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Environmentally Improved Reactive Dyeing of Nylon 6 Electrospun Nanofibres Using Ultrasonic Energy

Awais Khatri*, Shamshad Ali, Alishba Javeed, Farooq Ahmed

Colour Research Lab, Department of Textile Engineering, Mehran University of Engineering and Technology, Jamshoro 76060, Pakistan.

*Corresponding author Email: awais.khatri@faculty.muet.edu.pk

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Abstract

Sustainable dyeing practices are essential for reducing the environmental footprint of the industry. This study investigates the potential of ultrasonic-assisted dyeing as an eco-friendly alternative to conventional exhaust dyeing of Nylon 6 electrospun polymeric nanofibres with reactive dyes. The findings reveal that ultrasonic dyeing not only enhances the colour yield (K/S of 1.72) as compared to the conventional method (K/S of 1.42) but also provides environmental benefits. The method resulted in a 28% reduction in the total dissolved solids (TDS) contents of dyeing effluent, 25% saving in the dye consumption, substantial reduction in the thermal energy (1200 kcal), reduction in the process time (10 min) and slightly better colourfastness performance. Moreover, ATR-FTIR, light transmission and SEM analyses were used during the research. These improvements offer both enhanced performance and ecological benefits. The findings are relevant for high-performance textile applications particularly functional apparels and smart textiles, where colour vibrancy, material integrity and environmental sustainability are crucial.

Keywords: Ultrasonic energy, Exhaust dyeing, Nylon 6, Electrospun nanofibres, Reactive dyes, Ecology

Introduction

The electrospun nanofibres have been used for a variety of applications, including high performance composites, sensing devices, effluent filtration, protective textiles, energy generation, and enzyme immobilization, due to the flexibility of their properties [1-4]. Additionally, these nanofibres have shown a great potential for usage in functional and fashionable garments due to their higher porosity and better breathability than conventional fibres [5]. Consequently, electrospun nanofibrous mats have successfully made their way to large scale manufacturing for being used in clothing and have shown significant durability when put through laundering [6]. Since aesthetic property is one of the fundamental needs for clothing, the success of employing electrospun nanofibrous mats for apparels has drawn the attention of scientists and experts to the coloration of electrospun nanofibrous mats. Consequently, the dyeability of electrospun nanofibrous mat has been effectively investigated [7].

One of the newest technologies utilized to increase the effectiveness of aqueous-based processes and increase their sustainability by using less water, lower energy consumption and reduced chemicals

load is ultrasonic (US) energy [8-10]. Mass transfer improvement, which would otherwise be achieved by using heavy concentrations of chemicals, higher temperatures and elongated process times, is the main advantage of employing US energy in textile dyeing [11]. The environmental benefit of US dyeing lies in thermal energy consumption, lowering dye and chemical requirement and effluent pollution load reduction, making it a more sustainable alternative to the conventional (CN) dyeing technologies. Moreover, the colour yield (*K/S* value) for dyed electrospun nanofibrous mats and quicker coloration have been reported with US aided dyeing methods [12].

US dyeing has gained increasing attention as a sustainable alternative method of textile coloration in the recent couple of decades. Several studies have explored its application on various textile materials including nanofibers to enhance dye uptake and process efficiency. In a prior study by Ali *et al.* [13], disperse dyes were dyed onto polyurethane nanofibres using US energy. The study's findings revealed notable increases in colour yield as well as substantial cost savings through reduction in the utilization of thermal energy, dyeing time, and dye quantity. Similarly, Jatoi *et al.* [14] reported US dyeing of nylon with disperse dyes to improve the colour yield and the effluent pollution load. However, these studies primarily focused on dyeing synthetic fibres with disperse dyes, leaving a research gap in using reactive dyes for US dyeing of nanofibers.

This study extends the scope of US dyeing to dye Electrospun Nylon 6 nanofibres with reactive dyes. The reactive dyes have gained popularity in the cellulose fibre dyeing industry due to their vivid and solid hues [15]. Wide selection of low-cost dazzling colours with great washing fastness are available. Reactive dyes' hydroxyl groups establish covalent bonds with fibre at the different active sites of cotton and wool [16-17]. Like wool protein polymer, the six terminal amino end groups in nylon make it easier to dye using anionic reactive dyes. The nucleophilic amino end groups of nylon 6 form covalent bonds with the fibre-reactive electrophilic groups of reactive dyes, particularly chlorotriazines and vinylsulphones based dye structures, during the dyeing process [18-19].

