



# Performance Characteristics in Textile Application of Photochromic Dye Capsules

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## ABSTRACT

Photochromic dyes which change their color with UV light, are water insoluble and sensitive to the environmental conditions. In order to be able to use these dyes in textile industry, the dyes are encapsulated with a polymer, and then the encapsulated photochromic dyes are applied onto the textile materials. The encapsulated photochromic dyes can show different properties according to the photochromic dye and polymer type, capsule size and the encapsulation method applied. In this study, the performance characteristics of different commercial photochromic dye capsules were investigated after the application onto cotton fabrics by pad-cure process. It was observed that the fabrics still provide UV protection at 50 + UPF level even after 20 washing cycles. The photochromic fabrics lost their color and UV protective properties at most 16% and 4%, respectively after 20 UV on-off cycles.

## 1. INTRODUCTION

People's expectations from textile materials have changed with the rise of living standards and the developments in technology. The demands for textiles are not only style and durability, but also functional properties. Textile materials, which have functionality in different fields such as medical, protective, sports etc. are getting more interesting and these textiles bring competitive power to the textile companies with their value-added. Photochromic textile materials, which are one of the functional textiles, can be used for both fashion effect and smart textiles such as UV sensors [1].

Photochromic dyes are colorless without UV light and they become colorful by changing their molecular structure under UV irradiation. This color changing is reversible thermally (T-type) or photochemically (P-type), thus the color disappears when the UV light source is removed [2]. Photochromic dyes are divided in many different classes while the photochromic dyes, which change their color based on pericyclic reactions, are more common to use in various industries such as glasses, cosmetics, plastics, textiles etc [1,3]. These photochromic dyes, which are spiropyrans, spirooxazines, naphthopyrans (chromenes), diarylethenes and fulgides, show photochromism effect by ring opening/closing reactions (Figure 1) [2,4,5]. These

dyes differ in terms of photochromic properties, resistance to photodegradation (fatigue resistance), etc., depending on their structures [6]. For example, diarylethenes and fulgides show P-type photochromism, while the other dyes show T-type photochromism [7]. Spirooxazines have higher fatigue resistance than spiropyrans [6]. Naphthopyrans is less sensitive to temperature than spirooxazines [8,9]. In this context, it should also be evaluated whether the photochromic dye has suitable properties for the usage area where it will be applied.

Photochromic dyes also provide UV protection property due to their color changes by absorbing UV light and can be used in the UV protective textile production [10]. UV rays above a certain rate is harmful to human health and precautions such as using sunscreen, dressing, etc. can be taken to protect against UV rays. Although textile materials provide UV protection by absorbing or reflecting UV rays, not all textile materials provide the same protection level. In order to evaluate the UV protection property provided by textile materials, the UV protection factor (UPF) value should be analyzed. UPF is the ratio of the average effective UV rays in the environment ( $ED$ ) to the average of the UV rays passing through the textile material ( $ED_t$ ) [11]:

**To cite this article:** Morsümbül S, Akçakoca Kumbasar EP. 2022. Performance characteristics in textile application of photochromic dye capsules. *Tekstil ve Konfeksiyon*, 32(2), 155-161.

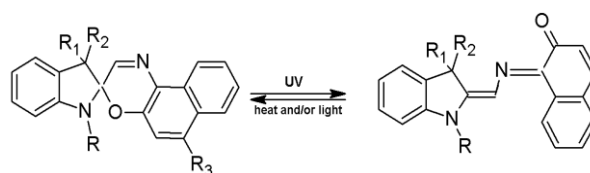
## ARTICLE HISTORY

Received: 25.05.2021

Accepted: 23.05.2022

## KEYWORDS

Photochromism, cotton, UV protection, washing durability, fatigue resistance



(R: alkyl; R<sub>1</sub>, R<sub>2</sub>:alkyl (usually both methyl); R<sub>3</sub>: H, halogen, etc.)

**Figure 1.** Reversible color change of photochromic dye (spirooxazine) with UV light [2]

$$UPF = \frac{ED}{ED_f} = \frac{\sum_{\lambda=290}^{\lambda=400} E(\lambda)S(\lambda)\Delta\lambda}{\sum_{\lambda=290}^{\lambda=400} E(\lambda)T(\lambda)S(\lambda)\Delta\lambda}$$

where  $E(\lambda)$  is the relative erythemal spectral effectiveness,  $S(\lambda)$  is the solar spectral irradiance in  $Wm^{-2}nm^{-1}$ ,  $\Delta\lambda$  is measured wavelength interval in nm,  $T(\lambda)$  is average spectral transmittance of the fabric specimen, and  $\lambda$  is the wavelength in nm.

