

Ultrasound-aided Dyeing of Cotton Fabric with Spirooxazines and Photochromic Properties

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Abstract: Photochromic fabric can change color when exposed to a certain light wavelength. The fabric can reversibly restore to its original color after the light disappears. In this study, two spirooxazines with photochromic properties, i.e., N-methyl-3,3-dimethyl-9'-hydroxy-spiro [2H-indole-2-[3H] naphtho [2,1-b] [1,4] oxazine] (Spirooxazine A) and N-methyl-3,3-dimethyl-9'-dichloro-s-triazine group spiro [2H-indole-2'-[3H] naphtho [2,1-b] [1,4] oxazine] (Spirooxazine B), were synthesized. The spirooxazines were characterized by Fourier transform infrared (FTIR) and ultraviolet (UV) spectrophotometers. Spirooxazines A and B were used to dye cotton fabrics under ultrasonic irradiation. The results show that cotton fabric dyed with Spirooxazine B has excellent photochromic properties, and better fading time and fatigue resistance.

Keywords: Spirooxazine, Photochromic properties, Cotton fabric, Dyeing, Ultrasonic irradiation

Introduction

There is considerable interest in photochromic dyes because of their ability to undergo ultraviolet (UV)-induced transformation, thus causing color change, generally from colorless to colored [1,2]. The dyes revert back to their original color, either thermally when the light source is removed, or by irradiation with different wavelengths of light. Currently, photochromic dyes are most widely applied in protective ophthalmic lenses, and could be potentially used in security printing, optical memory and switching, solar energy storage, nonlinear optics and biological systems, cosmetics, and electrophoretic displays [3-6]. There has also been considerable recent interest in the use of photochromic dyes for textiles in view of developing functional or smart products, such as those with the ability to screen UV radiation, used as UV sensors and camouflage, in security printing, for brand protection and authentication, as well as in design applications [7,8].

Among the photochromic dyes, spirooxazines have been extensively investigated due to their ability to impart intense photocoloration in appropriate application media, excellent fatigue resistance and ease of synthesis [9-14]. Spirooxazines are generally colorless, however, they become intensely colored when exposed to UV light, and once again become colorless when the light source is removed. There have been a number of reports on the use of commercial photochromic materials that contain spirooxazines for treatment on textiles by conventional dyeing and screen printing. Spirooxazines have also been incorporated into polymer matrixes to obtain photochromic fibers [15] and made into microcapsules for attachment onto textiles [16-21]. A thin layer of hybrid silica-photochromic dye was coated onto the surface of wool

to produce photochromic wool fabrics by using the sol-gel process, and the durability of the photochromic coated layer was improved by introducing epoxy groups into the silica matrix [11]. Currently, N-methyl-3,3-dimethyl-9'-hydroxy-spiro [2H-indole-2-[3H] naphtho [2,1-b] [1,4] oxazine] (denoted here as Spirooxazine A) a spirooxazine, is frequently applied to natural and synthetic fibers, such as wool, cotton, polyester, and nylon, to obtain photochromic properties [22-27]. However, no chemical bonds are formed between Spirooxazine A and textile fibers, consequently, Spirooxazine A would be removed after washing. Therefore, novel spirooxazine derivatives need to be developed to improve the photochromic effects of Spirooxazine A. In contrast, N-methyl-3,3-dimethyl-9'-dichloro-s-triazine group spiro [2H-indole-2'-[3H] naphtho [2,1-b] [1,4]oxazine] (denoted here as Spirooxazine B), a spirooxazine derivative that contains the -Cl group, can be chemically bonded onto textiles, such as cotton, wool, and silk, because the -Cl group can react with -OH in the textile fibers. However, there have been no reports on the synthesis of Spirooxazine B and dyeing of textiles with Spirooxazine B.

Ultrasonic irradiation is commonly used to enhance a wide variety of chemical and physical processes, such as cleaning, emulsification, degassing, and extraction. In recent years, ultrasonic energy has been also used in textiles to develop and increase the speed of textile processing [28]. It is well known that the use of ultrasonic energy to dye textiles as a means of cleaner production. Ultrasonic energy can improve dye uptake and enhance the dyeing rate under less extreme conditions (e.g., lower dyeing temperature and reduced chemical usage) because of the cavitation phenomena and other mechanical effects, such as dispersion, degassing, diffusion, and intense agitation of the liquid [29].

