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IMMOBILIZATION OF ESTERASE ENZYME ONTO SILICA NANOFIBERS FOR BIOMEDICAL APPLICATIONS

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Abstract: The search for new solutions to medical problems has led to a considerable development in the application of nanotechnologies to medicine. Silanization of substrates with hydroxyl ($-OH$), amino ($-NH_2$) or carboxy ($-COOH$) functional groups on the surface of nanofibers is a process currently being used in the engineering of biocompatible nanomaterials applicable to different types of wounds. This paper describes a successful procedure for immobilization of the esterase enzyme onto the surface of silica nanofibers through the silanization of surfaces with 3-aminopropyl triethoxysilane (APTES). The immobilized enzyme was detected by electron microscopy (SEM), atomic force microscopy (AFM), optical microscopy and specific histochemical reactions. The above-mentioned biomolecules were selected as a models for immobilisation another different types of enzymes, particularly proteolytic enzymes.

Keywords: nanofibers; immobilization; wound – dressing; debridement, enzymes.

1 INTRODUCTION

The current functional nanofibrous scaffolds produced by electrospinning have great potential in many biomedical applications, such as tissue engineering, wound dressing, enzyme immobilization, and drug delivery [1]. Electrospinning has attracted much attention and generated strong interest in recent years due to its versatility and great potential for application in various scientific fields. Nanoscale fibers are generated through a strong electric field applied to a polymer solution or melt. The non-woven nanofibrous mats produced in this way mimic extracellular matrix components much better than conventional techniques do. To date, more than 200 polymers have been electrospun for various applications, and the number of these is gradually increasing [2-5].

An important advantage of electrospinning is (1) the production of very thin fibers (on the order of a few tenths of a nanometer), with (2) a large surface area, and (3) easy

functionalization for various purposes. The possibility of large-scale production combined with the ease of the process makes the technique very attractive and broadly applicable. Moreover, there are more than ten well-established companies engaged in the nanofiber production today [6-7].

As regards the materials used for electrospinning, biodegradable polymers are used most frequently. The assumption is that covalent immobilization of biomolecules such as enzymes onto the nanofiber surface increases their long-term stability while decreasing their toxicity (at the lowest concentration needed *in situ* to exert biological activity). There is a wide variety of solid carriers (both natural and synthetic); these are functionalized to achieve a covalent bond between biomolecules. In general, the surface modification of a nanofiber mesh is made using various well-documented methods (plasma treatment, wet chemical methods, co-electrospinning of surface-active-agents, etc.) [8].

Chemical immobilization of bioactive molecules on the surface of nanofibers is preferred to physical immobilization, because the molecules are bound covalently to the nanofiber structure. Consequently, they are not leached from the surface of the modified nanofibers so easily, and stay bound for an extended period. It should be noted that a partial inactivation of the immobilized biomolecules can occur depending on the extent of the active *in situ* modification, as well as other factors, e.g. the structure and length of the spacer used [9].

Silanization was used to functionalize the nanofibrous surface (in order to create enough reactive groups on it) in all cases. The first step in the silanization was physisorption of alkylalkoxysilanes onto the surface, followed by rapid hydrolysis of the alkoxy group (methoxy or ethoxy groups), yielding the formation of hydroxyl and amino groups that can form a covalent bond to silanol on the nanofiber surface [10]. We decided to study the immobilization of esterase enzyme (as a model biomolecule) onto silica nanofibers made from TEOS. In the second part of our research we are going to focus on the different types of proteolytic enzymes with aim to prepare an effective type of wound-dressing that can remove necrotic tissue from the wound.

Our work is currently focused on the immobilization of proteolytic enzymes with the aim of treating difficult-to-heal wounds, particularly debridement (medical removal of dead, damaged, or infected tissue), by improving the healing potential of the remaining healthy tissue.

2 MATERIALS AND METHODS

2.1 Materials

Nanofibers were prepared from:

- TEOS (tetraethyl orthosilicate, 98 wt.%, Sigma-Aldrich),
- propan-2-ol (p.a. 99.9 wt.%, Penta CZ),
- distilled water,
- hydrochloric acid (2 mol/L, Sigma-Aldrich-Fluka).

We used:

- APTES (3-aminopropyl triethoxysilane, 98 wt.%, Sigma-Aldrich),
- propan-2-ol (p.a. 99.9 wt.%, Penta CZ),
- distilled water,
- acetic acid (p.a. 100 wt.%, Merck),
- glutaraldehyde solution grade II (25 wt.% in water, Sigma-Aldrich), and
- esterase from a porcine liver (≥ 15 units/mg solid, Sigma-Aldrich) to immobilize biomolecules on the silica nanofibers.

2.2 Synthesis of spinning solution

TEOS (300 mL) and propan-2-ol (330 mL) were added into an Erlenmeyer flask and mixed together for 15 minutes at ambient temperature. Subsequently, a solution of propan-2-ol (200 mL), 2M HCl (8 mL) and distilled water (60 mL) was prepared and added into the mixture under continuous stirring. In the next step, the solution was gently stirred at 200 rpm/min until the mixture was cooled back to ambient temperature (after 50 minutes). Then, the solution was stirred at 300 rpm/min, refluxed and heated in an oil bath (temperature 200°C) for 2 hours. Finally, the 400 mL of propan-2-ol was distilled. After distillation, the spinning solution was quickly cooled and stored in a 500 ml PE bottle at laboratory conditions [11, 12].

2.3 Preparation of pure silica nanofibers by electrospinning method

Silica nanofibers were electrospun from free liquid surface on the needless Nanospider™ device (Elmarco). The spinning solution was prepared by sol-gel method stated above (chap. 2.2). Electrospinning was run under standardized conditions (electrode distance 140 mm, string speed 0.2 mm/s, head speed 350 mm/s in 500 mm, tow 90/100 m³/h, voltage 57 kV, air humidity 33 %, laboratory temperature 23°C). A baking paper without silicone was used as a substrate for the silica nanofibers prepared. In this way, a silica nanofibrous sheet of specific weight (32 g.m⁻²) and mean fiber diameter (approximately 200-900 nm) was obtained. Subsequent thermal stabilization of the silica nanofibers was performed under 180°C for 2 hours [11, 12].

2.4 Functionalization of the pure silica nanofibers through APTES

We used silica nanofibers prepared from TEOS in the previously mentioned method (chap. 2.3.). For silanization reactions of pure silica nanofibers we applied the following procedure: 2 mL of APTES, 45 mL of propan-2-ol in dry conditions. Nanofibrous samples were immersed into the prepared solution at ambient temperature for 1 hour. The functionalized silica nanofibers were then rinsed twice with distilled water in order to remove the last traces of reactants, finally were dried in the oven at 40°C for 3 hours [11, 12].

2.5 Immobilization of the enzyme on functionalized silica nanofibers through glutaraldehyde

Functionalized silica nanofibers (0.5 g) subsequently reacted with the solution of glutaraldehyde (GA) for immediate immobilization of the enzyme. 4 mL of glutaraldehyde grade II (25 % solution, Sigma-Aldrich) and 26 mL of phosphate buffer (pH 7.2) were mixed together. Afterwards, the functionalized nanofibrous samples were immersed in the above-prepared solution at ambient temperature for 10 minutes then rinsed with 10 mL of the same buffer. Esterase (0.1 mg) was dissolved in 1 mL of a phosphate buffer (pH 7.2) for the immobilization. The weight of the functionalized silica nanofibers used (size 30 x 30 mm) was approx. 0.5 g. The samples were incubated in the esterase solution for 5 minutes, then rinsed with the phosphate buffer and dried at ambient temperature [13].

2.6 Electron microscopy - SEM

Images were taken with the Scanning Electron Microscope Carl Zeiss ULTRA Plus. The transparent samples (magnified 50000x) were gold-dusted in advance (to lead the charge out) and observed (through the In-Lens detector) in the form of secondary electrons SE1.

A measurement of nanofiber diameters was made with the SEM images (the number of randomly selected nanofibers n = 500 with

the 3 SEM images), using the SW NIS-Elements AR (Laboratory Imaging), and processed statistically through the program QCExpert (TriloByte Statistical Software, Pardubice, Czech Republic).

2.7 Atomic force microscopy – AFM

Atomic force microscope NanoWizard III from JPK was used for scanning of area 3D surface. The scan was performed in the non-contact mode. The scanning area was 2.3 x 2.3 µm. The sample was prepared with the use of a special reversible tape and put to the AFM. For data processing of the scan the JPK DataProcessing was used.

2.8 Optical microscopy

Images from the optical microscope were taken using the optical microscope Carl Zeiss Axio Imager M2. Epiplan NEOFLUAR 50x optical aperture 0.8 HD DIC with EC lens was used, the magnification was 500x. The images were processed using the software Axio Vision 4.8.2. The images from the optical microscope were used to verify histochemical dyeing of the esterase attachment on the nanofibers.

2.9 Quantification of the enzyme immobilized

Quantification of the attached enzyme was performed through the reaction with α-naphtyl acetate ($\geq 98\%$, Sigma-Aldrich) and o-dianisidine (95 %, Sigma-Aldrich). Subsequently, 1.8 mg of α-naphtyl acetate was weighed and dissolved in 6 mL of phosphate buffer (pH 7.2), and then 30 mg of o-dianisidine in 30 mL of phosphate buffer (pH 7.2) was added. The reaction of the enzyme proceeded upon insertion of the sample into the solution. The reaction was characterized by a color change of the sample, from white to colorless (pink), at the sites to which the enzyme was bound.

3 RESULTS AND DISCUSSION

3.1 Functionalization of nanofibers

For functionalization of the pure silica nanofibers and creation of covalently bound amino groups, we used APTES. The whole process of immobilization (from APTES to esterase enzymes onto silica nanofibers) is shown schematically in Figure 1.

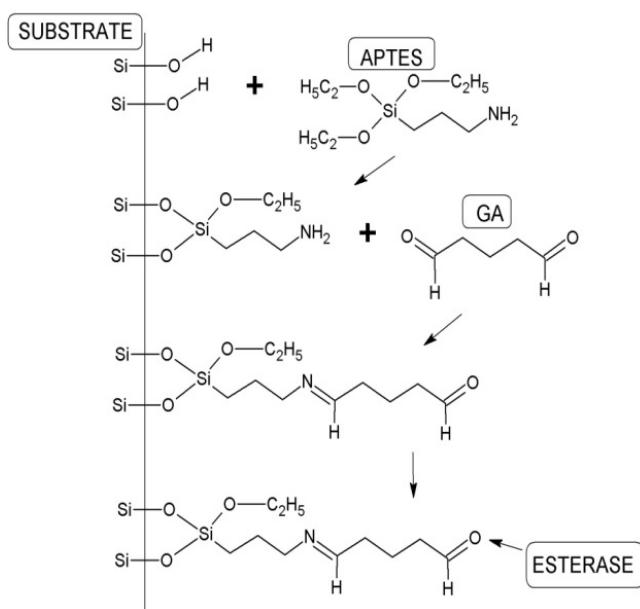


Figure 1 The scheme of the enzyme immobilization on the silica nanofibers

3.2 Measuring properties of nanofibers

Selected images of the SEM samples of the silica nanofibers with and without the immobilized esterase enzyme are shown in Figures 2a and 2b. In the case of the immobilization of the esterase enzyme, the surface of the silica nanofibers changed and was no longer smooth (see Figure 2b at a magnification of 50000x) – there were clearly visible structures with the size of 10-40 nm, covering continuously the whole surface of the silica nanofibers. In the case of non-modified silica nanofibers (Figure 2a), we can clearly see a smooth surface without any artefacts. The nanofibers have different thickness in both cases (Figures 2a, 2b).

The AFM NanoWizard III from JPK, was used in non-contact mode, the data were further processed using the JPK DataProcessing software. Cantilever Nanosensors (CPPP-NCHAuD-30) was used for measuring. Three different display modes were used (Figures 3a-f): mode “error trace”, mode “height measured trace 3D” and mode “phase trace”.

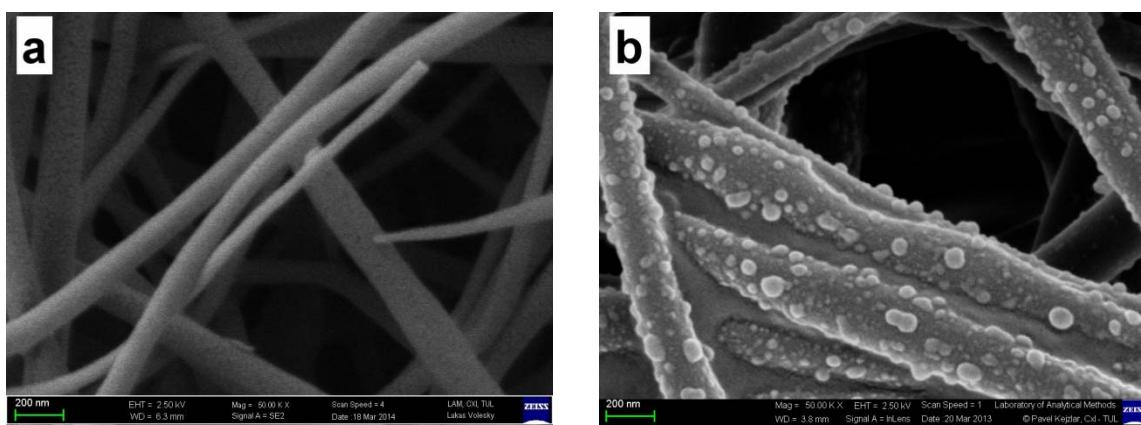


Figure 2 SEM images of functionalized silica nanofibers without immobilized enzyme (a) and silica nanofibers with the immobilized esterase enzyme dusted with gold (b)

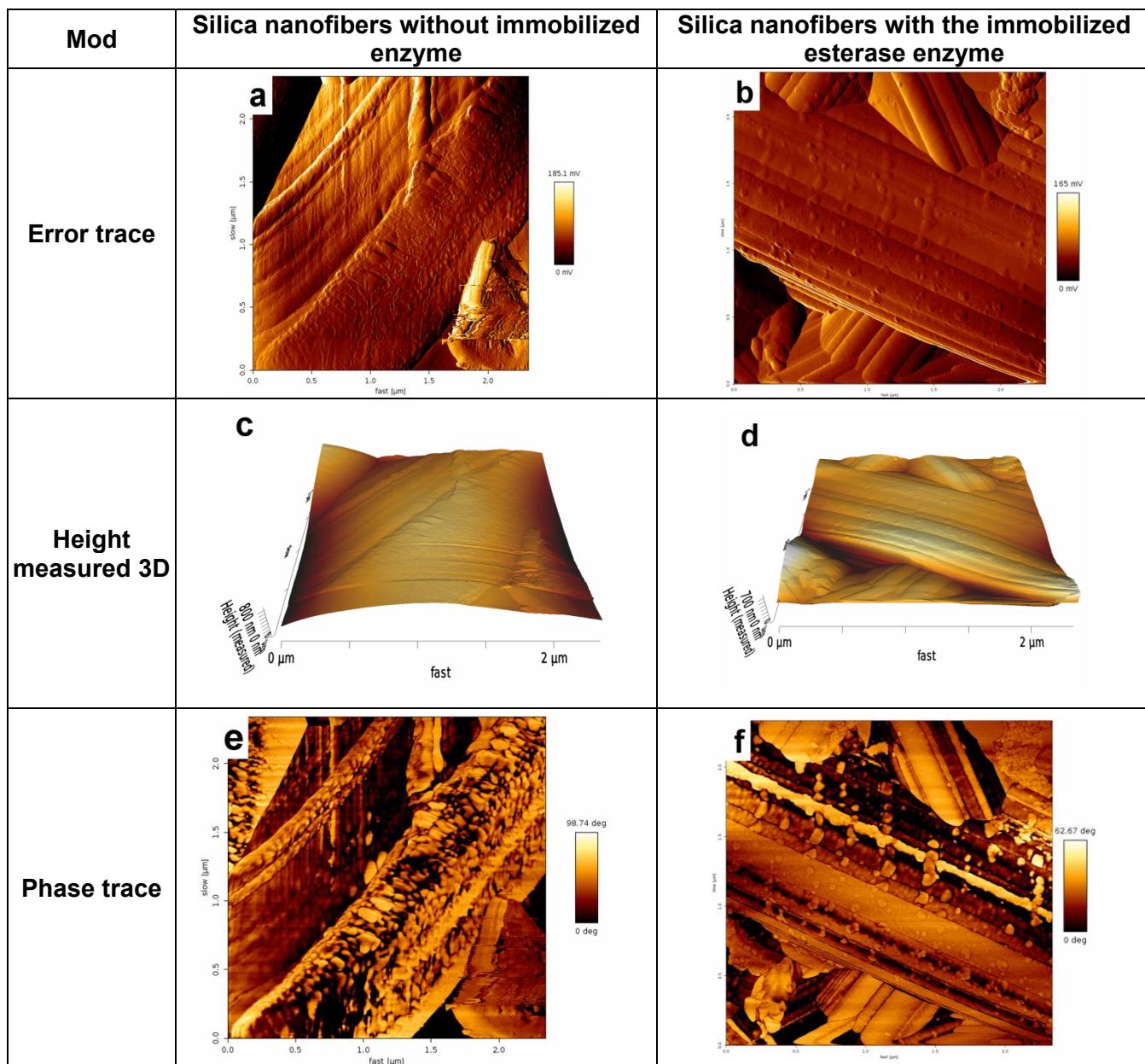


Figure 3 AFM results of the functionalized silica nanofibers without enzyme and with immobilized enzymes using three different display modes

The basic analysis of the AFM images was carried out in the programme JPK DataProcessing. The size of the bound esterase enzyme in nm was measured (Figure 4), also the distance between two neighboring molecules of the esterase bound on the surface of silica nanofibers (Figure 5) was measured. In Figures 4 and 5, we can see the profile axis marked in height measured mode (the marked area of measurements and obtained values). Figure 4

clearly confirms that the size of the bound enzyme is about 10 nm. The size of an immobilized enzyme depends on the cluster of esterase molecules and ranges from 10 nm to 40 nm. When we used the mode „height measured“, the molecular size of an esterase was 10 nm. In contrast, Figure 5 shows the distance between two neighboring molecules of bound esterase, which is about 280-290 nm.