The environmental benefits of colouring nylon 6 electrospun nanofibrous mats with reactive dyes with US assistance are also discussed as reducing thermal energy, amount of dye and effluent pollution. Batchwise reactive dyeing was used to colour nylon 6 electrospun nanofibrous mats utilizing heat energy (CN dyeing method) and US energy (US dyeing method). For the US dyeing method, the potential for thermal energy, processing time, and dye usage savings was examined. Additionally, dye effluents were examined for TDS levels to investigate the ecological advantages of the US assisted dyeing over the conventional method. The dyeing optimizations were carried out against the final colour yield. The dyed samples were also characterized for colourfastness, and morphological and chemical structures.

Materials and Methods

Materials

The Sigma Aldrich Nylon 6 polymer (pellets, 3 mm) was purchased and utilized exactly as it was delivered. Formic acid solvents were obtained from Daejung Chemicals, South Korea. The Drimaren Navy CL-R, a CI Reactive Black 5 (*bis*-sulphatoethylsulphone) dye and one of the most widely used reactive dye for navy and black shades, was used in this research. The dye and the Hostapal CV (non-ionic

detergent) were obtained from Archroma Pakistan Ltd. Both sodium hydroxide and acetic acid were of analytical grade. Deionized water was used for all the dyeings.

Dyeing Apparatus

On a ceramic hot plate, nylon 6 was dyed by conventional exhaust dyeing (Cimarec, Thermo Scientific). The dyebath was prepared in a 10 mL glass tube that was dipped into a 250 mL glass beaker of water to employ the CN dyeing. To ensure a clean dyeing process, this setup was covered with aluminium foil. The dye pots were manually shaken numerous times at regular intervals throughout the dyeing process to achieve the appropriate agitation during the dyeing process. Same procedure was adopted for the US dyeing method; however, the dyeing was carried out on an US bath (Elmasonic S 30/H, Germany). The US frequency was set to 37 kHz and the power input was set to 300 W.

Methods

Preparation of Nylon 6 Electrospun Nanofibrous Mats

For making nylon 6 electrospun nanofibrous mats, a Linary Electrospinning machine (Italy) was used. A polymer solution of 20% (w/w) nylon 6 polymer was created by dissolving a known amount of nylon 6 pellets in formic acid that was 100% concentrated. For completely dissolving the nylon 6 pellets, the nylon 6 polymer solution was mixed at room temperature for 12 h using a magnetic stirrer spinning at around 500 rpm. This produced a clear, uniform solution that was suitable for the electrospinning process.

For electrospinning [20], a capillary tip with a 0.6 mm inner diameter was attached to a 5 mL plastic syringe, which was used to deliver polymer solutions. The polymer solution was filled with a copper cable attached to a positive electrode (anode) and a negative electrode (cathode) fastened to a metal drum (collector). Electrospinning was carried out by positioning the plastic syringe in front of the collector in ambient conditions. The nylon 6 polymer solution's electrospinning was optimized as using a voltage of 15 kV and setting the tip-to-collector distance of 15 cm. The polymeric nanofibres were electrospun and deposited continuously on a surface of the rotating metallic drum for 8 h forming a 'fibrous mat'. The nylon 6 electrospun nanofibrous mats were dried in air to remove the residual solvent for 24 h. The thickness of nylon 6 electrospun nanofibrous mats obtained was in between 30-40 µm. The thickness was measured with a Digital Micrometer MCD 130-25 (Japan). The nylon 6 electrospun nanofibrous mats were then dyed with reactive dyes by batchwise method.

