


Imidazolium and Ammonium-Based Ionic Liquids for Reactive Dyeing

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ABSTRACT

In this study, the effect of ionic liquids on the dyeing of cotton fabrics with reactive dyestuffs was investigated. For this purpose, 100% cotton fabrics were treated with imidazolium (1-ethyl-2,3-dimethylimidazolium ethyl sulfate- E) and ammonium (methyl-tri-n-butylammonium methyl sulfate-B) based ionic liquids with three different concentrations. To assess the effect of the treatments, the cotton fabrics were characterized by FT-IR, SEM and DSC. After this treatment, the cotton fibers were dyed with 1.0% o.w.f reactive dye. The K/S values were determined to evaluate the effect of ionic liquids on the dyeability of cotton fabrics and fastness analyses to washing, water and rubbing were conducted to examine the end-use properties of dyed fabrics. The results revealed that ionic liquids settled on the fabric surface and after each ionic liquid modification, the increasing intensity was observed with increasing ionic liquid concentrations at broad bands according to the FT-IR analysis. It was determined that the K/S value of the modified cotton fabric increased with the increase in ionic liquid E concentration, especially. Considering the overall results, it is possible to state that ionic liquids which are implemented in this study are efficient in enhancing the dyeability of the cotton fabrics.

1. INTRODUCTION

The dyeing of textile materials that can differentiate their aesthetic appearance and texture creates a great impact on the environment due to the high amounts of water, energy, and chemical additives. The chemicals are toxic and not easily biodegradable [1] after dyeing processes, if the wastewater is not treated and dumped into rivers, this also causes severe ecological and human threats [2]. Many efforts have been made to find and optimize more ecologically friendly methods for conventional dyeing

processes. Recently, green solvents have been studied to enhance the dyeability of textile fibers. Ionic liquids are known as green solvents, which are highly effective in improving the UV resistance, thermal insulation, and flammability of different textile materials [3–7]. In recent studies, it is seen that ionic liquids are used for water/oil emulsion separation [8-13]. Ionic liquids have been used as alternative agents in many applications, which require chemical stability, thermal stability, and ionic conductivity. Ionic liquids are also implemented to dissolve cellulose to produce regenerated cellulose fibers [14–16]. Recently, the

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studies have been conducted with ionic liquids to enhance dye absorption and dyeing behavior of different textile fibers and facilitate the removal of dyes from aqueous solutions [17–19].

Dyeing processes of fabrics are enormously important for the marketing of textiles. This process is known as the aqueous application of dyes or pigments to textile materials, mainly using synthetic substances and chemical additives with an incredibly large amount of water. Most of the practical chemicals in this process are extremely toxic and not readily biodegradable [1, 20–24]. From this perspective, the better solution seems to be the innovation of both textile processing technologies and related chemistry to reduce the effluent pollution. In the recent years, in order to reduce water and chemical consumption and waste discharge, some processes have been employed for nonaqueous dyeing processes. For this purpose, ionic liquids can be used for the improvement in the substantivity and wettability of the fibers by penetrating active species [25, 26]. Thus, it is thought that darker dyeing can be obtained with less dyestuffs. Also, in recent studies ionic liquid becomes important to find solutions to reduce water consumption and discharge of polluting chemicals [27, 28].

In this study, the cotton fabric was firstly treated with two different ionic liquids (imidazolium and ammonium based) and then dyed with the reactive dyestuffs. The effect of ionic liquids was characterized by SEM, FTIR and DSC before dyeing. Then, the dyeing performances of the treated

cotton fabrics were investigated. After the dyeing procedure, fastness of the dyed samples and color strength analyses were determined. When this study is compared with the literature, it is realized that the imidazolium and ammonium-based ionic liquids have not been used for dyeing cotton products before. In addition, when the dyeing studies with ionic liquids were examined, it was determined that there were researches on color measurement and dye absorption in these studies. Apart from these previous studies, the end-use properties of the dyed fabrics were also evaluated by conducting fastness analyzes in this current research. The proposed process, being conducted at dyes lower than those usually employed, assures significant chemical savings. Thus, it is aimed to obtain dyeing in darker tones with the usage of lower dyestuffs.

