

FATIGUE STUDY OF SPIRO[INDOLINE-NAPHTHOXAZINES] 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 spiropyrans (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.

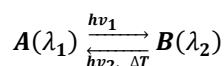


Figure 1 General scheme of photochromism from A to B [1, 2]

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.

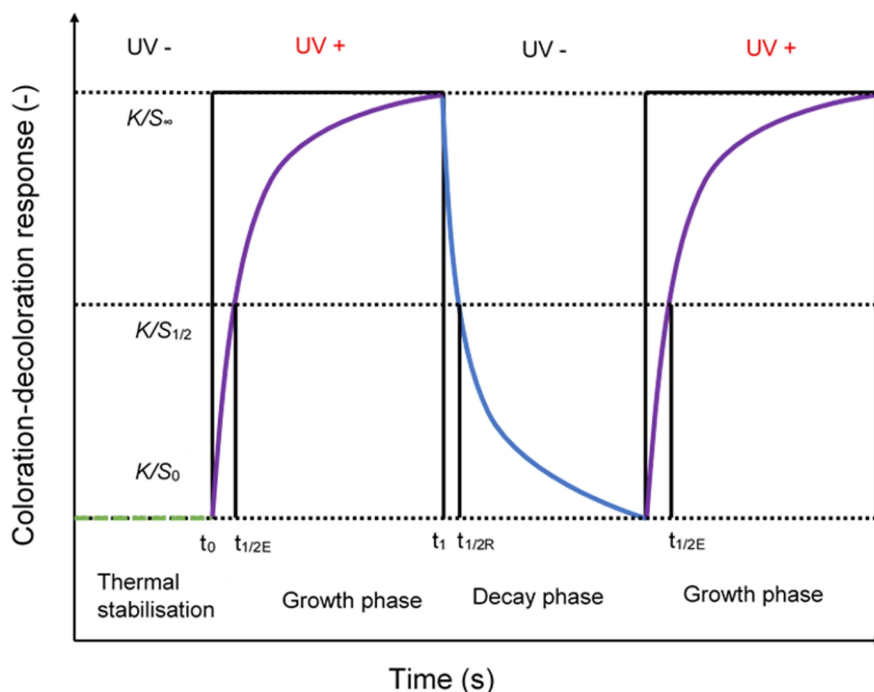


Figure 2 Kinetic phases of the photochromic cycle, where: t_0 = UV+; t_1 = UV-; $t_{1/2E}$ = half-life measure during growth phase; $t_{1/2R}$ = half-life measure during decay phase; $(K/S)_0$ = coloration value at beginning; $(K/S)_{1/2}$ = half of the total coloration value before reaching the equilibrium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

- 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.

- 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 $(0, t)$.

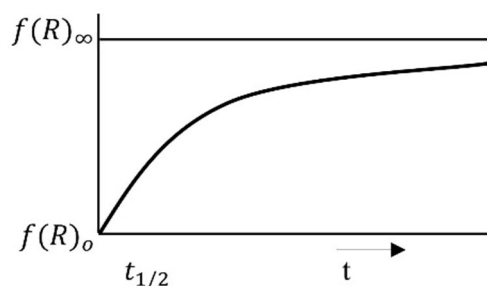


Figure 3 Photokinetic trace – exposure phase

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 $f(R)_0$ 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].

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

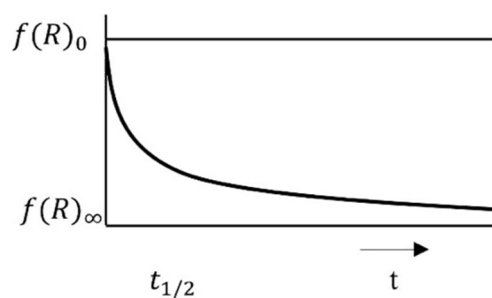


Figure 4 Photokinetic trace - decay phase

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:

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

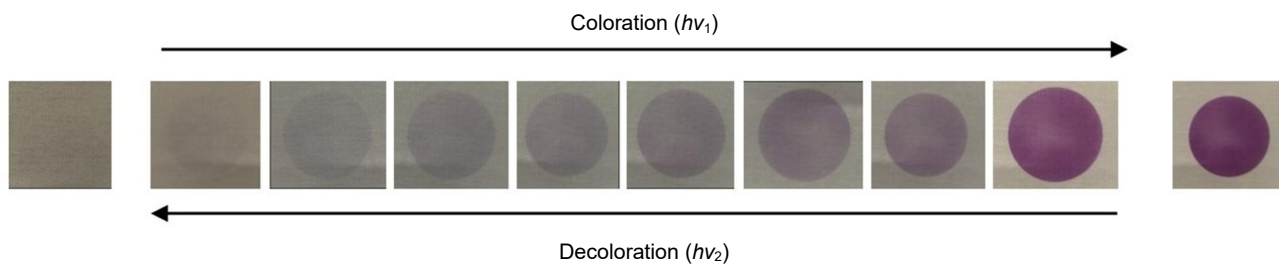


Figure 5 Coloration and decoloration of photochromic system

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 irradiance 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.

