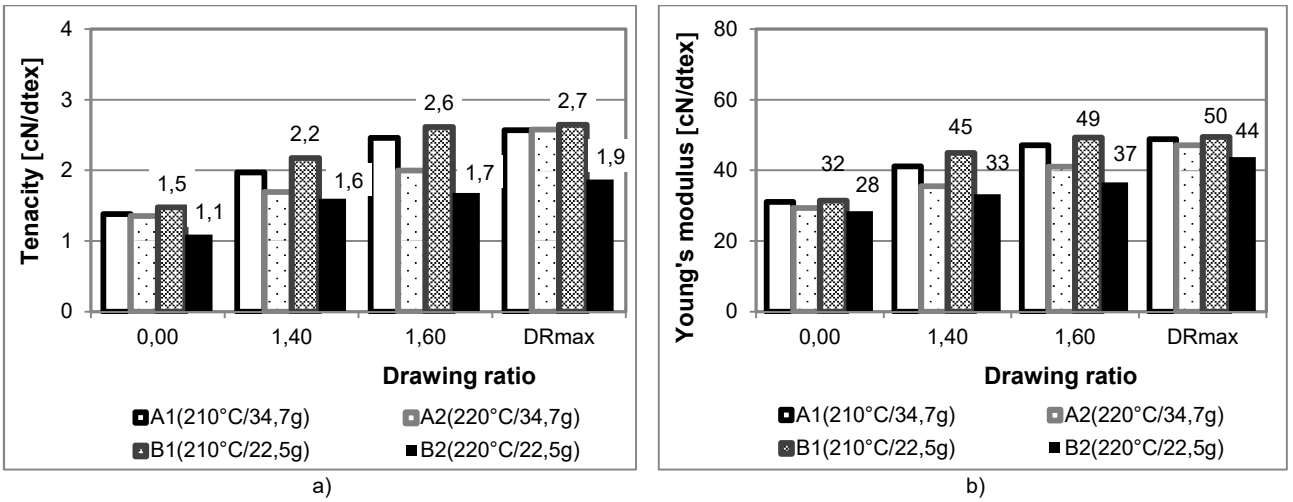

Figure 2 Dependencies of tenacity at break and Young's modulus on drawing ratio of PLA fibres

It was found that in the spinning field at spinning temperature of 220°C a lower total orientation of macromolecular chains had occurred, which resulted to lower tenacity fibres in comparison with PLA fibres at 210°C. At the same time, lower crystallinity had occurred in PLA fibres prepared at 220°C, resulting in lower Young's modulus and higher elongation of fibres. Fibres with a lower dose of 22.5 g/min/nozzle show more significant changes in the parameters of the supramolecular structure due to temperature than fibres with a higher melt dose of 34.7 g/min/nozzle, but the impact of PLA melt dosing per nozzle on the supermolecular structure and mechanical properties of fibres is not obvious. The reduction of the melt dosage from 34.7 g/min/nozzle to 22.5 g/min/nozzle had the most significant effect on the change in the overall fineness of the fibres.

It was also found that process of uniaxial deformation has significant influence on studied properties of PLA fibres. The highest maximum drawing ratio at uniaxial deformation was reached for fibres prepared at a spinning temperature of 220°C. Nevertheless, the tenacities at a spinning temperature of 210°C were comparable and higher at the maximum drawing ratio compared to the tenacities obtained at the maximum drawing ratio at 220°C. As the drawing ratio increased, all structural parameters increased. By comparing the structure of drawing fibres at the same drawing ratios (λ=1.4 and 1.6), it was found that the increase in the crystallinity due to the increase in the spinning temperature. The decrease of the orientation of macromolecules chains in the direction of fibre axis of fibre (birefringence) as well as the orientation of macromolecules chains in the surface layers of fibre (sound speed) occurs mainly by increasing the spinning temperature from 210°C to 220°C.

The tenacity of drawing fibres increases in the same order at the same drawing ratios: 220°C/22.5 g/min < 220°C/34.7 g/min < 210°C/34.7 g/min < 210°C/22.5 g/min. Reciprocally to the tenacity, the elongation of the fibres decreases.

From the achieved structural and mechanical properties of the fibres was found, that the best suitable spinning process from PLA Luminy LX175 is the spinning temperature of 210°C, dosing of PLA melt per nozzle 22.5 g/min.

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