Equalizing Effect of β-Cyclodextrin on Dyeing of Polyamide 6,6 Woven Fabrics with Acid Dyes

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ABSTRACT: Cyclodextrins (CD) are produced from starch by the action of cyclodextrin glycosyltransferase (CGTase) enzyme. Structurally, cyclodextrins consist of 6, 7, or 8 (α , β , and γ cyclodextrins, respectively) D-glucopyranosyl units connected by α -(1,4) glycosidic linkages. Having polar and hydrophilic outer sides and hydrophobic cavitation gives cyclodextrins a chance to form inclusion complexes with dyes in hydrophilic mediums. In this research, the equalizing effect of β -cyclodextrins in dyeing of polyamide 6,6 woven fabrics with 6 different acid dyes were investigated. From the experimental results, it was determined that the β -cyclodextrin shows a retarding and equalizing effect in dyeings carried out with the dyes that show interaction with β -CD. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 2660–2668, 2007

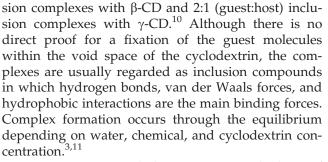
Key words: cyclodextrin; PA 6,6; inclusion complex; equalizing; dye

INTRODUCTION

Cyclodextrins are a family of oligosaccharides, which are obtained from degradation of starch by the cyclodextrin transglycosylase enzyme.¹ Cyclodextrins are cyclic oligosaccharides composed of 6, 7, and 8 or more glucopyranose units that are bonded to each other with α -1,4 linkages and these are named as alpha (α), beta (β), and gamma (γ) cyclodextrins, respectively.^{1,2} Depending on the number of glucose residues in the molecule, the rings of these dextrins have different internal diameters.³ Cyclodextrins have a truncated cone shape,⁴ and the cavity of cyclodextrin narrows towards the end, because the C6 atom can rotate with regards to the hydroxyl groups on C2 and C3 (Fig. 1).⁵

Polar and hydrophilic outer surface and hydrophobic cavitation of cyclodextrins enable them to retain hydrophobic compounds in a hydrophilic medium.^{6,7} As a result of this retention, they can form inclusion complexes with many other organic compounds.⁸ Because of their inclusion within the cyclodextrin structure, their vapor pressure is reduced and their release becomes more gradual and controlled.⁹ Such substrates usually form inclusion compounds with 1 : 1 stoichiometry; however, other stoichoimetries have also been reported. This is related to the diameter of the cyclodextrin. For example, crystal violet and methylene blue have been reported to form 1 : 1 inclu-

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For many years, cyclodextrins were studied out of curiosity, without knowing any significance for their practical and technical usage.⁴ Today, cyclodextrins are used in many areas such as food industry, pharmacy, cosmetics, environmental protection, and textile industry.^{2,12} An important factor concerning possible applications in the textile area is that the use of cyclodextrins does not cause any problems in waste water and they are biodegradable.¹³ Some of the literatures about the usage of cyclodextrins in polyamide dyeing are summarized below.

Shao et al.¹ have investigated the interactions between β -CD and 27 water soluble dyes in aqeous solutions by UV–vis spectrophotometry. The effects of pH and auxiliaries on the interactions were studied. Experiments showed that β -CD has a protective function to prevent dyes from being affected by salt and acids; and acid dyes in the presence of β -CD resulted in a high level of dyeing even in strongly acidic conditions.

Savarino et al.⁴ tested β -CD as a low environmental impact additive in dyeing processes. With this aim, a series of 12 dyes were synthesized and used for the dyeing nylon 6 and 6,6. Interactions between β -cyclo-



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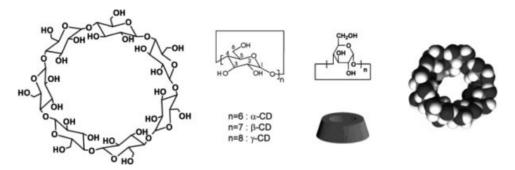


Figure 1 Chemical structure of cyclodextrins.

dextrin and dyes were studied by means of solubility isotherms. β -CD showed good leveling properties in the dyeing of polyamide fibers.

Savarino et al.¹⁴ have also researched the effects of additives on dyeing of nylon-6 with dyes containing hydrophobic and hydrophilic moieties. They have synthesized a series of disperse dyes with variable hydrophobic chain lengths and hydrophilic heads. The dyeing properties of polyamide fibers were assessed with dyeing isotherms. The positive effects of cyclodextrins on the dyeing uniformity were shown by tristimulus colorimetry.