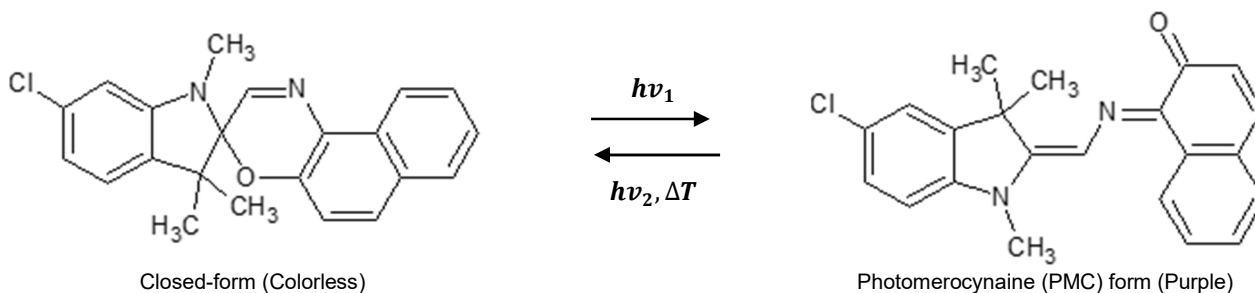


Figure 6 The photochromic transition of used pigment

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}.

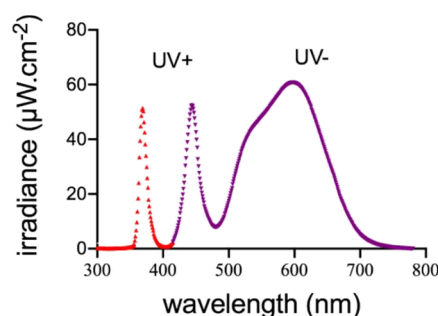


Figure 7 Spectral power distribution (SPD) of used light sources

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.

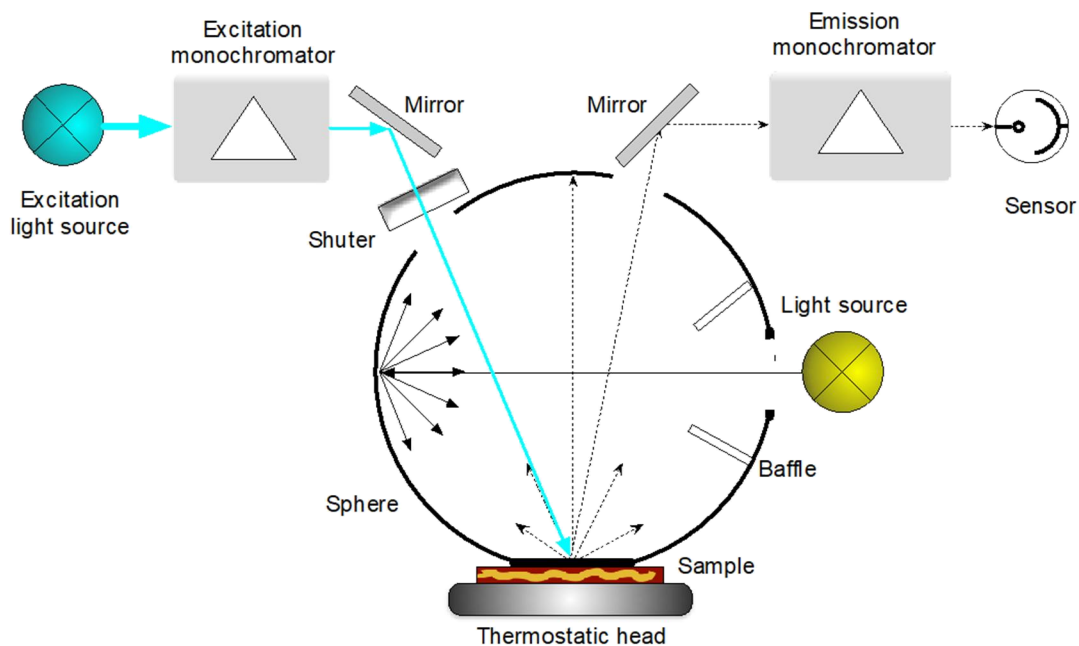


Figure 8 General scheme of FOTOCHROM 3 instrument [16]

3 RESULTS AND DISCUSSION

A new approach for measuring the fatigue resistance of the photochromic system has been described. The photodegradation of used chroimic 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 chroimic molecules under the UV influence. The experimental set-up was kept for five irradiance cycles in a continuous mode (Figure 9).