In the UV transmittance measurements, four data about UV protection properties of the fabric are obtained. These are the mean UPF, rated UPF, UVA and UVB transmittance values. Mean UPF is the UPF value obtained as a result of averaging the UV transmittance measurements made from 4 different points of a fabric. Mean UPF can be measured up to 2000 UPF, depending on the capacity of the measuring device.

The rated UPF value is the average UPF value of four testing fabrics reduced for the standard error in the average UPF, calculated for the 99% confidence level, and finally rounded down to the nearest multiple of five. If the rated UPF is less than the lowest individual UPF fabric measurement, the rated UPF is the lowest value of measured UPF rounded to the nearest multiple of five [12,13]. UVA and UVB transmittance values are the values that show how much percent the fabric transmits UVA and UVB rays, respectively.

Although many different standards are applied in the UPF analysis of textile materials, the most widely used standard is the Australian / New Zealand standard (AS / NZS 4399: 1996). According to this standard, which classifies the UPF values as in Table 1, textile materials with 40 UPF and above provide excellent protection against UV rays [10–12].

**Table 1.** Classification of UPF values according to AS / NZS 4399: 1996 standard [10–12]

UPF Range	Protection category	Effective UV Transmission (%)	UPF Rating
40 – 50, 50+	Excellent	≤ 2,5	40, 45, 50, 50+
25 – 39	Very good	4,1 – 2,6	25, 30, 35
15 – 24	Good	6,7 – 4,2	15,20

UV protection properties of textile materials depends on many parameters such as fiber type, fabric construction, additives (e.g. UV absorber) in the material etc. Color is also one of the most important properties that affect UPF of textiles. A number of authors has studied UV protection properties of textile dyes [14–21]. However, UV protection properties of photochromic dyes, which change their color

by absorbing UV light, on textile materials have not been studied sufficiently. Thus, in this study, the photochromic textiles were also evaluated from the point of UV protection.

Various application methods have been used to apply the photochromic dyes to textile materials such as embedding the dye in the polymer matrix during the spinning (photochromic yarn) [22–24] or screen-printing process (photochromic T-shirts) [1,25]. In addition to this, several researchers have studied on the application of spirooxazine or naphthopyran dyes onto polyamide, polyester, polyacrylonitrile and cotton fabrics by different methods such as exhaustion, padding or printing [26,27,36–38,28–35].

Although the first photochromic t-shirt was put on the market in 1989 [1], photochromic dyes could not attract the expected attention from the textile industry sufficiently due to their disadvantages such as low water solubility, sensitive structure to high temperature, low affinity to the textile materials and low diffusion ability to high crystalline structures such as synthetic fibers [28,30,33,36]. Thus, textile-dyeing yield of the photochromic dyes are low due to these properties. Microencapsulation can be applied as an alternative method to improve the application of photochromic dyes onto textile materials.

Microencapsulation is a process in which very small particles of liquid or solid material (core material) are coated with a continuous film of polymeric material (shell material) [39]. Capsule shell materials protect the core materials from external factors by wrapping the core material with one or more layers and improve the applicability of the core material by increasing its stability [40,41].

In this study, it was aimed to apply two different commercial photochromic dye microcapsules on cotton fabrics and evaluate the color build-up by UV irradiation, fatigue resistance and UV protective properties of the fabrics after the microcapsule application and consecutive laundering. It was observed that UV protective properties of cotton fabrics can be improved with the application of photochromic dye microcapsules and the photochromic fabrics can retain their properties even after 20 repeated washing and UV on-off cycles.

## 2. MATERIAL AND METHOD

The scoured and bleached interlock knitted 100% cotton fabric was obtained from a textile factory and was used as received (without any further treatment prior to the experimental part). Construction properties of the fabric were shown at Table 2. Thickness of the fabrics was measured on SDL ATLAS Digital Thickness Gauge according to ASTM D 1777-96 standard. Total porosity of the fabric was calculated by using Equation 1 [42,43].

$$\varepsilon = 1 - \frac{\rho_a}{\rho_b} \quad (1)$$

where  $\rho_a$  is the fabric density ( $\text{g/cm}^3$ ),  $\rho_b$  is the fiber density ( $\text{g/cm}^3$ ) and  $\varepsilon$  is the porosity. Fabric density is calculated by dividing the fabric mass per unit area, by fabric thickness. The mean density of cotton fibres is accepted as  $1.52 \text{ g/cm}^3$  [42].