Savarino et al.⁸ have showed the reactivity and effects of cyclodextrins in textile dyeing. Four- to tenfold improvement of color uniformity and minor changes of color yield have been found upon dyeing nylon 6,6 and microfiber nylon 6 fabrics in the presence of cyclodextrin when compared with dyeing without it.

Lijima and Karube¹⁵ investigated the interaction of acid azo dyes with dimethyl-and trimethyl- β -cyclodextrins spectrophotometrically. The binding ability was found to increase with the dimethyl derivative, when compared with original cyclodextrin, due to hydrophobic interactions.

In this study, by examining the change of the interaction between β -cyclodextrin and acid dye with regards to pH, temperature, and β -CD concentration, their possible usage as an equalizing agent in dyeing of PA 6,6 fabrics with acid dye was investigated.

EXPERIMENTAL

In the study, 100% PA 6,6 plain woven fabric (30 ends/cm and 50 picks/cm), which is ready to dye, was used. For spectral measurements, dye solutions were prepared with pure water. Rinsing after dyeings was carried out by using soft water ($0.5-1^{\circ}F$).

Six acid dyes were used in experiments; Telon Red GGW (C.I. Acid Orange 19), Telon Red BN (C.I. Acid Red 42), Supranol Marine R (C.I. Acid Blue 113), Isolan Dark Blue 2S-GL (C.I. Acid Blue 193), Telon Violet

3R (C.I. Acid Violet 42), and Telon Blue RR (C.I. Acid Blue 62) were kindly supplied by Dystar and their chemical formulas are given in Table I.

Cavamax[®]W7 (Wacker Chemie) was selected as β -cyclodextrin. Keriolan A2N-T (CHT), which has affinity for the dye with base polyglycol ether derivative, was used as the equalizing agent.

HT-type thermal dyeing machine was used for dyeing processes. Remission measurements were realized on X-rite SP78 model spectral photometer (with D 65 day light and 10° measurement angle), and absorbance measurements of dye solutions were performed on Shimadzu UV-1201 trade mark absorbency measurement device.

It is known that cyclodextrin molecules do not interact with all dyes, instead they form inclusion complexes with only dyes having appropriate molecular size and structure. For this reason, to determine the acid dyes that form inclusion complexes with β -CD, dyeing processes in 1 g/L dye were realized with 6 acid dyes, of which chemical structures are known. Dyeings were carried out by using liquors (liquor ratios 20:1), with and without cyclodextrin (4 g/L), and for which the pH was adjusted to 5 with the aid of buffer CH₃COOH (99%) (Smyras)/NaCH₃-COO.3H₂O (Riedel-de Haen).

For this aim, the fabric was added into the dye bath at 40°C and the temperature was gradually (2°C/min) raised to 100°C and held at that temperature for 60 min. Then, the bath was cooled and disposed. After dyeing, all samples were subjected to cold (5 min)-hot (10 min at 70°–80°C)-cold (5 min) rinsing with soft water, and then they were dried at room temperature. Two acid dyes that showed interaction with β -CD and two acid dyes that did not show interaction with β -CD were selected according to the results of spectrophotometric measurements of the fabric samples.

Then, the interaction of these dyes with β -cyclodextrin was investigated by the spectrophotometric method, which is a safer method and a determination of the complex formation was attempted. For the determination of interaction, aqueous solutions of dyes in certain concentrations at different pH values