2. MATERIAL AND METHOD

2.1 Material

In this study, 100% cotton fabric (warp/weft density of 29/25 yarns/cm and 240 g/m²) was used. 1-ethyl-2,3-dimethylimidazolium ethyl sulfate and methyl-tri-n-butylammonium methyl sulfate were obtained from Sigma-Aldrich Co. The chemical formulations of the ionic liquids are given in Figure 1. Dyestuffs kindly provided by Sigma Aldrich Co. (USA) included Cibacron Red FN-R (CI number: Reactive Red 238) as a reactive dye. Na₂CO₃, Na₂SO₄, and CH₃COOH were supplied by Merck (Germany).

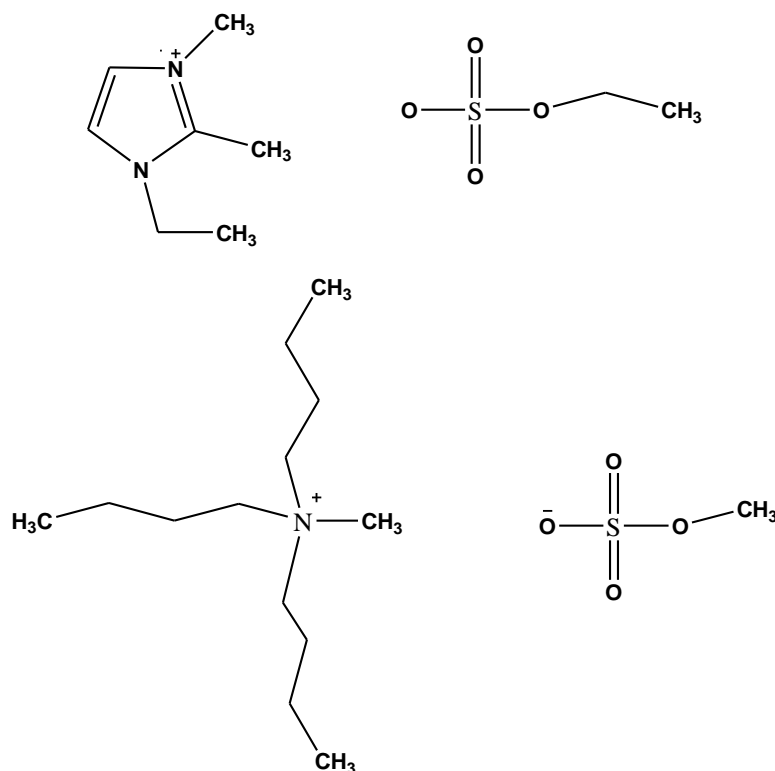


Figure 1. The chemical formulas of the ionic liquids such as a) 1-ethyl-2,3-dimethylimidazolium ethyl sulfate, b) methyl-tri-n-butylammonium methyl sulfate

2.2 Method

2.2.1 Fabric treatments

Before treatment of the cotton fabrics, 1-ethyl-2,3-dimethylimidazolium ethyl sulfate (E) was dissolved in ethanol 2, 6, and 10% w/v volumes and is stirred continuously with a magnetic stirrer until completely dissolved. The same process was implemented for methyltri-n-butylammonium methyl sulfate (B) to dissolved in ethanol in 10, 15, 20 wt%/v concentrations [5, 6]. Then, the cotton fabrics were treated in these solutions for 5min at ambient temperature. Then, the excessive solution is removed from the fabric and the cotton fabrics were left to dry at room temperature. Table 1 lists the sample codes of the untreated and treated cotton fabrics.

Table 1. Fabrics sample descriptions

Fabric code	Fabric details
C	Unmodified cotton fabric
CE2	Fabric modified with 2 % w/v E/ethanol
CE6	Fabric modified with 6 % w/v E/ethanol
CE10	Fabric modified with 10 % w/v E/ethanol
CB10	Fabric modified with 10 % w/v B/ethanol
CB15	Fabric modified with 15 % w/v B/ethanol
CB20	Fabric modified with 20 % w/v B/ethanol

2.2.2 Dyeing procedure

The cotton fibers were dyed 1.0% o.w.f by using reactive dyes. Dyeing process for the reactive dye is represented in Figure 2. 60 g/l Na₂CO₃ and 14 g/l Na₂SO₄ were used for the dyeing. The washing procedure after dyeing was washing at 50°C for 10 min with distilled water, washing at 50°C for 10 min with CH₃COOH, washing at 90°C with a cleansing agent (Eriopon - Huntsman) for 15 min., washing at 50°C during 10 min and washing at room temperature during 10 min, respectively. The liquor ratio was kept at 20:1.