Conventional Exhaust Dyeing of Nylon 6 Electrospun Nanofibrous Mats

The nylon 6 electrospun nanofibrous mats were cut into three square cm dimensions for dyeing with reactive dyes by conventional method. The nylon electrospun nanofibrous mat was dyed by preparing the dyebath with a varying reactive dye concentrations on mass of fibre (2, 2.5, 3, 3.5, and 4%) and a liquor-to-fibre ratio of 40:1. The dyeing temperature was raised (40, 50, 60, 70, and 80 ºC) and continued for varying durations (30, 40, 50, 60, and 70 min) to confirm the dye fixation on the nylon 6 electrospun nanofibrous mat. The dyeing was also experimented for varying dyebath pH 4, 5, 6, 7, and 8). Multiple dyeings with varying parameters were conducted for optimization. The dyed samples were gently rinsed with cold water (5 min) then washed in a bath of liquor ratio 60:1 using 2 g/L non-ionic detergent (Hostapal CV) at 60 °C for 30 min and then again rinsed by

batch-wise method, until no dye was bleeding. Finally, dyed nylon 6 electrospun nanofibrous mats were dried in air.

US Dyeing of Nylon 6 Electrospun Nanofibrous Mats

The US dyeing and washing-off of nylon 6 electrospun nanofibrous mats were accomplished in the exact same way as the CN dyeing detailed in the preceding section. However, the dyeing step was carried out in the US bath.

Testing

Colour measurement and testing

The Datacolour SF650 reflectance spectrophotometer (USA) was used to test the dyed nylon electrospun nanofibrous mats for the colour yield (*K/S* value), CIE *L* a* b* C** and h^o coordinates. The equipment was set up with a 9 mm sample aperture, an illuminant D65 with a 10° standard observer, and both the UV and specular components included. Five distinct locations of each sample were tested, and the average value has been reported. The colour yield is determined on basis of the reflectance value at maximum absorption by a Kubelka-Munk equation referred to as Eq. 1 [7].

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

Where K is the absorption coefficient, S is the scattering coefficient, and R is the reflectance of the coloured electrospun nanofibrous mat at its maximum absorption. Dyed samples were examined for colourfastness to mercury light (BS 1006:1990, UK-TN) on a light fastness tester in addition to being tested for colourfastness to washing (ISO 105 C10:2006) on a Gyrowash (James H. Heal, UK) (SDL Atlas, UK).

Thermal energy in dyeing process

According to reports, the main benefits of the US dyeing method over the CN dyeing method include reduced water use, dyestuff usage, effluent discharge, and thermal energy use [21]. The following equation was used to determine how much thermal energy was needed for the CN and the US methods.

$$Q = m \text{ x } c \text{ x } \Delta q \quad (2)$$

Where Q is the thermal energy content (in kcal), m is the mass of alcohol (in kg), c is the heat capacity of water (in Cal/g ^{o}C), and Δq is the change in temperature (in ^{o}C).

Effluent testing

To prepare a representative dyeing effluent, the optimum dyebath recipes of both the CN and US methods were diluted 100 times with deionized water. Using digital pH meter (HACH), the produced effluents were examined for pH and for TDS contents with a digital TDS meter (Mettler Toledo).

Morphology of nylon electrospun nanofibrous mats

On a scanning electron microscope (JSM 6380L, JEOL, Japan) operating at a 15 kV accelerating voltage, the surface morphology of electrospun nanofibrous mats was studied. Before SEM test, the sample was vacuum sputtered with gold. The average diameter distribution for each nylon electrospun nanofibrous mat was found using image analysis software (Image Pro® Plus, Version 5.1, Media Cybernetics, Inc.) from SEM images (5000x) in 100 different areas.

The chemical composition of nylon electrospun nanofibrous mats

On a Nicolet iS10 FT-IR spectrometer, a chemical structural investigation of nylon 6 electrospun nanofibrous mat was performed using attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy in the 4000-800 cm^{-1} range (Thermo Electron Corporation, USA).

Results and Discussion

Effect of Dyebath pH

The colour yield of dyed nylon 6 electrospun nanofibrous mats were evaluated for dyeings at five different pH levels (4, 5, 6, 7, and 8). The dyeing time and temperature were kept constant for 60 min at 80 °C using a 4% dye concentration. The dyeability of nylon 6 electrospun nanofibrous mats with reactive dye (CI Reactive Black 5) using both conventional and US dyeing methods is significantly influenced by the pH values of the dyebath, as shown in Fig. 1. It was discovered that the colour yield of nylon 6 electrospun nanofibrous mats was steadily improved along with a concurrent rise in the dyebath's pH from acidic to alkaline pH. It is evident that the nylon 6 electrospun nanofibrous mats are more dyeable at all pH values thanks to the US dyeing technique. At an alkaline pH, it was quickly elevated until it reached a colour yield of 1.54. Similar colour yield profiles for nylon 6 electrospun nanofibrous mats dyed using the CN dyeing method were achieved, with a maximum colour yield of 1.35. Significantly, when compared to the conventional approach, nylon 6 electrospun nanofibrous mats dyed with the US method yielded improved colour yield values. This was attributed to the continual cavitation that US energy caused in the dyeing solution, which allowed reactive dyes to de-aggregate and improved dye mobility to create a stir effect that improved disperse dye diffusion into electrospun nanofibrous mats [21].