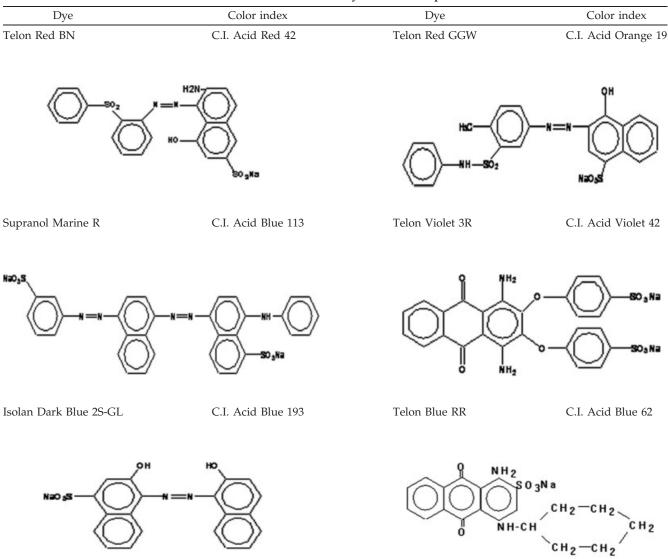


TABLE I Chemical Structures of Acid Dyes Used in Experiments

(pH 3, 5, and 8) were prepared in the presence or absence of β -CD and then spectral measurements were carried out. Dye concentration was specified as 0.1 g/ L in the study. β -CD concentration was selected as 4 g/L to observe the interaction more clearly. It was observed that β -cyclodextrin had no absorption in the range of 400–700 nm.

After the interactions between β -cyclodextrin and dyes were determined, isotherm dyeings were performed with two acid dyes (C.I. Acid Violet 42 and C.I. Acid Blue 62), which showed interaction with β -cyclodextrin, in two different dye concentrations (0.5 and 1 g/L), four different β -CD concentrations (0.5, 1, 2, and 4 g/L), at three different pH values (3, 5, and 8), and three different temperatures (60, 80, and 90°C).

The reason for conducting the dyeing processes at $60, 80, \text{ and } 90^{\circ}\text{C}$ is that the critical temperature region

view point of dye levelness; and it is important to limit the dye uptake of fibers in this critical region to obtain uniform dyeing. Dyeing processes were carried out in an isothermal way with liquors of which pH values are set [for pH 3 with buffer HCl (37%) (UPARC)/sodium citrate (Horasan Kimya), for pH 5 with buffer CH₃COOH (99%) (Smyras)/NaCH₃₋ COO.3H₂O (Riedel-de Haen), and for pH 8 with buffer NaH₂PO₄.2H₂O (Merck)/Na₂HPO₄.2H₂O (Merck)] and containing 0.5 and 1 g/L dye for 45 min at three different temperatures (60, 80, and 90°C) in liquor ratio 10:1. After dyeing, all samples were subjected to cold (5 min)-hot (10 min at 70-80°C)-cold (5 min) rinsing with soft water and then samples were dried at room temperature. Results were evaluated by measuring remission values of fabrics and absorbance values of dye liquors.

for PA 6,6 fibers is between 60 and 80°C from the

Dye uptake (%) was calculated with the aid of absorbance values. For this aim, absorbance values were measured at the maximum absorbance wavelength, which was determined before, by taking samples from the liquors at the beginning and at the end of the dyeing. For each sample, relative absorbance values, after dyeing for 45 min, were calculated by taking the initial absorbance values as 100. "Dye uptake values %" were calculated using these values with the aid of the formula below:

For determining the influence of β -CD on the levelness (L) of dyeing and to compare its effect with conventional equalizing agents, dyeings at pH 5 and 1 g/ L dye concentration (liquor ratio 20:1) either in the absence of auxiliaries or in the presence of β -CD (1 g/L) or equalizing agent (1 g/L) were carried out. Fabric was added to the bath at 40°C and the temperature was gradually (2°C/min) raised to 100°C and held constant for 60 min. Then, the bath was cooled and disposed of and the fabric was rinsed. Two acid dyes, which have interactions with β -CD (C.I. Acid Violet 42 and C.I. Acid Blue 62), were selected for these experiments. After dyeing, K/S values of samples were measured from 70 different points of the samples (n = 70) and L values were calculated according to the following formula:

$$L = \left[1 - \sqrt{\frac{\sum \left[\frac{(K/S)_i}{(K/S)} - 1\right]^2}{n - 1}}\right] \times 100$$

where $(K/S)_i$ is the sample's color yield measured from one point and (K/S) is the average of *n* measurements.

The higher *L* value means that the dyeing is more uniform (level). When L = 100, dyeing is absolutely uniform and when L = 0, dyeing is totally uneven.