The binder (Itobinder AG - anionic acrylic copolymer) was supplied by LJ Specialities Ltd. Two different types of commercial photochromic dye microcapsules (Violet-powder and Blue-liquid) were used in powder and liquid form. Trade names and structures of the capsules are not revealed for proprietary reasons.

The photochromic dye capsules (50, 75 and 100 g/l) were applied to the fabric by pad-cure process. Wet pick up ratio was set to be 100%. The binder concentration was 50 g/l. After padding, the samples were dried at  $100^\circ\text{C}$  for 3 minutes and cured at  $150^\circ\text{C}$  for 4 minutes in a laboratory scale tenter (Ataç, GK 40, Turkey).

The morphology of the fabrics was investigated by scanning electron microscopy (SEM, Thermo Scientific Apreo S). Each sample was coated with a 1 nm thick Au layer using sputter coater (Leica EM ACE600) prior to SEM observations.

Color measurements were carried out using a Colorlite SPH 870 spectrophotometer (with LED light source), processed using the ColorLite ColorDaTra Professional software, 400-700 nm, under D65 illumination and an observer angle of  $10^\circ$  with a  $45^\circ/0^\circ$  geometry. The samples were placed on the plate kept at constant temperature ( $20^\circ\text{C}$ ) by a peltier system and then the samples were irradiated by solar simulator (ABET Sunlite Solar Simulator) for 3 minutes (Figure 2). After that, the solar light switched off and color measurements were carried out as immediate as possible (within 3 seconds). The color value of unirradiated sample was regarded as the standard. Color build-up on irradiation of the samples was discussed by the difference between the standard and UV irradiated colored samples by using  $\Delta E^*$  (color difference) values.

The solar simulator has a Class A spectral match for the International Electrotechnical Commission (IEC) and the Japanese Industrial Standards (JIS) Committee standards and has an irradiance of AM 1.5G,  $100 \text{ mW/cm}^2$  (1 Sun).

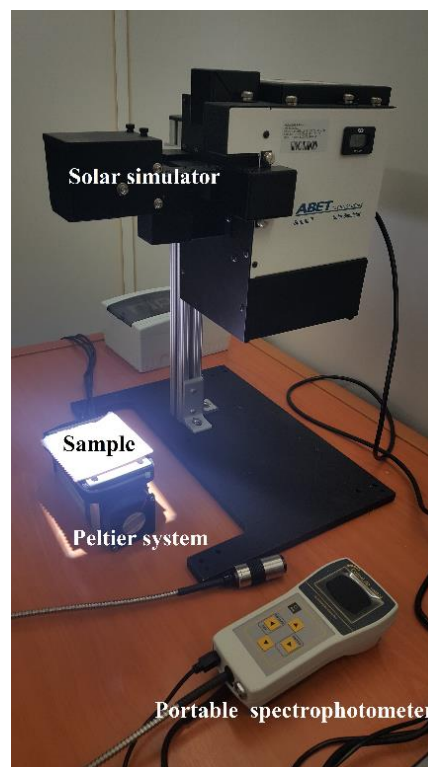


Figure 2. The color measurement setup

Mean UV protection factor (UPF) values of the fabrics were measured with Labsphere UV 2000F device according to standard AS/NZ 4399:1996.

Consecutive laundry washings were applied by ISO 105-C06:2010 (test method A2S) standard for testing the washing durability of the applications. The fastness assessment method with grey-scale is not a suitable for photochromic materials due to the dynamic color changes of photochromic dyes [34]. Therefore, the  $\Delta E^*$  and the mean UPF values of the samples were compared to evaluate the washing durability.

The fatigue resistance tests were carried out based on the literature [36,38]. The samples were irradiated by solar simulator for 2 min and then left in the dark for 5 min to fade back to their original unexposed states. This UV on-off cycle was repeated 20 times for each sample. Color and UV transmittance measurements were carried out after every 5 irradiation cycles.

