

Journal of Textiles, Coloration and Polymer Science https://jtcps.journals.ekb.eg/



An Overview of the Dyeing Process of Lyocell Fabric and its Blends

Heba Ghazal^a, Nadeen Khaleed^{a*}, Sohaila Shaker^a, and Ahmed G. Hassabo^{b*}

^a Benha University, Faculty of Applied Arts, Printing, Dyeing and Finishing Department, Benha, Egypt

^b National Research Centre (Scopus affiliation ID 60014618), Textile Research and Technology Institute, Pretreatment and Finishing of Cellulose-based Textiles Department, 33 El-Behouth St. (former El-Tahrir str.), Dokki, P.O. 12622, Giza, Egypt

Abstract

This study aims to investigate the lyocell fabric which become very popular in recent times among the researcher due to its higher strength, rigid crystalline morphology, being completely non-toxic, biodegradable manufactured by an eco-friendly non-polluting process, and its proximity to cotton. This study exhibits the effect of both synthetic dyes and natural dyes on lyocell fabric. Synthetic dyes include reactive dyes and vat dyes, while natural dyes include pomegranate peel dyes, Mexican marigold flowers, and gardenia dyes. Also dyeing lyocell with blended fabrics such as cotton and silk and polyamide.

Keywords: Lyocell Fiber, biodegradable, vat dyes, Pomegranate Peel, Mexican marigold flower, Gardenia dyes, blended fabrics.

Introduction

Lyocell is the generic term for "regenerated cellulosic fibers" which are obtained by spinning of dissolved wood pulp in different organic solvent. Lyocell fibers are derived from 100% natural wood pulp using a solvent spinning process. Lyocell was first made commercially available in 1988 by Acordis (then Courtaulds plc) under the brand name "Tencel®"[1]

The abbreviation CLY stands for lyocell and it is derived from the Latin word "lyein", meaning "dissolvable", and "cell", another Latin word meaning "cellulose"[2]. Lyocell fibers is defined as cellulosic fibers that are produced by regenerating cellulose into fiber form out of a solution in Nmethylmorpholine-N-oxide (NMMO)[3]. Solvent spun lyocell fibers consist of crystalline cellulose-II and amorphous cellulose, and have a higher degree of crystallinity (80%) in comparison with other regenerated cellulosic fibers, such as modal (49 %) and viscose (41 %)[4]. It is 100 % natural in origin, as it is made from wood pulp, and is fully biodegradable. Lyocell fibers are mostly used for apparel fabrics but it has been shown that, due to the fibrillating property very interesting nonwoven fabrics can also be made as well[5].

Lyocell fiber has several advantages over natural and manmade fibers. Since it is possible to recycle almost 99% of the organic solvent used in the obtaining of lyocell fiber and water is the only substance used in the coagulation bath without needing any acid or alkali, lyocell fiber can be considered harmless from toxicological and dermatologic aspects[6]. In addition, the dry tenacity value of lyocell fiber is much higher than those of other cellulosic fibers and it comes close to that of polyester fiber. Therefore, lyocell can be considered to be one of fibers which can be used blended with other fibers in order to manufacture strong yarns and fabrics. The most important characteristic property of lyocell fiber is its fibrillation behaviour, which arises from the increased fiber fragility due to the fact that the fiber has a crystallization degree around 90%[7].

Lyocell is 100% natural in origin as it is made from wood pulp and is fully biodegradable. Lyocell has an inherent soft handle and a tendency to fibrillate, which sets it apart from other regenerated cellulosic fibers[8]. The natural characteristics of lyocell, such as high moisture absorption and soft handle, give it a fresh feeling and impart wearing comfort, making it an ideal fiber for garments. As compared with viscose, lyocell has high strength in both the wet and dry states, with the dry tenacity approaching that of polyester[8]. Lyocell fibers can be dyed using the dye classes and procedures suitable for all other cellulosic fibers. Lyocell has (generally) much higher dyeability than all other cellulosic fibers and requires specialized dyeing conditions. Some of

*Corresponding author : Ahmed G. Hassabo, E-mail: aga.hassabo@hotmail.com, Tel. 01102255513 Receive Date: 05 July 2023, Accept Date: 06 August 2023 DOI: 10.21608/JTCPS.2023.221105.1211

^{©2024} National Information and Documentation Center (NIDOC)

the multifunction reactive dyes are suitable for controlling fibrillation[9]. The unique crystalline structure of lyocell is responsible for its propensity to fibrillate. Fibrillation occurs when the fiber is subjected to mechanical action in the wet state. The phenomenon is called "primary fibrillation"[10].

Swelling of the porous regions of the fiber breaks the hydrogen bonds linking the crystalline units and forcing them apart. Because of the mechanical action, the outer crystalline regions may peel away, and "Secondary fibrillation" produces peels called fibrils. This property of fibrillation can be advantageous for creating a variety of fabric aesthetics and appearances ranging from a fully fibrillated fiber (the peach-skin effect) to a clean, smooth appearance by suppressing fibrillation. At the same time, fibrillation can change the handle of the cloth and, if fibrillation is not controlled, these microfibers get entangled and give rise to 'pilling'. Thus, the processing of lyocell requires utmost care and is more challenging than the processing of other regenerated fibers[10].

Lyocell is completely non-toxic, biodegradable manufactured by an eco-friendly non-polluting process. Moreover, Lyocell fibers are claimed to offer environmental advantages over other regenerated fibers concerning the recyclability of the solvent and the renewable source of cellulosic starting material[8].

Lyocell shares many properties with other cellulosic fibers such as cotton, linen, ramie, and rayon. It is soft, absorbent, comfortable, very strong when wet or dry, and resistant to wrinkles; it can be machine- or hand-washed or drycleaned, it drapes well, and it can be dyed with many colors, as well as simulating a variety of textures like silk. Fabrics made of 100% lyocell or blends have a luxurious and silky hand with vibrant colors. Unlike the previous generation of cellulosic fibers, the new generation of lyocell has a tenacity that withstands rigorous processing. Across the fashion spectrum, it has been embraced by well-known designers and retailers. Lyocell fiber blends well with other natural and synthetic fibers such as cotton, polyester, lycra, or wool adding comfort and performance[11].

The principles of the lyocell production process are simple[12]. The wood pulp is firstly wetted out with dilute aqueous amine oxide to penetrate the pulp fibers fully. The subsequent removal of the excess water under heat and vacuum is a very effective way of making a homogeneous solution with a minimum of undissolved pulp particles and air bubbles[13]. The solution is highly viscous at its operating temperature (90–120 °C) and must be processed in similar high-pressure equipment to that used in melt polymer systems[13]. The fibers are formed by spinning into an air gap and then coagulating in a water/amine oxide bath. They are then washed and dried and cut. The wash liquors are recovered, purified, concentrated, and then recycled. The process description below applies to the two commercial-scale operations developed bv Courtaulds and Lenzing. Courtaulds and Lenzing developed their production methodology independently[13]. The process description in the scheme (1) applies to both systems, but the process engineering is different. Courtaulds chose to follow the tow route, whereby the fiber is made as a continuous tow band through washing before being dried, mechanically crimped, and finally cut into staples. This is much more reminiscent of the technology used for the manufacture of Courtaulds' acrylic fiber 'Courtelle'. Lenzing chose a different following the traditional approach, viscose methodology of cutting the fiber while wet and washing after cutting, allowing the crimp to develop during the relaxed washing step. The biggest benefit of this new production technology relates to the environmental impact which needs high levels of process control to manage the environmental aspects of production, as chemicals such as carbon disulphide, caustic soda, and sulphuric acid are utilised. Lenzing has shown in its viscose production worldwide that this can be controlled, but the lyocell process is fundamentally cleaner.