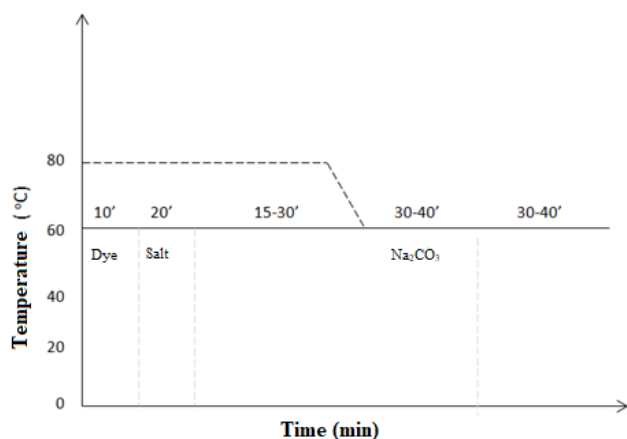


Figure 2. Dyeing process reactive dye

2.2.3 Characterization

The morphological structure of the cotton fabrics was examined by an FESEM FEI Nova Nanosem 650 model scanning electron microscope. SEM images were taken with a voltage of 5 kV. Before the examination, the cotton fibers were coated with Au-Pd alloy by sputter coating.

A Perkin Elmer Spectrum BX instrument ATR-FTIR was used to define the effect of ionic liquid modification on cotton fabrics. The spectra were taped in the range of 650–4000 cm⁻¹ wave number with a resolution of 2 cm⁻¹.

A Perkin Elmer Pyris Diamond DSC was utilized for differential scanning calorimetric. DSC experiments were performed on all samples at a heating rate of 5°C/min, using dry nitrogen (N₂) as the carrier. Scans are started from a temperature of 20°C, reaching a final temperature of 80°C. The melting temperature of the ionic liquid is in the range of 50-60°C. DSC analysis was carried out at temperatures up to 80°C in order to see the melting peaks clearly and to ensure a homogeneous melting

Color strength values of dyed samples were evaluated using a CM-3600d model Minolta Spectrophotometer (D65 daylight and 10°). All samples were stated at under standard atmospheric conditions (65% relative humidity and 20°C). Color strength values were calculated by the Kubelka-Munk equation: (Equation 1).

$$K/S = (1 - R)^2 / 2R \quad (1)$$

Where, S is the absorption coefficient, K is the scattering coefficient and R is the reflectance.

The color difference (ΔE^*) is evaluated by the following: (Equation 2).

$$\Delta E^* = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{1/2} \quad (2)$$

where ΔE^* is the color difference among the sample and the standard. ΔL^* signifies the difference among darkness (where min. $L^* = 0$) and lightness (where max. $L^* = 100$), Δa^* the difference among red (+a*) and green (-a*), Δb^* is the change among blue (-b*) and yellow (+b*).

Fastness to washing, fastness to water and fastness to dry and wet rubbing of the fabrics were evaluated according to ISO 105-C06 A1S, ISO 105-E01 and ISO105-X12. The tests were performed using the subsequent instruments: SDL Atlas Linitest for washing, SDL Atlas M231/PR1 Perspirometer for water fastness and SDL Atlas rubbing fastness tester for dry and wet rubbing. SDC multifibre tape fabric was used as adjacent material. In the washing fastness tests, ECE non-hosphate detergent was used.

3. RESULTS AND DISCUSSION

Figure 3 and 4 show SEM micrographs of the cotton fabrics. SEM images present that cotton fibers have a rough surface structure. This is due to the existence of impurities and some micro-cracks possibly caused by stretching in the yarn spinning and fabric manufacturing processes. Subsequently ionic liquid treatment, there becomes an

observable coating layer both in the gaps and on the surface of the yarns [5, 6]. The coating layer becomes more featured with increasing volume of the ionic liquid, particularly for B coded ionic liquid-modified cotton fabrics (Figure 4d).