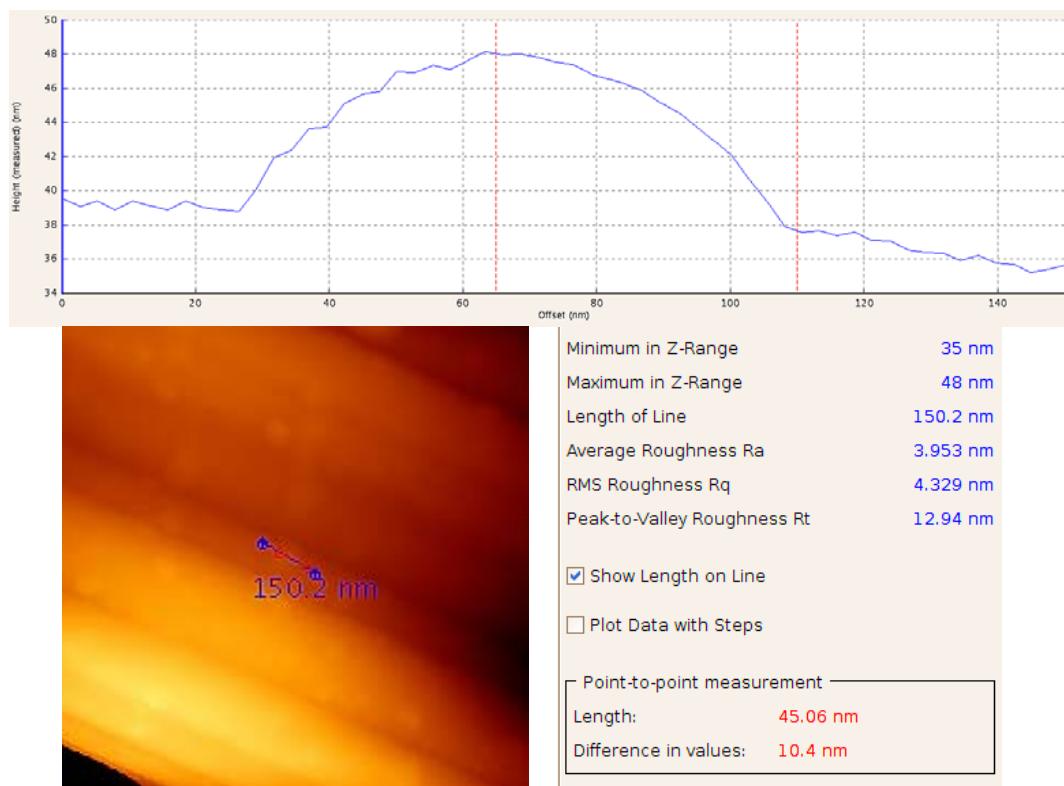


Figure 4 Size of the bound enzymes using AFM methods (scanning area 1.5 x1.5 μm)

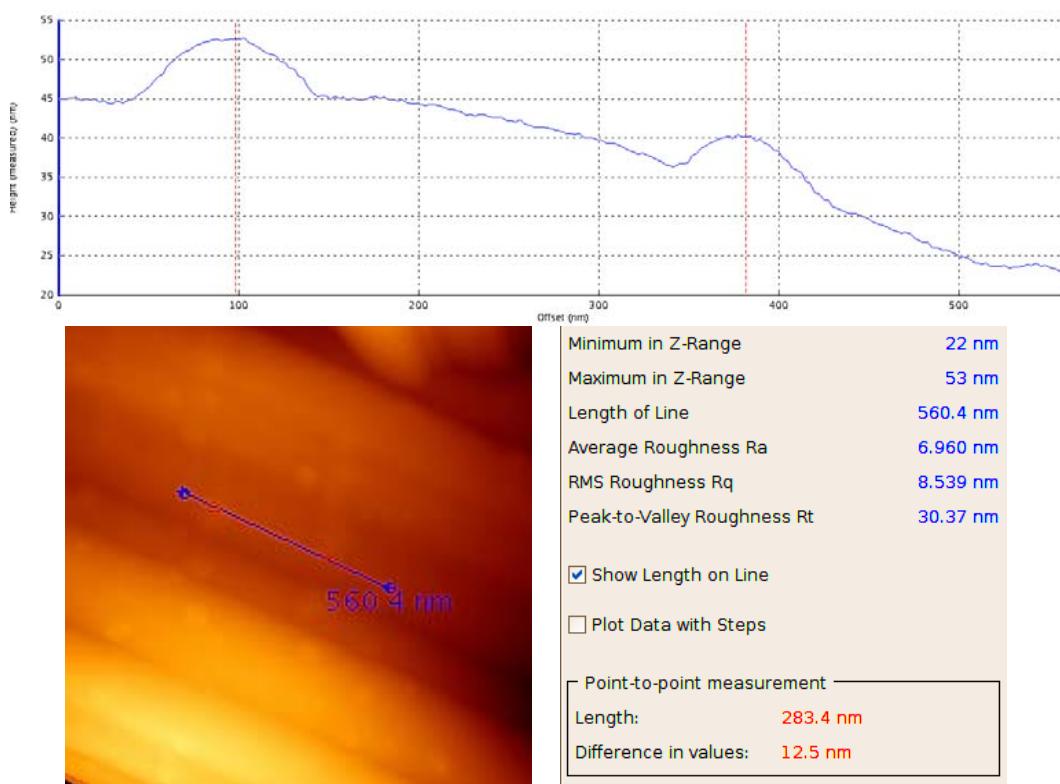


Figure 5 Analysis of the AFM image – distance between 2 molecules of bounded enzymes (scanning area 1.5 x1.5 μm)

3.3 Verification of enzyme immobilization

The enzyme immobilization was verified by the regularity and the intensity of the color reactions with o-dianisidine. Successful immobilization of enzymes was confirmed in all the prepared samples. The immobilized enzymes were observed with a scanning electron microscope (see Figure 2b), AFM (see Figures 3b, d, f, 4 and 5) and an optical microscope (see Figure 6). In the case of silica nanofibers, the histochemical reaction was quite evident. The SEM and optical microscopy observations also confirmed the formation of bigger enzyme units on the surface of every single fiber. The observed enzyme units are shown in Figure 6. The dimension of enzyme units led to an increase in the diameter of nanofibers. The diameter of the silica nanofibers increased by 44.8 % (increase in percentage of the mean).

These results implied that the silica nanofibers contained enough (-OH) groups for immobilization. Figure 6 was taken with the optical microscope Carl Zeiss Axio Imager M2, EC Epiplan NEOFLUAR lens 50x, 1.4 megapixel CCD camera AxioCam ICC1, optical aperture 0.8 HD DIC, mode NG 100.

3.4 Silica nanofibers with immobilized enzymes

Enzymes (particularly proteolytic) immobilized onto the nanofibrous substrate can be used

mainly in wound-treatment (e.g. after a debridement). A big advantage of proteolytic enzyme immobilization onto silica nanofibers is direct contact *in situ* with necrotizing tissues.

Silica nanofibers can copy the wound precisely – preventing the air bubble formation. They are dissolved slowly, and with their dissolution they release molecules in the form of silica. The immobilized enzymes are released steadily with the dissolution of the silica nanofibers (complete degradation of silica nanofibers ends within 10 days). Compared with current wound-dressings, the tested nanofibers offer some significant advantages: (1) low price and (2) efficacy of treatment after debridement. It is a patient-friendly treatment since the method is painless and therefore suitable for burn marks and difficult-to-heal necrotic wounds, where the removal of necrosis is the primary step in treating the wound. Silica nanofibers are an excellent carrier – quite inert towards the human organism. Some big advantages of the carrier include: its (1) excellent wetting behavior, (2) biodegradability, (3) non-cytotoxicity (nanofibers do not contain any residues of dissolving agents or other residues), (4) easy handling, (5) stability in the given environment and (6) low production costs.

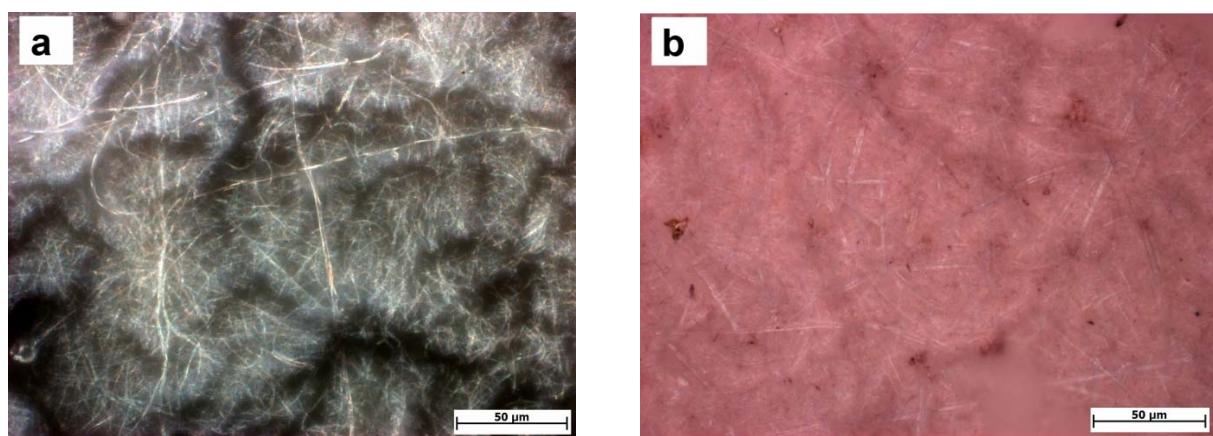


Figure 6 Histochemical reaction – functionalized silica nanofibers without immobilized enzyme (a), the proof of esterase enzyme immobilization on the silica nanofibers - small dark red spots (b). Magnification 500x

4 CONCLUSION

Functionalized silica nanofibers were prepared and examined with a focus on the immobilization of esterase enzyme. As a model biomolecule the esterase enzyme, which belongs to hydrolase enzymes, was used. Although the esterase enzyme immobilized, the surface character of the silica nanofibers with the immobilized esterase was ragged and the conglomerates of enzymes were from 10 to 40 nm. The successful immobilization of esterase enzyme onto tested silica nanofibers was confirmed through SEM, AFM, optical microscopes and histochemical reactions via o-diasidine.

As another biomolecules are currently tested proteolytic enzymes from group animal, plant and bacterial, the whole results will be published later. Mentioned proteolytic enzymes are selected with the aim of fast and effective treating difficult-to-heal wounds particularly after debridement.

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IMOBILIZACE ENZYMU ESTERÁZA NA KŘEMIČITÁ NANOVLÁKNA PRO BIOMEDICINSKÉ APLIKACE

Translation of the article

Immobilization of esterase enzyme onto silica nanofibers for biomedical applications

Nanovlákna z oxidu křemičitého (vyrobená z TEOS) po funkcionálizaci s APTES (zavedení -NH₂ funkční skupiny) a glutaraldehydu byla následně využita pro imobilizaci enzymů, konkrétně esterázy. Uvedený enzym patřící mezi hydrolázy byl vybrán jako modelová biomolekula. Imobilizovaná esteráza kopíruje povrch křemičitých modifikovaných nanovláken, místy na nich vytváří konglomeráty velikosti 10 až 40 nm. Úspěšná imobilizace esterázy na testovaných křemičitých nanovlákních byla potvrzena pomocí SEM, AFM, optického mikroskopu a histochemické reakce prostřednictvím barviva o-diasidine. Dalším cílem naší práce je imobilizace proteolytických enzymů na křemičitá nanovlákna s cílem rychlého, efektivního a bezbolestného odstraňování debridmentu.

2D AND 3D SOFTWARE FOR THE DESIGN OF CLOTHING PRODUCTS

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Abstract: This article focuses on the options and uses of 2D and 3D design programs in the technical preparation for production when creating clothing model designs, creating technical documentation and presenting fashion collections. The subjects of the solution are examples of graphic processing for use in textile and clothing design of products connected with the presentation of possible outputs and procedures when presenting a company.

Key words: textile design, clothing design, colorways, Modular system, draping, V-stitcher, dress on a 3D virtual model

1 INTRODUCTION

We can find the term "design" in all areas of product development, thus in the development of the product's appearance too. It is considered a link between art and technology. It includes the visual appearance and the shape of ordinary objects and also machines, interiors, furniture, home accessories, packaging technology and packaging processing of products, fonts, posters, advertisement, etc., design of the environment and fashion design [1].

Design is connected with graphics that have great power to immediately express something for which a plethora of words would be needed. It can immediately stimulate interest that would not be possible otherwise.

Graphic design is based on the knowledge of the relationships between the image and font styles of a period, the social group, the function and communicated meaning; on the knowledge of the rules of legibility; on the knowledge of the emotive effect of shapes and colours on the human psyche; on the knowledge of the social mechanisms and communication conventions. It significantly differs from free creation [2].

The graphic designer's sphere of activity used to mainly be based on paper. Today,

when communication takes place in computer monitors and other audiovisual media, the creation of computer programs, ways of social behaviour and information technologies are included in the graphic designer's tasks [3].

2 TEXTILE AND CLOTHING DESIGN

When designing, the designer must visualise a certain need and then try to think what would fulfil it. Such a need can be defined in several ways: it can be a functional need, an aesthetic need, the social or psychological need or, as is most common nowadays, an economic imperative from the producer [4].

The development and design of new textile materials plays an important role in the sphere of fashion design because the design of clothing is also subject to their qualitative characteristics.

Textile design represents the overall "appearance of a pattern" on a textile and textile structures. Each pattern accentuates and emphasizes the content and purpose of the material.

Clothing design characterises the overall "artistic impression" of clothing, which corresponds to current fashion requirements. It includes the clothing cuts

worked-out in detail and uses the kinds of materials in the latest colours and patterns. Permanent and fast change, especially in women's fashion, has brought about a significant shift in the textile industry and design, which is the basis for textile and particularly design creation.

In connection with 'design', the term 'virtual design' was introduced; it is closely connected with computer technology and 2D and 3D software that allow the development of imaginative designs and the quick and easy development and modification of original designs of textiles, clothing and their accessories, film and theatre costumes and advertising.

3 2D AND 3D SOFTWARE FOR CLOTHING PRODUCT DESIGN

Initially, Design Software allowed only the creation of 2D drawings – designs, technical drawings, etc. Development in this field allowed the creation of designs using 3D simulation, which provides a realistic view of the created product.

Mass customisation, as paradigm in clothing production, combines the advantage of both mass production and customized production using 2D and 3D software very usefully [5].

All the currently offered Design Software of individual production fields have much in common and you can achieve the required result by using practically any of them. Only individual procedures and their details differ. Software for clothing design is offered by CAD system producers, especially Lectra Systèmes, Koppermann Computersysteme GmbH, Gerber Technology Inc., and others that are, as well as a significant part of pre-production, detailed in Table 1. These are the latest programs that allow the designers in the textile and clothing industries to work more productively and effectively and to provide feedback and cooperate with customers all around the world.