The solvent used in the production is reclaimed to an efficiency of higher than 99.8% and reused within the production. The process is outlined schematically in the **scheme (1)**, which is based on the Courtaulds production where the fibers are washed, finished, dried, and crimped as a continuous tow before cutting to the required staple length. In Lenzing production, the fibers are cut immediately after spinning and washed as a cut fiber bed[13].



Scheme 1. The Lyocell production process

Dyeing of lyocell fabric by utilizing synthetic dyes

Lyocell is a cellulosic fiber and can therefore be dyed with the same dyestuff types as other cellulosic fibers, such as reactive dyes. However, its fundamental dyeing properties generate a unique profile, and dye application methods are needed that are specific to lyocell[13]. The dye yield, measured as color value, of lyocell, is exceptionally high. Generally, the dye yield obtained on lyocell is more reminiscent of mercerized cotton than other manmade cellulosic. The high dye yield, though, does mean that steps have to be taken to improve migration and leveling properties. In exhaust dyeing, this means the adoption of high-temperature migration application methods with slower alkali addition to ensure level dyeing. In cold pad batch reactive dye application, methods that slow the fixation rate are beneficial to allow the dyes more time to penetrate the fiber fully[13].

Dyeing lyocell fabric by utilizing reactive dyes

Khan et al. managed to dye lyocell by using the bifunctional and polyfunctional reactive dye (CI Reactive Red 282) and (CI Reactive Red 286). The dyeing was carried out at four different shade% and PH 10.5. Before the dyeing procedure, a mild scouring occurred which is sufficient to remove the possible dust, dirt, or spin finish chemicals that adhere to lyocell. The other chemicals used in this experiment e.g. wetting agent, sequestering agent, detergent, Glauber's salt, sodium hydroxide, and sodium carbonate. The dyeing process was carried out by using the concentration of dyebath liquor 1:10 at 60°C for 45min then the samples had been washed using 2g/l detergent for 10 min[14].

The result of this study is that the exhaustion% of bifunctional reactive dye in lyocell is greater than that of polyfunctional reactive dye. But a significant amount of bifunctional reactive dye was lost in the effluent during the wash-off process which made a significant difference in fixation%. The case of polyfunctional reactive dye showed more than 90% fixation of the exhausted dye. But the overall fixation of the applied dye is less than that of the bifunctional reactive dye. It was observed that polyfunctional reactive dye showed better fixation with darker shades more than 3% shade. All the dyed samples showed very good to excellent color fastness[14].

Another study done by Aijaz et al., to dye Lyocell fabrics with Reactive dyeing via exhaust dyeing technique involves two major steps, i) exhaustion, where textile substrate initially attracts the dye molecules in the presence of an electrolyte, and ii) fixation, where textile substrate captures the absorbed dye molecules (i.e. the substrate makes covalent bonding with dye molecules in presence of strong alkali). In the entire dyeing process, the textile substrate remains immersed in the dye bath. This way, there is simultaneous dye build-up on the fiber surface and consistent decrement in the dye concentration. Moreover, regulated dye bath temperature is responsible for the controlled movement of the dye molecules for even dye diffusion and smooth dye transport within the fiber polymer system[15, 16].

The dyeing procedure was carried out using ultrasonic (US) energy which produces a higher color yield compared to conventional (CN) dyeing techniques. the use of US energy would result in more promising and eco-friendly dyeing outcomes for lyocell fabrics via the exhaust process. Moreover, we observed that the use of ultrasonic energy for dyeing lyocell fabric via pad ultrasonic batch route showed an excellent dyeing performance. It's reported that ultrasound-assisted exhaust reactive dyeing (US exhaust dyeing) of lyocell fabric presents the comparative analysis of dyeing outcomes and effluent discharge with the CN exhaust dyeing process. For CN exhaust dyeing, the lyocell fabric samples were immersed in the dye bath with a liquor ratio of 15:1 containing dye (2% o.m.f) and NaCl (10-70 g/L) for 30 min. Later on, Na2CO3 (2-10 g/L) was added to the dye bath, and the process was further carried out for dye fixation for 60 min. Dyeing was carried out in high temperature (HT) dyeing machine[17]. After dyeing, the wash-off process to remove unfixed dye from the surface of the substrate as shown in scheme (2)



The effect of temperature on the dyeing outcomes of lyocell fabric samples dyed with CI Reactive Red 195 and C I Reactive Blue 250 using CN exhaust dyeing technique. It was witnessed that both dyes initially showed increasing in the color yield with increasing temperature from 30 to 60 °C followed by a decline at 70 °C and onwards. Since, enhanced temperature could hasten the rate of dye hydrolysis, thus, reduced dye fixation was observed at elevated temperatures. However, when lyocell fabric samples were dyed with the assistance of US energy, the dyeing results obtained were comparatively higher for all samples for both dyes than those dyed with the CN exhaust dyeing process. optimum dyeing results for two dyes CI Reactive Red 195 (color yield 15.66, dye fixation 86.87%) and CI Reactive Blue 250 (color yield 16.99, dye fixation 86.77%) were achieved at 50 °C and were attributed to the improved rate of mass transfer and dye reaction owing to cavitation phenomenon. Beyond 50°C, no significant improvement in the dyeing performance of samples was noticed, these findings were ascribed to the higher dye hydrolysis and reduced dye substantivity due to the enhanced rate of process reaction at higher temperatures in the presence of US energy. Therefore, 50°C temperature was selected for further analysis. It is concluded that Colorfastness to light and washing results were good[17].

under optimized conditions of two processes, USdyed samples presented higher dyeing performance (color yield up to ~7%, dye fixation up to 5%) compared to conventionally dyed samples. Additionally, substantial savings in terms of energy (10 °C) and chemical consumption (20 g/L NaCl and 02 g/L Na₂CO₃) were observed for the US dyeing process, which makes the US dyeing process relatively more economical alternative for the successful reactive dyeing of lyocell fabric. Moreover, a ~33% higher production rate was achieved[18].

Although dyeing of lyocell fabric with reactive dyes has resulted in better color yields than cotton there is a problem with the durability of these dyes because substrate and water both have hydroxyl sites because reactive dyes react with these hydroxyl sites. Therefore, there is always a risk of dye hydroxylation during the dyeing process[13]. Therefore, to solve this problem, it is very important to choose dyes that do not react with hydroxyl sites of water and have strong color yield as well as excellent fastness properties and easily reproducible characteristics. Since vat dyes have long been used to dye cellulosic fibers due to their excellent fastness properties, brighter shades, and high color yield[19].

Dyeing lyocell by utilizing vat dye

Vat dye is an anthraquinone-based waterinsoluble dye having a coplanar and multi-ring system, which is an ideal potential dyestuff that can successfully overcome the major problems associated with reactive dyes. The chemical structure of the vat dyes in a reduced state facilitates the diffusion of dye molecules into the substrate surface. The dye molecules adsorbed on the substrate through Vander Waals forces and diffused in the substrate are then oxidized to permanently trap them in a fiber polymer matrix[20]. This permanent hold of dye molecules within the substrate offers excellent fastness properties when compared to reactive dyes. Besides, vat dyes have been used commercially to dye various cellulosic fibers for long times because of their wide shade range, excellent reproducibility, and exceptional fastness properties, i.e. light and rubbing[21].