In this study, two spirooxazines, namely, Spirooxazines A and B, are synthesized. The two spirooxazines are characterized

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by using FTIR and UV. Cotton fabrics are dyed with these two spirooxazines under ultrasonic irradiation. The degree of photochromism, fading time and fatigue resistance of the cotton fabrics dyed with the two spirooxazines are compared. Influence of the concentration of Spirooxazine B on color strength of the dyed cotton fabrics is also investigated.

Experimental

Materials

Plain weave 100 % cotton fabric (58×40 count/cm², 84 g/m²) in white color was used. 2,7-dihydroxynaphthalene (97 %), 1,3,3-trimethyl-2-methyleneindoline (97 %), and cyanuric chloride (97 %) were purchased from Aladdin Reagent Co., Ltd. All chemicals were of analytical grade.

Synthesis of Spirooxazines

The synthesis process of Spirooxazines A and B is shown in Figure 1.

Synthesis of 1-nitroso-2,7-dihydroxynaphthalene

2,7-dihydroxynaphthalene (5.6 g) and sodium nitrate (NaNO₂, 2.8 g) were added to an aqueous solution of sodium hydroxide at -5 °C in a three-necked flask and cooled by using ice. NaNO₂ (2.5 g) and sulfuric acid (12 ml, 50 %) were added under stirring for 70 min. The mixture was stirred at -3 °C for 60 min. The precipitation was filtered and rinsed with deionized water until neutrality was achieved. After that, the precipitation was dried in a vacuum oven at 50 °C. A red-brown colored product was subsequently obtained (95 % yield).

Synthesis of Spirooxazine A

1-nitroso-2,7-dihydroxynaphthalene (5.7 g) was dissolved in ethanol and the solution was heated to 90 °C under a nitrogen atmosphere for 15 min. 1,3,3-trimethyl-2-methyleneindoline (5.2 g) in ethanol was dropwise added into the solution during a time period of 30 min. The mixture was

then stirred for 5 h. After that, activated carbon (5 %) was added and the mixture was stirred for 15 min. The mixture was filtered and the filtrate was evaporated under a reduced pressure to remove the ethanol. The precipitation was placed overnight and then rinsed in ethanol. Finally, the resultant white products were dried in an oven and these products were named as Spirooxazine A (32 % yield).

Synthesis of Spirooxazine B

Cyanuric chloride (0.369 g) was dissolved in acetone (20 ml) and the solution was cooled to 0-5 °C in an ice bath. Then, Spirooxazine A (0.688 g) was added to the mixture and stirred at 5 °C for 2 h. After that, sodium carbonate (NaCO₃, 20 g/l) was added and the mixture was stirred for 1 h. Subsequently, deionized water was added to the mixture which was then filtered. Finally, the precipitation was dried and the resultant product was named as Spirooxazine B (70 % yield).

Dyeing Cotton Fabrics with Spirooxazines A and B

Prior to dyeing, the cotton fabrics were cleaned in deionized water, ethanol, and acetone under ultrasonic irradiation for 10 min to remove grime and other impurities on the surface of the fabrics. Spirooxazines A and B dyes (0.8 g/l) were dissolved in ethanol, respectively. The cleaned cotton fabrics were placed into the solutions and dyed at 50 °C for 30 min under ultrasonic irradiation.

Washing Cotton Fabrics Dyed with Spirooxazines A and B

The cotton fabrics that were dyed with Spirooxazines A and B were washed in 0.37 % detergent with 10 steel balls at 40 °C for 45 min in accordance with the standard method AATCC 61-2003 (Colorfastness to Laundering), and then rinsed in deionized water twice, and finally cured at 100 °C.

Characterization

FTIR spectra of Spirooxazines A and B were recorded as KBr discs with a Nicolet 6700 FTIR spectrophotometer. UV absorption spectra of Spirooxazines A and B that were dissolved in ethanol were measured by using a UV/Vis spectrophotometer (WFZ UV-4802) with the wavelength changed from 200 to 400 nm.

