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INVESTIGATION OF THE EFFECT OF INCLUSION COMPLEXES ON THE DYEING OF SOYBEAN FIBERS

Gülşah Ekin KARTAL[1*]
Mehmet KERTMEN[2]
Murat DEMİR[1]

[1]Dokuz Eylü lÜniversitesi Mühendislik Fakültesi Tekstil Mühendisliği Bölümü, İzmir, Türkiye
[2]İskur TekstilEnerji Tic. ve San. A.Ş., Kahramanmaraş, Türkiye

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ABSTRACT: This study was conducted to assess the dyeability of soybean fibers. The dyeing process was performed at three different temperatures (20°C, 50°C, and 90°C) using two types of dyestuffs: acid and reactive dyes. The entire dyeing procedure was repeated in the presence of cyclodextrin to investigate whether cyclodextrins influence dye uptake. Color measurements of the dyed fibers were conducted based on the CIE-Lab color system. Additionally, washing fastness test was carried out to assess the colorfastness of the dyed fibers. To examine potential differences in the morphological structure of the fibers, fiber images were observed under an optical microscope. Furthermore, FT-IR analysis was performed to investigate potential bonding interactions between cyclodextrins and the fiber structure. The findings indicated that soybean fibers are not suitable for dyeing at high temperatures, as this leads to structural deterioration in the fibers. In contrast, the use of cyclodextrins enhanced dye uptake and resulted in higher color strength and color uniformity, particularly at 50 °C. The presence of cyclodextrins also contributed to a slight improvement in washing fastness, especially in cotton and wool multifiber samples. Based on the color yield and fastness tests conducted, the optimal dyeing temperature was determined to be 50°C, and the use of cyclodextrins was found to be effective in improving dyeing performance without causing fiber damage.

Keywords: Soybean fiber, Dyeing, Reactive, Cyclodextrin

İNKLÜZYON KOMPLEKSLERİNİN SOYA LİFLERİNİN BOYANMASI ÜZERİNDEKİ ETKİSİNİN ARAŞTIRILMASI

ÖZ: Bu çalışmada, soya liflerinin siklodekstrin varlığında boyanabilirliği değerlendirilmiştir. Boyama işlemi, asit ve reaktif boyalar olmak üzere iki tip boyarmadde kullanılarak üç farklı sıcaklıkta (20°C, 50°C ve 90°C) gerçekleştirilmiştir. Tüm boyama proseslerindesiklodekstrinlerin boya alımını etkileyip etkilemediğini araştırmak için siklodekstrinler yardımcı madde olarak kullanılmıştır. Boyanmış liflerin renk ölçümleri, CIE-Lab renk sistemine göre gerçekleştirilmiştir. Ayrıca, boyanmış liflerin yıkamaya karşı renk haslığı değerlendirilmiştir. Liflerin morfolojik yapısındaki potansiyel farklılıkları incelemek için optik mikroskop altında lif görüntüleri incelenmiştir. Ayrıca, siklodekstrinler ve lif yapısı arasındaki potansiyel bağlanma etkileşimlerini araştırmak için FT-IR analizi gerçekleştirilmiştir. Analizler sonucunda, liflerde yapısal bozulmalara yol açtığı için soya liflerinin yüksek sıcaklıklarda boyamaya uygun olmadığı görülmüştür. Çalışma, siklodekstrin kullanımının boya emilimini artırdığını ve özellikle 50°C'de daha yüksek renk kuvveti ve renk homojenliği sağladığını ortaya koymuştur. Siklodekstrinlerin varlığı, özellikle multifiberde yer alan pamuk ve yün liflerinde yıkama haslığında hafif bir iyileşmeye de katkıda bulunmuştur. Yapılan renk verimi ve haslık testlerine dayanarak, optimum boyama sıcaklığı 50°C olarak belirlenmiş ve siklodekstrin kullanımının lif hasarına neden olmadan boyama performansını iyileştirmede etkili olduğu görülmüştür.

Anahtar Kelimeler: Soya lifi, Boyama, Reaktif, Siklodekstrin

***SorumluYazarlar/Corresponding Authors:** gulsah.kartal@deu.edu.tr*
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1. INTRODUCTION

Soybean fibers are among the renewable protein-based fibers and are increasingly gaining attention in sustainable textile production. Although they were first produced in the mid-20th century, their low strength has limited their production. Today, soybean fibers are being reconsidered as an alternative to natural protein-based fibers such as wool and cashmere due to their environmental advantages, cost-effectiveness, and improved processing technologies [1]. When their chemical structure is examined, it is known that the ratio of acidic amino acids in soybean fibers is higher than in wool, and the dominant amino acid is glutamic. However, the low content of sulfur-containing amino acids (e.g., cysteine) results in limited disulfide bonds and weakens the cross-linking mechanism of the fibers. This situation is a determining factor in the dyeing process and mechanical properties [2, 3].

The thermal stability of soybean fibers is limited; their strength decreases significantly at 160°C, turns dark yellow at 200°C, and brown at 300°C and enters the carbonization process. The topic of soybean fibers and their response to high temperatures is complex and requires expert knowledge. These fibers, primarily composed of proteins and polysaccharides, cannot withstand high temperatures. The denaturation of proteins at high temperatures disrupts their structure, leading to a loss of elasticity and durability. Moreover, the weakening of amide bonds, structural changes in lignin compounds in the cell wall, and increased enzymatic activity all contribute to fiber degradation. The disruption of lignin polymerization and water loss make the fibers more brittle and sensitive. These interactions significantly weaken the mechanical properties of soybean fibers, reducing their durability under high temperatures. [2, 4]. Therefore, it is recommended to avoid temperatures exceeding 100°C during dyeing and heat treatments. Temperatures above 110°C cause hardening in the fiber structure, while dyeing at low temperatures can cause difficulties in terms of color yield and homogeneity [5, 6]. In this context, one of the most significant difficulties in the dyeing processes of soybean fibers is to be sensitive to high temperatures while at the same time achieving the desired color intensity and homogeneity.