Hussain et al. managed to dye lyocell fabric utilizing Vat dye with three different colors namely Novasol Blue GF, Novasol Gold Yellow RK, and Novasol Red GB with 0.5% shade. About 100% desized lyocell woven fabric samples of weight 5 gm each were taken. the high-temperature (HT) dyeing machine was used for dyeing purposes. The dyeing process was carried out at a liquor ratio of 1:50, the fabric samples along with dye and auxiliaries (dispersing agent, leveling agent, protective colloid, Sodium hydrosulfite, caustic soda, and hydrogen peroxide) were put into the dye bath and treated for 35 min at 70°C. After that, the samples were taken out and dipped into a new bath containing hydrogen peroxide for the oxidation process. Then oxidation process was carried out for 20 min at 40°C and washed in cold water. Finally, the soaping process was carried out to remove unfixed dyes and residual chemicals for 15 min at 90°C. [21]

Lyocell fabric was dyed by the conventional high-temperature dyeing method. The temperature of the dyeing bath was observed in the range from 50 to 90°C with an interval of 10°C for a constant dyeing time of 30 min. The effect of temperature was observed by evaluating the color strength (K/S) value of the dyeing results was observed beyond 70 °C. This dyeing behavior of lyocell fabric with increments in the dyeing temperature was attributed to the enhanced rate of dye diffusion. Enhanced temperature leads to increased kinetics of dye molecules which helps maximum dye molecules to diffuse into the polymer system of the fabric. Moreover, at the temperature of 70°C and above, the diffusion of dye molecules achieves equilibrium, therefore, there was not a significant change in the color strength of the samples[21].

Table 1	(K/S)	and lio	htness y	values	of	samnles
I abit I.	11/0/	anu ng	nuncss	values	UI.	sampics

	9 · · · · · · · · · · · · · · · · · · ·		
	l *	K/S	
Yellow	52.63	25.89	
Red	19.93	26.09	
Blue	16.87	22.6	

Furthermore, it was observed that the blue dye produced a higher color yield compared to the yellow and red dye. The improved dyeing performance of blue dye was attributed to the smaller size of blue dye molecules.

Dyeing behavior was analyzed concerning dyeing time. The dyeing time was observed from 10 to 50 min with the time interval of 10 min, It was observed that dyed samples showed an improving trend with increasing dyeing time up to 40 min. By increasing the time of dyeing beyond 40 min, there was no significant improvement in the dyeing behavior of the samples. It could be assumed that after 40 min the equilibrium was achieved, thus, dyeing results did not change with further increase of time. Optimized dyeing results including color yield (K/S) and lightness values of samples dyed under optimal dyeing conditions are provided in Table (1). It was observed that samples dyed with yellow vat dye offered the highest K/S value and the least ratio of white dots when compared to samples dyed with red and blue vat dyes[21].

Results showed the 70°C temperature and 40 min of dyeing time achieved optimum dyeing performance. Dyed lyocell samples also showed excellent colorfastness to washing, rubbing, and light as provided in **Table (2)**. Surface morphology analysis showed no major change in the morphology of dyed and undyed samples. Moreover, dyed lyocell samples exhibited comparatively lower effluent weight than cotton and the most widely used cellulosic fiber, making lyocell dyeing relatively more economical and environmentally friendly[21].

Rubbing					
Dye/Dyeing	Dray	Wat	Lightfas	Change	
method	Dry	wei	tness	in color	
Conc-red	4	3/4	6	4/5	
Conc -blue	4/5	4/5	7	4/5	
Conc-yellow	4/5	4/5	7	4/5	

Table 2. Colorfastness results

Dyeing lyocell fabrics by utilizing Natural Dyes

In recent years, there is a continuous intensification of environmental contaminations due to the discharge of toxic and non-biodegradable effluents from the textile manufacturing industries, particularly the coloration industry[22]. The commercially used synthetic dyes (manmade through chemicals) have threatened the worldwide environment. Therefore, the roles of synthetic dyes have been criticized widely[23]. In these aspects, environmentalists have made renaissance attention, to look promptly for eco-friendly qualified products. which are produced from natural resources. In this study, lyocell fabric is investigated with natural dyes extracted from pomegranate peel by using different techniques such as pre-mordanting, meta-mordanting (simultaneous mordanting), and after-mordanting (post-mordanting)[24].

Dyeing lyocell by utilizing Pomegranate Peel Dyes

Rehman et al. managed to dye lyocell fabric utilizing the dried pomegranate powder (5 g) were taken in a thimble (sample container) of apparatus. The solvents 120 mL (ethanol) and 80 mL of distilled water with a liquor ratio of 1:40, in a round bottom flask (solvent container) were heated to 95 °C. The vapors were passed through the tube and raised into the condenser. At the top, the vapors were condensed and dripped down into the thimble. The thimble was drained by the suction effect when the condensed solvent reached the top level of the siphon. The extracted material flew back into the round bottom flask and started mixing with the clean solvent. The

working of the apparatus was continued for 100 min. The extract was purified through a rotary evaporator and subsequently, the filtrate dye solution was used for dyeing the samples of lyocell fabric. It was observed that the color of the dye extracted was brownish-yellow. The weight of the extracted dye was 120 mL[24].

Dyeing process: The fabric samples were mordant before dyeing. Mordanting is a procedure that fixes the dyestuff to the textile materials. Without mordanting the dye uptake ability decreases. Three different methods were used for mordanting of lyocell fabric samples. The differences between these three methods are summarized in **Table (3)**. After mordanting, a dyeing process was carried out to add colors to the sample fabrics.

The lyocell fabric was dyed by exhaust method with extracted dye from pomegranate peel. lyocell fabrics were dyed for 1 h with material to liquor ratio (MLR) of 1:40 in a water shaker dyeing machine, at 90°C. After the dyeing process, the dyed fabric samples were treated with soaping agents and washed with water. Subsequently, they were dried in an oven at 26 °C temperature for 24 h. In three different mordanting methods, pomegranate peel was used with ferrous sulfate and copper sulfate for dyeing lyocell fabric samples at 90°C[24]. **table (4)** shows the dye absorption concentration on the surface of Tencel fabric by using K/S values.

The values of dyed lyocell fabrics. It explains that a lower value of L* has higher darker shades and a higher value of L* has lighter shades for lyocell fabric. Due to chemical mordants, the highest color value (K/S = 4.72) was found with ferrous sulfate through the pre-mordanting method and the lowest color value (K/S = 1.57) with copper sulfate. It is concluded that dyed lyocell fabrics have good light fastness (4-5 grade) with mordant ferrous sulfate whereas copper sulfate mordant shows fair light fastness (3-4 grade) results. It is also observed that the results through the pre-mordanting method are of better quality. It is because the samples show no change in color or no color fading[24].

Table 3. different methods used for mordanting of lyocell fabrics

		., .				
Methods of mordanting	Chemicals treatment	pH	Temp. (°C)	Liquor ratio	Time (min)	Rinsed
Pre-mordanting method	Fabric samples were first mordanted with ferrous and copper sulphate and then used for dyeing	Neutral	Boiling	1:40	60	Dyed samples were rinsed with water and dried at room temperature
Simultaneous- mordanting method	The mordants and dye were added to the dyeing bath simultaneously	Neutral	Boiling	1:40	60	Dyed samples were rinsed with water and dried at room temperature
Post mordanting method	 (i) Fabric samples were first dyed with pomegranate dye (ii) After-dying process, samples were corrided out with wordpate 	Neutral	Boiling	1:40	60	Dyed samples were rinsed and dried at room temperature

Table 4. values of dyed lyocell fabrics

Mordant	Mordant Methods	K/S	L*
	Pre-mordanting	4.72	44.95
FeSO ₄	Simultaneous mordanting	1.95	78.59
	Post-mordanting	2.45	76.63
	Pre-mordanting	3.07	54.51
CuSO ₄	Simultaneous mordanting	1.57	86.55
	Post-mordanting	2.05	82.16

Dyeing lyocell by utilizing natural Gardenia on the lyocell fabric pretreated with tannic acid

Guizhen et al., manage to dye lyocell fabric with natural Gardenia as a yellow colorant by using tannic

acid as a mordant. Extraction of Gardenia dye: Dried gardenia of 25 g was soaked in 1000 ml distilled water and heated and stirred at 70 °C for 30 min. And then the extract was cooled to room temperature and filtered with a funnel and filter paper. The resulting filtrate was set at a constant volume of 800 ml and used as a dye solution. Then Pretreatment of lyocell fabric with tannic acid was carried out. Tannic acid has many hydroxyl groups, which can be easily combined with cellulose fiber through hydrogen bonds and van der Waals force. Tannic acid was used as a mordant for pre-mordanting lyocell fabric before dyeing with Gardenia dye[25]. The pre-mordanting treatment was conducted with a tannic acid solution of various concentrations at 40 °C for 180 min with a bath ratio of 1:50 in the thermostatic shaker bath. Then the lyocell fabric was removed and then washed and dried in an oven at 80 °C. The treated and untreated lyocell fabrics were dyed with Gardenia extract in the thermostatic shaker bath with a bath ratio of 1:40 for different time intervals. The dyeing temperature was varied between 60 and 90 °C. The dye bath pH was adjusted from 3.5 to 9.5 using 0.1 M HCl and 0.1 M NaOH solutions. After the dyeing was finished, the dyed fabrics were removed and washed under running water, and then dried in an oven at 80°C[25].