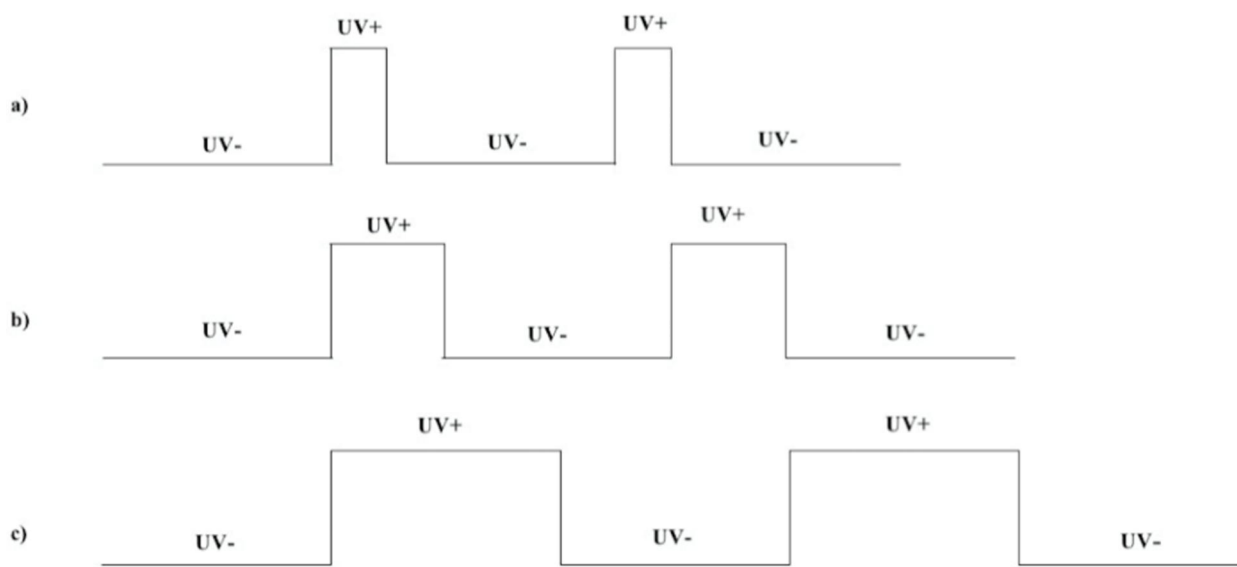


Figure 9 Schematic of the used experimental set-up

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.

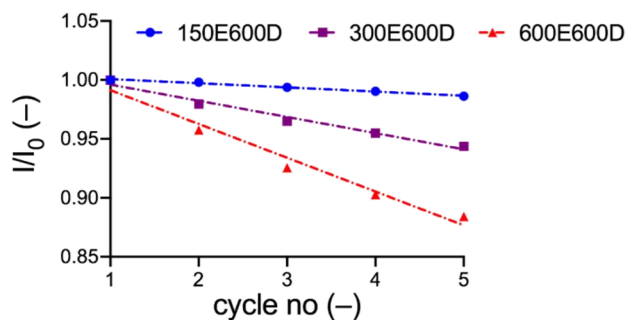


Figure 10 Dependence of I/I_0 and exposure time t of the individual cycle

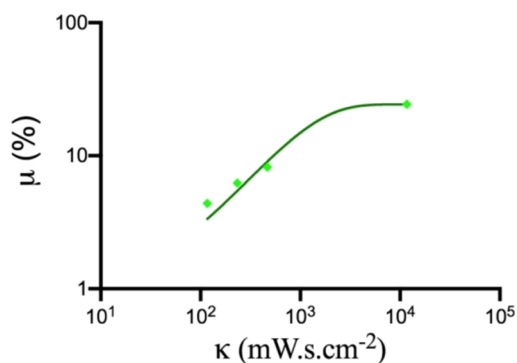


Figure 11 Three exposure times with 600 s decoloration time

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 [%]
	1 th cycle		2 nd cycle		3 rd cycle		4 th cycle		5 th 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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