In recent years, various studies have been carried out on the dyeability of soybean fibers with different dyes and process conditions. It has been stated that soybean fibers have good affinity with reactive and acid dyes, but dyeing at high temperatures can deteriorate the fiber structure and cause color changes. For example, Kalaycı et al. (2024) dyed soybean fibers with black mulberry fruit extract and examined the effects of microwave dyeing techniques on color yield and fastness properties. Their studies reported that microwave-assisted dyeing gave satisfactory results in a shorter time compared to traditional methods [7]. Similarly, Tahir et al. (2024) examined the effect of dyeing temperature on soybean fibers and found that fibers underwent significant color changes and their mechanical properties weakened at temperatures above 100°C [1]. Recent studies have investigated the dyeing behavior of soybean fibers and their blends using a variety of dyes and conditions [8-13]. For instance, Cao et al. examined the kinetics of acid dyeing on soybean/casein/PVA blend fibers [9], while Liu et al. studied dye fixation in soybean/flax yarns using reactive dyes [8]. However, limited research has addressed the role of environmentally friendly additives such as cyclodextrins in improving dye uptake and fastness properties in pure soybean fibers.

Among the solutions to increase sustainability in textile dyeing processes, the molecular encapsulation method draws attention. This method allows the improvement of specific physicochemical properties by trapping a guest molecule in the inner cavity of a supramolecular central molecule [14, 15]. Cyclodextrins (CDs) are the primary molecules widely used in forming such inclusion complexes and provide various advantages in textile dyeing processes. Cyclodextrins obtained by enzymatic hydrolysis of starch can form complexes with hydrophobic guest molecules thanks to their inner cavity structures, thus increasing the solubility and stability of textile dyes [16-18]. In particular, β-cyclodextrin (β-CD) contributes to developing resistance against external effects by exhibiting strong binding ability to textile surfaces [19, 20].

Cyclodextrins in the dyeing process provide more homogeneous dyeing by increasing the solubility of low-solubility dyes. These properties allow the desired color tones to be obtained with less dye, water, and auxiliary chemical use and contribute to sustainable dyeing applications [21, 22]. In addition, cyclodextrins can help achieve more uniform coloration by forming temporary complexes with dye molecules, thereby controlling their gradual release and preventing sudden or uneven dye absorption on the fiber surface. The binding mechanism of cyclodextrins to textile surfaces is based on parent-cavity interactions, and the stability of the complex depends onthe cyclodextrin cavity size [23, 24]. Significantly, when used as auxiliary materials during direct dyeing of cotton fabrics, cyclodextrins increase the dyeing efficiency by forming complexes with low-solubility dyes [25-28].

Studies on cotton and wool fibers have demonstrated that cyclodextrins can enhance dye solubility and improve both color yield and color fastness through the formation of inclusion complexes with dye molecules [21, 23, 24]. Bezerra et al. (2020), in their comprehensive review, summarized findings from several studies indicating that β-cyclodextrins facilitate better dye-fiber interactions and lead to improved washing fastness values [23].However, studies examining the effect of cyclodextrins on soybean fibers are limited. The primary purpose of this study was to investigate the effect of cyclodextrins on the dyeability of soybean fibers dyed with acid and reactive dyes at different temperatures, and it was evaluated whether cyclodextrins could be used as an alternative additive in the dyeing process of soybean fibers.

In this study, the effects of cyclodextrins on the dyeability of soybean fibers are evaluated. The research scope analyzed cyclodextrins' effects on color yield, fastness, and fiber morphology in dyeing processes at different temperatures (20°C, 50°C, and 90°C) using acid and reactive dyes. The findings obtained aim to contribute to sustainable dyeing practices by providing new information on the effect of cyclodextrins on the dyeing process of soybean fibers.

2. MATERIAL AND METHOD

2.1. Material

β-CD (1134.98 g/mol) was supplied by Sigma–Aldrich, USA. The dyestuffs used in this study Lanasol Blue 3R (a reactive dye, originally produced by Huntsman) and Nyloset Blue E-2RF (an acid dye) were generously supplied by SetaşKimya A.Ş. for research purposes. Na_2CO_3, Na_2SO_4, and CH_3COOH were supplied by Merck (Germany). Soybean fibers were supplied by the ISKUR Group, Türkiye.