It was observed that the k/s value of the dyed sample with pre-mordant treatment was much higher than that of the untreated sample (k/s value 4.56), and the k/s value increased obviously with an increase in tannic acid content. By measuring the effect of pH, temperature, and dyeing time, it's concluded that the obtained optimum dyeing conditions were as follows: dye bath pH concentration of 4 g/L, dyeing temperature of 90°C, and dyeing duration of 60 min. The maximum color strength of the lyocell fabric dyed under optimal conditions reached 7.45 which indicated the optimistic dyeing processes of Gardenia dye to the lyocell fiber modified with tannic acid. The good washing and rubbing fastness as noted in Table (5) indicates that tannic acid pre-mordanting is helpful in the dyeing of natural Gardenia dye with lyocell fiber, which provides an ecological dyeing method for green fiber lyocell[25].

 Table 5. Washing and rubbing fastness of lyocell

 fabric dyed with Gardenia extract

Fabric sample	Washing fastness	Wet rub- bing fast- ness	Dry rubbing fastness
With premordanting	3–4	3–4	4
Without premordanting	2–3	2	3

Dyeing lyocell by utilizng Mexican marigold flower

Rashdi et al. managed to extract natural dye from the petals of a Mexican marigold flower and applied it to the lyocell fabric with five different mordanting agents.

Extraction of marigold flower dye: A water bath shaking machine was used for dyeing lyocell fabrics, and the Soxhlet apparatus was selected to extract the natural dye. First, petals were removed from Mexican marigold flowers. These petals were washed with water to remove dust particles and other impurities. The petals were dried in the daylight for 48 hours. After drying, the dried petals were crushed into fine particles (powder) with the help of a grinder machine. The extracted dye in powder form was used for the further process. **Scheme (3)** shows the preparation steps of the raw material.

A Soxhlet extractor was used for the filtration of the solvent and residue to achieve better and more efficient separation[26]. An organic solvent (ethanol) was also used during the extraction method. The evaluated amount of marigold powder (F) and dignified volume solvent (S) was taken in a certain F/S ratio. The raw material (ground powder of flower) was kept in the thimble of Soxhlet extractor and a condenser was filled with a high flow rate of water over it. The extraction was carried out for 6 hours continuously. The volume of the solution attained was measured. Furthermore, a rotatory evaporator was used for the evaporation of the solvent and the dye extracted was weighed[27]. Scheme (4) shows the mechanism of the Soxhlet extraction method. The dried Mexican marigold flower powder (10 g) was taken in the thimble of an apparatus. In a round bottom flask, the ethanol (240 ml) and distilled water (160 ml) with a liquor ratio of 1:40 were heated at a temperature of 95°C. The vapors were passed through the tube and elevated into the condenser. At the top, the vapors were condensed and dripped down into the thimble. The thimble was shattered by the suction effect when the condensed solvent was grasped at the top of the siphon. The extracted material winged back into the round bottom flask and started mixing with the clean solvent. The extracted dye was purified through the rotatory evaporator and the filtered dye solution was used for dyeing the lyocell fabric samples. It was observed that the tint of the dye extracted was dark orange in color[28].



(a) Petals in cut form (b) Washing of petals (c) Dried petals in sunlight (d) Powder of petals Scheme 3. Preparation steps of the raw material



Scheme 4. schematic representation of a Soxhlet extractor method.

Dveing procedure: Two different mordanting methods, that is, pre-mordanting and postmordanting, were used for dyeing the lyocell fabrics. The samples of lyocell fabrics were mordanted before and after the dyeing processes. Five different mordanting agents, (ferrous sulfate, copper sulfate, potash alum, stannous chloride, and potash dichromate, were used separately during the dyeing of lyocell fabric. Each mordant (4%) was dissolved in distilled water to make the liquor ratio of 1:40. The samples were dipped into the mordant solution and the dyeing process was operated for half an hour in a dyeing machine at 90°C. After dyeing, the dyed fabric samples were taken out and soaping was done. Dyed samples were washed several times with a cold and then with hot water to remove the unfixed surface dye. The washed samples were dried out in the hot air oven[29].

Table (6) shows the k/s values of dyed lyocell fabric samples at 90°C. It is observed that the optimum results of dyeing lyocell fabric were achieved at acetic pH 4–6 using five mordents and the color shade values of lyocell fabrics with mordanting agent ferrous sulfate was a better choice for dyeing lyocell fabric by post-mordanting method since it has good color efficiency (K/S 7.812), and colorfastness to washing (4–5), rubbing (4–5), light (4–6), and perspiration (4–5). Thus, the dye extracted from the marigold flower showed good dyeing and colorfastness properties. Thus, a dye extracted from marigold flowers may have a bright future because of its environmental safety and protection[29].

 Table 6. K/S values of dyed lyocell fabric

 Mordanting agent
 Mordanting

 K/S

Mordanting agent	mMethod	K/5
FeSO₄	Pre-mordanting	6.395
FeSO ₄	Post-mordanting	7.812
Sncl ₂	Pre-mordanting	1.057
Sncl ₂	Post-mordanting	1.314
CuSO₄	Pre-mordanting	0.846
CuSO ₄	Post-mordanting	0.301
K ₂ Cr ₂ O ₇	Pre-mordanting	4.495
K ₂ Cr ₂ O ₇	Post-mordanting	1.787
KAI(SO ₄) ₂ 12.H ₂ O	Pre-mordanting	0.261
KAI(SO ₄) ₂ 12.H ₂ O	Post-mordanting	0.247

Dyeing of lyocell/cotton blended fabrics

the dyeing behavior of lyocell and its blends, with different reactive dyes using the exhaust method, was studied. Lyocell-blended fabrics gave greater color strength and less fibrillation than 100% lyocell. In another study, color yields of lyocell, viscose, and cotton, after exhaust dyeing with reactive dyes, were examined. It was found that lyocell had a greater color yield value than cotton. When dyeing with sulfur and direct dyes, the results were the same.

Tuba et al., examined the exhaust dyeing properties of Tencel/cotton blended fabrics using different processes. In this study, scouring, with/without bleaching, followed by defibrillation (biopolishing treatment), and peroxide residue removal along with dyeing, using different enzymes such as Cellusoft Combi and Terminox Ultra 10L (Novozymes), in rope form was carried out with knitted fabrics containing cotton and Tencel in different ratios (100% combed cotton, 25% cotton/75% Tencel-brand lyocell fiber, 50% cotton /50% Tencel, 75% cotton/25% Tencel and 100% Tencel yarns were used)[30].

Before dyeing, Scouring processes were carried out for 45 min at 90°C with 2 g/L sodium carbonate and 1 g/L wetting agent using a liquor ratio (LR) of 20:1. The test fabrics were treated with hydrogen peroxide for bleaching. The LR for bleaching was 15:1 with H₂O₂, NaOH (1 mL/L), and stabilizer (2 mL/L). Bleaching was applied to all blended fabrics except 100% Tencel because the cotton fibers contained more impurities than Tencel fibers[30].