The programs of Techline by Macenauer, then Office Word by Microsoft, but also Corel DRAW and Illustrator can be used for graphic work in the pre-production phase.

Table 1 Software products for textile and clothing design

Name of the system	Lectra Systems		Koppermann Computersysteme GmbH		Gerber Technology Inc.	
Textile design software	KALEDO	Kaledo Knit	TEX-DESIGN™	TEX-LINE™	Artworks Studio Standart Edition or Professional or Expert	ColorBook
		Kaledo Weave		TEX-KNIT™		Colorways
		Kaledo Suite		TEX-CHECK™		Palete
		Kaledo Collection		TEX-DRESS™		Draping
Fashion design software					Fashion Studio 2000	Color Reduction and Cleaning
						Design and Repead
						Easy Coloring
						Easy Knit
						Easy Weave
						Staryboard and Kataloguing

4 EXPERIMENT

The aim of the experiment is to create a design for a clothing product for contracts using the design software of Artworks Studio Standart Edition, Fashion Studio 2000 and AccuMark by Gerber Technology and software V-Stitcher by Browzwear using the virtual presentation functions in 2D and 3D. Artworks Studio interfaces with Adobe Photoshop, especially in the first phase, i.e. the design of the textile pattern, and V-Stitcher interfaces with AccuMark system. The following outputs can be achieved by using these design programs and other graphics programs.

4.1 Design of pattern and colours of a textile

The pattern of an existing textile can be computerised using a scanner or camera. An alternative is to create a new pattern from scratch using a tablet, as in Figure 1, and tools of the programs adjusted for that purpose.

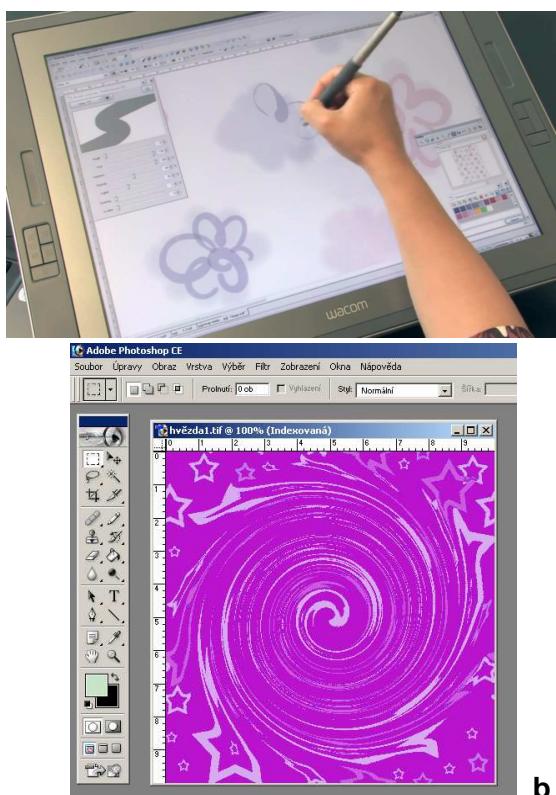


Figure 1 Creating design of textile pattern a) using tablet, b) in Photoshop program

The interaction of Photoshop with Artwoks allows the pattern to be repeated, checked for continuity and enables colour reduction of the designed textile to the required minimum. The task of repeating the design and pattern depends on creating a fluid continuation of the pattern in its repeated print on the textile. Fashion Studio 2000 also allows the creation of a design, or the modification of weave or knitting repeat with the choice of colours of threads and loops and simulation of weaving and knitting.

When designing textiles, you can work with a colour library in a part of the ColorBook program (RGB, HSV, CMYCK) and create Pallets of colours for the given fashion season and patterned collections of clothing products. The exact number of colours and shades can be determined in ColorBook or by storing scanned textile patterns to the Pallet and determining the numbers and shades of colours in the pallet.

New colour designs can quickly and easily be created from a patterned textile design using Corways program, see Figure 2, by:

- combining the colours included in the textile pattern,
- choosing new colours of the textile pattern using the colour library or pallet.

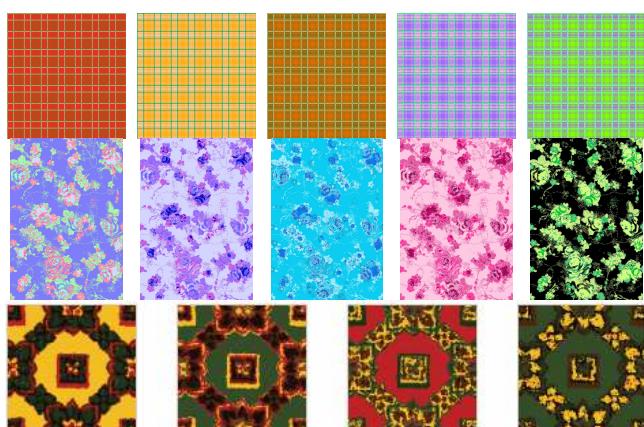


Figure 2 Models and repeats of textile patterns with a change of colour design in Colorways

Designs created either way allow the creation of material catalogues that are offered during the presentation of the clothing model samples at contracts. In the presented

patterned materials, the scale of the pattern can be changed in a design program. In accordance with such created material designs, textiles can be produced anywhere in the world.

The catalogues of materials and presentations of materials can be offered in electronic form too. Their printed version can be used in magazines, books, etc. An example of the presentation of materials is shown in Figure 3.

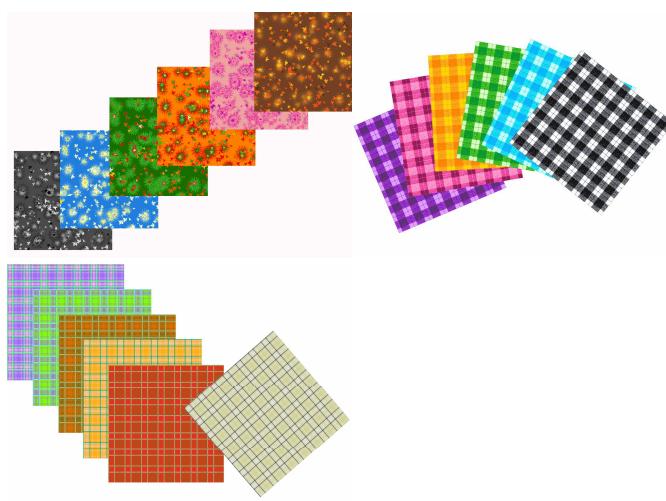


Figure 3 Changes of textile designs in Artworks Studio-Colorways and possible presentations

4.2 Design of a clothing model

Design – a sketch of a clothing model is a drawing of the product on a figure showing details, accessories and decorations. It is influenced by the current fashion trends, created in A4 format and equipped with a sample of the material from which it will be made.

Designers with specialized, aesthetic and technical skills design the model and prepare the collection for market. When preparing the market collection, the designer's work is based on fashion trends for the given season (spring - summer, autumn - winter), or on previous models that were successful. Clothing models can also be created using special design software. Models created by manual sketching or from a photograph can be computerised using a scanner or camera. They can also be created from scratch using a tablet, as in Figure 4a, or in Photoshop,

Figure 4b, etc. and subsequently modified interactively using the program tools.

Presentations and catalogues of product collections for a given season and for contracts can be made from such model designs, or they can be used for fashion magazines, books and company's web pages.

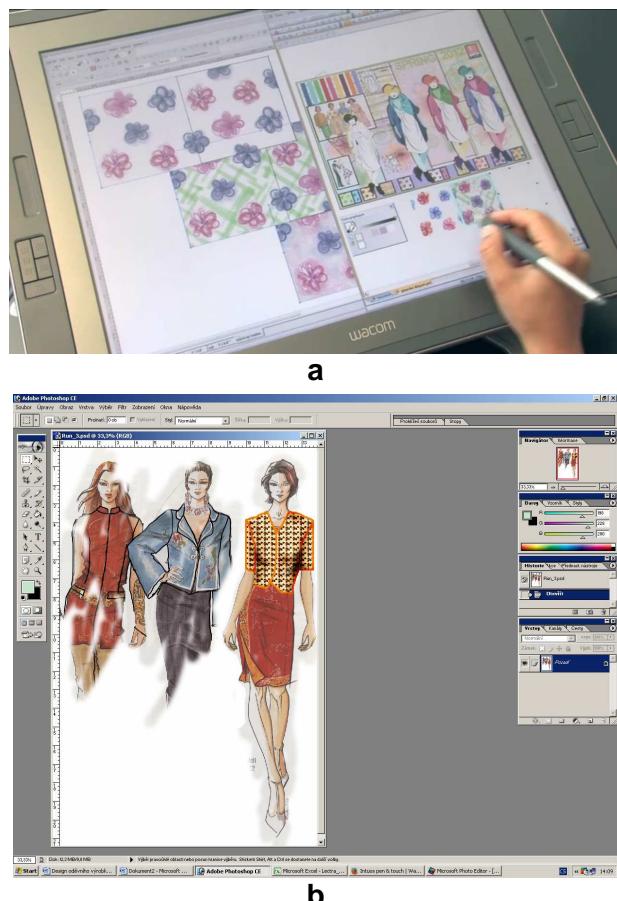


Figure 4 Designing clothing models a) with a tablet, b) in Photoshop

4.3 Creation of technical drawing

A technical drawing depicts a clothing product from front and back views, or it can also be a detail of a certain part. It is made on a separate sheet with the style number in accordance with producer's standard practice. It can be supplemented with technical data and figures that define the dimensions and measuring points on the clothing. A 'technical description', which briefly describes the product and gives details that are not evident from the technical

drawing at first sight, is a part of the technical drawing. The technical drawing is prepared by the department of technical preparation of production. It is used in all stages of production and sale.

Currently, a technical drawing can be made using the program tools of Techline, Word, Corel DRAW, Illustrator, Fashion Studio, etc. The implementation of a suitable development program into the area of

technical documentation creation is the priority when creating a modular system as in Figure 5, which has to make the creation of technical drawings easier.

An example of a technical drawing, including a description of technical operation details and cuts in marked points using MS-Word is shown in Figure 6.

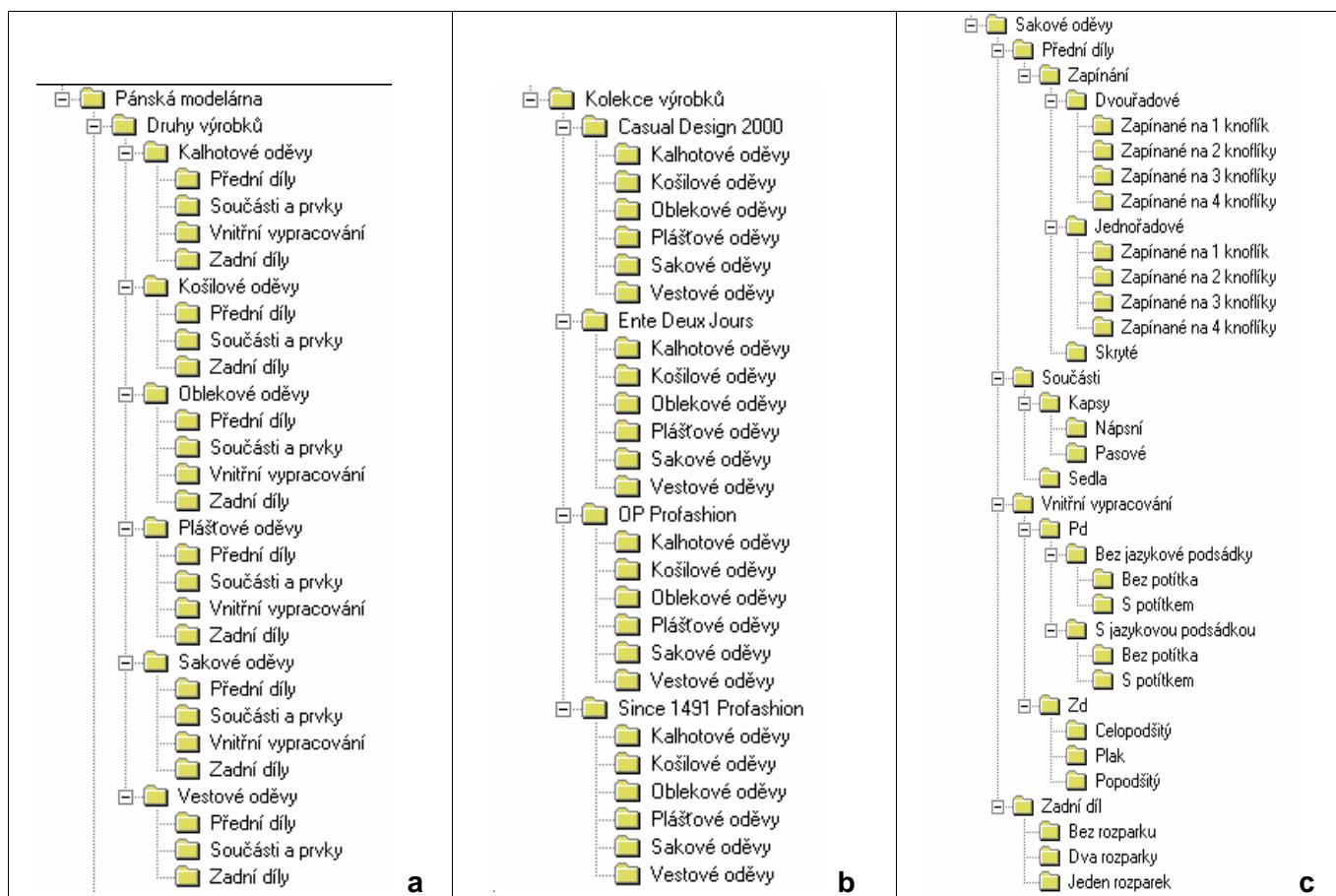


Figure 5 Modular system – a) Kinds of processed products, b) Product collection, c) Standard solution with respect to clothing types and their equipment [6]



a

BACK PART lining

35. Fold the back turn-up crease and sew the boundary seam of the back part facing together with the back part lining.

36. Iron the seam turn-ups and sew through. Sharp sew the hanging tag.

Drawing of the operation	Cut B-B	Seam
		 Dorsal

Figure 6 a) Technical drawing of the clothing product; b) Drawing of the operations; Cut B-B [7]

4.4 3D simulation of clothing onto a virtual figure

The significance of 3D simulation is in the fact that new textures can be simulated in a 2D

image of the object using an original photograph (of clothing, fashion accessories, parts of interior or domestic objects, etc.) and the specific effects, i.e. shading and gathering

of the material can be maintained. This is based on a special image of an object that is dissected into segments and created using masks and grids. The specific effect is achieved using these masks and grids and by suitable combination and modifications of textures. A wide range of modifications, colour combinations and styles of the products can be achieved using textures.

A condition for creating 3D simulation is a photograph which depicts the object in light colours, preferably white, without any pattern. This is because of the pattern not being marked on the surface of the object's newly created texture.

Examples of 3D simulations on a clothing product model and the individual steps are characteristic for software from Gerber Technology in Artworks Studio - module Draping. Draping is created in Drape, where

a mask and grid are created, and Texture, where the textiles from which the model has to be made are defined.

The mask precisely copies the silhouette of a given part of the model, see Figure 7a. The perimeter of the grid is always larger than the mask and it is created for the same parts of the clothing as the masks, Figure 7b. In the grids, the definition of 4 points on the perimmetter and the selection of grid density are important. In the next step, the mask and grid of each part on the clothing are connected using Assign Surface, as in Figure 7c. Particular textiles that have to be simulated in individual parts of the clothing model are defined in the Texture part. Also here assigning of Textures to relevant parts of the grid using Assign Texture is shown in Figure 7d.