2.2. Dyeing Procedure

The soybean fibers were dyed with acid and reactive dyes at a concentration of 1.0% (based on the weight of the fibers). The dyeing processes were performed at three different temperatures (20 °C, 50 °C, and 90 °C) to investigate the influence of temperature on dye uptake and fiber integrity. The liquor ratio was maintained at 20:1 for all dyeing processes. For reactive dyeing, 1% Na_2CO_3 and 10% Na_2SO_4 were added to the bath, and dyeing was carried out for 60 minutes under the selected temperature conditions. After dyeing, the fibers were rinsed with distilled water at 50 °C for 10 minutes, treated with CH_3COOH at 50 °C for another 10 minutes, followed by a second rinse at 50 °C and a final rinse at room temperature for 10 minutes. For acid dyeing, the dye bath was adjusted to pH 4.5 using 1% CH_3COOH, and the same dyeing and washing durations and temperatures were applied as in the reactive dyeing procedure. No salts were used in acid dyeing. In cyclodextrin-assisted dyeing, β-cyclodextrin was added to the dye bath at a concentration of 10 g/L, and no additional chemicals such as Na_2CO_3 or Na_2SO_4 were used. The cyclodextrin was pre-dissolved in the dye bath before adding the fibers and dye. This approach was applied to both reactive and acid dyes at all three temperatures. The total dyeing time remained 60 minutes, and the liquor ratio was also 20:1. In cyclodextrin-assisted reactive dyeing without alkali, covalent bonding between dye and fiber is not expected due to the absence of alkaline conditions. Instead, dye-fiber interactions are primarily physical, facilitated by the inclusion complexation ability of β-cyclodextrins. These interactions involve hydrogen bonding and improved dispersion of dye molecules, leading to enhanced dye uptake and color uniformity even without fixation through chemical bonding. After dyeing, the fibers underwent a washing protocol comprising several steps. Initially, they were rinsed with distilled water at 50°C for 10 minutes. Subsequently, they were rinsed with CH_3COOH at 50°C for another 10 minutes, followed by an additional 10-minute rinse at 50°C and a final rinse at room temperature for 10 minutes. The liquor ratio was maintained throughout this process at 20:1. When cyclodextrins were used as an auxiliary agent, Na_2CO_3 and Na_2SO_4 were not employed. Instead of these chemicals, cyclodextrin was added to the dyestuff and fibers at the beginning of the dyeing process.

2.3. Instrumentation

The morphological characteristics of the samples were assessed using optical microscopy, employing an Olympus BX-43 microscope set at 100× magnification.

Color measurements of the fibers were conducted using a Minolta CM 3600 D spectrophotometer (D65 illuminant, specular inclusion, 10° observer angle). The K/S and ΔE values were determined through the following calculations [22].

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

In the given context, K represents the scattering coefficient, S stands for the absorption coefficient, and R signifies the reflectance.

The discrepancy in color is denoted by ΔE* and is computed as:

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

where ΔE* represents the CIELab color difference between the batch and the standard. Here, ΔL*, Δa*, Δb*, and consequently ΔE* are expressed in compatible units. ΔL* signifies the variation between lightness (where the maximum L* = 100) and darkness (where the minimum L* = 0), Δa* represents the difference between green (–a*) and red (+a*), and Δb* indicates the difference between yellow (+b*) and blue (–b*).

The washing fastness of the dyed fibers was assessed in accordance with ISO 105-C06 A1S standards. ISO 105-C06 A1S is a standard that evaluates the color fastness of textile products during washing. This test determines how resistant the fiber's color is to effects such as fading and multifiber contamination during the washing process. The test examines the color change and transfer after washing the fiber at a specific temperature and detergent; the results are scored from 1 to 5 (1 = feeble, 5 = excellent resistance). This standard is used primarily in the textile sector for quality control and durability evaluations and ensures that the products have a long life after washing. The specific tests were administered utilizing the SDL Atlas Linitest for washing fastness during 30 min at 40°C. An SDC multifiber strip fabric was employed as an adjacent material during the evaluations. The fastness assessments were conducted with adjacent multifiber fabrics and analyzed based on gray scales. ECE non-phosphate standard detergent was utilized in the washing fastness trials.

A Perkin Elmer Spectrum BX instrument equipped with ATR-FTIR capability was employed to investigate the impact of β-CD on the dyeing procedure of soybean fibers. Spectra were recorded within the wave number range of 650–4000 cm^{-1} with a resolution of 2 cm^{-1}. FT-IR analyses were conducted on fibers dyed with reactive dyestuffs at 50°C, considering microscope images and color measurement results, to ascertain whether cyclodextrins influence the dyeing process of soybean fibers.

3. RESULTS AND DISCUSSION

3.1. Optical Microscope Analysis

After dyeing at three different temperatures in the presence of two different dyestuffs and cyclodextrin, optical microscope images of the soybean fibers were captured. The resulting fiber images are presented in Figure 1.