In Process 1 (P1), fabrics containing 100%, 75%, 50%, and 25% Tencel were fibrillated by scouring, then enzymatically de-fibrillated and then dyed at 2% owf dye concentration, all in the same bath. In Process 2 (P2), after scouring, peroxide bleaching was performed, followed by enzymatic defibrillation, bleach residue removal, and dyeing (2% owf), all in the same bath. Process 3 (P3), unlike P2, was used to increase the efficiency of peroxide residue removal by catalase enzyme addition during defibrillation, followed by bleach cleanup and bath dyeing[30].

Then Dyeing Lyocell and cotton blended fabric samples with reactive dyes (Remazol Navy Blue dye) using an LR of 20:1.

Process 2 is the conventional dyeing process. After dyeing, the fabric samples were thoroughly rinsed with cold and warm water, then washed for 30 min at 95 °C at an LR of 10:1. The fabric samples were then rinsed with warm and cold water again[30].

It was observed that the Tencel ratio in the mixture increased, the K/S value increased, and this increase was most prominent using the P1 method. P1 could be used instead of P2 with the fabrics having Tencel content of 75% and greater. For fabrics having 50% or greater cotton content, P3, a more economical and ecological process, can be used as an alternative to P2. Bleaching was necessary to

obtain higher K/S values in fabrics containing 50% to 75% cotton.

These cases showed that the P3 process could be used instead of P2 for fabrics having 50% or greater cotton content. Moreover, after peroxide bleaching, it was also observed that dyeing could be continued by the addition of catalase enzyme in the same dye bath, thereby removing a rinse step from the process. The need for peroxide bleaching increased as the cotton ratio in the blended fabrics increased. Although this bleaching was not required for the fabric containing 75% Tencel, fabrics containing 50% to 75% cotton needed this treatment, and better color depth resulted in these fabrics[30].

the treated blended fabrics showed overall color differences from 3 to 5 washing fastness results. Moreover, no significant effect of the fiber ratios on wash fastness values was observed.

When the crock fastness values of the processes were evaluated, there were no differences in the dry crock fastness values, whereas the wet crock fastness values for P1 were better than those for P2 and P3. In summary, the P3 process could be used as an alternative to the conventional P2 process in fabrics with 25% to 50% Tencel content in blends with cotton, and fabrics having 75% or greater Tencel content in blends with cotton could be treated with P1 as an economic and environmentally-friendly alternative to the conventional P2 process[30].

Another study done by Aravin et al., to dye Lyocell/cotton blended fabrics (40:60) with salt-free dyeing by using Polyvinylamine Chloride (PVAmHCl) as a pretreatment agent in improving its dyeability with reactive dyes and in achieving evenness of dye uptake[31].

PVAmHCl has been used as a physical modifying agent. Due to its wide range of properties, PVAmHCl has found use in catalysis, liquid chromatography, treatment of wastewater, recovery of oil, and in polymeric dyes. It has been used in the modification of cotton for salt-free dyeing. PVAmHCl arises from the presence of a large number of cationic sites (NH+3Cl-). Nucleophilic sites involving primary amino groups within the PVAmHCl molecule are of particular value for achieving salt-free dyeing of cotton with reactive dyes. As the pH increases, the proportion of NH+3Cl groups in the molecule decreases and that of the NH2 groups increases[31].

Preparation of Fabric: The fabric sample was desized using the acid desizing method. The fabric was scoured by the alkali method using a standard procedure. Then, it was subjected to a bleaching process using hydrogen peroxide as the bleaching agent. Then The padding method was used for the pretreatment of cotton with PVAmHCl (2.5-20 gpl). The pH (7-7.5) of the pretreatment solution was maintained by the buffer potassium dihydrogen phosphate and sodium hydroxide. Padding was carried out using two dips (4 min each) and two nips.

Fabric samples were predried at room temperature and then baked at 102°C for 12 min in a rapid baker. Padding was done at different concentrations of PVAmHCl. Then Exhaust dyeing was carried out at a liquor ratio of 1:30 and pH (10-11)[31]. Dyeing of the fabric pretreated with different concentrations of PVAmHCl was carried out at 80°C for 60 min. Fixation was conducted for 20 min using Na2CO3. The Dye and chemicals are given in **Table (7)**

SI No.	Dye and Chemicals	Functions		
1	CI RX Red 120A (Generic name) Reactive Red HE-3B (Commercial name)	Dyeing		
2	Polyvinylamine Chloride (PVAmHCI)	Pretreatment		
3	Potassium dihydrogen Phosphate (KH ₂ PO ₄)	To maintain pH		
4	Sodium Carbonate (Na ₂ CO ₃)	Fixing agent		
5	Sodium Hydroxide (NaOH)	Swelling agent		
6	Sodium Chloride (NaCl) Caustic Lye	Exhaustion agent		

Table 7. Functions of Dye and chemicals used.

It was observed that a higher K/S value was found with 10% polyvinyl amine chloride. As the concentration of polyvinyl amine chloride increased above 10%, the K/S value decreased[31]. The K/S value of the untreated sample was comparatively lower than the K/S value of samples treated with 5-10% polyvinyl amine chloride. The K/S value was found to be less than that of the conventional method when 2.5% polyvinyl amine chloride was used. The observations indicate that the pretreatment of Lyocell/cotton fabric increases dye uptake. This decrease in K/S value may be for the following reasons. When an excess of polyvinyl amine chloride is padded on the fabric, the bonding between the fiber and some cationic polymers becomes weak, and repulsion forces also exist within the cationic polyvinyl amine chloride. This would lead to the presence of unbound polymer in the dye bath, thereby hindering the absorption of dye and possibly causing it to flocculate. It can be seen from Table (8) that dye reactivity on pretreated fabric was greater due to the presence of primary amino groups provided by the Polyvinylamine Chloride. This confirms the effectiveness of pretreatment in enabling the fabric to be dyed without salt[31].

The wash fastness was excellent for all samples the salt-free dyeing, confirming the from effectiveness of dye fixation due to pretreatment with PVAmHCl. Rubbing fastness was also observed to be good when compared with that obtained by conventional dyeing. The tensile strength of conventionally dyed fabric and the pretreated samples were found to be almost the same. There was an increase in the crease recovery angle of the fabric because There might be cross-linking of PVAmHCl between the cellulosic molecules. These cross-links hinder the molecular and fibrillar slippage and stabilize the structure, thereby increasing the crease recovery angle. The increase in flexural

57

rigidity shows that the fabric became slightly stiffer as a result of the treatment of the fabric with PVAmHCL.

Table 8	. K/S	value o	of control	and	treated	samples.
---------	-------	---------	------------	-----	---------	----------

PVAmHCI concentration (g/l)	K/S Value
2.5	12.3
5.0	15.1
10.0	18.8
15.0	16.1
20.0	12.5
Conventional	13.7
Untreated & no salt	8.3

By using this pretreatment method, the following advantages were observed: elimination of salt as an electrolyte, maximum fixation of dye, minimum hydrolysis of dye, low volume of water requirement during the wash-off process, significant savings in process costs, and environmentally friendly[31].

Dyeing of lyocell/silk/polyester blended fabrics

H D Joshi et al. decided to dye lyocell, silk, and polyester by using the two-bath dyeing method. They have been dyed with bifunctional reactive dyes, acid dyes, and dispersed dyes in bulk.

Pretreatment of Lyocell/silk union fabrics was given a degumming treatment with 5 g/l Marseilles soap, 0.375 g / 1 sodium carbonate, and 0.2 g / 1 sodium hydroxide using a liquor ratio of 20:1 on a winch machine at 80 °C for 6 h. This was followed by rinsing treatment with plain water (40 °C). Then scouring and bleaching were carried out and the blended fabrics were treated with 5 g / l hydrogen peroxide (50%), 5 g/l sodium carbonate, and 1 g/l nonionic detergent (at alkaline pH) with a liquor to goods ratio of 20:1 at 80 °C for 1 h on a jigger machine. The scoured fabrics were washed with a solution of 1 g/l acetic acid to neutral pH. After that enzyme treatment of all the blended fabrics was carried out using 1 g / l (wetting agent/ detergent), 0.63 g/l (cellulase enzyme), and 1 g/l acetic acid using a liquor ratio of 20:1, pH 5.5 at 55 °C for 45 min. The temperature was then raised to 80 °C to deactivate the enzyme[32].