Table 2. Construction properties of the fabric used in the application studies

Yarn count	Course per cm	Wales per cm	Stitch density	Thickness	Mass per unit area	Total porosity
12 tex	20	40	800 stitch/cm <sup>2</sup>	0,085 cm	0,023 g/cm <sup>2</sup>	83%

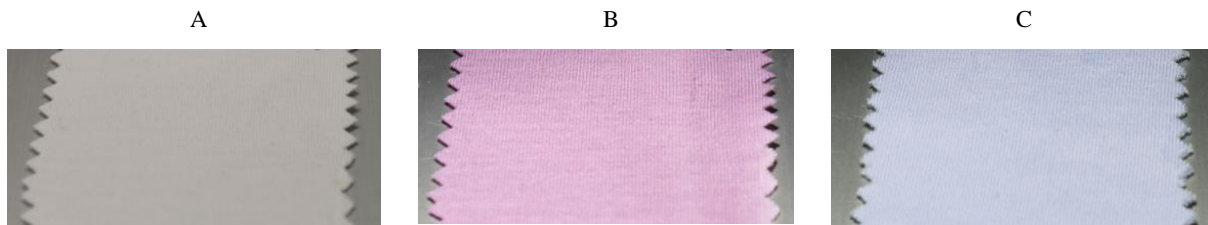
### 3. RESULTS AND DISCUSSION

The photochromic capsule applied samples were in a colorless state (white) in the absence of UV light. Reversible violet and blue colors were developed after UV irradiation (Figure 3) and the samples reverted to the colorless state when the UV irradiation source is removed.

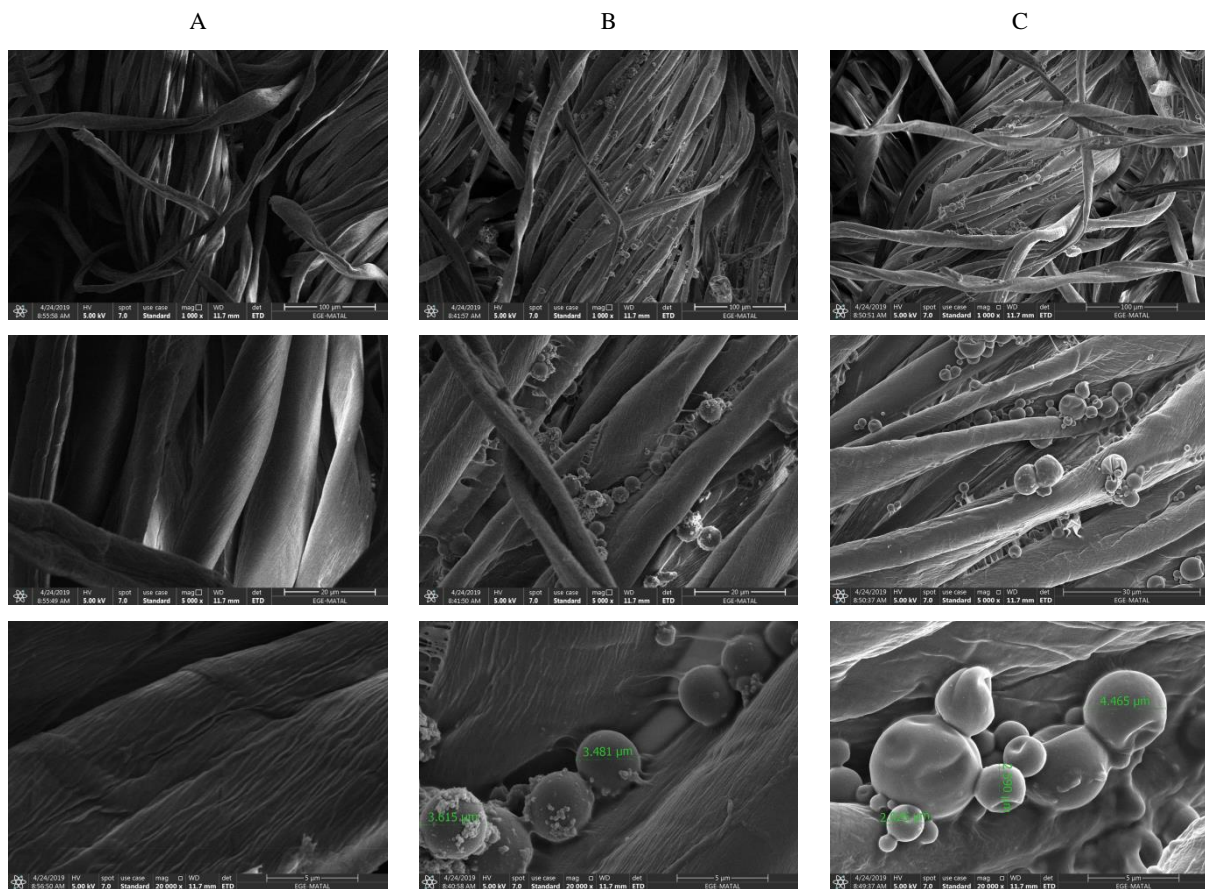
SEM images of the fabrics (Figure 4), recorded at three different magnifications (x1000, x5000 x20000), clearly showed that the microcapsules adhered on cotton fibers without obvious breakage during the pad-cure process. The particle size of the photochromic microcapsules ranged from 2 to 5  $\mu\text{m}$  (Figure 4).