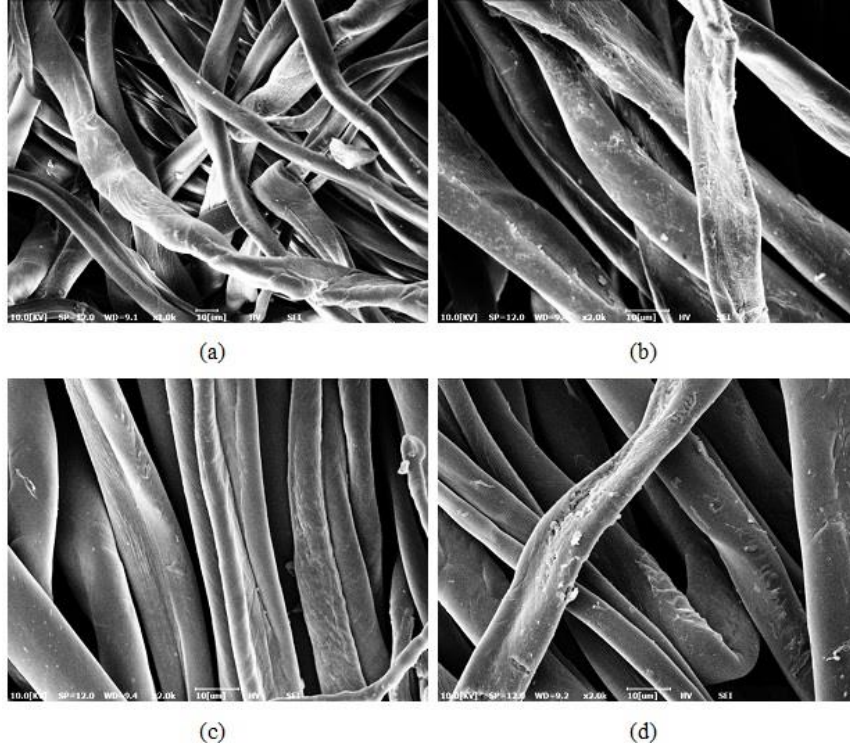


Figure 3. SEM images of (a) C, (b) CE2, (c) CE6, (d) CE10

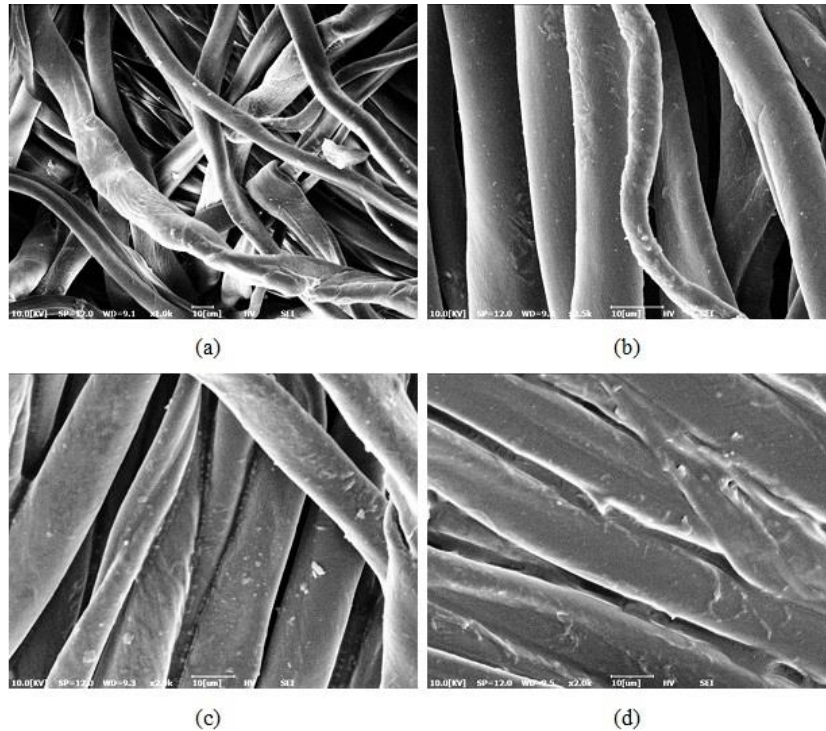


Figure 4. SEM images of (a) C, (b) CB10, (c) CB15, (d) CB20

Figures 5a and 5b represent the FTIR spectra of E and B modified cotton fibers in comparison with the untreated one, respectively. As seen in Figures 5a and 5b, natural fiber shows characteristic functional groups related to cellulose, hemicellulose in cotton fiber. After each ionic liquid treatment, the increasing peak intensity was observed with increasing ionic liquid concentrations associated with the introduced amino groups into the fibers. Increasing amino group content in the fiber increases the functional groups and this produces an increase in the intensity of peaks [29, 30]. The peak located in the broadband between 3000 and 3600 cm^{-1} which can be related to the $-\text{OH}$ stretching vibrations of cellulose [31]. The peak located at 2909 cm^{-1} is assigned to the characteristic C-H stretching vibrations in cellulose and hemicellulose [32]. While modification of cotton fiber with E increased the intensity of the peak (2909 cm^{-1}) with increasing concentrations, the modification of cotton fiber with B broadened and increased the intensity of the peak (2909 cm^{-1}). The band at 1644 cm^{-1} can be related to the water content in fibers [33] and the peak intensity increased with increasing ionic liquid concentration in each ionic liquid treatment. The observed small peaks between 1550 and 1640 cm^{-1} can be attributed to C = N stretching vibrations [34]. The intensity of the