Dyeing procedure: The lyocell union fabrics were dyed with reactive dyes, acid dyes, a direct dye, and a dispersed dye to give solid shades as well as two-color effects. The silk portion of the union fabrics was dyed with acid dyes. Dyeing was carried out in open-width form on a jigger dyeing machine at a liquor-to-goods ratio of 20:1 according to the process given in the **Schemes (5,6,7,8)**. After dyeing, the fabrics were rinsed thoroughly with water and 1 g/1 nonionic detergent at 50°C, followed by a cold-water wash. The polyester portion was dyed first using disperse dye in a jet dyeing machine at 120 °C for 30 min[32].



Scheme 5. Dyeing profile for dyeing silk portion of lyocell/silk blend with acid dyes



Scheme 6. Dyeing profile for dyeing lyocell with reactive dyes (Coracion bifunctional and Corrective MCT)



Scheme 7. Dyeing profile for dyeing lyocell with direct dyes



Scheme 8. Dyeing profile for dyeing lyocell with reactive dyes (Corafix and Levafix bifuntional)

Resin finishing of all the dyed fabrics was carried out to impart a smooth, lustrous handle to the fabrics. The resin finishing recipe employed was: (low formaldehyde cross-linking agent; Clariant) 50 g/1, polyethylene emulsion 40 g/1, reactive softener 10 g /1, magnesium chloride 7.5 g/1, Polysol (polyvinyl acetate 48% for imparting a stiffening effect) 10 g/1. Finishing was carried out at 70% padding mangle expression at 170 °C at a fabric speed of 35 m/min. A resin finishing treatment is necessary for the processing of lyocell to minimize post-laundering fibrillation[33].

Results of this study show that exhaustion and fixation of reactive dyes take place best under neutral conditions. Under alkaline conditions, the reactive dye uptake on silk is low as compared with the neutral medium. However, in the absence of an electrolyte, the uptake of reactive dyes on silk decreases considerably. Using a low electrolyte concentration in subsequent reactive dyeing can reduce cross-staining on silk. In the case of cellulosic fibers, dyeing with reactive dyes is carried out in two stages: in the primary exhaustion stage, the reactive dye is taken up as a direct dye, while, in the secondary exhaustion stage, fixation takes place in the presence of an alkali. In the case of silk, the fixation of the reactive dye takes place in the neutral medium. For lyocell/silk fabrics, although the silk was dyed with acid dyes so that the dyeing sites were occupied, there was probably some amount of acid dye desorption, and those dyeing sites were occupied by the reactive dyes, which gives rise to the staining effect. Although maximum exhaustion and fixation with reactive dyes on silk take place under neutral conditions, the presence of electrolytes in alkaline conditions does result in dye fixation[32].

Lyocell has been dyed in lighter hues (yellows), while silk has been dyed in darker hues (reds, blues). It's observed that there is no cross-staining. the results of blended fabrics of lyocell with silk and polyester give bright shades with reasonably good fastness properties and cross-dyeing and the solid shades give good color[34].

Dyeing of lyocell/cotton/polyamide blended fabrics

Shan et al., managed to dye cotton/polyamide/ lyocell fabrics with short wet-steaming low-carbon cleaner pad dyeing technology with reactive dyes and compared it with traditional pad dyeing.

Traditional pad dyeing of cotton/polyamide/lyocell fabrics

Cotton/polyamide/lyocell fabrics are usually dyed by the two-phase technology during continuous pad dyeing with reactive dyes. **Scheme (9)** showed the dyeing process. In the first step, the reactive dye solution (adjust pH to 4.5 by $C_2H_4O_2$) was poured into the pad dyeing machine. The fabric sample was passed through the rolling mill, with two-dip two nips to ensure that the dye solution on the fabric was about 70 %, and then put into the drying machine for drying (100°C) for 1 min. The second step was to steam the sample with saturated steam for 3 min for fixing the reactive dye. The third step, putting the sample into the dryer to dry (at 100°C) for 1 min. In the fourth step, the acidic solution was changed into an alkaline solution (Na₂CO₃ of 30 g/L, Na₂SO4 of 200 g/L) and step two was repeated. The fourth step was to wash the stained fabric samples. The fabric sample was washed with cold water and then washed the dyed fabric with hot water (60-70°C) for 1 min and 2 g/L soap solution (bath ratio of 1:50, 100°C) for 5 mins. Finally, the floating color was removed with hot water (60-70°C) for 1 min and cold water, and the fabric was dried at 100°C for 2 min[35].

Short wet-steaming low-carbon cleaner pad dyeing

The fabric sample was dipped in the dye solution (adjust the pH to acid citric acid) and nipped the fabric off the roller of a rolling mill, then the above steps were repeated to make sure the quality of the dye solution from the fabric was 90 % of the weight of the initial fabric. Then the fabric was directly steamed (at 100–102°C) for 2 mins with a steamer at the saturated vapor pressure so that the polyamide fiber was dyed. Then the alkaline solution (Na₂CO₃ of 10 g/L, NaOH of 10 g/L, and Na₂SO₄ of 200 g/L, respectively) was poured into the clean rolling mill[35].

The fabric passed through the rolling mill twice and was steamed at the saturated vapor pressure $(100-102^{\circ}C)$ for 2 mins, to realize the dyeing of cotton and lyocell fibers. After dyeing, the dyed fabric sample was washed with cold water and then washed with hot water (60-70°C) for 1 min and 2 g/L soap solution (bath ratio of 1:50, 100°C) for 5 mins to dismiss the excess dye from the fabric. Finally, the fabric was dried with a drying machine $(100^{\circ}C)$ for 2 mins after washing with hot water (60-70°C) for 1 min and cold water. **Scheme (10)** showed the dyeing process.



Scheme 9. Schematic diagram of the traditional two-phase pad dyeing process



Scheme 10. Schematic diagram of short wet-steaming low-carbon cleaner pad dyeing process.

In this study, reactive dye with three shades (red, yellow, blue) was used to test the dyeability of cotton/lyocell and polyamide. The pH value of the initial dye solution was 3 in the acid condition and 9 in the alkaline condition. Reactive red 195 dye was selected for this experiment as reactive dyes had preferable water solubility, diffusivity, and acid resistance to reduce the interaction due to diverse technological conditions on the dyeing of different fibers, to obtain better union dyeing properties for cotton/polyamide/lyocell fabric[35].

The process of steaming the fabric allowed the dye molecules to enter the fiber, and then the fabric was put into the dryer for drying. Therefore, the fabric could absorb more alkaline agents when it was dipped and rolled with the alkaline fixing solution.

Reactive red 195 solutions with different concentrations were prepared. The pH value of the initial dye solution was adjusted to 3.7 with citric acid. Then the dyed fabrics were conducted with two kinds of alkaline fixing solution respectively and the steaming time of both acid and alkali was 2 mins.

Mechanism of dyeing cotton/lyocell/polyamide fabric

Cotton's chemical structure was similar to that of lyocell. Cotton and lyocell fibers swelled fast when the dye solution met them due to their hydrophilicity, which widened the pores and filled the dye molecules into the fiber[36]. Some dye molecules were adsorbed on the surface of cotton and lyocell fibers via the van der Waals force and hydrogen bonding, whereas fractional dye molecules were distributed on the surface of polyamide fiber via the Coulomb force, van der Waals force, and hydrogen bonding[37]. Despite the electrostatic affinity between polyamide and dyes, certain dyes migrated with water from polyamide to more hydrophilic cotton and lyocell. Due to the formation of hydrogen bonds between amido groups in polyamide molecular chains, the aggregation structure of polyamide fiber macromolecules exhibited a certain degree of crystallinity and direction. The strong intermolecular chain force at the crystallization zone restricted the spatial movement of molecules.