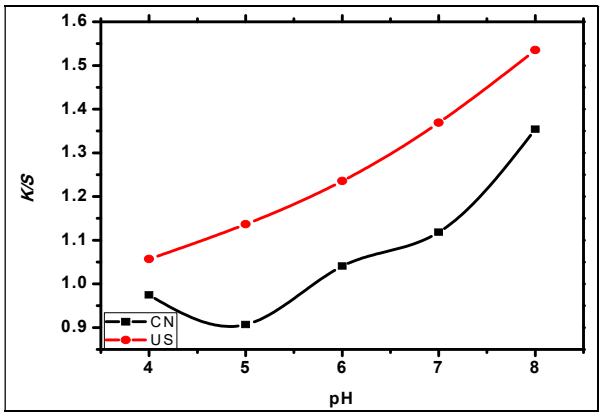

Figure 1. Effect of pH on colour yield (K/S) in both conventional (CN) and US (US) dyeings

Effect of Dyeing Temperature

Dyeing of nylon 6 electrospun nanofibrous mats was caried out with five different temperatures (40, 50, 60, 70, and 80 ºC) at an alkaline pH, maintaining the dyeing time constant for 60 min using a 4 % dye concentration. Fig. 2 displays the colour yield of nylon 6 electrospun nanofibrous mats coloured using the batchwise approach with CI Reactive Black 5 reactive dye at varying temperature. It was discovered that the colour yield of nylon 6 electrospun nanofibrous mats gradually improved with a concurrent rise in dyeing temperature from 40 °C to 80 °C. The colour yield of dyed nylon 6 electrospun nanofibrous mats was significantly higher in the US procedure between 70 ºC and 80 ºC dyeing temperature. From 40 ºC, the colour yield for nylon 6 electrospun nanofibrous mats coloured using the US method steadily increases. The temperature was then quickly increased to 80 °C, reaching a colour yield of 1.54 for CI Reactive Black 5. For the CN dyeing method, similar colour yield profiles for nylon 6 electrospun nanofibrous mats with coloured fibres were achieved, with a maximum colour yield of 1.35 for CI Reactive Black 5.

In contrast to the conventional approach, nylon 6 electrospun nanofibrous mats dyed with the US method yielded improved colour yield values. This was explained by the continual cavitation in the dyeing solution caused by US energy, which allowed for the de-aggregation of reactive dyes and an improvement in dye mobility, creating a stir effect that increased reactive dyes diffusion into the electrospun nanofibrous mats [14].