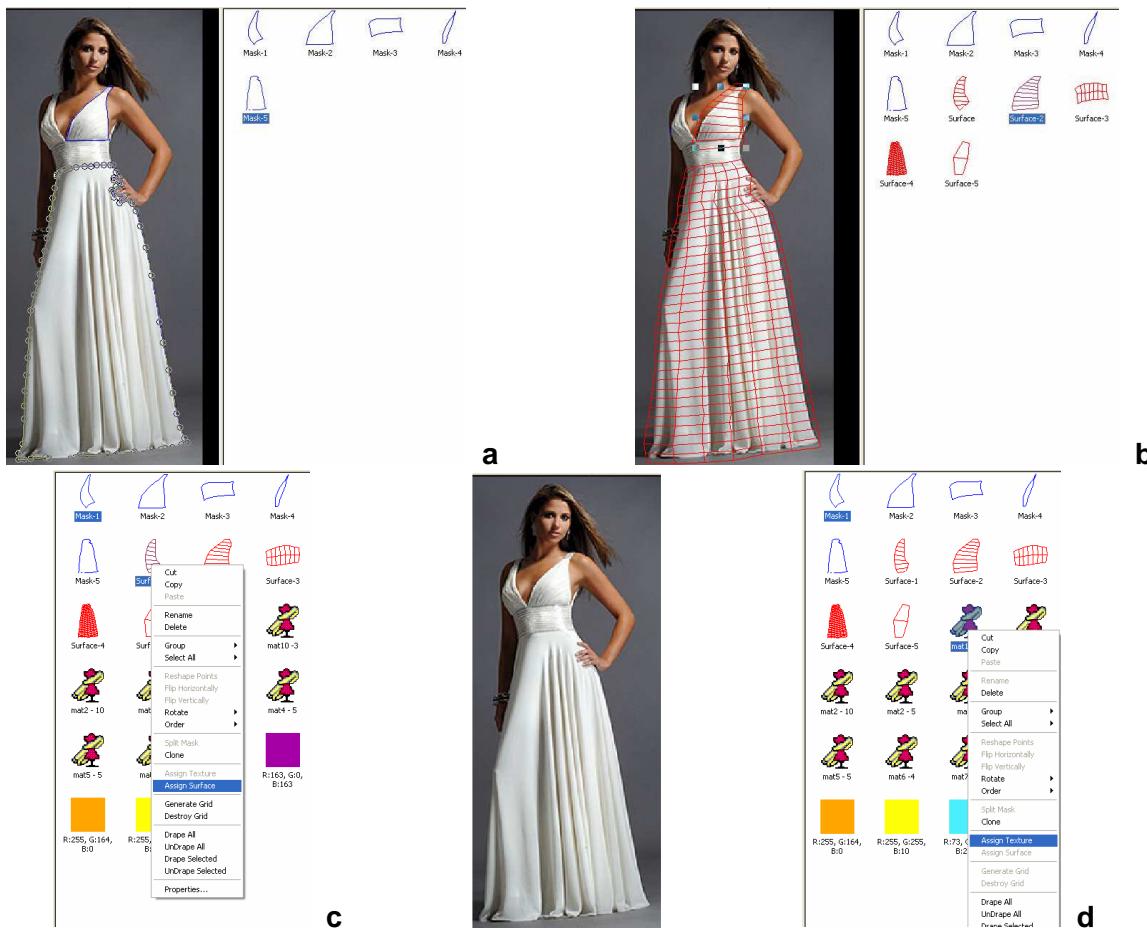


Figure 7 a) Creation of mask, b) grids for 3D simulation; c) assignment of the mask and grid, d) Assignment of the mask and texture of a selected part of the clothing

A 3D simulation depicting the texture on the clothing model is defined using the Drape all function. The final model including the texture is depicted in Figure 8a. The path of creating draping for the first to the nth model of the clothing is displayed in the tree in the left part of Draping Figure 8b. The difference is always in the combinations of assigned grids and textures depending on segmenting. The set of clothing models when changing the texture is shown in Figure 9.

Based on the above mentioned procedures using relevant software, collections of clothing models, accessories and textile materials can be created for certain periods for contracts, which can be presented in

electronic form or in printed form in catalogues.

4.5 V-Stitcher

V-Stitcher is a program which is based on 2D templates forming the clothing model and which allows 3D visualization of clothing and assessment of the clothing fitting on a virtual figure of a man, woman, boy or girl, whose dimensions can be modified. It allows textiles to be simulated in clothing based on actual parameters such as, for example: type and construction of textile, square density, thickness and friction, flexural rigidity, shear rigidity, expansion and shrinkage.

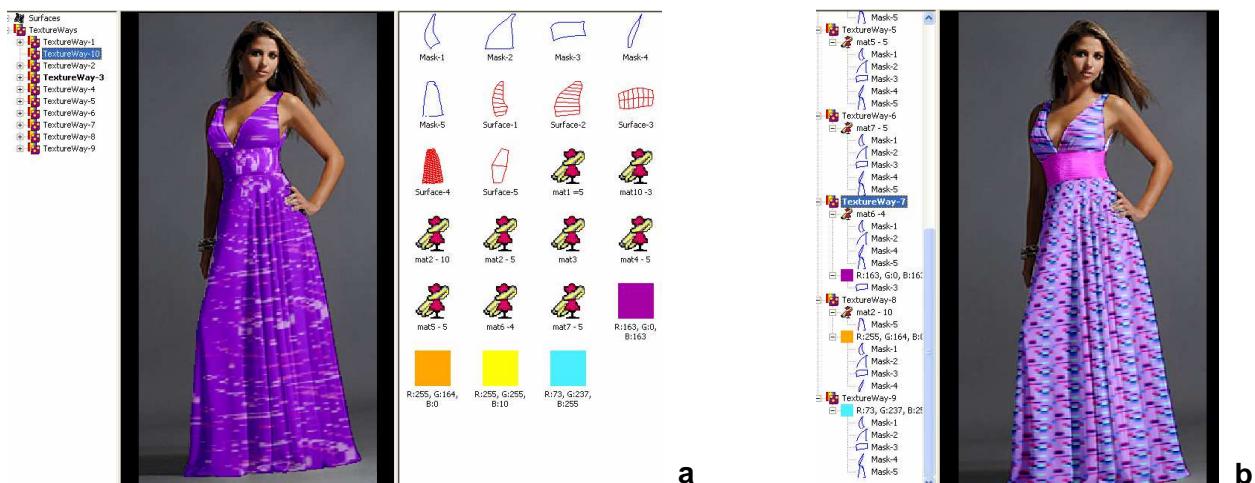


Figure 8 a) Imaging of a new texture on clothing model; b) Change and combination of textures for the first to nth clothing model



Figure 9 3D simulation by the change of texture in Artworks Studio – Draping

The procedure for virtual depiction of the clothing created in 2D and its transfer to 3D imaging needs:

- the creation of a database of the clothing source,
- the creation of the size range, i.e. labelling the sizes and definition of dimensions.
- the import of the cut parts from the AccuMark system, see Figure 10a, or from another CAD system in .dxf format.
- the definition of the figure – setting the size of the figure, modification of dimensions, as in Figure 10b.
- the grouping of the cut parts on a figure, the selection of the type of clothing parts to be wrapped on a figure and the definition of the position of the point of equilibrium for simulation Figure 11.
- the sewing of the cut parts together with no need to define seam turn-ups, see Figure 12,
- the allocation of textile materials to individual parts of the clothing, see Table 2.

Table 2 Properties of the textile Default

Property	Unit	Weft	Warp
Square density	g/m ²	300	
Friction	-	0.2	
Thickness	mm	1.0	
Flexural rigidity	dyn*cm	100	100
Elastic hysteresis	deg/cm	0	0
Expansion	N/m	500	500
Linearity of expansion	%	50	50
Shear rigidity	N/m	50	
Linearity of shear rigidity	%	50	
Shrinkage	%	0	0

*Note: deg - degree,
dyn – unit of force,
1N = 100 000 dyn

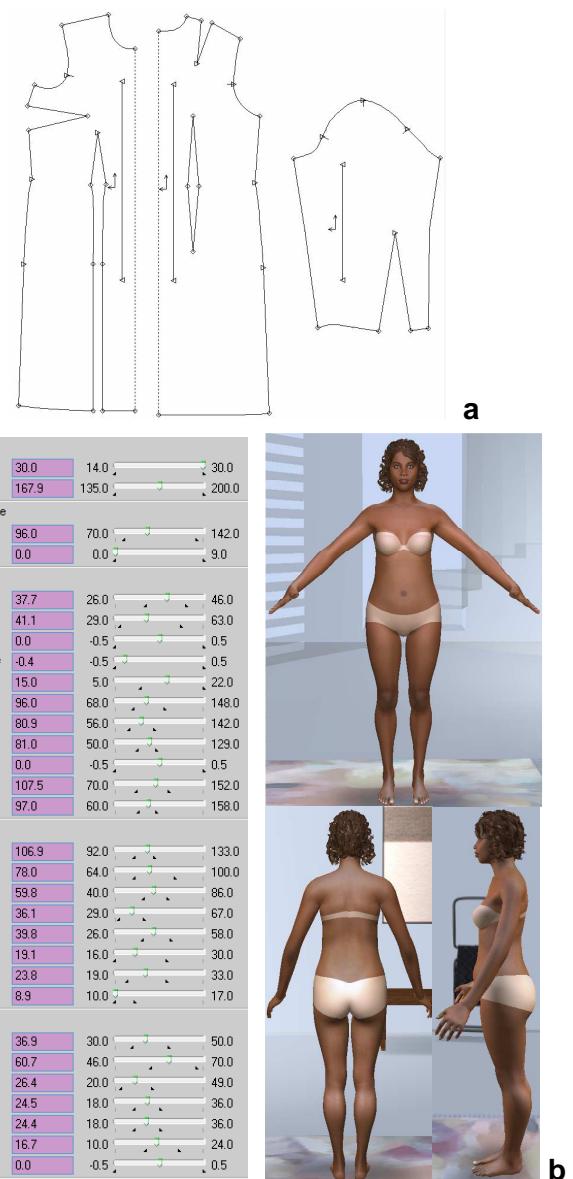


Figure 10 a) Display of the cut parts of women's dress, b) Display and definition of dimensions of virtual figure

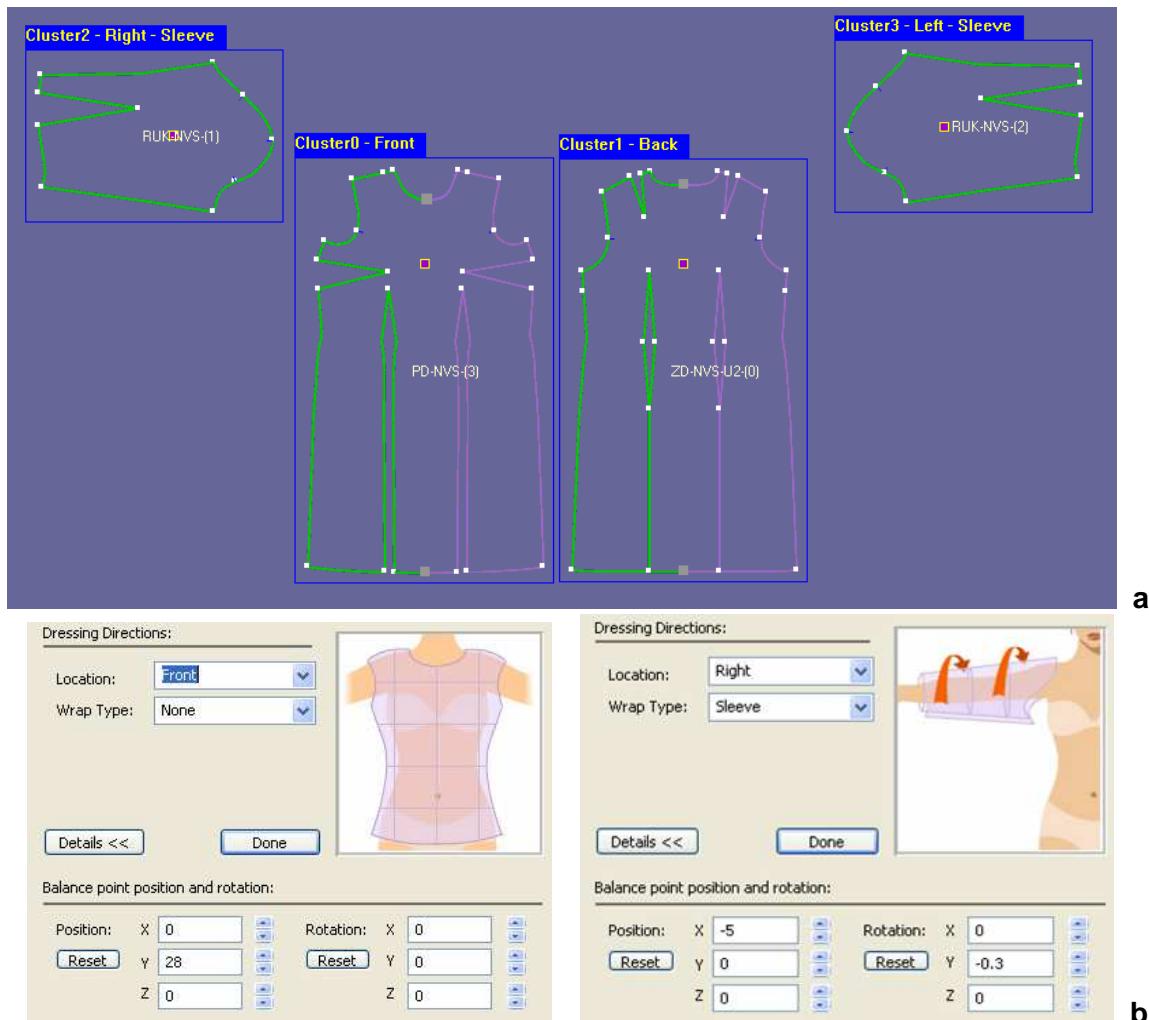


Figure 11 a) Grouping of the parts, b) Choice of the type of clothing parts wrapping on a figure

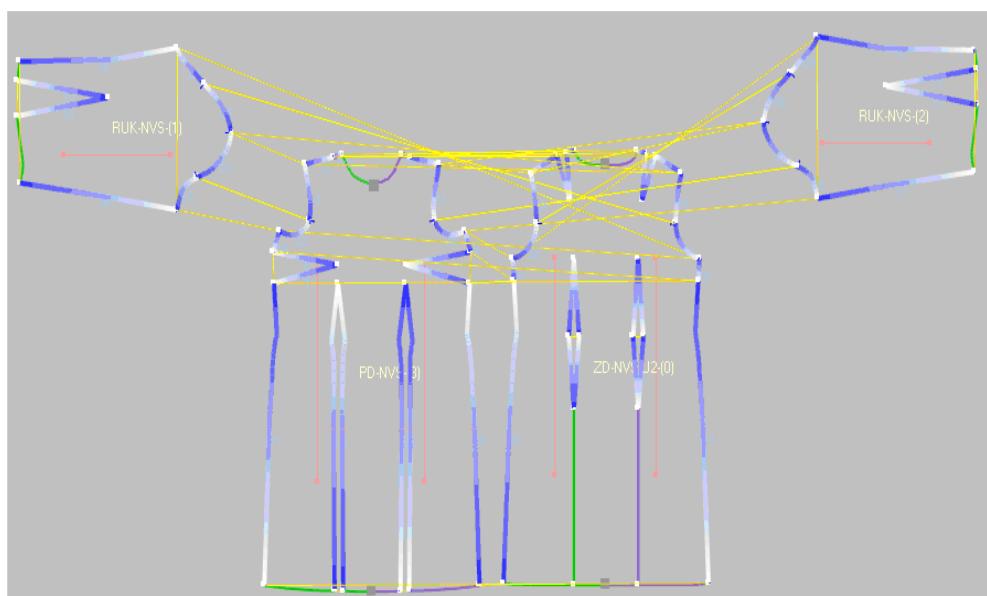


Figure 12 Example of virtual sewing of the parts of women's dress together

The definition of properties of the textile is of key importance when verifying the clothing on a figure. A textile with the properties from the V-Stitcher catalogue named Default was applied for the simulation of clothing.

- The imaging of women's dress on a 3D virtual figure in various poses, Figure 13. The influence of textile parameter selection on the simulated clothing on a virtual figure is shown in Figure 14.

V-Stitcher allows us to look at the clothing on a model in real time and to assess its fitness in static and dynamic postures by using a set of simple and intuitive 3D tools. It allows the assessment of the quality of cut documentation and suitability of the chosen textile from which the product should be made.