As shown in scheme (11), During steaming, the molecular chain movement accelerated and the intermolecular connection force broke down in the polyamide fiber's amorphous region. The dye molecules entered the polyamide fiber from its surface, and the amido gen group changed into an amido gen cation through an acid. The amido gen cation then joined the dye anion through an ionic connection under the influence of the Coulomb force. As the molecular chain movement intensified during steaming, the monochlorotriazine active group on the dye molecule underwent a nucleophilic substitution reaction with amidogen, and the intermolecular connection force was dismantled in the amorphous area of polyamide fiber.



Scheme 11. The schematic diagram of dye fixing on cotton/lyocell fiber and polyamide fiber

As shown in scheme (12), The alkali and waterabsorbing fabrics were steamed at saturated vapor pressure, allowing the alkali to enter the amorphous area of cotton and lyocell fibers with water. The dye molecule of reactive red 195 has a high relative molecular weight and a strong van der Waals interaction with the fiber. During alkali steaming, the vinyl sulfone sulfate group on dye molecules was converted into a stable vinyl sulfone group. To achieve dyeing of the dye on the three fibers[38], part of it interacted with the main hydroxyl group on the fiber molecule of cotton and lyocell, while part of it reacted with the amido gen group on the polyamide fiber. Because of the higher reactivity of the vinyl group sulfone sulfate compared to the monochlorotriazine group, the unreacted or reacted amidogen group on polyamide interacted with the vinyl sulfone group on the dye molecule to create a covalent link, resulting in a darker polyamide fiber[39].



Scheme 12. dye fixing reaction diagram of fibers with monochlorotriazine group and vinyl sulfone sulfate group

It was observed that the K/S value (6.1) of cotton/lyocell yarns increased with the rise of alkali concentration in the fixing solution, but polyamide was hardly affected. The high alkalinity of the fixing solution accelerated the formation rate of cellulose anion and expedited the rate of bonding reaction. This promoted the complete fixation reaction between cotton/lyocell fibers and dye molecules that entered the fiber. However, polyamides mainly existed in the form of anions, and the dye bound with van der Waals force might be desorption by a

hydrogen bond. The optimum condition of dyeing when the pH value was 3–4, the steaming time of both acid and alkali was 4 mins, and both NaoH and Na₂CO₃ were 10 g/L in the alkaline fixing solution containing 200 g/L Na2SO4 by wet-steaming pad dyeing technology[35].

This study exploited a new short wet-steaming cleaner pad dyeing technology, which not only improved the dye utilization and color yield of cotton/polyamide/lyocell fabric but also enhanced the union dyeing property of cotton/lyocell blended yarns and polyamide yarns. Moreover, the processing time, power, heat consumption, and carbon dioxide emission were reduced, so it promoted the lowcarbon development of dyeing for industrial crops and products in the printing and dyeing industry. Compared with other types of reactive dyes, bifunctional reactive dyes with monochlorotriazine and vinyl sulfone sulfate groups (M-type) emerged as the best dyeing effect. Therefore, it was better to employ bifunctional reactive dyes more in production practice, which brought the cotton/polyamide/lyocell fabric with higher dyeing depth and decreased the consumption of dyes and energy sources, and achieved sustainable chemical dyeing of cotton/ polyamide/ lyocell fabric[35].

Conclusion

Conclusively, lyocell fabrics play a significant role in sustainable dyeing. The impact of various dyes on lyocell fabric and its blended fabrics lead to enhancing the dyeability and fastness properties. All these studies indicate that the most effective dye is the vat dye which obtained the highest k/s value of 25.89. The obtained outcomes have significant practical ramifications for the implementation of dyeing by using short wet-steaming cleaner pad dyeing technology which promoted the low-carbon development for the dyeing industry.

Conflicts of interest

There are no conflicts to declare

Funding sources

There is no fund to declare

Acknowledgments

The authors are gratefully-grateful to acknowledge the Faculty of Applied Arts, Benha University. Furthermore, the authors are gratefully grateful to acknowledge the Central Labs Services (CLS) and Centre of Excellence for Innovative Textiles Technology (CEITT) in Textile Research and Technology Institute (TRTI), National Research Centre (NRC) for the facilities provided.

References

- ŞArdaĞ, S., "Investigation of Mechanical Properties of Fabrics Woven with Tencel/Cotton Blend Yarns". *Textile and Apparel*, **29**(2) 162-170 (2019)
- Bilir, T.B. and Şardağ, S., "The Investigation of Performance Properties of Tencel Cotton Blended Yarn". Uludağ University Journal of The Faculty of Engineering, 1(22) 13-28 (2017)
- Jeyaraj, J.M., Arumugam, M. and Kulandaiappan, V., "A Study on the Functional Properties of Silk and Polyester/Lyocell Mixed Fabric". *Matéria (Rio de Janeiro)*, 20 924-935 (2015)
- Colom, X. and Carrillo, F., "Crystallinity Changes in Lyocell and Viscose-Type Fibers by Caustic Treatment". *European Polymer Journal*, 38(11) 2225-2230 (2002)
- Široký, J., Blackburn, R.S., Bechtold, T., Taylor, J. and White, P., "Attenuated Total Reflectance Fourier-Transform Infrared Spectroscopy Analysis of Crystallinity Changes in Lyocell Following Continuous Treatment with Sodium Hydroxide". *Cellulose*, **17** 103-115 (2010)
- Owen, G., "Innovation in the Man-Made Fibers Industry: Corporate Strategy and National Institutions. Chapter 9 Innovation in Fibers: The Carbon Fiber and Tencel Stories. Prepared for Seminar at Spru", (2012)
- Badr, A.A., El-Nahrawy, A., Hassanin, A. and Morsy, M.S. "Comfort and Protection Properties of Tencel/Cotton Blends". New Orleans
- Borbély, É., "Lyocell, the New Generation of Regenerated Cellulose". Acta Polytechnica Hungarica, 5(3) 11-18 (2008)
- 9. Hohbery, T. and Thumm, S., "Melliand Textilber". *Engl. Edn*, 6 E124 (1998)
- 10. Hussain, T., "Developments in the Processing of Lyocell Fabrics". *Department of Textile Chemistry, National Textile University, Pakistan,*
- Adnan, M. and Moses, J., "A Study on the Thermophysiological and Tactile Comfort Properties of Silk/Lyocell Blended Fabrics". *Matéria (Rio de Janeiro)*, 25(2020)
- Farrington, D.W., Lunt, J., Davies, S. and Blackburn, R.S., "Poly (Lactic Acid) Fibers". *Biodegradable and* sustainable fibers, 6 191-218 (2005)
- Taylor, J., "Controlling Fibrillation-Experiences of the Dyeing and Finishing of Lyocell Fibers". *Coloration Technology*, 131(6) 424-433 (2015)
- Khan, M.E., "Comparative Study on Dyeing of Cotton, Modal and Lyocell Fabrics Using Bifunctional and Polyfunctional Reactive Dyes". *Journal of Textile Science and Technology*, 6(1) 40-48 (2019)
- Larik, S.A., Khatri, A., Ali, S. and Kim, S.H., "Batchwise Dyeing of Bamboo Cellulose Fabric with Reactive Dye Using Ultrasonic Energy". *Ultrasonics* sonochemistry, 24 178-183 (2015)