RESULTS AND DISCUSSION

Investigation of interaction between β-CD and acid dyes

Dye uptake speed of polyamide fibers during dyeing with acid dye is not constant throughout the dyeing. Dye uptake is slow until critical temperature is reached and then the dye uptake speeds up. It is necessary to increase the temperature slowly or to fix the temperature for sometime (stepping) to achieve uniform dyeing, since 60–80% of dye is uptaken in this temperature region. Addition of equalizing agent into the liquor during dyeing increases the critical temperature 20–30°C. Besides, increasing critical temperature, adding equalizing agent also slows down the dye uptake and even slightly decreases the amount of dye taken by the fibers.¹⁶ Briefly, when an equalizing agent having affinity to the dye is added to the dyeing liquor, it slows down the dye uptake and also influences the amount of dye uptake, and dye uptake realized in the presence of equalizing agent can be less than the dyeing performed without any auxiliary agent.

When it is considered that β -CD used in our study interacts with the dye, color yields of the dyeings performed in the presence of β -CD with dyes forming inclusion complexes with it are expected to be less than dyeings carried out without the usage of β -CD. For this reason, dyeings were performed by using six different acid dyes with and without β -CD and the dye showing a change in its color yield is thought to interact with β -CD. From Figure 2, effects of β -CD usage on relative color yield in dyeings carried out with various dyes can be seen.

As it can be seen from Figure 2, color yields of dyeings carried out with C.I. Acid Orange 19, C.I. Acid Red 42, C.I. Acid Blue 113, and C.I. Acid Blue 193 did not change much in the presence of β -CD; however, a 20-25% decrease was established in the color yields of dyes C.I. Acid Violet 42 and C.I. Acid Blue 62 when β -CD was used. Taking these results into consideration, C.I. Acid Violet 42 and C.I. Acid Blue 62, which are thought to interact with β -CD, and C.I. Acid Blue 193 with no interaction were selected and spectrophotometric measurements of these dyes were performed in the visible region, because visible region spectrophotometric measurements of dye solutions prepared in the presence and absence of β -CD give more definite information whether there has been an interaction between β -CD and the dye molecule or not.

When β -CD forms an inclusion complex with a dye molecule, high electron density in the cyclodextrin cavity mobilizes the electron system of the guest compound. As a result of this, characteristic changes are observed in the visible spectrum of the dye. Hence, by means of the UV–vis spectrophotometric measure-

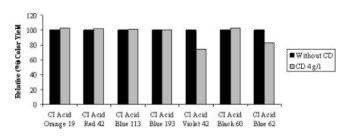


Figure 2 Relative (%) color yields of dyed samples with various acid dyes.

ments of dye solutions in the presence or absence of β -CD, we can know whether the interactions are between β -CD and the dye molecule or not.¹ Thus, no change is expected in the spectrum of dye that does

not interact with cyclodextrin. Spectrophotometric measurement results of dyes C.I. Acid Violet 42, C.I. Acid Blue 62, and C.I. Acid Blue 193 are given in Figure 3.

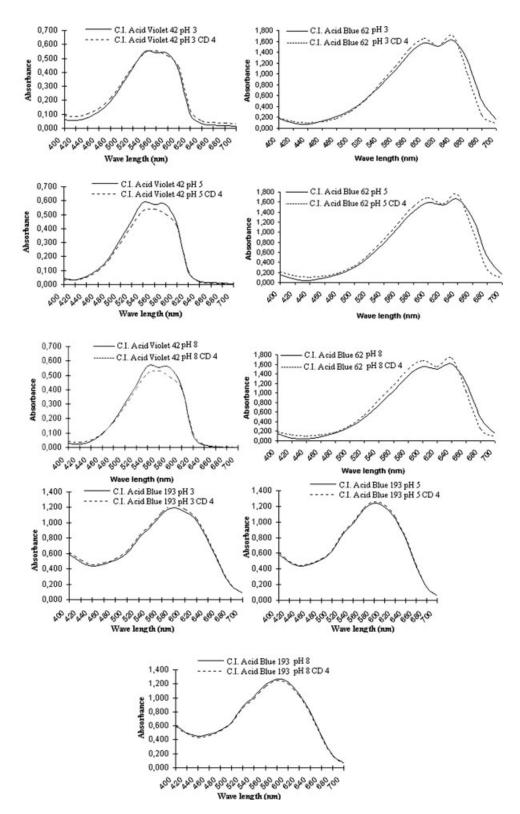


Figure 3 Absorbance measurements of dye solutions in the presence and absence of β -CD having different pH values (pH 3, 5, and 8) in visible region (400–700 nm).