Color measurements of the untreated cotton fabric and those dyed with Spirooxazines A and B were carried out by using a color spectrophotometer (Color i5), illuminated with pulsed xenon (D65 calibrated) with a small aperture plate to measure the specular reflectance and processed by using software. Lightness (L^*), redness/greenness (a^*), yellowness/blueness (b^*), color difference (ΔE^*) and color strength (K/S) values were used to express the color. The color characteristics of the dyed fabrics were evaluated before and after the UV irradiation. For measurement of the photocoloration, the UV light source was set up horizontally and the fabrics

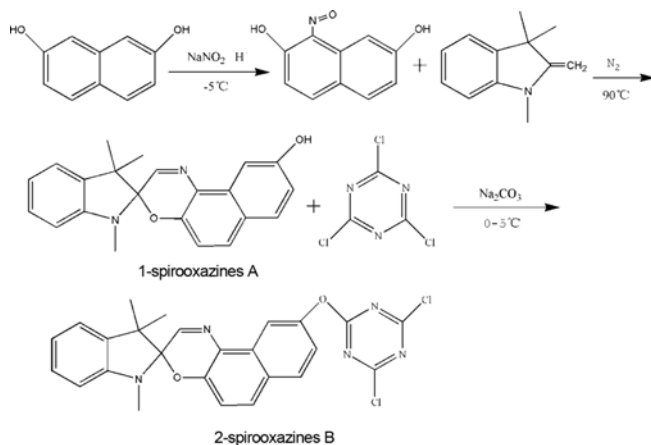


Figure 1. Synthesis process of spirooxazines A and B.

were irradiated under the UV light for 1 min, which used a pulsed xenon bulb with a maximum emission of 254 nm. This was sufficient to develop the full color. After UV irradiation, every fabric appeared to be visually uniform.

To evaluate the fading behavior of the dyed fabrics after UV irradiation, the cotton fabrics dyed with Spirooxazines A and B were irradiated under UV light for 1 min and the ΔE^* values were obtained by color measurements at intervals after removal from the UV light source. Time was recorded as fading time when ΔE^* tended to 0. Fatigue resistance of the dyed cotton fabrics was measured by testing the fading cycle of the dyed fabrics. The dyed fabrics were irradiated with UV light for 1 min and then left in the dark to fade back to their original unexposed states. The ΔE^* values were evaluated after every five irradiation cycles and compared with the values obtained after the original UV exposure. The coloration and decoloration photos of the cotton fabrics dyed with Spirooxazines A and B were taken by a digital camera.

Results and Discussion

FTIR Spectra of Spirooxazines A and B

FTIR spectra of Spirooxazines A and B are shown in Figure 2. In Figure 2(A), the peak at 3230.23 cm^{-1} is ascribed to the -OH groups of Spirooxazine A. The peaks at 1242.66 cm^{-1} and 1080.67 cm^{-1} are attributed to the stretching vibration of the functional groups $\text{C}_{\text{spiro}}\text{-O-C=}$. The absorption band at 979.63 cm^{-1} is due to the stretching vibration of $\text{C}_{\text{spiro}}\text{-O}$ [30]. The stretching vibration of C=N is observed at 1632.59 cm^{-1} . The peaks at 1618.00 cm^{-1} and 1527.39 cm^{-1} are assigned to the stretching vibrations of C=C in the naphthalene ring. The characteristic absorption peak of N=O (aromatic monomer) at 1531.25 cm^{-1} is observed. The peaks at 1489.39 cm^{-1} and 1449.41 cm^{-1} are assigned to the symmetrical and asymmetrical stretching vibrations of the naphthalene ring. The peaks at 1300.15 cm^{-1} and 1242.66 cm^{-1} are assigned to the stretching