- Udrescu, C., Ferrero, F. and Periolatto, M., "Ultrasound-Assisted Dyeing of Cellulose Acetate". Ultrasonics sonochemistry, 21(4) 1477-1481 (2014)
- Babar, A.A., Peerzada, M.H., Naveed, T. and Dayo, A.Q., "Exhaust Reactive Dyeing of Lyocell Fabric with Ultrasonic Energy". *Ultrasonics Sonochemistry*, 58 104611 (2019)
- Khatri, M., Ahmed, F., Jatoi, A.W., Mahar, R.B., Khatri, Z. and Kim, I.S., "Ultrasonic Dyeing of Cellulose Nanofibers". *Ultrasonics Sonochemistry*, 31 350-354 (2016)
- Robinson, T., Chandran, B. and Nigam, P., "Removal of Dyes from a Synthetic Textile Dye Effluent by Biosorption on Apple Pomace and Wheat Straw". *Water research*, 36(11) 2824-2830 (2002)
- Hakeim, O.A., Nassar, S.H., Raghab, A.A. and Abdou, L.A.W., "An Approach to the Impact of Nanoscale Vat Coloration of Cotton on Reducing Agent Account". *Carbohydrate polymers*, **92**(2) 1677-1684 (2013)
- Memon, R.H., Syed, U., Harijan, K., Mahar, F.K., Babar, A.A., Hussain, S., Hussain, B. and Khatri, Z., "Vat Dyeing of Lyocell Fabric with High Temperature Dyeing Technique". *The Journal of The Textile Institute*, **113**(11) 2335-2341 (2022)
- Gupta, V.K., "Application of Low-Cost Adsorbents for Dye Removal–a Review". Journal of environmental management, 90(8) 2313-2342 (2009)
- Kechi, A., Chavan, R.B. and Moeckel, R., "Dye Yield, Color Strength and Dyeing Properties of Natural Dyes Extracted from Ethiopian Dye Plants". *Textiles and Light Industrial Science and Technology*, 2(3) 137-145 (2013)
- Rehman, F., Naveed, T., Nadeem, M. and Wei, W., "Study on Tencel Fabric Dyeing with Pomegranate Peel Natural Dyes". *Asian Journal of Chemistry*, 29(10) 2279-2284 (2017)
- Ke, G., Mulla, M.S., Peng, F. and Chen, S., "Dyeing Properties of Natural Gardenia on the Lyocell Fabric Pretreated with Tannic Acid". *Cellulose*, **30**(1) 611-624 (2023)
- Bhande, R. and Giri, P., "Extraction of Garcinia Indica Oil from Kokum Seed". *International Journal* of Engineering Technology, Management and Applied Sciences, 5(6) 723-727 (2017)
- Toussirot, M., Nowik, W., Hnawia, E., Lebouvier, N., Hay, A.E., De La Sayette, A., Dijoux-Franca, M.G., Cardon, D. and Nour, M., "Dyeing Properties, Coloring Compounds and Antioxidant Activity of Hubera Nitidissima (Dunal) Chaowasku (Annonaceae)". *Dyes and Pigments*, **102** 278-284 (2014)

- Leon, K., Mery, D., Pedreschi, F. and Leon, J., "Color Measurement in L* a* B* Units from Rgb Digital Images". *Food research international*, **39**(10) 1084-1091 (2006)
- 29. Rashdi, S.Y., Naveed, T., Sanbhal, N., Almani, S., Lin, P. and Wei, W., "Lyocell Fabric Dyed with Natural Dye Extracted from Marigold Flower Using Metallic Salts". *Autex Research Journal*, **20**(3) 352-358 (2020)
- Toprak, T., Şardağ, S. and Anis, P., "A New Environmentally-Friendly Method of Dyeing Lyocell/Cotton Blended Fabrics". *AATCC Journal of Research*, 5(5) 23-30 (2018)
- Periyasamy, A.P., Dhurai, B. and Thangamani, K., "Salt-Free Dyeing-a New Method of Dyeing on Lyocell/Cotton Blended Fabrics with Reactive Dyes". *Autex Res J*, 11(1) 14-17 (2011)
- Joshi, H.D., Joshi, D.H. and Patel, M.G., "Dyeing and Finishing of Lyocell Union Fabrics: An Industrial Study". *Coloration Technology*, **126**(4) 194-200 (2010)
- Schulz, F. and Ottenbach, A., "Melliand Textilber". Engl. Edn, 10 155 (1997)
- Bates, I., Ibbett, R., Reisel, R. and Renfrew, A.H.M., "Protection of Lyocell Fibers against Fibrillation; Influence of Dyeing with Bis-Monochloro-S-Triazinyl Reactive Dyes". *Coloration Technology*, 124(4) 254-258 (2008)

- Zhang, S., Ren, Y., Zhao, H., Han, L., Yang, G., Liu, Y. and Fang, K., "Short Wet-Steaming Low-Carbon Cleaner Pad Dyeing of Cotton/Polyamide/Lyocell Fabric with Reactive Dyes". *Industrial Crops and Products*, **197** 116556 (2023)
- Mu, B., Liu, L., Li, W. and Yang, Y., "High Sorption of Reactive Dyes onto Cotton Controlled by Chemical Potential Gradient for Reduction of Dyeing Effluents". *Journal of environmental management*, 239 271-278 (2019)
- Shirvandi, Z., Atashkar, B., Zolfigol, M.A. and Rostami, A., "Transition-Metal-Catalyzed One-Pot Selenylation of Electrophilic Arylating Agents Using Triphenyltin Chloride/Se as a Phenylselenating Agent". Organic & Biomolecular Chemistry, 20(22) 4625-4634 (2022)
- Funar-Timofei, S., Fabian, W.M.F., Kurunczi, L., Goodarzi, M., Ali, S.T. and Vander Heyden, Y., "Modelling Heterocyclic Azo Dye Affinities for Cellulose Fibers by Computational Approaches". *Dyes and Pigments*, 94(2) 278-289 (2012)
- Wang, M., Yi, N., Fang, K., Zhao, Z., Xie, R. and Chen, W., "Deep Colorful Antibacterial Wool Fabrics by High-Efficiency Pad Dyeing with Insoluble Curcumin". *Chemical Engineering Journal*, 452 139121 (2023)

نظرة عامة على عملية صباغة نسيج الليوسيل وخلطاته

هبه غزال¹، سهيلة شاكر¹، نادين خالد¹، أحمد جمعه حسبو²*

¹ جامعة بنها ـ كلية الفنون التطبيقية ـ قسم طباعة المنسوجات والصباغة والتجهيز ـ بنها ـ مصر

² المركز القومي للبحوث (60014618 ID Scopus) ، معهد بحوث وتكنولوجيا النسيج ، قسم التحضيرات والتجهيزات للألياف السليلوزية -الجيزة - مصر

* aga.hassabo@hotmail.com البريد الإلكتروني aga.hassabo

الملخص

تهدف هذه الدراسة إلى التحقيق في نسيج الليوسيل الذي أصبح شائعا جدا في الأونة الأخيرة بين الباحثين نظرا لقوته العالية ، ومورفولوجيته البلورية الصلبة ، وكونه غير سام تماما ، وقابل للتحلل الحيوي تم تصنيعه بواسطة عملية صديقة للبيئة غير ملوثة ، وقربه من القطن. تظهر هذه الدراسة تأثير كل من الأصباغ الاصطناعية والأصباغ الطبيعية على نسيج الليوسيل. تشمل الأصباغ الاصطناعية الأصباغ التفاعلية وأصباغ ضريبة القيمة المضافة ، بينما تشمل الأصباغ الطبيعية أصباغ قشر الرمان وزهور القطيفة المكسيكية وأصباغ الغردينيا. أيضا صباغة الليوسيل مع القوسيل مع المخلوطة مثل القطن والحرير والبولي أميد.

الكلمات الدالة: ألياف الليوسيل ، قابلة للتحلل ، قشر الرمان ، زهرة القطيفة المكسيكية ، أصباغ الغردينيا ، الأقمشة المخلوطة.