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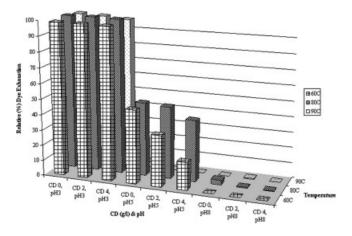


Figure 4 Relative (%) dye exhaustion values of dyeings realized with 0.5 g/L C.I. Acid Violet 42.

In our study, dyeings were performed at pH 3, 5, and 8; thus, in spectrophotometric measurements, interactions were investigated at these pH values. It can be said that there is no interaction between C.I. Acid Blue 193 (dyes for which λ_{max} and A_0 values do not change) and β -CD according to the measurement results. On the other hand, it was determined that there was 20 nm increase in the maximum absorbance wavelength of C.I. Acid Violet 42 in the presence of β -CD for all pH values (pH 3, 5, and 8), and maximum absorbance wavelength of 540 nm has increased to 560 nm (Batochromic effect). No significant change in absorbance values was observed at pH 3; however, there was a hyphochromic effect (decrease in absorbance value) at pH 5 and 8. In the presence of β -CD, for all pH values, hyperchromic effect (increase in absorbance value) was observed in the maximum absorbance values of C.I. Acid Blue 62.

It is not possible to say that molecular size of dye by itself determines the interaction with β -CD. Even though, suitability of molecular structure to the cavity of β -CD is a precondition, the position and the effect

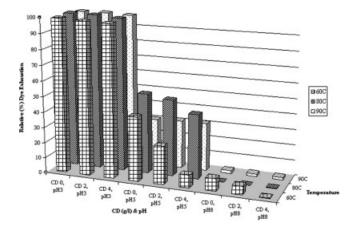


Figure 5 Relative (%) dye exhaustion values of dyeings realized with 1 g/L C.I. Acid Violet 42.

of substituents in the molecular structure of dye are important for inclusion complex formation. In molecular structures of C.I. Acid Orange 19 and C.I. Acid Red 42 dyes, there are no substituent group on the phenyl ring. Therefore, during the synthesis of these dyes, an important fraction of cis-compound could be formed. The spectrum of cis-azobenzene remains unchanged in the presence of β -CD, because the cisconfiguration does not fit the cavity of β -CD.¹ It is thought that the reason C.I. Acid Blue 113 does not interact with β -CD is that even though it contains substituent in the phenyl rings, it has big molecules, which cannot enter into the cavity of β -CD. Thus, for the formation of a good complex, the dye molecule to be bound must fill the cavity of the cyclodextrin and it must be in contact with the walls of the cavity.⁵

The reason why C.I. Acid Blue 193 does not interact with β -CD can be due to the fact that it does not have a free phenyl ring that can form an inclusion complex with β -CD. On the other hand, in the presence of β -CD, change is observed in the spectrums of dyes C.I. Acid Violet 42 and C.I. Acid Blue 62, because it is thought that β -CD may form inclusion complex on the anthraquinone rings of these dyes, and for C.I. Acid Violet 42 also on the phenyl rings containing substituent groups. On the basis of these results, C.I. Acid Violet 42 and C.I. Acid Blue 62, which are thought to interact with β -CD, were selected to be used in the study.

Change of the interaction between β -CD and dye with according to the pH, temperature, and β -CD concentration

In aqueous solutions, cyclodextrins take the dye molecules into their cavities that have a hydrophobic character, and they release dye molecules as the free molecules attracted by the polymer. The interaction between dye molecule and cyclodextrin changes with

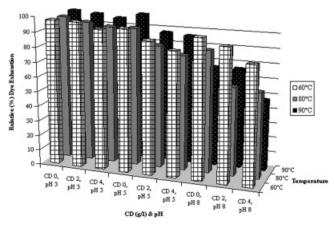


Figure 6 Relative (%) dye exhaustion values of dyeings realized with C.I. 0.5 g/L Acid Blue 62.

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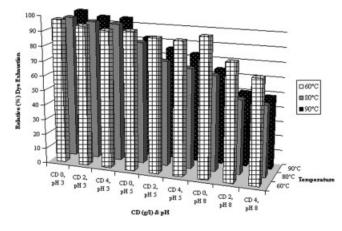


Figure 7 Relative (%) dye exhaustion values of dyeings realized with 1 g/L C.I. Acid Blue 62.

the factors such as pH and temperature of the medium.

Relative (%) dye exhaustion values of dyeings performed with C.I. Acid Violet 42 and C.I. Acid Blue 62 are given in Figures 4–7.