vibration of C-O . The peak at 1185 cm^{-1} is assigned to the stretching vibration of Ar-O . The peaks at 898.21 cm^{-1} , 842.04 cm^{-1} , and 744.58 cm^{-1} are ascribed to the bending vibrations of the aromatic ring. All of these absorption peaks indicate that a spiro group exists in the products. Moreover, the results confirm the successful synthesis of Spirooxazine A. In Figure 2(B), the peaks at 1249.39 cm^{-1} and 1084.48 cm^{-1} are attributed to the stretching vibration of the functional groups of $\text{C}_{\text{spiro}}\text{-O-C=}$. The absorption band at 976.44 cm^{-1} is due to the stretching vibration of $\text{C}_{\text{spiro}}\text{-O}$. The peak at 2964.80 cm^{-1} is assigned to the stretching vibration of the C-H bonds. The peak at 1635.96 cm^{-1} is attributed to the stretching vibration of C=N , while those at 1618.17 cm^{-1} and 1515.68 cm^{-1} to the stretching vibration of C=C in the naphthalene ring. The peak at 1413.46 cm^{-1} belongs to the stretching vibration of CH_3 , while those at 1360.49 cm^{-1} and 1299.52 cm^{-1} are caused by the stretching vibration of Ar-N . The peak at 1160.62 cm^{-1} is caused by the stretching vibration of Ar-O , and that at 976.44 cm^{-1} is assigned to the stretching vibration of C-Cl . The peaks at 898 cm^{-1} , 860 cm^{-1} , and 834 cm^{-1} are attributed to the bending vibration of the C-H in the naphthalene ring. The results demonstrate that the cyanuric chloride group has been successfully grafted onto Spirooxazine A, and Spirooxazine B is formed by the chemical reaction between Spirooxazine A and cyanuric chloride.

UV Spectra of Spirooxazines A and B

UV spectra of Spirooxazines A and B are illustrated in Figure 3. In their ring-closed colorless form, the two spirooxazines generally have an absorption peak of moderate intensity in the UVA region (320-400 nm), weak absorption in the UVB region (290-320 nm), and very strong UVC absorption (220-290 nm). It can be observed that Spirooxazines A and B have two peaks when λ_{max} is 240 nm and 345 nm in the UV region respectively. This can be considered as a consequence of the absorption of Spirooxazines A and B. In addition, Spirooxazines A and B show intense UVC absorption,

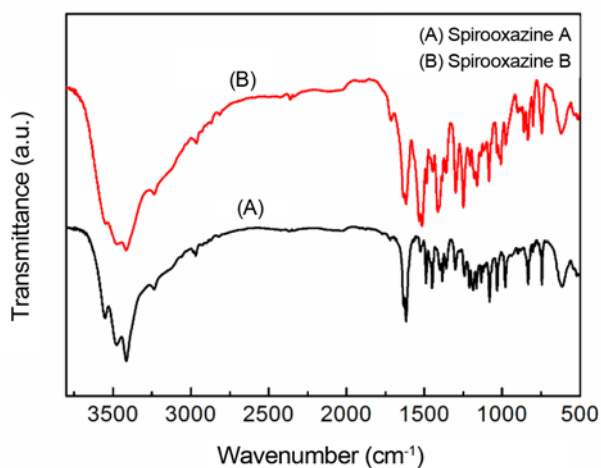


Figure 2. FTIR spectra of spirooxazines A and B.

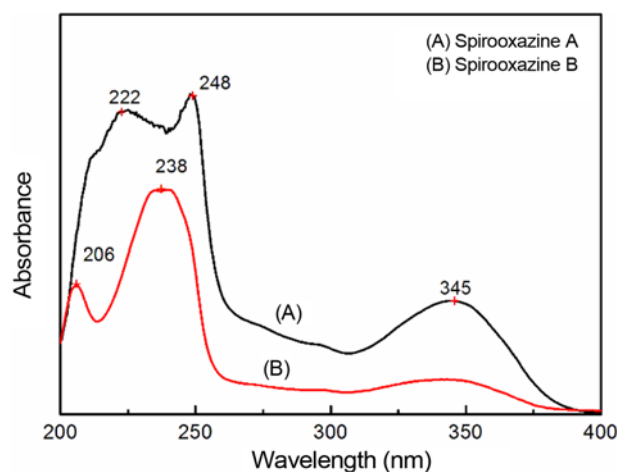


Figure 3. UV spectra of spirooxazines A and B.

especially at around 240 nm, which is much stronger than that in the UVA and UVB regions. The strong absorption at 240 nm is caused by the electronic transitions of indoline of the molecular structure in the spirooxazine. The absorption peak at around 340 nm is attributed to the electronic transitions of naphtha of the molecular structure in the spirooxazine. A weak peak at 206 nm is also observed which is due to the absorption peak of ethanol.