peaks between 1500 and 1288 cm^{-1} which are related to the CH_2 symmetric bending and vibrations of C-H and C-O groups of the aromatic rings [35] in hemicellulose, increased with increasing concentrations. The intensities of the absorption peak at 1250–1200 cm^{-1} increased, which indicated N = C-H bonds due to the ionic liquid modifications [36]. The strong peak at 1013 cm^{-1} , which can be assigned to the stretching vibrations of C-O and O-H in cellulose change significantly with each ionic liquid modification [37]. With the increasing concentrations, the main peak (1013 cm^{-1}) and its shoulders became distinct. Also, small peaks between 938 and 731 cm^{-1} can be related to the β -glycosidic linkages between monosaccharides and CH_2 rocking in cellulose [38–40], became more distinct and visible after each ionic liquid treatment with increasing concentrations. The peaks at 625–767 cm^{-1} are observed afterward modification, which indicates out of plane NH bending [41]. These changes in functional groups successfully confirm the ionic liquid modification of cotton.

Figure 6a and Figure 6b present DSC curves of E and B-modified cotton fabrics compared with the unmodified one. Endothermic and exothermic peaks are visible on the cotton DSC curve.

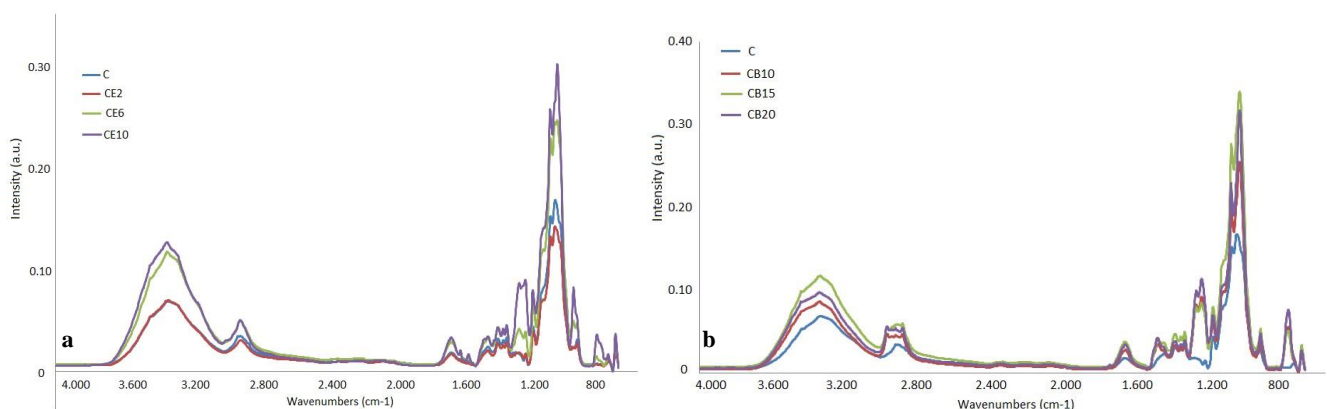


Figure 5. a. FTIR spectra of the unmodified and E modified cotton fibers, **b.** FTIR spectra of the unmodified and B-modified cotton fibers