Figure 13 Imaging of women's dress on a virtual figure in various poses and the scale of pressure [8]

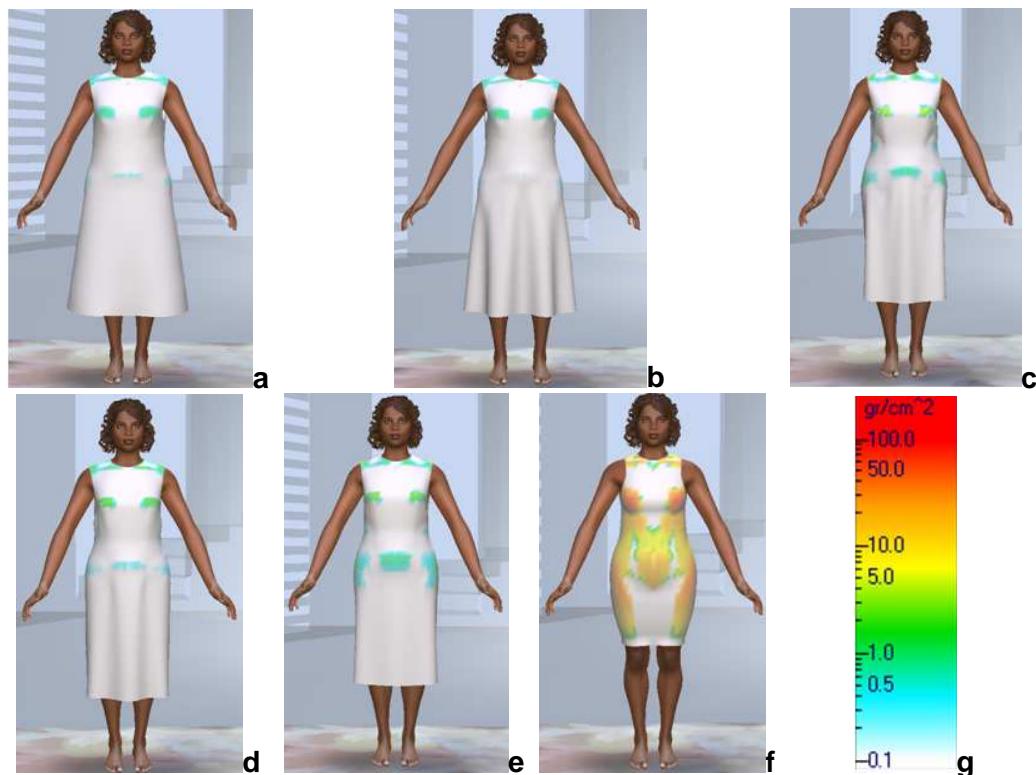


Figure 14 Influence of flexural rigidity on simulated clothing a) 2000 dyn*cm, b) 100 dyn*cm; Influence of shear rigidity c) 300 N/m, d) 5 N/m; Influence of shrinkage e) 3 % warp, 2 % weft, f) 30 % warp, 20 % weft; g) Scale of pressure [8]

5 CONCLUSION

The results of the experiment illustrate rational procedures and methods of marketing and selling clothing product collections using 2D and 3D software. They also provide possible solutions of the outputs and their application in the technical preparation of production.

The designers, model makers and technologists are those who, by using their specialized, aesthetic and technical education and skills, create the image of a company. By their action, they express the company's identity, which can be understood as an attempt at external differentiation, uniqueness and distinctiveness that is visible in aspects such as design, culture, behaviour of an organization, organization's philosophy, communication and products.

Due to rational procedures within companies, the internal structure of the company, the quality of the products and services on offer, leads to changes in their price and how new and innovative products are promoted.

We need to realize that thanks to the number of compatible programs, electronic communication can be carried out between

individual workplaces within a company as well as between companies, and common tasks and activities can be solved faster and qualitatively better at all stages of production, especially during new product development or innovation.

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2D A 3D SOFTWARE PRO DESIGN ODĚVNÍHO VÝROBKU

Translation of the article
2D and 3D software for design of clothing products

Článok je zamieňen na možnosti a uplatnenie 2D a 3D design programov v technické príprave výroby pri tvorbe návrhov modelov oděvu, tvorbe technické dokumentace a prezentaci módnich kolekcí. Předmětem řešení jsou ukázky grafického zpracování, jak pro účely textilního, tak i oděvního navrhování výrobků, spojené s ukázkou možných výstupů a postupů prezentujících firmu. Hlavním důvodem řešení je podpořit tvůrčí činnost návrhářů a techniků uplatněním design programů a poukázat na nekonečné možnosti řešení, kterých lze dosáhnout modifikacemi v krátkém čase a v patřičné kvalitě. Tvůrčí činnost a umění použitím moderních grafických software výrazně ovlivní i komunikaci mezi jednotlivými výrobními, řídícími útvary i obchodními partnery a kvalitu nabízených služeb a služeb vnímaných zákazníkem.

EFFECT OF SEWING SPEED ON SEAM STRENGTH

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Abstract: The seam strength of super imposed seam and lapped seam have been investigated at five different sewing machine speeds from 2900 r/min to 4800 r/min on a single needle lockstitch machine. Three types of fabric, categorized as heavy weight, medium weight and light weight of fabric are used for the experiment. The effect of sewing machine speed on all the three categories of fabric has been observed. Results show that the seam strength decreases at different rate in lapped seam and superimposed seam. Seam strength in case of heavy weight fabrics decreased 17.1 % and 23.2 % for superimposed and lapped seam respectively. In medium weight fabric seam strength decreased by 21 % and 25.6 % respectively for super imposed seam and lapped seam. While the light category of fabric showed the maximum damage due to sewing machine speed, where the seam strength for superimposed seam decreased by 26% and in case of lapped seam this decrease is 31.3 %. It is observed that superimposed seam strength is always higher as compared to lapped seam. There is a strong linear relation with sewing speed and seam strength.

Key words: seam strength, lockstitch.

1 INTRODUCTION

The quality of a garment not only depends upon its appearance but also on its technical properties and seam strength. For getting a quality product it is necessary to select the appropriate type of seam and sewing conditions. Different features that are present inseams are elasticity, stability, strength and appearance. Seam whether applied on upholstery or garments face repeated loading in daily use. There are many parameters that affect seam strength like direction of fabric, stitch type, type of sewing thread, needle type and sewing machine speed. There can be other parameters that affect seam strength but mostly the above cited parameters play a cardinal role in impacting the seam strength. Seam performance properties are influenced by seam designs, seam processing variables and fabric characteristics. Seam efficiency has been defined as the ratio of seam strength to the strength of fabric un-sewn expressed as percentage of fabric strength [1].

In order to save time sewing machine workers operate sewing machines at high

speed. In sewing production of heat is a natural phenomenon due to friction of sewing machine needle, sewing thread and fabric subjected to be sewn. During high speed sewing heat and dynamic loading are mainly responsible for the reduction of strength in sewing thread which in turn causes the early breakdown of seams [2]. In high speed sewing the sewing thread has to pass through a needle's eye, the fabric and bobbin case mechanism 50-80 times before becoming part of the seam [2]. Literature review gives us strong evidence that strength of sewing thread decreases after sewing. The strength of sewing thread decreases 60 % after sewing but then other researchers revealed that 30-40 % strength of sewing thread decreases after sewing in case of cotton thread and argued that structural damage, thermal and dynamic loading are responsible for this reduction of sewing thread [3, 4]. Different measures are adopted in order to decrease needle heat like the use of forced air, oiling of sewing thread by different means (by using felt, wick etc.). In another study on mercerized cotton threads the impact on sewing thread is a 30 %

reduction in sewing thread and around 20 % loss in initial modulus and breaking elongation has been reported [5]. However in a recent study conducted by Adnan Mazari [6], it has revealed that needle heat causes the breakdown of sewing thread which in turn is responsible for the reduction of seam strength. He has further investigated that temperature of sewing machine needle is reduced up to 20°C by the installation of cooling fan and in this way tensile properties of sewing thread will be 8 % better than the sewing thread used without cooling fan.

The strength of constituent yarns plays a great role in the strength of the fabric but other factors like warp and weft geometry, fabric geometry, weave design, weaving conditions and fabric finishing treatments also are equally important to the strength of the fabric. It is true that fabric quality and direction play great role for the production of a quality garment but there are other parameters that are equally important like selection of suitable sewing conditions [7] optimization of sewing conditions, proper selection of a sewing thread and actual performance of the garment during wearing. Lapere and Courtney [8] described that the strength of woven fabric decreased after sewing operation hence serviceability of a stitched article depends on the fabric's technical features as well as on the seams.

The quality and efficiency of seam is related to many factors like type of fabric, stitch density, type of stitch, thread type, seam type and sewing conditions. There have been many studies which explain their relationship but there is a gap in the area of sewing machine speed and its impact on seam strength while taking into account the weight of fabric subjected to be sewn. The aim of this research is to compare seam strength of super imposed seam and lapped seam while operating sewing machine at different sewing

speeds and to check whether the impact of sewing machine speed is same on light weight, medium weight and heavy weight fabrics.

2 EXPERIMENTAL PART

The fabric used in this research work is 100 % cotton fabric and the type of weave is 2/1 Z twill weave. The fabric has been classified in three categories; heavy weight, medium weight and light weight fabric (6 Ounces/yard, 9 Ounces/yard, 12 Ounces/yard). The technical detail of fabric has been given in Table 1.

A lock stitch machine of JUKI having a model of DDL-8300 N is used in this research. By using different pulleys which have diameters 120 mm, 110 mm, 100 mm, 90 mm and 80 mm respectively, the revolutions of sewing machine were calculated by using tachometer.

Sewing thread plays a very cardinal role in the seam strength. Three different types of sewing threads were used for the three categories of fabric. The detail of sewing thread is given in Table 2.

Super imposed seam and lapped seam have been used in this research work.

Super imposed seam is formed by combining two or more pieces of fabric with normally equal seam allowance edge by super imposing one piece of fabric to the other. Lapped seam is formed by overlapping one piece of fabric to the other piece of fabric. Fabric must extend on both right and left sides of the lapped area. Five replicates of all samples have been taken in order to eliminate noise factor and on the basis of average value all the results have been deduced. The seam strength has been calculated according to ASTM D 1683-02 (ASTM 2007).

Table 1 Properties of fabrics

Fabric weight	Fabric type	Weave	Mass/Unit Area (g/m ²)	Fabric thickness (cm)	Ends/cm	Picks/cm
LW (light weight)	100 %cotton Denim	2/1 Twill	203	0.035	27	22
MW (medium weight)	100 %cotton Denim	2/1 Twill	305	0.06	35	20
HW (heavy weight)	100 %cotton Denim	2/1 Twill	406	0.1	36	21

Table 2 Thread properties

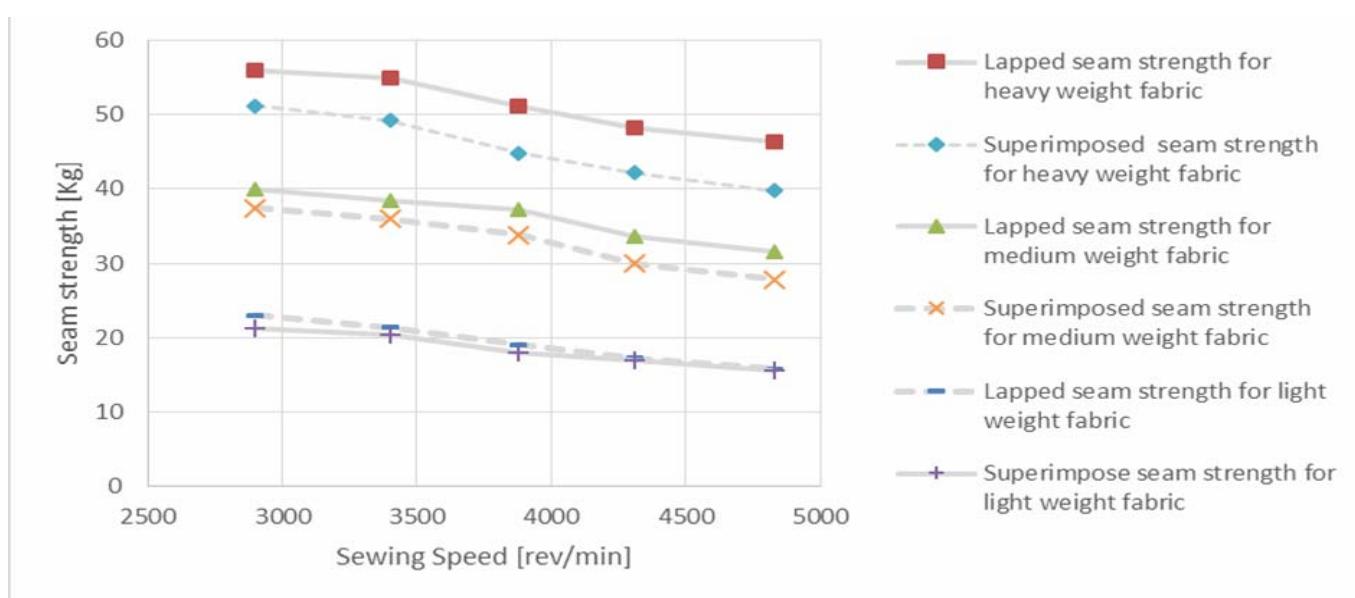
Thread	Thread type	Producer	Thread Count (tex)	Twist (t/m)	Twist direction (ply/single)	Coefficient of friction, μ (-)
A (for heavy fabric)	100 % cotton Ring Spun	COATS	30*3	480	Z/S	0.18
B (for medium weight fabric)	100 % cotton Ring Spun	COATS	30*2	680	Z/S	0.16
C (for light weight fabric)	100 % cotton Ring Spun	COATS	20*2	480	Z/S	0.13

3 RESULTS AND DISCUSSION

Figure 1 shows the effect of sewing speed on seam strength. It is clear from the image that the seam strength decreases with the increase on sewing speed. The effect is much dominant at speed higher than 3000 r/min, which might be due to the reason that the

needle heat is above 220°C at 3000 r/min and rises linearly after this speed [9].

It is visible from the Figure 1 that lapped seam is much stronger as compared to superimposed seam which can be because ,the superimpose seam are thicker as compared lapped seam and layer size increases the needle temperature and causes weaker needle thread [6].

**Figure 1** Effect of sewing speed on seam strength

Figures 2-4 show the percentage loss of seam strength as compared to seam strength measured for 2900 r/min of sewing machine. It is observed that superimposed seam shows nearly 8 % less seam strength as compared to lapped seam. The percentage difference of

seam strength is much higher at higher speeds of sewing. The percentage loss of seam strength is much higher at light weight fabric as compared to seam strength measured at 2900 r/min.

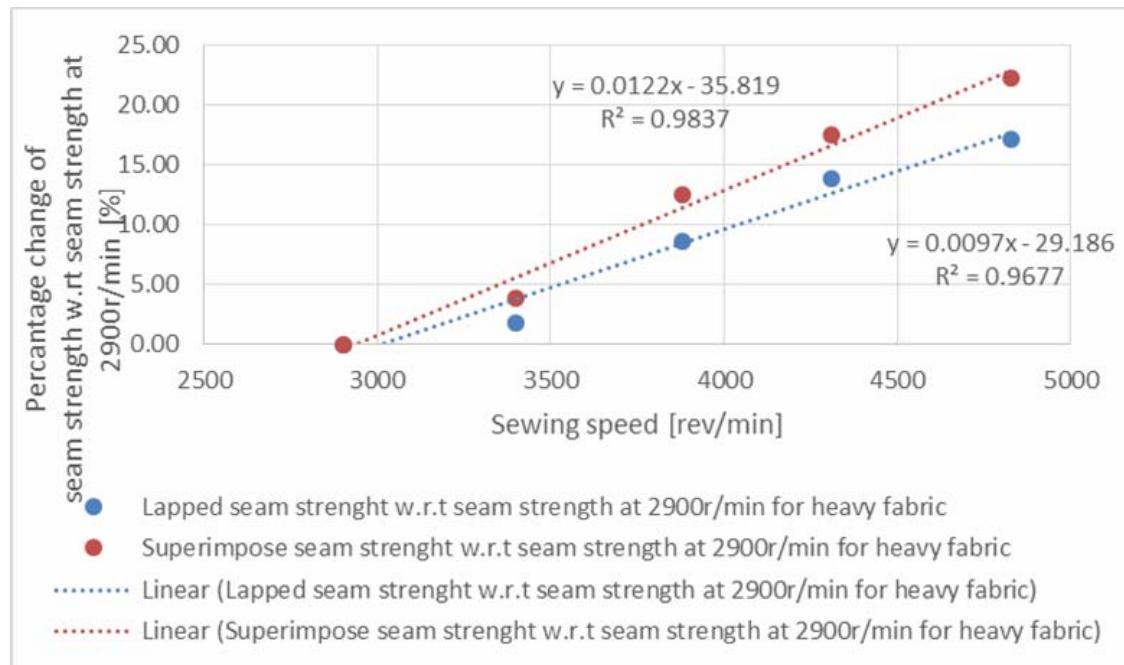


Figure 2 Percentage loss in seam strength as compared to seam strength at 2900 r/min for heavy weight fabric