Photochromism of Cotton Fabrics Dyed with Spirooxazines A and B

In order to evaluate the photochromism, color measurements of the cotton fabrics dyed with Spirooxazines A and B after washing were carried out by using a color evaluation instrument with a separate UV source. The color was evaluated from reflectance measurements converted into L^* , a^* , b^* , ΔE^* , and K/S values by using CIE Lab color space, and the results are shown in Table 1. The ΔE^* represents the color difference of the dyed cotton fabric before and after UV irradiation. Images of the original and the dyed cotton fabrics before and after UV irradiation were taken by using a digital camera and are presented in Figure 4.

The cotton fabrics dyed with Spirooxazines A and B changed from white to blue under UV irradiation. Lightness (L^*) was highest for the original cotton fabric, however, the lightness decreased after the cotton fabrics were dyed with Spirooxazines A and B. There were no significant changes in the lightness for the dyed fabrics before and after UV irradiation. In addition, the background color of the dyed fabrics was higher than that of the original fabric. Two factors may influence the background color of the dyed fabrics. One is the degree of thermochromism, thus resulting in the formation of the planar ring-opened and colored form

of the spirooxazine dyes, which become fixed within the crystalline structure of the cotton, thus giving rise to a permanent color. The other factor may be the level of thermal instability of the spirooxazine dyes at elevated temperatures, thus leading to colored decomposition products.

Regardless of whether the dyed cotton fabrics were exposed to UV irradiation, the blueness (b^*) is stronger than the original cotton fabrics. The blue color of the cotton fabric dyed with Spirooxazines A and B before UV irradiation is similar. Moreover, the blueness in the cotton fabrics dyed with Spirooxazines A and B after UV irradiation is stronger than that before the UV irradiation. The cotton fabrics dyed with Spirooxazines A and B have a moderately more pale blue color before UV irradiation and then develop a deep blue color after UV irradiation because the spiro in the molecules of Spirooxazines A and B change into cyanines, which become blue after the colorless Spirooxazines A and B are exposed to UV irradiation. These results suggest that the cotton fabrics dyed with Spirooxazines A and B have excellent photochromism. However, it could be observed that the color intensity and color difference of the cotton fabric dyed with Spirooxazine B are much higher than that dyed with Spirooxazine A after UV irradiation, as shown in Table 1, although the concentrations of Spirooxazines A and B in the dyeing solution were the same. The results indicate that the cotton fabric dyed with Spirooxazine B develops into a deeper blue color under UV irradiation than that dyed with Spirooxazine A after washing as shown in Table 1 and Figure 4. This phenomenon can be explained by a weak van der Waals force which interacts between Spirooxazine A and the cotton fibers. Therefore, Spirooxazine A can be easily removed from cotton fibers after washing. However, the C-Cl groups in cyanuric chloride are active because of the

Table 1. Color properties of original cotton and cotton dyed with spirooxazines A and B before and after UV exposure

Sample		K/S	L^*	a^*	b^*	ΔE^*
Original		0.53	93.20	0.07	3.33	-
Spirooxazine A	Before irradiation	0.52	83.99	1.35	-4.60	-
	After irradiation	0.54	82.09	0.38	-6.28	2.72
Spirooxazine B	Before irradiation	0.55	83.92	1.30	-4.28	-
	After irradiation	0.83	74.82	-6.46	-11.49	13.96

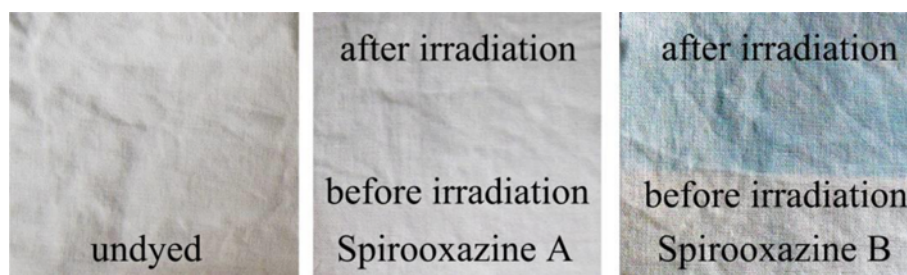


Figure 4. Photos of cotton fabrics dyed with spirooxazines A and B.