When the figures are examined, it can be seen that slowing down the dye uptake of β -CD was obtained at 60°C more distinctively, and this effect decreased as the temperature increased (80 and 90°C). The reason, for the decrease in the interaction between β -CD and C.I. Acid Violet 42 and C.I. Acid Blue 62 dyes as temperature increases (from 60 to 90°C), is thought to be due to the fact that inclusion complex that dye molecule forms with β -CD is more stable at a lower temperature, because kinetic energy of the dye molecules is less. Thus, it will be more difficult for β -CD to hold the dye molecules as the temperature of the medium increases, since mobility of the dye molecules will also increase. However, when Figures 4-7 are examined, it can be said that the interaction between dye and cyclodextrin at 80 and 90°C is important only at pH 5 for C.I. Acid Violet 42 and at pH 5 and 8 for C.I. Acid Blue 62.

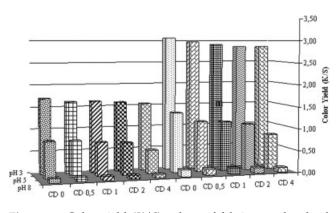


Figure 9 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Violet 42 at 80°C in different pH values and β -CD concentrations.

For both of the dyes, it is seen that slowing down the dye uptake of β -CD is not so apparent in the dyeings realized at pH 3, and dye uptake significantly decreased (that is amount of dye in the liquor increased) at pH 5 as the concentration of β -CD increased especially at 60°C and also for C.I. Acid Blue 62 at 80°C. It is determined that β -CD slowed down the dye uptake slightly in the dyeing process with C.I. Acid Violet 42 at pH 8, and on the other hand, it showed a significant slowing down effect for C.I. Acid Blue 62. All these results are valid for both color depths of 0.5 and 1 g/L.

The reason that β -CD did not show retarding effect at pH 3 is the increased number of functional groups to which dye can bond as a result of the imino ($-NH_2-$) groups in the structure of the fiber transforming into amido groups ($-NH_2+-$) with positive charges, and because of this reason, the dye uptake occurs rapidly. Thus, slowing down dye uptake by cyclodextrin cannot be observed distinctively.

Although observing an evident effect on the dyeing with C.I. Acid Blue 62 at pH 8, the reason for the effect being weak for C.I. Acid Violet 42 is that this

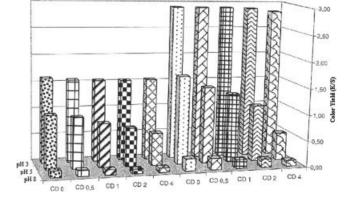


Figure 8 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Violet 42 at 60°C and in different pH values and β -CD concentrations.

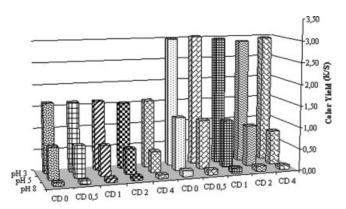


Figure 10 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Violet 42 at 90°C in different pH values and β -CD concentrations.

dye has a disulphonate structure and at this pH, fibers show anionic character (-charge) because of the dissociation of carboxyl groups in the structure of PA fibers and hence the dye uptake is already slow because of the electrostatic repulsion between the fiber and dye molecule. It is known that when acid dye in the structure of disulphonate dissociates in aqueous medium, it gains two sulfo $(-SO_3^-)$ groups with (-) charge, and since fiber also has (-) charge in dyeings carried out at pH 5, the repulsion force between fiber and dye molecule are higher with regards to the C.I. Acid Blue 62 having monosulphonate structure. In this case, slowing down of the dye uptake, which is already little and slow, by β -CD cannot be observed clearly.

From these results, it can be said that the interaction between C.I. Acid Violet 42 and β-cyclodextrin occurs most obviously in dyeings carried out at 60°C and pH 5. For example, in the dyeing carried out in color depth of 1 g/L at 60°C and pH 5, dye uptake is 41.41% without cyclodextrin, and it is 24.72% in the presence of 4 g/L cyclodextrin, that is it by almost half. The interaction between β -cyclodextrin and C.I. Acid Blue 62 is found to be the most noticeable in dyeings carried out at 60 and 80°C and pH 8 and especially pH 8. For example, in the dyeings carried out at 60°C, pH 5, and with 0.5 g/L color depth when β -cyclodextrin was not used dye uptake was 94.75%, and in the presence of 4 g/L β -cyclodextrin dye uptake was 83.05%, in other words dye uptake decreases $\sim 10\%$. These results also support the spectrophotometric measurement results obtained to determine the interaction between the dye and cyclodextrin.