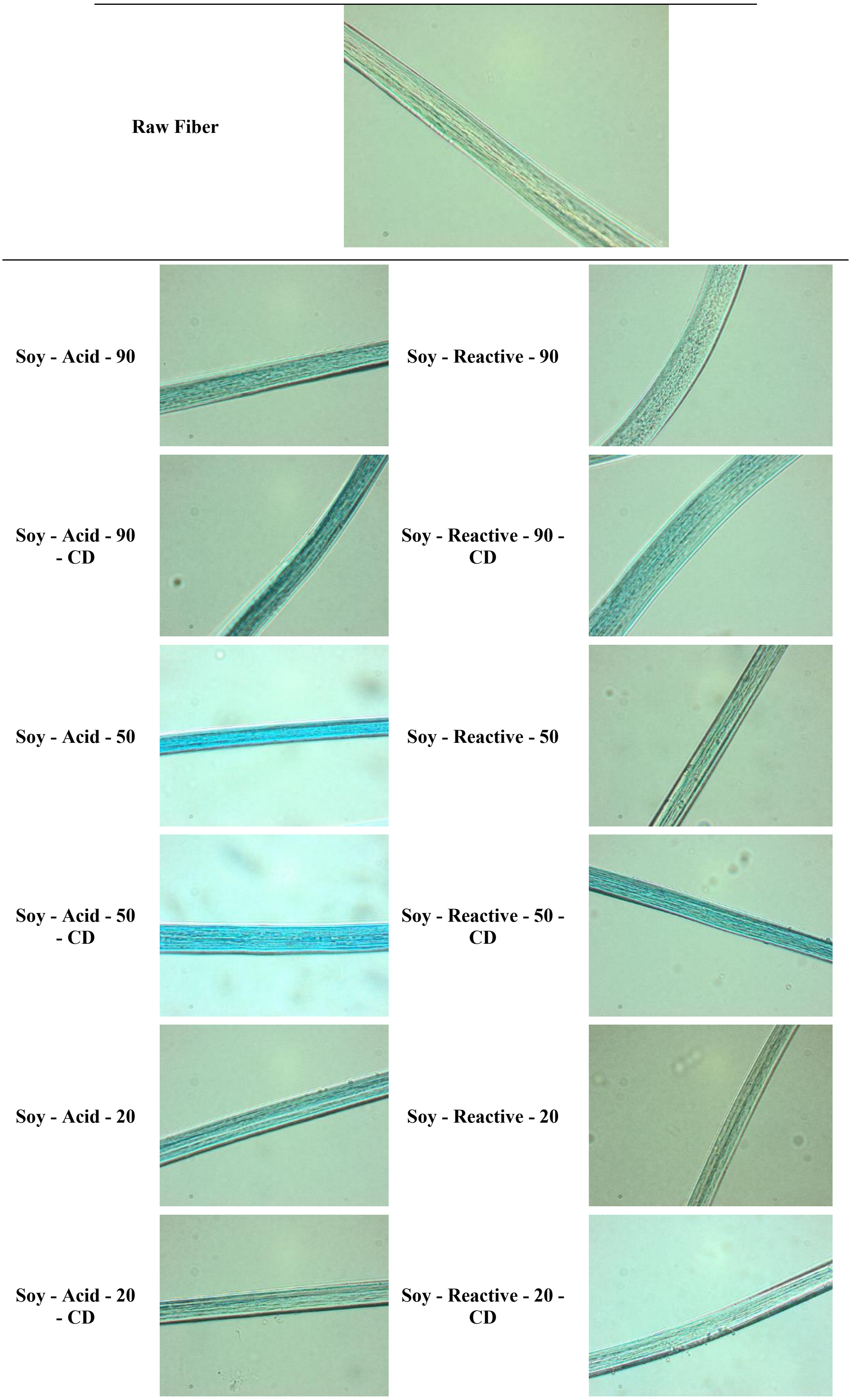


Figure 1. Optic microscope images of dyed soybean fibers

Upon examining the figures in Figure 1, it is evident that the structure of the soybean fibers deteriorates as the temperature increases. Moreover, when the temperature was increased to 90°C during the experiment, the fibers hardened, also according to the literature [5, 6].

This observation confirms that soybean fibers are unsuitable for dyeing at high temperatures, as reported in the literature. However, when comparing the two types of dyestuffs (acid and reactive), it was noted that neither caused any fiber splitting. The molecular structure and interaction mechanisms of cyclodextrins complement the fibers' characteristics. Cyclodextrins adhere to the fiber surface and enhance the bonding of dye molecules to the fibers. This results in a more uniform dye distribution and prevents fiber structure deterioration. Additionally, cyclodextrins are typically natural and biocompatible, minimizing the likelihood of damaging textile fibers. Their low potential for causing fiber damage ensures the preservation of fiber structure and durability [18, 20]. Consistent with the literature, the presence of cyclodextrins during the dyeing process does not degrade the fiber surface. Optical microscopy images revealed noticeable surface changes in soybean fibers dyed at elevated temperatures, particularly at 90 °C, where fibers exhibited increased rigidity and irregularity. While these visual observations suggest potential thermal effects, it is important to note that optical microscopy provides only limited insight into sub-surface or molecular-level changes.In samples dyed with β-cyclodextrin, the fibers exhibited more uniform coloration and smoother surface texture compared to conventionally dyed counterparts. Although this may indicate a stabilizing effect, the precise mechanism of interaction between cyclodextrins and the fiber matrix cannot be directly confirmed through surface imaging alone. Existing studies have demonstrated that cyclodextrins can improve dye dispersion, reduce aggregation, and promote more even dye-fiber interactions [23, 24]. Thus, the potential protective role of cyclodextrins proposed in this study is supported by both visual observations and prior literature.

3.2. Color Measurement and FastnessProperties

In Table 1, the color values obtained as a result of dyeing soybean fibers with acid and reactive dyestuff are given. In addition, the total color difference (ΔE) values of the trials using cyclodextrin were also calculated. All spectrophotometric measurements were conducted in triplicate (n = 3). Standard deviations are provided for K/S, R%, and CIELAB parameters (L, a*, b*) to reflect measurement variability. λ_max was constant within each dye type as 620 nm for acid dyes and 625 nm for reactive dyes.

When evaluating the color measurement results of soybean fibers after dyeing, it was observed that the fibers became darker as the dyeing temperature increased, especially with acid and reactive dyestuffs. This effect was more pronounced with reactive dyestuffs, which interact with temperature to bind to the textile fibers and generate color. The intensity of this reaction can vary based on factors such as the dyestuff's chemical structure, the chemicals used in the process, the dyeing temperature, and the duration. Generally, higher temperatures lead to improved performance of reactive dyes and the attainment of darker colors [28, 29]. As expected, lower L* values were observed in samples dyed with cyclodextrins, indicating darker and more intense shades. Slight increases in a* values suggested minimal shifts toward the red component, consistent with the deeper blue-violet tone perceived in cyclodextrin-assisted dyeing. These findings align with the increased K/S and ΔE values, confirming the enhanced dye uptake and improved color depth in the presence of cyclodextrins.