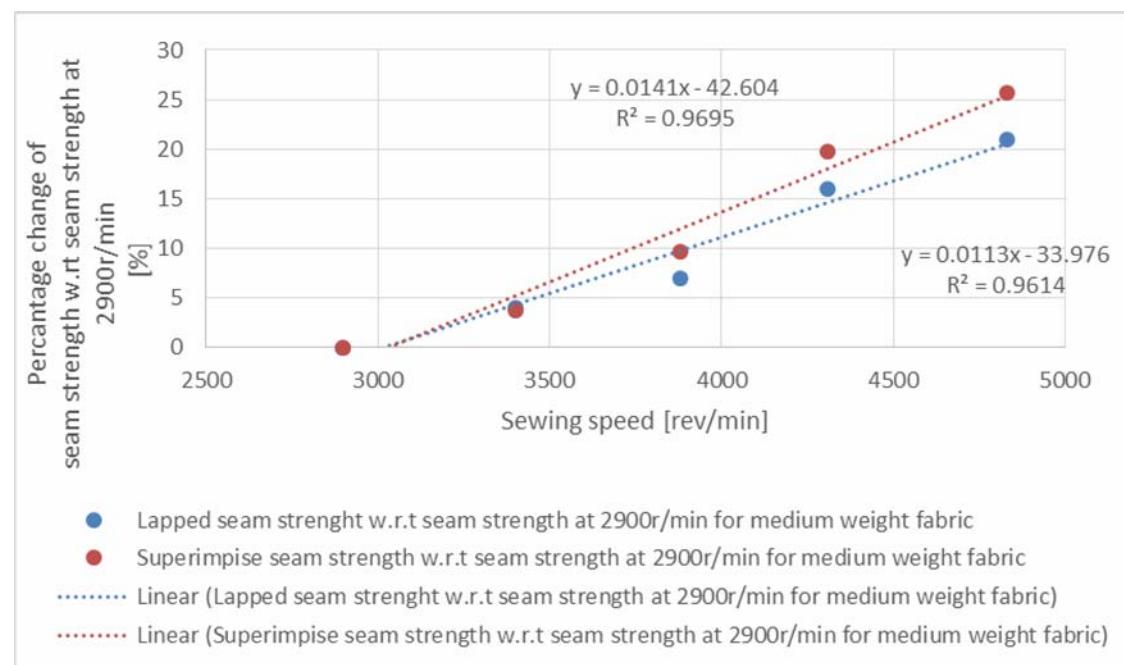


Figure 3 Percentage loss in seam Strength as compared to seam strength at 2900r/min for medium weight fabric

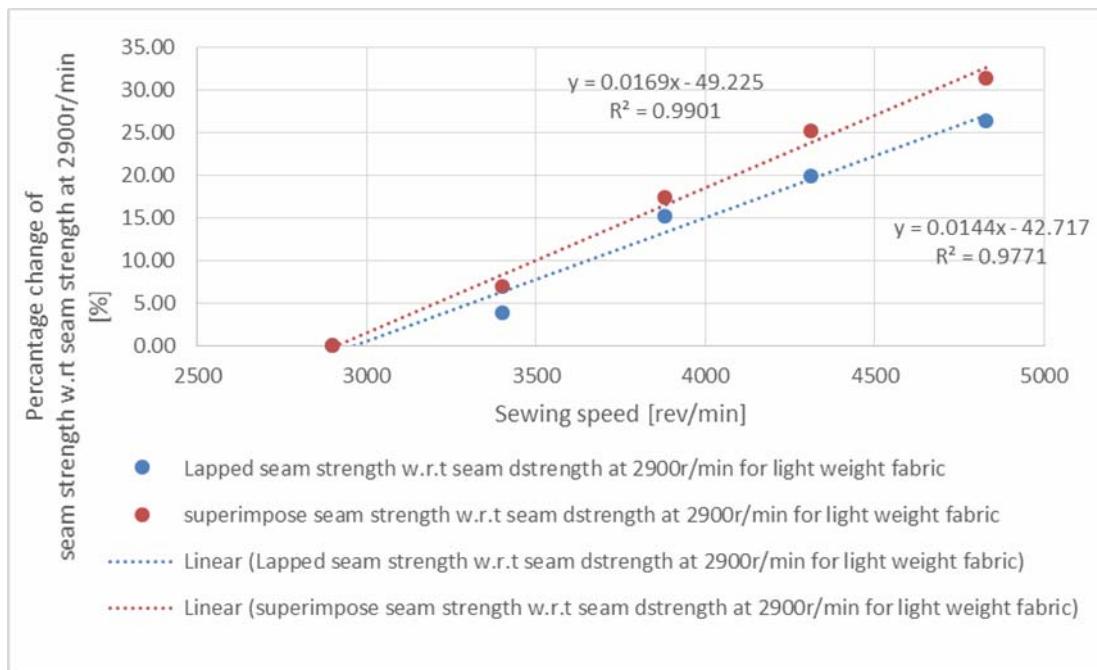


Figure 4 Percentage loss in seam strength as compared to seam strength at 2900 r/min for light weight fabric

4 CONCLUSION

The following includes the major outcomes from this research:

- There is a strong linear relation with sewing speed and seam strength.
- Superimposed seam possess weaker seam strength as compared to lapped seam strength.
- Seam strength in case of heavy weight fabrics decreased 17 % and 23 % for superimposed and lapped seam respectively. In medium weight fabric seam strength decreased by 21 % and 26 % respectively for super imposed seam and lapped seam.
- The light weight category of fabric showed the maximum damage at higher sewing machine speed, where the seam strength for lapped seam decreased by 26 % and in case of superimposed seam this decrease is 31.3 %.
- Lapped seam appeared to be much stronger for different weight of fabrics and also at different speeds of the machine.

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VPLYV RÝCHLOSTI ŠITIA NA PEVNOSŤ ŠVOV

Translation of the article
Effect of sewing speed on seam strength

Pevnosť švov, lemovacích švov a preplátovaných švov bola skúmaná pri piatich rôznych rýchlosťach jednoihlového šijacieho stroja od 2900 r/min do 4800 r/min. Na experiment boli použité tri druhy materiálu kategorizované ako ľahká, stredná a ľahká hmotnosť textílie. Sledoval sa efekt rýchlosťi šijacieho stroja na všetkých troch druhoch materiálov. Výsledok ukazuje, že pevnosť vo šve klesá pri preplátovaných švoch and lemovacích švoch rôzne. Pozorujeme že pevnosť lemovacích švov je vždy vyššia v porovnaní s pevnosťou preplátovaných švov. Existuje silná lineárna závislosť medzi rýchlosťou šitia a pevnosťou švov.

VÝSKUM PRÍPRAVY ELEKTROVODIVÝCH NANOSÓLOV S PODIELOM CNT A ELEKTROVODIVÝCH ADITÍV PRE TEXTILNÉ APLIKÁCIE

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Abstrakt: V príspevku je uvedená stručná charakteristika uhlíkových nanotrubíc (CNT) a ich využitie pri aditivácii v nanosóloch pre zvýšenie elektrickej vodivosti. V ďalších častiach príspevku sú uvedené podmienky prípravy elektrovodivých (EV) nanosólov s podielom CNT a iných elektrovodivých aditív (mikročastice) a poznatky z ich aplikácie na vybrané typy textílií zo syntetických a prírodných vlákien resp. ich zmesí. Popísané sú podmienky overenia prípravy EV nanosólov s rôznymi koncentráciami a typmi CNT a elektrovodivých mikročastic. Zdôraznený je význam využitia ultrazvuku pri dispergácii aditív v roztokoch nanosólov aj pri ich aplikácii na textílie. Zároveň sú uvedené výsledky merania elektrických vlastností textílií dvomi skúšobnými metódami a posúdenia vplyvu údržby textílií praním na textílie upravené elektrovodivým nanosolom. V závere sú zhodnotené najúčinnejšie textílie z hľadiska ich úpravy EV nanosolom.

Kľúčové slová: nanotechnológie, nanosóly, uhlíkové nanotrubice, elektrovodivé sadze, elektrický povrchový odpor, elektrická vodivosť.

1 ÚVOD

Najviac vyskytujúca sa forma čistého uhlíka na zemi je grafit. Je to ľahká vrstevnatá pevná látka, ktorá dobre vedie elektrický prúd. Neobyčajne rýchlo sa rozvíja oblasť kompozitných materiálov spevňovaných nanotubicami – CNT (carbon nanotubes), ktoré majú výnimočné mechanické vlastnosti, najmä vysokú pevnosť v ľahu a malú hmotnosť.

CNT sú jedným z mechanicky najpevnejších materiálov. Ich vysoká pevnosť, tuhost a modul pružnosti im definujú ideálne mechanické vlastnosti, ktoré sa zúročia pri

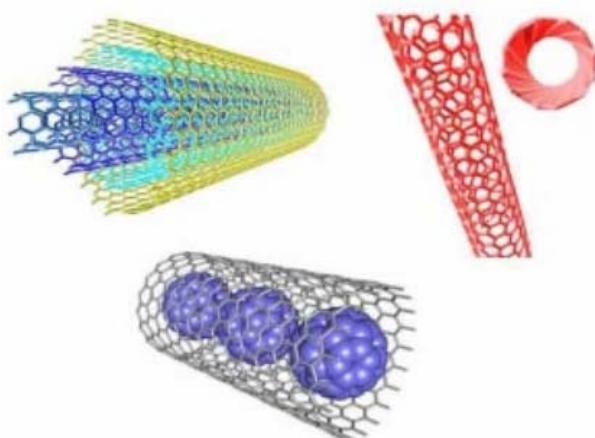
príprave nanokompozitných materiálov pre široké spektrum použitia. Základné charakteristiky uvádzame len z dôvodu ich porovnania s doteraz viac používanými uhlíkovými vláknami aj v textilných kompozitoch, zatiaľ čo CNT sú vo vzťahu k textilu úplne novou surovinou. Zatiaľ boli zistené informácie o používaní CNT na aditiváciu elektrovodivých polymérnych systémov vo vlákne resp. polymérnych vodivých vrstiev na povrchu textílie [1]. Porovnanie vlastností klasických uhlíkových vlákien a uhlíkových nanotrubíc je uvedené v Tab. 1.

Tab. 1 Porovnanie vlastností uhlíkových vlákien a CNT

	Modul pružnosti v ľahu [GPa]	Pevnosť v ľahu [GPa]	Predĺženie [%]	Hustota [g/cm ³]
Uhlíkové vlákna	270 - 390	2,1	1,5	2,1
Jednostenné uhlíkové nanotrubice (SWCNT)	1 000 – 4 500	< 100	15 - 23	1,3 – 1,4
Viacstenné uhlíkové nanotrubice (MWCNT)	800 - 900	< 150	-	1,3 – 1,4

Prvými objavenými a skúmanými nanotrubicami boli viacvrstvové – multi-walled carbon nanotubes (MWCNT), ktoré sa skladajú z dvoch a viac grafitových vrstiev. Grafitové vrstvy sú od seba vzdialené 0,34 nm tak isto ako v grafeite. Tieto vrstvy sú stočené do valca, pričom vnútorná časť tvorí voľný priestor. Priemer MWCNT sa pohybuje medzi 2-25 nm, vnútorný otvor má rozmer 1-8 nm, dĺžka sa pohybuje v nanometroch.

Ďalším typom sú jednovrstvové nanotubice – single-walled carbon nanotubes (SWCNT), kde je jedna vrstva grafitu stočená do valca, ktorý má priemer 1-2 nm a jeho dĺžka môže dosahovať až niekoľko μm . Jednotlivé typy CNT sú schematicky znázornené na Obr. 1.



Obr. 1 Druhy CNT – viacvrstvové MWCNT, jednovrstvové SWCNT typ peapod [2]

Jedna z mala nevýhod nanotubíc je ich nedokonalá čistota, resp. obsah amorfného uhlíka, grafitových štruktúr, fullerénov, zvyškov katalyzátorov a pod. Nanotubice sú nerozpustné, čo znamená, že metódy čistenia musia byť založené na mikro alebo nanofiltrácii.

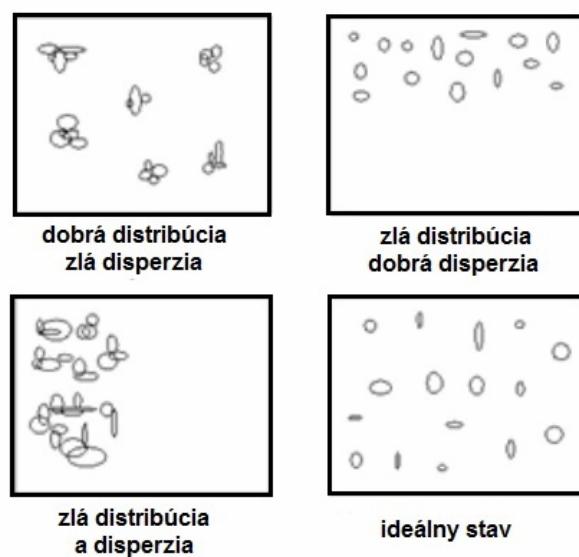
Príprava nanokompozitov s CNT je centrom záujmu súčasného výskumu. Existuje mnoho typov nanokompozitov s CNT, ale u väčšiny nie je doriešený problém dispergácie a distribúcie CNT v matrici (Obr. 2).

Kvalitná disperzia CNT sa uskutočňuje pomocou ultrazvukového miešania nanotubíc vo vhodnom rozpúšťadle. Množstvo CNT v rozteku záleží na použitom

rozpúšťadle, čase, frekvencii a amplitúde ultrazvukového miešania.

Jednou z variant použitia rozpúšťadlovej metódy je priame miešanie CNT v rozpúšťadle a následné miešanie matrice do rozteku. Dôležitým aspektom je chemická inertnosť rozpúšťadla a matrice. Výhodou takého materiálu je elektrická vodivosť bez straty mechanických vlastností.

Modifikácia (funkcionalizácia) uhlíkových nanotubíc sa uskutočňuje v disperznom prostredí prostredníctvom zlúčení obsahujúcich funkčné skupiny. Počas funkcionalizácie sa modifikátor adsorbuje na povrch uhlíkových nanotubíc. Priebeh deaglomerácie CNT pri rôznych podmienkach disperzného prostredia, použitia rôznych modifikátorov a rozpúšťadiel ako aj ultrazvuku sú schematicky znázornené na Obr. 2.



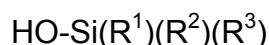
Obr. 2 Schopnosť deaglomerácie CNT do matrice [2]

V optimálnom prípade dôjde k chemickému naviazaniu modifikátora na povrch CNT prostredníctvom slabších Van der Waalsových väzieb, alebo silnejších kovalentných väzieb. Unikátné fyzikálne a chemické vlastnosti uhlíkových materiálov a hlavne CNT, akými sú: vysoká pohyblivosť nosičov náboja, vysoká pracovná teplota, tepelná vodivosť, prierazná pevnosť, biokompatibilita, nízka dielektrická konštantá,

odolnosť voči radiácii, chemická inertnosť a možnosť koncentrovať vysoký výkon na malej ploche, sú motiváciou pre výskum posúvajúci možnosti ich využitia v rôznych oblastiach.

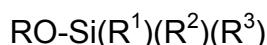
1.1 Popis prípravy silanizovaných CNT

Jednou z možností prípravy nanosólov prostredníctvom sól-gél reakcií je príprava anorganicko-organických komplexov s použitím organosiloxánov, ktoré sa po následnej aditivácii s použitím CNT môžu využiť ako nanosóly so zvýšenými elektrovodivými vlastnosťami. V nasledujúcich schémach je stručne uvedený popis prípravy silanizovaných CNT s využitím vhodných typov organosilánov. Počas hydroxylácie môže prebiehať reakcia uhlíkových nanotrubíc so zlúčeninami silanolu všeobecného typu:

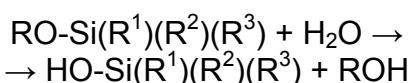


kde každé $\text{R}^1 - \text{R}^3$ je funkčná časť, ktorú možno vybrať zo skupiny, ktorá sa skladá z vodíka (H), hydroxylu (OH), tiolu (SH), nasýtených a nenasýtených alifatických uhlíkovodíkov, cykloalifatických uhlíkovodíkov, aromatických uhlíkovodíkov, amínov, amidov, esterov, éterov, epoxidov, silylu, germylu, stanylu a ich kombinácií.

V niektorých príkladoch takéto zlúčeniny silanolu vznikajú hydrolýzou siloxánových zlúčenín typu:

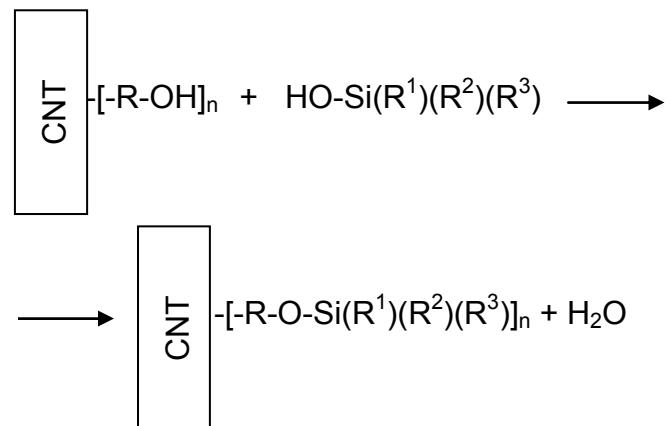


pričom:



kde R je uhlíkovodíková funkčná skupina.