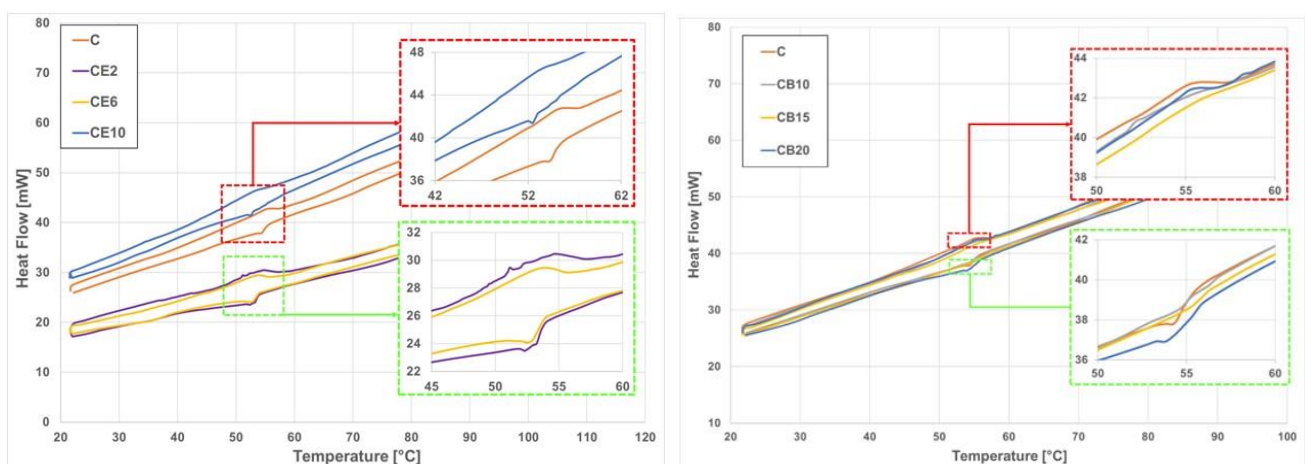


Figure 6. a. DSC curves of the unmodified and E modified cotton fibers, **b.** DSC curves of unmodified and B modified cotton fibers



Table 2 lists DSC data of the unmodified and modified cotton fabrics. The endothermic peaks are recorded in the range of 53.4–55.7°C for B-modified cotton fabrics. This can be attributed to strong-bond water loss and cotton wax melting [42]. The endothermic peaks are noticeable for the unmodified cotton, CE2, CE6, and CE10 fabrics at 55.1, 54.6, 53.4, and 53.0°C, respectively. It was observed that B treatment of cotton led to less variation in the endothermic and exothermic peaks. In other words, E treatment of cotton fiber led to lower phase change temperatures.

Table 3 depicts the color strength results of dyed cotton fabrics.

Table 2. DSC data of the ionic liquids

	The endothermic peak (°C)	The exothermic peak (°C)
C	55.1	54.3
CE2	54.6	52.3
CE6	53.4	52.8
CE10	53.0	52.5
CB10	53.4	54.5
CB15	55.7	54.3
CB20	54.9	54.0

Table 3. Color strength results of dyed cotton fabrics

	R%	K/S	ΔE*
C	8.6	4.9	NA
CB10	12.7	3.0	7.4
CB15	15.7	2.3	12.1
CB20	16.5	2.1	13.4
CE2	5.7	6.9	5.3
CE6	6.1	7.2	5.6
CE10	6.4	7.7	6.3