Table 1. Color measurement results of dyed soybean fibers

	Fibers	R%	K/S	ΔE	L*	a*	b*
	Soy - 90	2.99 ± 0.08	15.75 ± 0.42	NA	38.7 ± 0.4	1.42 ± 0.1	-17.74 ± 0.3
	Soy - 90 - CD	1.57 ± 0.05	30.86 ± 0.55	20.68	33.4 ± 0.3	1.95 ± 0.1	-28.89 ± 0.4
AcidDyes	Soy - 50	3.21 ± 0.09	14.59 ± 0.37	NA	42.3 ± 0.4	2.15 ± 0.1	-30.03 ± 0.3
	Soy - 50 - CD	2.57 ± 0.08	18.47 ± 0.46	4.31	40.1 ± 0.4	2.83 ± 0.1	-33.20 ± 0.4
	Soy - 20	3.43 ± 0.10	13.59 ± 0.35	NA	44.5 ± 0.5	1.87 ± 0.1	-30.09 ± 0.4
	Soy - 20 - CD	2.25 ± 0.07	21.23 ± 0.51	11.53	39.6 ± 0.4	2.45 ± 0.1	-37.69 ± 0.4
	Soy - 90	5.01 ± 0.12	9.01 ± 0.28	NA	46.2 ± 0.4	0.95 ± 0.1	-16.19 ± 0.3
	Soy - 90 - CD	3.70 ± 0.10	12.53 ± 0.33	5.25	44.3 ± 0.3	1.12 ± 0.1	-15.49 ± 0.3
ReactiveDyes	Soy - 50	5.00 ± 0.11	8.90 ± 0.26	NA	47.1 ± 0.4	0.83 ± 0.1	-16.73 ± 0.3
	Soy - 50 - CD	4.63 ± 0.10	9.82 ± 0.31	1.54	45.9 ± 0.3	1.04 ± 0.1	-16.01 ± 0.3
	Soy - 20	12.39 ± 0.20	3.09 ± 0.21	NA	58.7 ± 0.5	0.51 ± 0.1	-5.58 ± 0.2
	Soy - 20 - CD	11.27 ± 0.18	3.49 ± 0.25	2.75	57.4 ± 0.5	0.64 ± 0.1	-7.91 ± 0.2

NA: Not applicable

The significant increase in K/S values observed in cyclodextrin-assisted dyeing can be explained by the inclusion complex formation between β-cyclodextrin (β-CD) and the dye molecules. β-CD has a toroidal structure with a hydrophobic inner cavity and a hydrophilic outer surface, which enables the encapsulation of hydrophobic aromatic moieties of dye molecules. This inclusion complexation increases the solubility and stability of dye molecules in the aqueous dye bath, reducing their aggregation and leading to a more controlled and gradual release onto the fiber surface. Consequently, better penetration and more uniform dye distribution are achieved, which is reflected in the higher K/S values.

In addition to inclusion complexation, hydrogen bonding interactions may also contribute to this enhancement. The hydroxyl groups of β-CD can form hydrogen bonds with functional groups of the dye molecules, such as $–SO_3^-$, $–NH_2$, or –OH groups, improving the effective dye concentration available for adsorption on the fiber surface. Although this study did not directly analyze these molecular interactions, similar mechanisms have been reported in previous research on cyclodextrin-assisted dyeing of protein and cellulosic fibers [23, 26, 27]. These findings collectively support the hypothesis that β-CD not only acts as a solubilizing and dispersing agent but also facilitates stronger dye-fiber interactions, ultimately resulting in improved color yield.

Therefore, darker dyeing results are expected. Additionally, when evaluating the color measurement results across all temperatures and dyestuff types, it was observed that soybean fibers appeared darker blue in the presence of cyclodextrin. The color strength (K/S values) increased when cyclodextrins were used as auxiliary substances compared to conventional dyeing methods. Furthermore, when examining the ΔE values, it was evident that there was a noticeable color difference between fibers dyed with cyclodextrin and those dyed using the conventional method. Cyclodextrins are oligosaccharides with hydroxyl (-OH) groups that form internal cavities in the ring structure of glucose units and can make hydrogen bonds on their outer surface. These structures allow dyes to be transported to the fibers more efficiently through encapsulation. The internal cavity of cyclodextrins increases the solubility of lipophilic dye molecules in particular and increases the binding rate of dyes to the fibers. The hydrophilic groups on their outer surfaces strengthen the interaction with the fibers by ensuring that the dyes dissolve better in water. These mechanisms allow cyclodextrins to increase dye uptake in the textile dyeing, allowing more efficient and environmentally friendly results. Consequently, it can be concluded that cyclodextrins enhance dye uptake and contribute to improved color intensity [18, 23].

The images of the dyed soybean fibers in Figure 2 play a crucial role in validating the quantitative results of the color measurements and fastness tests.

These images provide a comparative view of how different dyeing conditions influence the final appearance of the fibers, with fibers dyed in cyclodextrins showing enhanced color uniformity compared to conventionally dyed fibers. Additionally, the figure confirms the findings from the microscopic analysis, with fibers dyed at 90°C appearing more degraded. These visual comparisons, therefore, further validate the quantitative results of the color measurements and fastness tests.The washing fastness values are shown in Table 2 as a result of dyeing of soybean fibers.

When evaluating the washing fastness results, both acid and reactive dyes generally produced good fastness grades (4) on soybean fibers. However, lower grades (2–3) were recorded for wool fibers in the multifiber strips, especially with reactive dyes. This can be explained by the different affinities of the dyestuff groups. Acid dyes exhibit strong affinity for protein-based fibers such as wool, forming ionic and hydrogen bonds, which results in better fastness. In contrast, the reactive dye used in this study is not specifically designed for wool dyeing under neutral conditions. Reactive dyes require alkaline conditions to form covalent bonds; under neutral pH, their interaction with wool fibers occurs mainly through weak physical adsorption. Additionally, reactive dyes are prone to hydrolysis during dyeing. Hydrolyzed dye molecules lose their reactive sites and can only adhere to the fiber through weak van der Waals or hydrogen bonds, which are easily removed during washing. This behavior likely contributed to the lower washing fastness grades observed for wool fibers when reactive dyes were used.