Tieto zlúčeniny silanolu reagujú s uhlíkovými nanotrubicami, funkcionálizovanými hydroxylom, podľa reakčnej schémy:



Popísaná reakcia sa nazýva „silanizácia“ a produkty tejto silanizácie sa označujú ako uhlíkové nanotrubice funkcionálizované silánom. Všeobecne platí, že každá uhlíková nanotrubica s hydroxylovými skupinami by sa mohla použiť v kroku silanizácie [3].

V niektorých príkladoch, kde aspoň jedna z $\text{R}^1 - \text{R}^3$ skupín na uhlíkových nanotrubiciach funkcionálizovaných silánom, je funkčná časť vzájomne pôsobiaca s polymérom. Uhlíkové nanotrubice funkcionálizované silánom sa miešajú s vhodným polymérnym prekurzorom, ktorý následne polymerizuje in situ s uhlíkovými nanotrubicami, funkcionálizovanými silánom. V niektorých príkladoch, kde polymérne prekurzory sú epoxidové prekurzory, aspoň jedna z $\text{R}^1 - \text{R}^3$ sa vyberie zo skupiny obsahujúcej amíny, epoxidové kruhy, kyseliny karboxylové, tioly, izokyanáty a ich kombinácie, pričom pri vulkanizácii dochádza ku kovalentnej integrácii s výslednou epoxidovou matricou.

Stručné uvedenie tvarových a veľkosťných charakteristík CNT, ich schopnosť deaglomerácie v rôznych disperzných podmienkach, ako aj schopnosť silanizácie povrchu CNT v podmienkach hydrolýzy pri príprave sólového roztoku boli uvedené ako východiska, na ktoré sa zamerali výskumné práce popísané ďalej v príspevku aj s uvedením konkrétnych výsledkov pri príprave elektrovodivých textílií.

2 EXPERIMENTÁLNA ČASŤ

2.1 Použité materiály

V experimentálnych prácach boli použité rôzne typy uhlíkových materiálov pre prípravu elektrovodivého nanosólu. V prvom rade to boli uhlíkové nanotrubice a potom, pre porovnanie elektrickej účinnosti, to boli uhlíkové mikročasticie (elektrovodivé sadze) vhodné pre aditiváciu polymérneho systému vytvoreného z organosilánov.

Uhlíkové nanotrubice (CNT)

typ U - MWCNT, pôvodom z USA,

- vonkajší priemer 30-50 nm,
 - vnútorný priemer 5-10 nm,
 - dĺžka 10-20 µm, čistota $\geq 95\% \text{hm.}$,
 - elektrická vodivosť $\geq 10^{-2} \text{ S/cm}$
- typ G** - MWCNT, pôvodom z Grécka,
- vonkajší priemer 10-40 nm,
 - veľkosť povrchu $150-250 \text{ m}^2/\text{g}$,
 - dĺžka 1-25 µm,
 - čistota min. $93\% \text{hm.}$,
 - elektrická vodivosť $\geq 10^{-2} \text{ S/cm}$

Timrex KS 44 - uhlíkové sadze (uhlík v prášku, CSA: 7782-42-5), pôvodom zo Švajčiarska

- veľkosť častic $40-51,5 \text{ }\mu\text{m}$,
- veľkosť povrchu $9,0 \text{ m}^2/\text{g}$,
- čistota min. $99,9\% \text{hm.}$,
- elektrická vodivosť nezistená

Organosiloxánové prekurzory a pomocné látky

Pre prípravu nanosólových roztokov boli použité nasledovné prekurzory:

TEOS - trietoxysilán (CAS 78-10-4)

VTES - vinyltriethoxysilán (CAS 78-08-0)

Pomocné látky pre hydrolyzu, ukončenie reakcie a rozpúšťadlá

HCl - katalyzátor

izopropanol – rozpúšťadlo,

TEG - trietylenglykol (prostriedok na ukončenie reakcie)

Textilné materiály

Pre aplikáciu pripravených elektrovodivých nanosólových roztokov a vyhodnotenie účinnosti zmeny elektrických vlastností boli použité nasledovné textilné materiály:

- 100 % bavlnená (ba) tkanina, väzba plátnová, plošná hmotnosť 120 g/m^2

- 100 % polyesterová (PES) tkanina, väzba: plátnová, plošná hmotnosť 125 g/m^2
- zmesná tkanina PES/ba (50/50), väzba plátnová, plošná hmotnosť 130 g/m^2

2.2 Skúšobné metódy

a/ Skúšobná metóda podľa STN EN 1149-1:

„Ochranné odevy. Elektrostatické vlastnosti. Časť 1: Skúšobná metóda na meranie povrchového odporu“

Hodnotenie povrchového odporu bolo vykonávané na zariadení TeraOhmeter, typ TO 03 s elektródou TE 50. Meranie sa vykonávalo v klimatizovanej komore pri teplote 22°C a relatívnej vlhkosti 30 % na vzorke s priemerom 10 cm po priložení kalibrovanej meracej sondy na povrch vzorky textílie. Na každej analyzovanej vzorke bolo vykonaných 10 meraní. Smerodajná odchýlka bola stanovená v rozmedzí 2,5-5,7.

b/ Meranie elektrickej vodivosti textílií metódou BDS

Na stanovenie elektrickej vodivosti sa použil širokopásmový dielektrický spektrometer Concept 40, fy Novocontrol, Nemecko. Prístroj umožňuje merať vodivosť v rozsahu 10^{-3} až 10^7 Hz . Vzorka textílie bola vložená medzi dve zlaté elektródy s priemerom 2 cm. Na každej analyzovanej vzorke bolo vykonaných 10 meraní. Smerodajná odchýlka bola stanovená v rozmedzí 1,2 – 3,1.

2.3 Organické siloxánové zlúčeniny a príprava nanosólov

Chémia sól-gél procesov je založená na hydrolyze a polykondenzácii molekulových prekurzorov, ako sú napr. rôzne alkoxidy (silány, zirkónaty a pod.) [3]. Tieto reakcie vedú k vytvoreniu anorganických resp. anorganicko-organických trojrozmerných sietí, ktoré môžu byť aplikované vo forme kompaktných blokov, náterov a tenkých filmov. Použitie neagresívnych rozpúšťadiel a nízkych teplôt taktiež umožňuje zavedenie organických molekúl. Rôzne anorganické a organické komponenty môžu byť navzájom kombinované a štruktúrované na nanorozmerovej škále. Vzniknuté organicko-anorganické hybridy vykazujú široké

spektrum najrozmanitejších fyzikálnych a úžitkových vlastností.

Pre prípravu nanosôlov sme využili sól-gél metódu prostredníctvom procesu kyslej kohydrolyzy organosiloxánových prekurzorov, TEOS : VTES v pomere 1:1. Vybrané typy organosiloxánov sme zmesovali za neustáleho miešania. Následne po pridaní 0,01 N roztoku HCl, za neustáleho miešania roztoku a súčasného chladenia, prebehla kohydrolyza prekurzorov. Do pripraveného kohydrolyzátu sme postupne pridali vybrané CNT typu U, typu G, resp. aditívum Timrex KS 44.

Pre ukončenie prebiehajúcej reakcie sme do roztoku postupne pridávali potrebné množstvo trietylénglykolu (TEG). S cieľom dosiahnutia lepšej dispergácie aditivovaných častíc v roztoku sme využili proces sonikácie (2x15 min) vplyvom ultrazvuku.

Po vykonaní úpravy v postepe prípravy nanosôlu, ako aj času sonikácie v ultrazvuku, bol pripravený základný elektrovodivý nanosól typu EVN2-TEG, s ktorým sme pracovali pri ďalších experimentoch.

Sonikácia bola použitá aj pri následnej aplikácii pripraveného elektrovodivého roztoku na vybraný typ textílie, pri rôzne zvolených časoch pôsobenia ultrazvuku na textíliu ponorenú do roztoku nanosôlu, za účelom zabezpečenia homogénnej dispergácie elektrovodivých nano resp. mikročastíc na povrchu textílie.

2.4 Podmienky aplikácie roztoku nanosôlu na textílie

Aplikácia roztoku nanosôlu na textílie v laboratórnych podmienkach bola vykonávaná v sklenených kadičkách. Do roztoku nanosôlu bola vložená vzorka textílie kruhového tvaru s priemerom 10 cm tak, aby vzorka počas celej doby aplikácie bola ponorená v roztoku. Následne bola kadička s roztokom a vzorkou textílie vložená do sonikačného zariadenia, kde prebiehalo pôsobenie ultrazvuku na roztok s CNT. Pôvodne v rozsahu 15-60 min, po vykonaní základných hodnotení bol zvolený najvhodnejší čas sonikácie 30 min.

Po vykonaní sonikácie boli vzorky textílie vysušené pri teplote 120°C po dobu 5 min a následne bola vykonaná fixácia za nasledovných podmienok:

- ba textília: $t = 150^{\circ}\text{C}/2 \text{ min}$
- 100 % PES a PES/ba textília: $t = 180^{\circ}\text{C}/2 \text{ min.}$

3 VÝSLEDKY A DISKUSIA

3.1 Využitie CNT v elektrovodivých nanosôloch

Základným experimentom bolo zvolenie vhodnej koncentrácie a obsahu vybraného typu CNT v nanosóle a čas sonikácie (dispergácie) CNT v roztoku nanosôlu.

Za základné koncentrácie boli zvolené nasledovné: 1,5; 3,0 a 5,0 g/l CNT typu U alebo typu G. Z hľadiska posúdenia vplyvu časového faktora pri sonikácii v ultrazvuku boli zvolené nasledovné časy sonikácie roztoku nanosôlu: 15, 30, 45 a 60 min.

Po vykonaní množstva experimentov v oblasti prípravy EV nanosôlov s rôznym obsahom CNT typu U a G, ako aj posúdenia optimálneho času vplyvu ultrazvuku pri dispergácii CNT v nanosóle, za účelom dosiahnutia homogénneho a stabilného roztoku nanosôlu (stabilita min. 24 hod.) boli za konečné podmienky zvolené nasledovné:

- koncentrácia 3 g/l CNT vybraného typu
- čas sonikácie roztoku nanosôlu 30 min
- CNT typu G – dosiahla sa homogénnejšia štruktúra roztoku aj nánosu na textíliu.

Homogénnosť štruktúry nánosu CNT z EVN2-TEG roztoku bola hodnotená vizuálne a predpoklad vyššieho stupňa homogenity pri CNT typu G bol priebežne potvrdzovaný aj pri meraní povrchového odporu. V prípade CNT typu G boli jednotlivé merania reprodukovateľné s odchýlkou ± 1 rád a v prípade typu U s odchýlkou $\pm 2-3$ rády.

Zistené priemerné hodnoty (x_i) povrchového odporu štandardných neupravených textílií boli nasledovné:

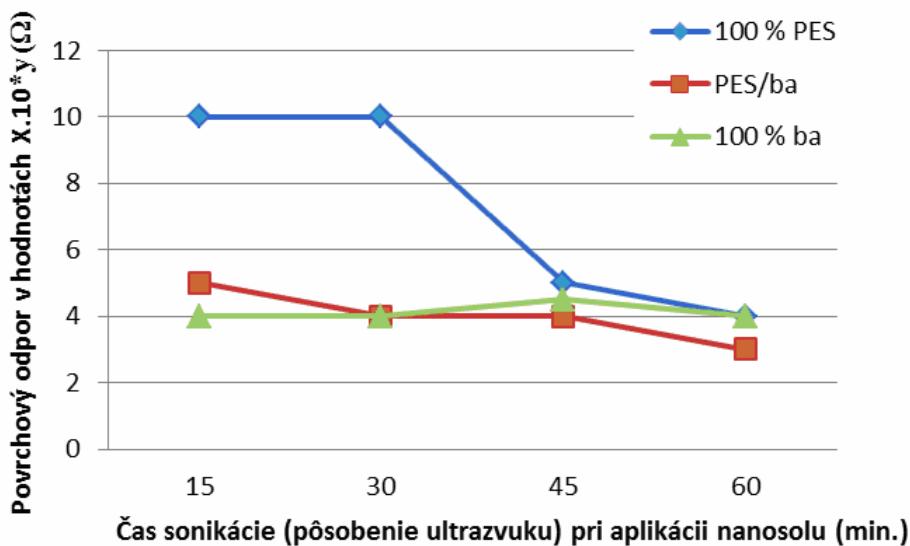
textília zo 100 % PES vlákien $4,80 \cdot 10^{10} \Omega$

textília zo 100 % ba $2,35 \cdot 10^{12} \Omega$

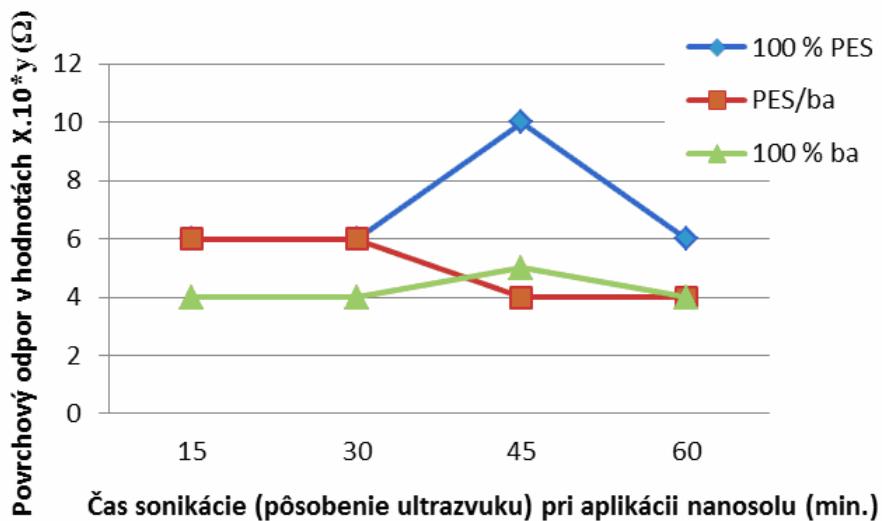
textília zo zmesi PES/ba (50/50) $1,18 \cdot 10^{11} \Omega$

Následne sme pristúpili k overovaniu vplyvu časového faktora pri sonikácii EV nanosólu počas jeho aplikácie na vybrané typy textílií zo 100 % PES, 100 % ba a zmesi PES/ba vláken.

Výsledky merania povrchového odporu vybraných druhov textílií upravených EVN2-TEG nanosolom s obsahom CNT typu U a G sú uvedené na Obr. 3 a 4.