NA: Not applicable

The cotton fabric was modified with different amounts of ionic liquid and then dyed by reactive dye under the same dyeing procedure. The effect of ionic liquid concentration on the K/S value was resulted and is shown in Table 3. The dyeability of the cotton fabric was studied with regard to the K/S and R% values. The higher K/S values means the darker color tone. Therefore, from Table 3, the K/S value of untreated cotton fabric is inferior to that of samples treated with CE ionic liquid. This indicates that the shadow depth of the untreated cotton fabric is shallower than the treated samples. The peak located in the broadband between 3000 and 3600 cm⁻¹ which can be related to the –OH stretching vibrations of cellulose [31]. The increasing intensity was observed in the FT-IR spectra and this suggests that reactive dyestuffs may be more easily bonded to cotton. It was determined that the K/S value of the modified cotton fabric increased with the increase in the volume of ionic liquid E. This also indicates that E-treated fabrics can have darker clear tone depths under the same dyeing conditions.

When the K/S values of the dyed fabrics were examined after the treatment with another ionic liquid, which is coded CB, it was determined that the color strength was decreased with the increment of the concentration of ionic liquid. When the FT-IR results are examined, after each ionic liquid modification, the increasing intensity was observed with increasing ionic liquid concentrations at broad bands. The intensity of the peaks between 1500 and 1288 cm⁻¹ which are related to the CH₂ symmetric bending and vibrations of C–H and C–O groups of the aromatic rings [35] in hemicellulose, increased with increasing concentrations. Due to this situation, it is thought that the color strength decreases.

The fastness to washing, water and rubbing values of the untreated and CE and CB treated cotton fabrics are listed in Table 4.

It can be determined from the Table 4 that the water, washing and rubbing fastness of cotton fabrics treated by E and B at the lowest concentrations show no difference when compared to the untreated one. However, these fastness results of the cotton fabric treated by E and B at the highest concentrations present an increase of half or one grade. Fastness values were found to be lower in the untreated fabric. Some dye molecules do not diffuse into the interior part of the cotton fibers and are only adsorbed on the surface. As a result, dye molecules are released easily from fibers when the fabric is wet. However, the better fastness values of dyed fabrics after pretreatment with ionic liquids indicate that the dyestuffs may have better adhered to the fiber.

4. CONCLUSION

In this study, the dyeing performances of the modified cotton fabric were investigated. This study reveals that E and B treatment could produce a remarkable modification on cotton and thus affect the cotton dyeing behaviors. The characterization studies showed that ionic liquids settled on the fabric surface and after each ionic liquid modification, the increasing intensity was observed with increasing ionic liquid concentrations according to the FT-IR analysis. These changes in the functional groups can be validated that the ionic liquid modification of the cotton successfully. According to the SEM images, after ionic liquid modifications, a coating layer was observed both on the surface and in the gaps of the yarns. The color depth of the dyed fabrics after E treatment was higher than that of the untreated wool. There was a half or one-grade change in washing and water fastness for E and B-treated cotton fabrics. These changes and results in the surface features of cotton fibers improved their dyeing performances. Moreover, the proposed process, being conducted at dyes lower than those usually employed, assures significant chemical savings. In ionic liquid studies, different ionic liquids can be used for different substrates.

Concentrations were determined by considering the solubility of ionic liquids in ethanol. In future studies, it is

planned to use ionic liquids with different solubility properties in different solvents.

Table 4. Fastness to washing and water test results of the cotton fabrics

	Fastness to washing							Fastness to water							Fastness to rubbing	
	Color Change	CA	CO	PET	PA	PAC	WO	Color Change	CA	CO	PET	PA	PAC	WO	Dry	Wet
C	4-5	4-5	4-5	5	5	5	5	4-5	3-4	3	4	3-4	3	4	5	4
CB10	4-5	5	4-5	5	5	5	5	4-5	4	3-4	4	3-4	3-4	4	5	4
CB15	5	5	5	5	5	5	5	4-5	4	3-4	4-5	4	3-4	4	5	4-5
CB20	5	5	5	5	5	5	5	5	4-5	3-4	4-5	4	4	4-5	5	5
CE2	5	5	4-5	5	5	5	5	5	4	3	4	3-4	3-4	4	5	4-5
CE6	5	5	4-5	5	5	5	5	5	4-5	3-4	4-5	4	3-4	4	5	4-5
CE10	5	5	5	5	5	5	5	5	4-5	3-4	4-5	4	3-4	4-5	5	4-5

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