Table 2. Washing fastness test results of dyed soybean fibers

	Fibers	A02	WO	PAN	PET	PA 6.6	CO	CA
Acid Dyes	Soy - 90	4-5	3	4-5	4-5	4-5	4	4-5
	Soy - 90 - CD	4-5	3-4	4-5	4-5	4-5	4-5	4-5
	Soy - 50	4-5	3	4-5	4-5	4-5	4	4-5
	Soy - 50 - CD	4-5	3-4	4-5	4-5	4-5	4-5	4-5
	Soy - 20	4-5	3	4-5	4-5	4-5	4	4-5
	Soy - 20 - CD	4-5	3-4	4-5	4-5	4-5	4-5	4-5
Reactive Dyes	Soy - 90	4-5	2-3	4-5	4-5	4-5	3-4	4-5
	Soy - 90 - CD	4-5	3	4-5	4-5	4-5	4	4-5
	Soy - 50	4-5	2-3	4-5	4-5	4-5	3-4	4-5
	Soy - 50 - CD	4-5	3	4-5	4-5	4-5	4	4-5
	Soy - 20	4-5	2-3	4-5	4-5	4-5	3-4	4-5
	Soy - 20 - CD	4-5	3	4-5	4-5	4-5	4	4-5

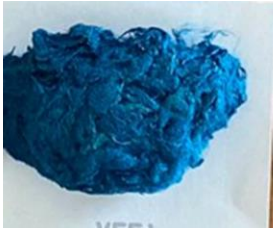
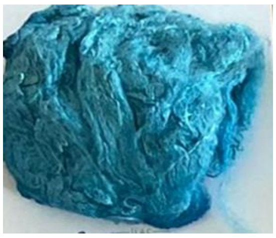
Raw Fiber			
Soy - Acid - 90		**Soy - Reactive - 90**	
Soy - Acid - 90 - CD		**Soy - Reactive - 90 - CD**	
Soy - Acid - 50		**Soy - Reactive - 50**	
Soy - Acid - 50 - CD		**Soy - Reactive - 50 - CD**	
Soy - Acid - 20		**Soy - Reactive - 20**	
Soy - Acid - 20 - CD		**Soy - Reactive - 20 - CD**	

Figure 2. Images of soybean fibers dyed at different temperatures and in the presence of cyclodextrin

3.3. Effects of Cyclodextrinon Chemical Properties

Figure 3 illustrates the FTIR spectra of β-CD, while Figure 4 displays the FT-IR spectra of dyeing with β-CD juxtaposed with conventional dyeing of soybean fibers.

The FTIR spectrum of β-CD displayed a distinct and intense peak at 2,854 $cm^{-1,}$ corresponding to the asymmetric/symmetric stretching of C–H bonds. Another prominent peak at 1,650 cm^{-1} represented the deformation bands of H–O–H bonds in water within β-CD. Peaks observed at 1,153 cm^{-1} and 1,029 cm^{-1} indicated the stretching of C–H bonds and C–H and C–O bonds, respectively. Additionally, the absorption of C–O–C vibration was noted at 1,153 cm^{-1} [30].

Soybean fibers have a complex chemical structure comprising carbohydrate, protein, and fat components. When analyzing the FTIR spectrum of soybean fiber, strong and broad absorption of hydroxyl groups is observed at around 3300 cm^{-1}. In comparison, absorption of methyl and methylene groups occurs at approximately 2900 cm^{-1}. The absorption of the C-O-C glycoside bond takes place at approximately 1050 cm^{-1}. Additionally, the FTIR spectrum of soybean fiber includes characteristic absorption bands of amide and acid groups of proteins, absorption bands of aromatic rings of lignin, and absorption bands of CH_2 bonds in oil. The amide group absorption is typically found between approximately 1650-1550 cm^{-1}, while the acid group absorption occurs in the 1700-1600 cm^{-1} range. The absorption bands corresponding to the aromatic rings of lignin are observed within the range of 1600-1500 cm^{-1}. The absorption bands related to CH_2 bonds in the oil are present in the range of 3000-2800 cm^{-1} [5, 31]. When comparing the FTIR spectra, no significant differences are observed between the spectra in the presence of cyclodextrin, except for a new and more intense peak detected solely in the 2361 cm^{-1} band. This peak corresponds to CO_2 and results from the measurement conditions [5]. Based on this data, it can be concluded that the presence of cyclodextrin does not form any new bonds with soybean fibers. However, it is believed that the color yield increases through physical bonding.

The FTIR spectrum of β-cyclodextrin exhibited characteristic broad O–H stretching around 3380 cm^{-1}, C–H stretching at 2925 cm^{-1}, and strong C–O–C stretching vibrations in the 1020–1150 cm^{-1} region, consistent with its cyclic oligosaccharide structure [23]. The untreated soybean fibers showed typical protein-based peaks, including amide I (C=O stretching) at ~1630 cm^{-1}, amide II (N–H bending) at ~1530 cm^{-1}, and amide III (C–N stretching) at ~1235 cm^{-1} [1]. After β-CD treatment, no new peaks were observed in the soybean fiber spectrum, confirming the absence of covalent bond formation between β-CD and the fibers. However, a slight broadening and shift in the O–H stretching band (from 3380 to ~3365 cm^{-1}) and minor intensity changes in the amide I and II regions were detected. These subtle changes may indicate hydrogen bonding or van der Waals interactions between the hydroxyl groups of β-CD and the amino or carbonyl groups of soybean fibers. The absence of new chemical bonds suggests that β-CD was physically adsorbed onto the fiber surface rather than chemically grafted, which is consistent with reports on β-CD application to wool and cotton fibers [21, 24]. Although FTIR analysis alone cannot fully confirm the interaction mechanism, the observed spectral changes, together with the enhanced K/S values, support the hypothesis that such physical interactions contributed to better dye dispersion and uniform adsorption on the fiber surface.