As stated by Little and Christie [34], the washing fastness evaluation by comparing with grey scales used for classical textile materials is not suitable for photochromic textile materials that change their color with UV irradiation. In this

context, considering the studies in the literature, the difference between the color values of the fabrics before and after UV irradiation was evaluated after repeated washings in order to examine the washing durability of the microcapsules [34]. Figure 5 illustrates  $\Delta E^*$  values of the samples after padding and consecutive laundering tests. It was observed that the color build-up on UV irradiation of the samples increased with capsule concentration. The level of photocoloration developed by UV irradiation of the samples was observed to decrease at most by 11% after 20 washings. However, some samples have also showed an increase in color values after repeated washing cycles. An explanation for this result may be proposed based on the loosening of the binder structure on the fabric after washings. It provides a more favorable environment for the color change of the photochromic dye by disappearing the film effect of the binder on the microcapsule surface and also increasing the deaggregation of the microcapsules [34].



**Figure 3.** Images of the photochromic microcapsules applied samples before (A) and after UV irradiation (B: violet powder and C: blue liquid)



**Figure 4.** SEM images of the untreated fabric (A) and the photochromic microcapsules applied samples (B: violet powder and C: blue liquid) with different magnifications (x1000, x5000 x20000)

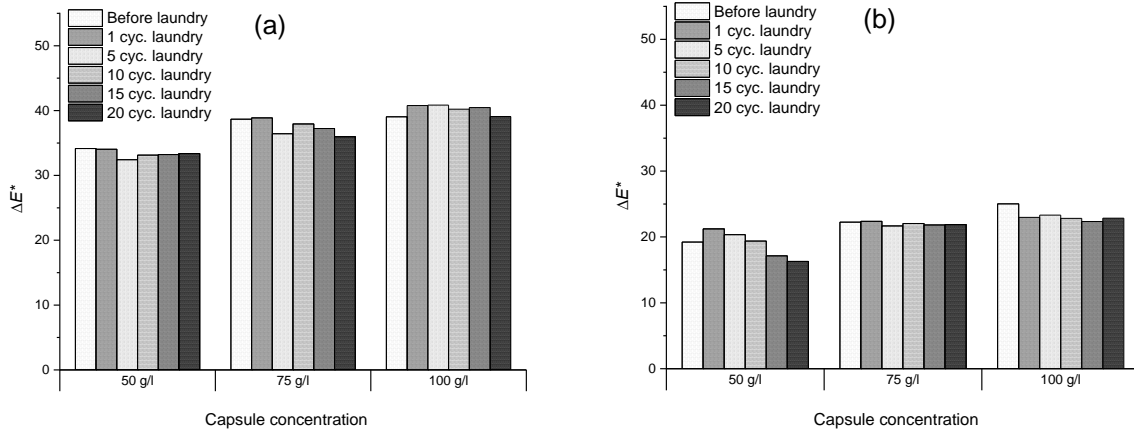


Figure 5.  $\Delta E^*$  values of the samples (a: for violet-powder, b: for blue-liquid) after padding and consecutive laundering tests.

One of the aims of this study is to evaluate the UV protection performance of photochromic textile materials. 50+ mean UPF values were obtained as a result of the photochromic capsule applications as seen in Figure 6, while the mean UPF value of the untreated fabric was 29 (before laundry tests). The mean UPF values also increased with the increasing capsule concentration. In addition, 50+ UPF values were again observed in all photochromic fabric samples even after 20 laundering cycles, while the mean

UPF value of the untreated fabric was about 25-30 UPF after the consecutive laundry tests.

$\Delta E^*$  and UPF values of the samples for every 5 UV on-off cycles were given in Figure 7. Based on the  $\Delta E^*$  values, the samples applied violet-powder and blue-liquid capsules (100 g/l) showed high fatigue resistance after 20 UV on-off cycles, retaining 97% and 84% of their photochromic response, respectively. The samples also showed approximately 4% loss for violet-powder and 0.5% loss for blue liquid in UPF values after 20 UV exposure cycles.