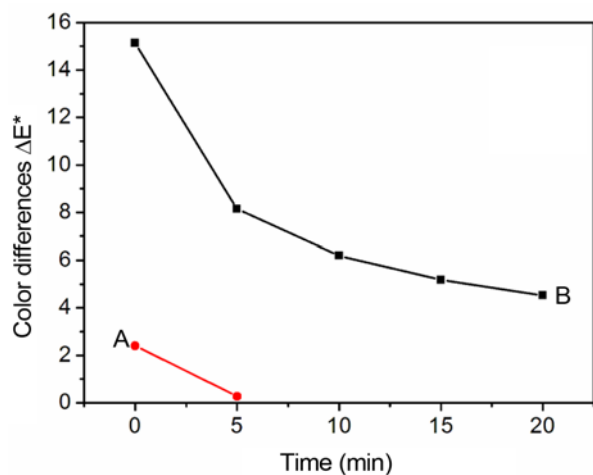


Figure 5. Color difference of cotton fabrics dyed with spirooxazines A and B for different fading times.

strong electron withdrawing effect of the triazine ring. The chlorine atom of cyanuric chloride in the molecules of Spirooxazine B can be replaced by hydroxyl group and C-O-C bonds are formed with an -OH group on the cotton fibers [31]. The formation of covalent bonds between Spirooxazine B and the cotton fibers leads to the improved adhesiveness of Spirooxazine B on cotton fibers. Therefore, Spirooxazine B can be firmly attached to the cotton fibers. Accordingly, the photochromic effect of the cotton fabric dyed with Spirooxazine B is superior to that of the fabric dyed with Spirooxazine A after washing.

Fading Time of Cotton Fabric Dyed with Spirooxazines A and B

The fading characteristics of cotton fabrics dyed with Spirooxazines A and B were investigated by irradiating the dyed fabrics under UV light for 1 min. The ΔE^* (ΔE^* refers to the color difference between the dyed fabrics before and after irradiation) over fading time of up to 20 min in the dark at room temperature was evaluated and the result is shown in Figure 5. The cotton fabrics dyed with Spirooxazines A and B after UV exposure faded after the UV source was removed. The decoloration process of the cotton dyed with Spirooxazine A was markedly faster than that of the cotton dyed with Spirooxazine B. The cotton fabric dyed with Spirooxazine A almost recovered to a white color after 5 min of fading time, while the fabric dyed with Spirooxazine B showed a reduction in color differences with increase of fading time, and it was still blue after 20 min of fading. The fading time of the fabric dyed with Spirooxazine B was significantly longer than that with Spirooxazine A. The slower fading of the fabrics dyed with Spirooxazine B implies that they are in a more restrictive polymeric environment that inhibits the reversion to colorless closed form, a process which involves a significant change in molecular geometry.

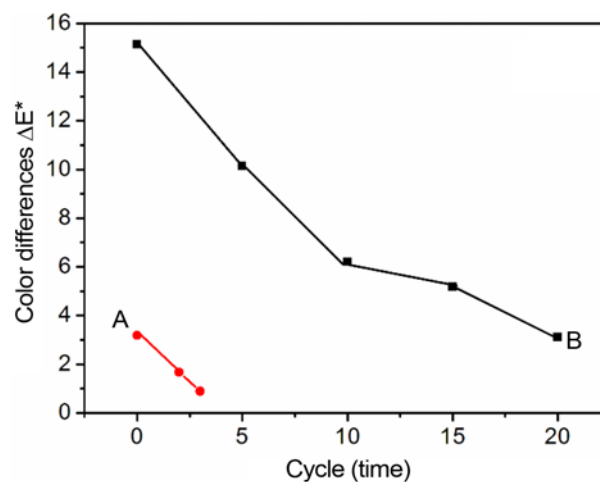


Figure 6. Fatigue resistance of cotton fabrics dyed with spirooxazines A and B.

Therefore, the cotton fabrics dyed with Spirooxazine B show a more durable photochromic effect than those dyed with Spirooxazine A.