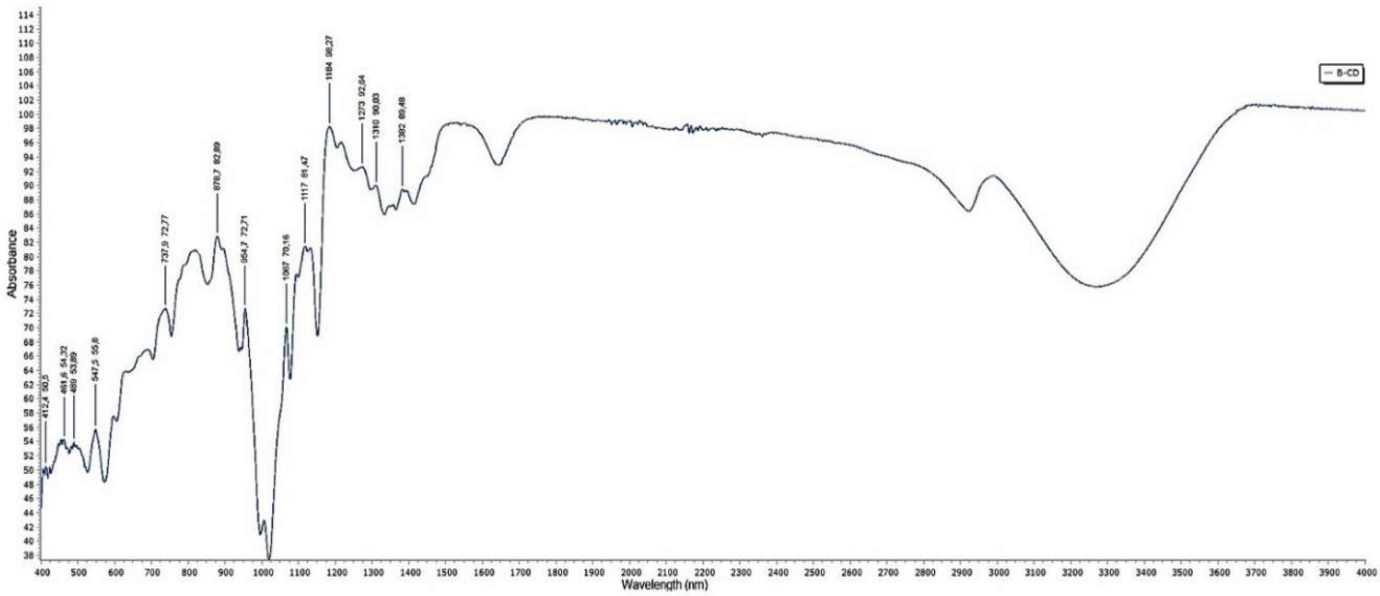


Figure 3. FT-IR spectra of β-CD

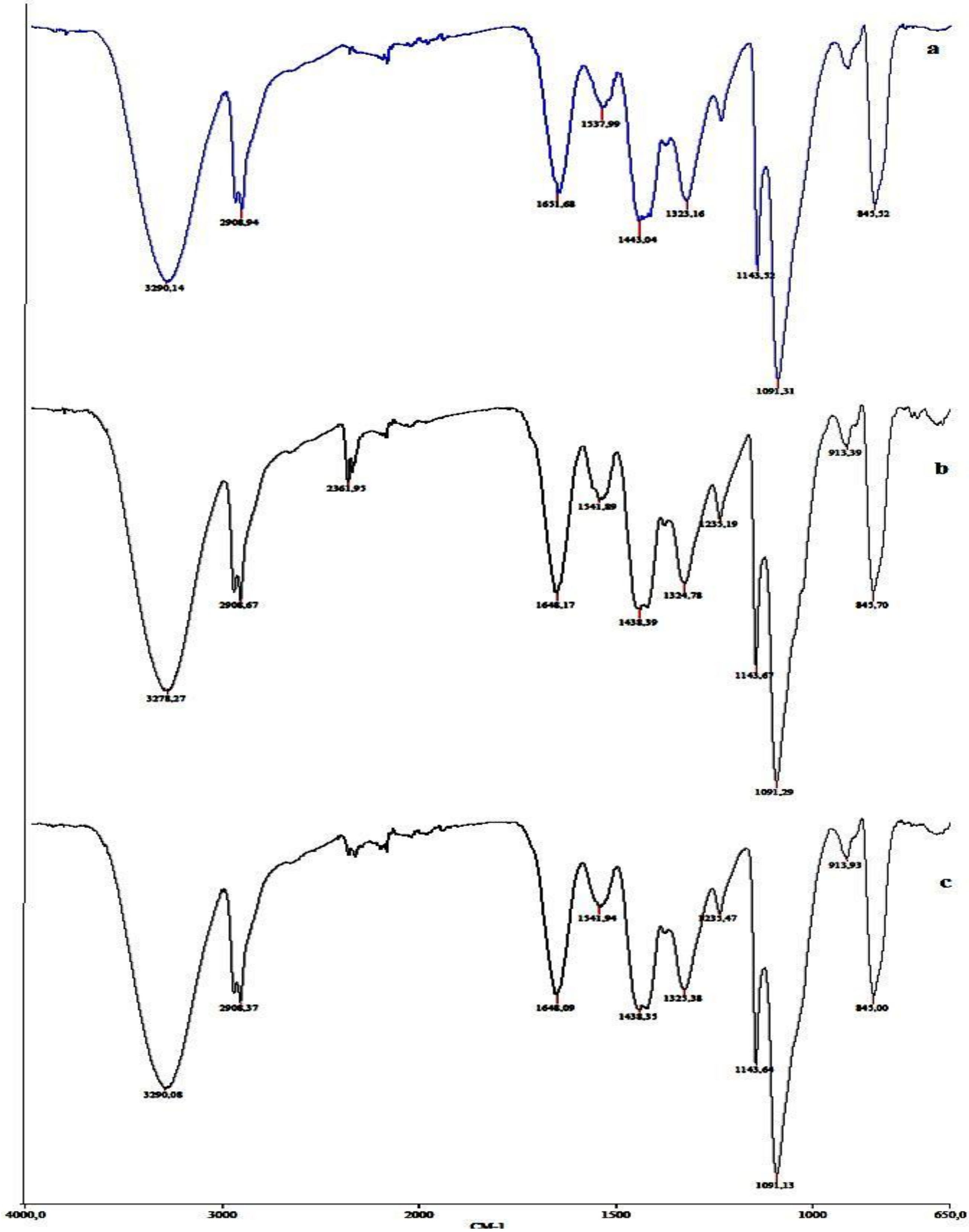


Figure 4. Fourier Transform–infrared (FTIR) spectra were obtained for (A) soybean fiber, (B) dyeing with β-CD, and (C) dyeing without β-CD.

3.4. Effect of Dyeing Temperature on the Soybean Fibers

When evaluating the effect of dyeing temperature on the soybean fibers, it was observed that increasing temperature generally led to deeper shades, particularly in reactive dyeing processes. Higher temperatures enhance dye diffusion and fixation, contributing to increased color yield. However, optical microscopy revealed that dyeing at 90 °C caused notable structural deterioration in the fibers, confirming literature reports that soybean fibers are sensitive to high temperatures. The observed decrease in fiber integrity at 90 °C is in line with the findings of Tahir et al. (2024), who reported significant weakening of soybean fibers at temperatures above 100 °C due to thermal degradation of protein structures [1]. Similarly, Choi et al. (2005) emphasized the importance of moderate dyeing temperatures to preserve fiber morphology.The best balance between color yield and fiber integrity was observed at 50 °C, which emerged as the most suitable dyeing temperature. The addition of cyclodextrins had a pronounced positive effect on dye uptake and color uniformity at all temperatures. The K/S values were consistently higher in samples dyed with cyclodextrin compared to those dyed conventionally. Moreover, cyclodextrin-assisted dyeing resulted in slightly better washing fastness, particularly in cotton and wool components of the multifiber strips [12]. These improvements are attributed to the inclusion complex formation between β-cyclodextrin and the dye molecules, which enhances dye solubility and enables a more gradual and uniform dye release onto the fiber surface, thereby improving penetration and retention. Our findings regarding the enhanced color yield and fastness with β-cyclodextrin are consistent with the review by Bezerra et al. (2020), which reported similar improvements in dye performance on cotton and wool fibers. Moreover, studies by El-Sayed et al. (2021) and Cireli&Yurdakul (2006) demonstrated that cyclodextrins improve dye solubility and uniformity, supporting our interpretation of their role in promoting more effective dye-fiber interactions, even under neutral pH [21, 23, 24]. A clear distinction was also noted between acid and reactive dyes. Acid dyes generally resulted in more intense shades at lower temperatures, likely due to their better affinity with protein-based fibers. Reactive dyes showed