Dyeing experiments were actually performed by using 0.5, 1, 2, and 4 g/L β -CD and without the presence of β -CD; however, dye uptake values (%) realized with 2 and 4 g/L β -CD and without the usage of β -CD are not shown in the figures to prevent confusion. For this reason, the effect of the change in β -CD

8.00

7,00

6,00

5,00

3.00

2.00

Color Yield (K/S

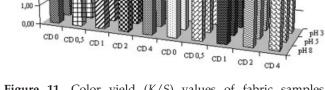


Figure 11 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Blue 62 at 60°C in different pH values and β -CD concentrations

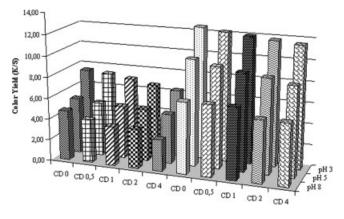


Figure 12 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Blue 62 at 80°C in different pH values and β -CD concentrations.

concentration on the dyeing results can be seen in Figures 8–13 in more detailed form (for 0, 0.5, 1, 2, and 4 g/L β -CD).

The figures show that the interaction between β -CD and dye decreases as the temperature increases from 60 to 90°C. Additionally, it can be seen that β -CD does not affect the color yield of dyed fabrics much at pH 3, and as the β -CD concentration increases, color yields of the dyed fabrics decrease for dyeings performed with C.I. Acid Blue 62 at pH 5 and 8 and with C.I. Acid Violet 42 at pH 5. All these results are in agreement with the absorbency measurement results.

Comparing the leveling effect of β -CD with equalizing agent

Results of experiments performed with the aim to compare the effects of β -CD and equalizing agent on the dyeing levelness are given in Table II.

From Table II, it can be seen that both β -CD and equalizing agent show positive effects on the level-

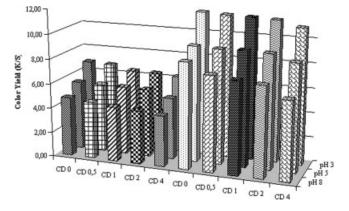


Figure 13 Color yield (*K*/*S*) values of fabric samples dyed with C.I. Acid Blue 62 at 90°C in different pH values and β -CD concentrations.

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| TABLE II Equalizing Effect of β-CD and Leveling Agent on PA 6,6 Dyed with Acid Dyes | | | |
|---|-------------------|-----------|------------------------|
| | Levelness (L) (%) | | |
| Dyes | No β-CD | With β-CD | With leveling agent |
| C.I. Acid Violet 42 | 95.12 | 95.97 | 96.11 |

90.11

83.89

71.91

ness of dyeings carried out with dyes that have interactions with β -CD (C.I. Acid Violet 42 and C.I. Acid Blue 62). The reason for the leveling effect of β -CD and equalizing agent being less in dyeing carried out with C.I. Acid Violet 42 with regards to the dyeing with C.I. Acid Blue 62 is that the dyeing performed with C.I. Acid Violet 42 without any auxiliary agent is already uniform.

CONCLUSIONS

The interactions between β -CD and 3 acid dyes were investigated spectrophotometrically. Although there were changes in the spectrums of C.I. Acid Blue 62 and C.I. Acid Violet 42 in the presence of β -CD, the spectra of C.I. Acid Blue 193 did not change. As a result of this study, it was seen that slowing down the dye uptake effect of β -CD decreased as the temperature increased from 60 to 90°C, and for dyeings performed at pH 3, this effect was not noticeable, and as the concentration of β -CD increased, dye uptake distinctively decreased in the dyeings carried out especially at 60°C for C.I. Acid Blue 62 at pH 5 and 8 and for C.I. Acid Violet 42 at pH 5. With the aid of these results, it can be said that the dye molecule will be held by β -CD in the critical region (between 60 and 80°C) during a normal dyeing process performed by starting at 40°C and raising the temperature to 100°C (pH around 5) and it will be released again at the end of the critical region. This shows us that β -CD can be used as an equalizing agent in the dyeing processes performed with acid dyes having interaction with it.

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