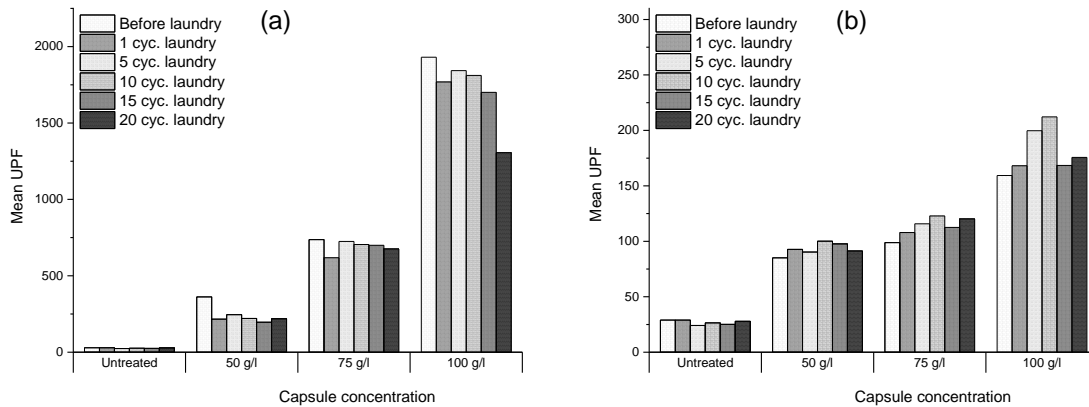


Figure 6. Mean UPF values of the samples (a: for violet-powder, b: for blue-liquid) after padding and consecutive laundering tests.

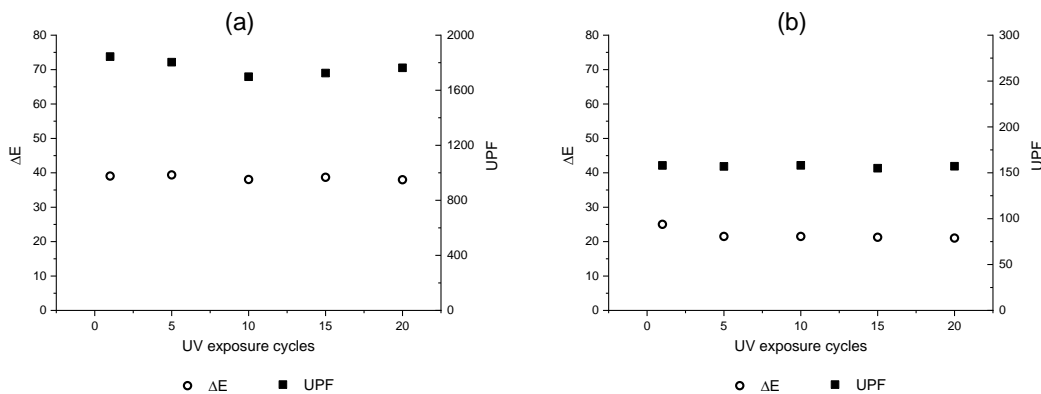


Figure 7. Fatigue resistance of the samples (a: for violet-powder, b: for blue-liquid).

#### 4. CONCLUSION

Application of two different microcapsules on cotton fabrics and evaluation the performance properties of the fabrics were presented in this study. Photocoloration degree of the samples decreased at most by 11% after the washings however, the color values of some samples increased after repeated washing cycles. An explanation proposed is that the binder structure on the samples loosens around the microcapsules with washings, and thereby the conversion between close and open ring forms of the dyes facilitates. The capsule concentration affected the color and UV protection properties of the fabrics positively and this effect is more pronounced for UPF values. Application of the photochromic dye microcapsules was found to improve UV protective properties of cotton fabric.

In general, the advantages of this study are as follows:

- Application conditions of the photochromic microcapsules on cotton fabric are presented.
- The performance properties of the photochromic microcapsules on cotton fabric were evaluated.
- It has been revealed that photochromic textile materials can be applied not only in creating a fashion effect, but also in the production of UV protective clothing as a functional textile material.
- It has been shown that the photochromic fabrics maintained their color and excellent UV protection (50+ UPF) properties after repeated washing and UV on-off cycles.

#### ACKNOWLEDGEMENT

The authors would like to gratefully acknowledge Ege University, scientific research projects through the project no. 18-TKUAM-003 for financial support to this research project.

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