Fatigue Resistance of Spirooxazines A and B Dyed Cotton Fabric

Fatigue resistance is one of the important properties for evaluating the service life of photochromic materials. Fatigue resistance was characterized by the number of times that reversible discoloration takes place or the length of its cycle life. The cotton fabrics dyed with Spirooxazines A and B were irradiated under UV light for 1 min and then faded over a period of time in the dark at room temperature. The cycle of UV exposure and fading was repeated 20 cycles, and then the ΔE^* was measured. The ΔE^* obtained after UV exposure is presented in Figure 6. The result shows that the ΔE^* is decreased with an increase in the number of cycles of UV exposure and fading. For the cotton fabric dyed with Spirooxazine A, the ΔE^* is decreased to 0.88 after 3 cycles. However, for cotton fabrics dyed with Spirooxazine B, the ΔE^* is still about 10 after 5 cycles and decreased to 5.01 after 20 cycles. The cotton fabrics dyed with Spirooxazine A would change color after about every 3 cycles, while the cotton fabrics dyed with Spirooxazine B would change color after more than 20 cycles. This indicates that the cotton fabric dyed with Spirooxazine B shows higher fatigue resistance.

Effect of Concentration of Spirooxazine B on Photochromism of Dyed Cotton Fabric

Spirooxazine B was selected for further investigation in dyeing cotton fabric because it has stronger photochromism. In an attempt to improve the degree of photocoloration, the influence of the concentration of Spirooxazine B in the dyeing solution on the color strength of the dyed cotton

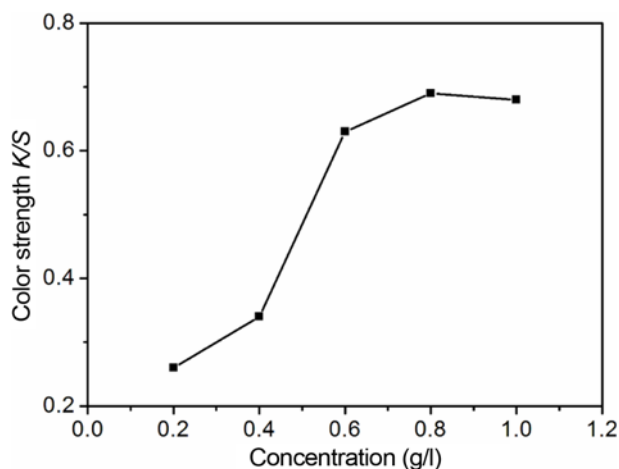


Figure 7. Influence of concentration of spirooxazine B on color strength of dyed cotton fabrics after UV irradiation.

fabric was studied. Figure 7 shows the color strength of the dyed fabrics after 1 min of UV exposure as a function of the dye concentration. The results show that the background color is relatively unaffected. The color strength increases as the concentration of Spirooxazine B is increased from 0.2 to 1.0 g/l. This is because the higher dye solution concentration may result in enhanced dye adsorption by the cotton fabrics and consequently, a higher level of dye diffusion into the cotton fabrics during dyeing. However, no significant increase can be observed for color strength when the concentration of Spirooxazine B is more than 0.8 g/l. One reason may be that Spirooxazine B in its highly non-planar ring-closed form is unable to exhaust further when the concentration is raised, in contrast to the more coplanar ring-opened forms. In addition, Spirooxazine B may be located close to the surface of cotton fabric, thus leading to the suppression of photochromism as a result of aggregation or self-absorption effects at higher concentrations. Therefore, there is no advantage gained when applying Spirooxazine B at a higher concentration than 0.8 g/l.

Conclusion

Two spirooxazines have been successfully synthesized from dihydroxynaphthalene. Spirooxazines A and B are applied to dye cotton fabric under ultrasonic irradiation. The FTIR spectra have confirmed the structure of Spirooxazines A and B. The results show that Spirooxazines A and B have photochromic properties. In addition, the cotton fabric dyed with Spirooxazine B shows better photochromic performance, and develops a higher degree of photocoloration after UV exposure. The cotton fabric dyed with Spirooxazine B dye also has a longer fading time and greater fatigue resistance. The color strength of the cotton fabric dyed with Spirooxazine B is increased when the concentration of Spirooxazine B is

increased in the dyeing solution, but only up to 0.8 g/l.

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