more significant improvements in dye uptake at higher temperatures, which is expected due to their temperature-dependent fixation behavior. However, without alkali in the CD-assisted reactive dyeing process, fixation was driven mainly by physical interactions rather than covalent bonding. Despite this, cyclodextrins still facilitated enhanced dye-fiber interaction for both dye classes, indicating their versatility as auxiliary agents. Compared to previous work by Liu et al. (2022), who focused on fixation efficiency in soybean/flax blends, our study provides new insight into pure soybean fibers and suggests that cyclodextrin may serve as an eco-friendly alternative to alkali-based fixation systems in certain cases [8].

4. CONCLUSION

This study demonstrated that β-cyclodextrin significantly enhances dye uptake and improves color uniformity in soybean fiber dyeing with both acid and reactive dyes. The highest K/S value increased from 15.75 to 30.86 (acid dyeing at 90 °C), while ΔE values confirmed notable color differences between cyclodextrin-assisted and conventional dyeing (ΔE up to 20.68). Washing fastness results also improved by approximately half a grade, particularly in wool and cotton components of multifiber strips, indicating stronger dye adhesion to the fibers.

The inclusion of β-cyclodextrin did not cause any observable fiber damage, as confirmed by optical microscopy, and helped maintain smoother fiber surfaces even at elevated temperatures. FTIR analysis indicated no covalent bonding between β-CD and soybean fibers, suggesting that the improved dyeing performance is primarily due to physical interactions, such as inclusion complexation and hydrogen bonding, which increase dye solubility and enable more controlled dye transfer.

The novel contribution of this study lies in its focus on pure soybean fibers, which are more thermally sensitive than natural protein-based fibers, and in evaluating β-cyclodextrin as an environmentally friendly auxiliary under neutral dyeing conditions. The findings suggest that β-cyclodextrin can serve as a potential alternative to conventional alkali-based auxiliaries, reducing the environmental load of dyeing processes. These findings underscore the promising potential of cyclodextrins as environmentally friendly auxiliaries in textile dyeing processes. This discovery opens up new avenues for sustainable textile production, offering hope for a more eco-friendly future. Future studies could delve deeper into the long-term durability of these effects and the practicality of using cyclodextrins in large-scale dyeing operations.

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