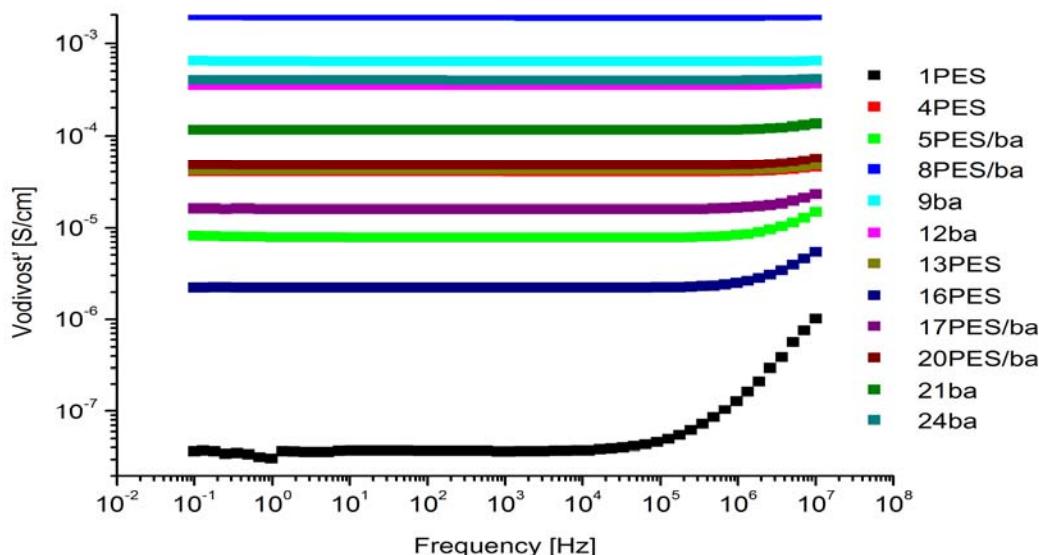
Obr. 3 Povrchový odpor textílií po aplikácii nanosólu EVN2-TEG s obsahom 3 g/l CNT typu U



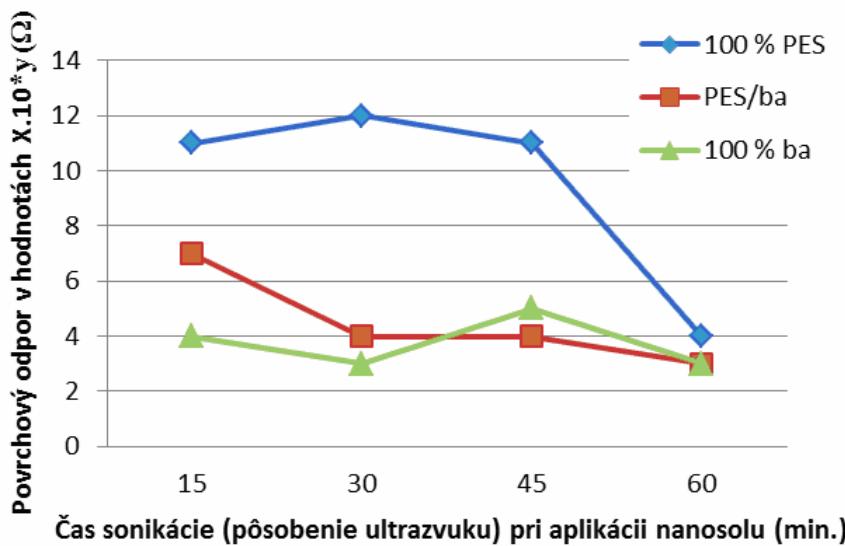
Obr. 4 Povrchový odpor textílií po aplikácii nanosólu EVN2-TEG s obsahom 3 g/l CNT typu G

Na vybraných vzorkách textílií boli vykonané aj skúšky merania elektrickej vodivosti metódou BDS. Pri príprave EV nanosólom upravených textílií bol zachovaný štandardný postup t.j. použitá koncentrácia 3 g/l CNT typ G, čas sonikácie nanosolu 30 min a čas sonikácie pri aplikácii nanosolu na textíliu tiež 30 min.

V rámci experimentu bol overovaný aj potenciálne pozitívny vplyv predúpravy povrchu vybraných textílií s využitím nízkoteplotnej plazmy s cieľom iniciovania vzniku voľných radikálov na povrchu textílií pred následnou aplikáciou EV nanosolu. Výsledok zmeny hodnoty povrchového odporu je uvedený na Obr. 6.



Obr. 5 Hodnoty elektrickej vodivosti, zistených metódou BDS, na upravených vzorkách textílií s použitím nanosolu EVN2-TEG.



Obr. 6 Povrchový odpor textílií predupravených plazmou (čas – 100 s) po aplikácii nanosolu EVN2-TEG s obsahom 3 g/l CNT typu G

3.2 Využitie elektrovodivých sadzí v elektrovodivých nanosóloch pri ich aplikácii na textílie

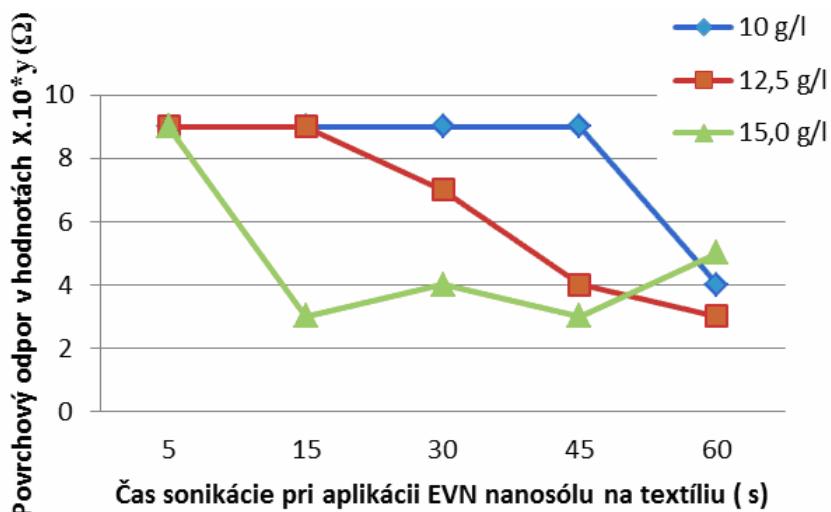
V rámci ďalších experimentov boli detailne rozpracované aktivity súvisiace s voľbou koncentrácie aditíva Timrex KS 44 v nanosóle EVN2-TEG: 10 g/l, 12,5 g/l a 15 g/l. Bol zvolený štandardný čas sonikácie pri príprave nanosólu v rozsahu 30 min. Následne bol sledovaný vplyv časového faktora pri sonikácii počas aplikácie nanosólu v čase: 5, 15, 45 a 60 min. Výsledky sú uvedené na Obr. 7 a 8.

Na základe vyhodnotenia dosiahnutých výsledkov je možné konštatovať, že pri

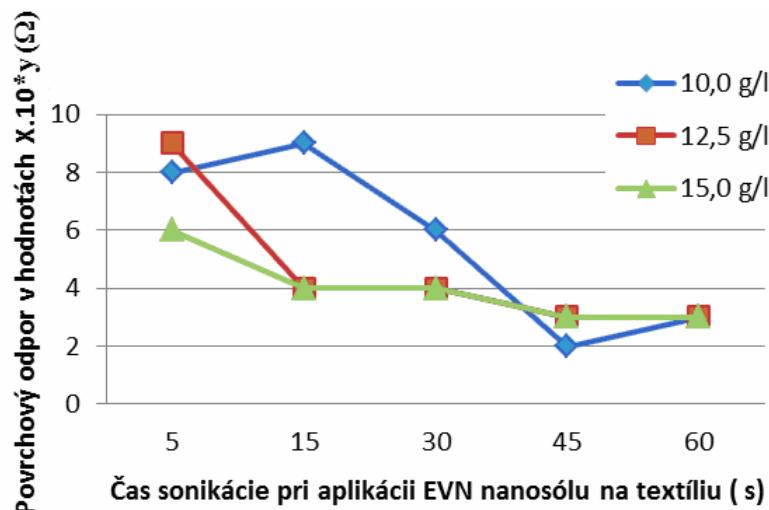
textíliach zo 100 % ba, resp. PES/ba sa dosahuje veľmi nízky elektrický povrchový odpor na úrovni 10^2 - 10^3 Ω už pri koncentrácií 12,5 g/l, optimálne pri koncentrácií 15 g/l aditíva Timrex KS 44.

3.3 Hodnotenie účinnosti elektrovodivej úpravy textílií po praní

Na vzorkách textílií upravených nanosólom s aditívom Timrex KS 44 bola sledovaná aj permanentnosť úpravy praním vzoriek textílií na zariadení Linistest. Výsledky sú uvedené v Tab. 2.



Obr. 7 Závislosť povrchového odporu od času sonikácie a koncentrácie aditíva Timrex KS 44 na 100 % bavlné

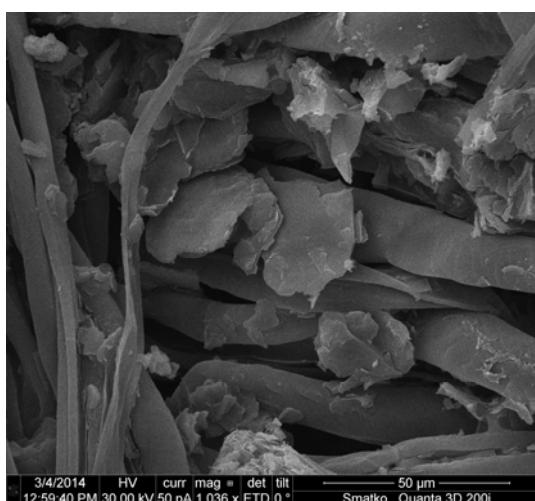


Obr. 8 Závislosť povrchového odporu od času sonikácie a koncentrácie aditíva Timrex KS 44 na zmesi PES/ba

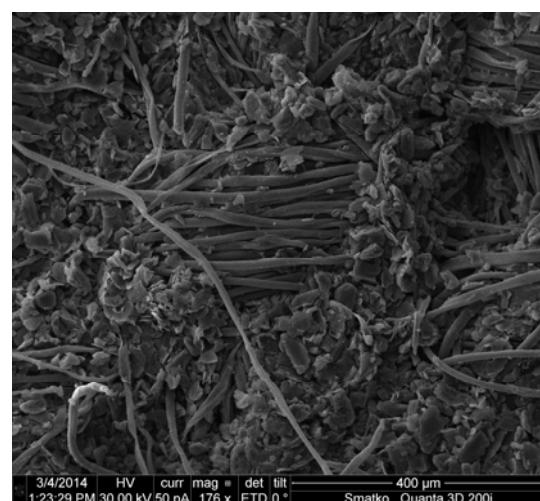
Tab. 2 Hodnoty povrchového odporu na textíliach po 5. praní v porovnaní s pôvodnými hodnotami po úprave EVN2-TEG nanosólom s obsahom aditíva Timrex KS 44.

Typ textílie	Obsah aditíva Timrex KS 44	Povrchový odpor textílie po úprave (Ω)	Povrchový odpor textílie po 5. praní (Ω)
100 % ba	10,0 g/l	$3,52 \cdot 10^5$	$5,18 \cdot 10^{11}$
PES/ba	10,0 g/l	$8,76 \cdot 10^4$	$1,47 \cdot 10^9$
100 % ba	12,5 g/l	$8,72 \cdot 10^3$	$2,62 \cdot 10^{10}$
PES/ba	12,5 g/l	$1,45 \cdot 10^3$	$1,25 \cdot 10^4$
100 % ba	15,0 g/l	$1,00 \cdot 10^4$	$4,86 \cdot 10^6$
PES/ba	15,0 g/l	$2,33 \cdot 10^3$	$1,79 \cdot 10^9$

Poznámka: Na každej analyzovanej vzorke bolo vykonaných 10 meraní. Smerodajná odchýlka bola stanovená v rozmedzí 2,5 – 3,6.



Obr. 9 Makroštruktúra povrchu 100 % bavlnenej textílie upravenej nanosólom s podielom elektrovodivých sadzí



Obr. 10 Makroštruktúra povrchu PES/ba textílie po aplikácii elektrovodivého nanosólom s podielom elektrovodivých sadzí

4 ZHRNUTIE

Vykonané experimenty preukázali vhodnosť použitia uhlíkových nanotrubíc aj elektrovodivých uhlíkových sadzí pre aditiváciu elektrovodivých nanosólov pripravených z organosilánov.

Vzhľadom na použité dva typy CNT je možné konštatovať, že pre prípravu a aplikáciu boli vhodnejšie nanotrubice typu G, ktoré vytvárali po aplikácii EV nanosólu na povrchu textílie homogénnejšiu vrstvu (vizuálne hodnotenie) a tým aj homogénnejšie elektrovodivé prostredie. Tento predpoklad bol potvrdený aj pri meraní elektrického povrchového odporu na vzorkách textílií, keď pri aplikácii EVN2-

TEG nanosólou s CNT typ G boli dosiahnuté reprodukovanéjšie výsledky.

Dosiahnuté výsledky potvrdili, že po aplikácii EVN2-TEG nanosólou je možné dosiahnuť uvedenou úpravou podstatné zníženie povrchového odporu na textíliach zo 100 % ba a zmesných PES/ba textílií rádovo až o 6-7 stupňov oproti neupravenej štandardnej textílii. Pri 100 % PES textílii je tento pokles menej výrazný len cca o 5 stupňov (Obr. 3 a 4).

Ako významný faktor sa preukázal aj čas sonikácie (dispergácie) CNT prostredníctvom ultrazvuku, ktorý je potrebný minimálne v rozsahu 30 min, aby sa dosiahla homogénnejšia úprava na povrchu textílie,

ktorá sa pozitívne prejavila v reprodukovateľnosti výsledkov merania povrchového odporu (Obr. 3 a 4).

Významná zmena elektrických vlastností na upravených textiliach bola potvrdená aj meraním parametra elektrickej vodivosti metódou BDS, ktorý dosiahol na 100 % ba a zmesných PES/ba textiliach hodnotu až 10^{-3} S/cm (Obr. 5).

Využitie nízkoteplotnej plazmy na predúpravu povrchu textilií pred aplikáciou EV nanosólu ďalej významne neznížilo hodnotu povrchového odporu (Obr. 6).

Pravdepodobne podstatnejší je vplyv predúpravy plazmou na 100 % PES textíliu, kde sa dosiahla zmena povrchového odporu ale až po 60 min sonikácie roztoku EVN2-TEG pri aplikácii na textíliu.

Overenie ďalšieho komerčného elektrovodivého aditíva na báze elektrovodivých uhlíkových mikročastíc pre prípravu elektrovodivých nanosólov potvrdilo dosiahnutie požadovaných elektrických vlastností. Dosiahla sa podstatne homogénnejšia vrstva na povrchu textílie (vizuálne hodnotenie) a lepšia reprodukovateľnosť pri meraní povrchového odporu (Obr. 8). Na zmesnej textílii PES/ba bola po úprave nameraná hodnota povrchového odporu na úrovni $10^2\text{-}10^3 \Omega$ (Obr. 8). Určitou nevýhodou je zvýšený obsah aditíva až na úroveň 12,5-15,0 g/l, aby boli dosiahnuté porovnatelné hodnoty povrcho-vého odporu s textíliou upravenou nanosólom obsahujúcim CNT.

Pri uvedených koncentráciách aditíva, najmä na 100 % ba a zmesnej PES/ba textílií, boli dosiahnuté aj po 5 praniach hodnoty povrchového odporu na úrovni $10^4\text{-}10^5 \Omega$. (Tab. 2). Uvedená skutočnosť potvrdzuje dostatočnú úroveň permanentnosti úpravy textílie vhodnej pre technické účely.

5 ZÁVER

Dosiahnuté výsledky potvrdzujú reálnosť prípravy elektrovodivých nanosólov na báze

organosilánov po vykonaní ich aditivácie s použitím CNT resp. elektrovodivých mikročastíc. Overenie ich aplikácie na vybrané typy textílií zo 100 % PES, zmesných PES/ba a 100 % ba vlákien zároveň potvrdilo, že najvyššia účinnosť zmeny elektrických povrchových vlastností sa dosiahla na 100 % ba a zmesnej PES/ba textílií. Významným faktorom pri príprave elektrovodivých nanosólových roztokov s podielom CNT a iných elektrovodivých mikročastíc je použitie ultrazvuku na zabezpečenie dostatočnej dispergácie častíc v roztoku a dosiahnutie stability roztoku vhodnej pre jeho aplikáciu na textíliu. Z hľadiska reprodukovateľnosti merania povrchového odporu a vodivosti sa dosiahli lepšie výsledky pri aplikácii elektrovodivých nanosólov s CNT menších rozmerov (vonkajší priemer 10-40 nm) a s dĺžkou 1-25 μm .

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Európska únia
Európsky fond regionálneho rozvoja



Agentúra
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Podporujeme výskumné aktivity na Slovensku/
Projekt je spolufinancovaný zo zdrojov EÚ

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RESEARCH OF PREPARATION OF ELECTROCONDUCTIVE NANOSOLS CONTAINING CNT AND ELECTROCONDUCTIVE ADDITIVES FOR TEXTILE APPLICATIONS

Translation of the article

**Výskum prípravy elektrovodivých nanosolov s podielom CNT a elektrovodivých aditív
pre textilné aplikácie**

The contribution presents short characteristic of carbon nanotubes (CNT) and employment of their possibilities in additivation in nanosols for enhancement of electrical conductivity. Conditions of preparation of electroconductive nanosols containing carbon nanotubes and other electroconductive additives (nanoparticles) as well as experience in their application on selected types of fabrics made from synthetic and natural fibres and/or their blends are given in the following parts of the contribution. Conditions of evaluation of the prepared electroconductive nanosols with various concentrations and types of CNT and electroconductive microparticles are described. Importance of ultrasound application on additive dispergation in nanosol solutions and their application on the fabrics is emphasized. Besides, results of measurement of electrical properties of the fabrics using two test methods and results of evaluation of influence of washing on fabrics finished with the electroconductive nanosol are presented and discussed. The most efficient fabrics are evaluated in the conclusion from a viewpoint of their finish with electroconductive nanosol.