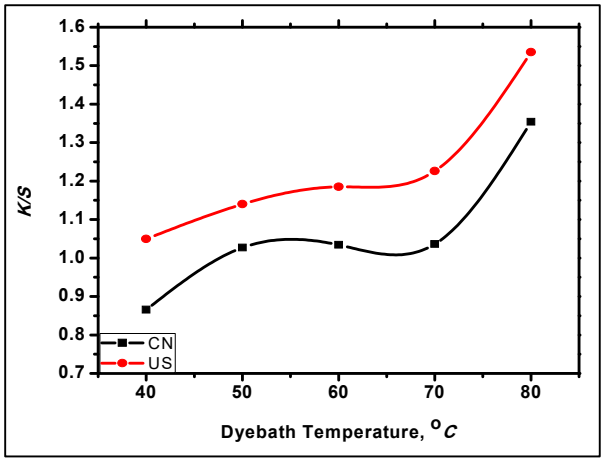

Figure 2. **Effect of dyebath temperature on colour yield (K/S)**

Dyeing Time Effect

The colour yield of dyed nylon 6 electrospun nanofibrous mats was measured for dyeings at five different dyeing periods (30, 40, 50, 60, and 70 min) at alkaline pH. The dyeing temperature was kept constant at 80 ºC using 4% dye concentration. The colour yield values of dyed nylon 6 electrospun nanofibrous mats using the batchwise approach are shown in Fig. 3. The findings showed that dyed nylon 6 electrospun nanofibrous mats gradually improved in colour yield as dyeing duration increased from 30 to 70 min. Similar to the pattern of findings shown in Fig. 4, colour yield results for nylon 6 electrospun nanofibrous mats coloured using the US approach were likewise superior to the conventional method for dyeing duration (Fig. 5). The highest colour yield achieved for the US approach was 1.72, and the highest colour yield for the conventional method was 1.42. The US cavitation, which causes disintegration of the aggregated reactive dyes and promotes dye mobility, was blamed for the improved colour yield findings in the US approach.

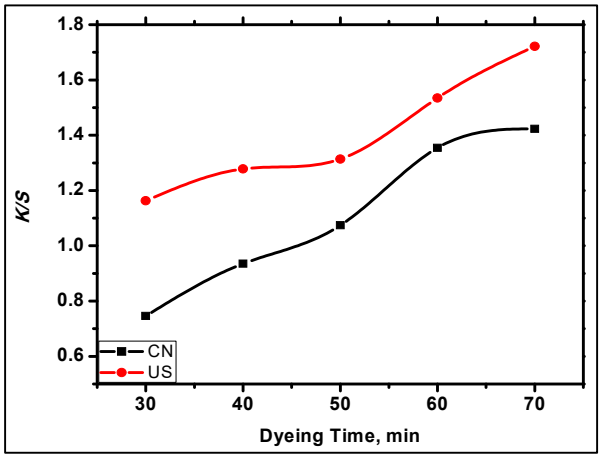

Figure 3. **Effect of dyeing time on colour yield (K/S)**

Effect of Dye Concentration (Colour Buildup)

Using the previously optimized circumstances, the impact of dye concentration on colour accumulation of the reactive dye (CI Reactive Black 5) was examined. The colour buildup characteristics at five distinct dye concentrations of 2, 2.5, 3, 3.5. and 4 % were assessed based on colour yield. Results shown in Fig. 4 show that the colour yield increases as dye concentration rises. However, the colour yields obtained even at 4% dye concentration were not very high. That may be because the active dye sites (protonated amino groups in this case) are significantly unavailable at the alkaline pH condition [22]. Moreover, the colour yield of nylon 6 electrospun nanofibrous mats dyed with the US approach are greater than those made using the conventional method; at a dye concentration of 4%, US dyeing yielded a colour yield that was 64.1% higher. The US

cavitation produced during exhaust dyeing is responsible for this increase in colour yield with sonication. Using conventional and US techniques, the profiles of colour yield findings show the uniform behavior of both dyes at optimized conditions.

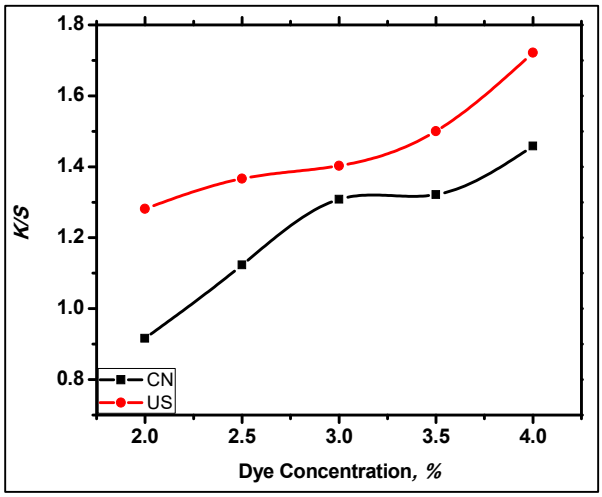

Figure 4. **Dye concentration's impact on colour yield (K/S)**

Colourimetric Values of Dyed Nylon 6 Electrospun Nanofibrous Mats

Table 1 compares the colourimetric values of the electrospun nanofibrous mats dyed by CN and US methods. It was noted in both instances that the lightness values (L^*) dropped as reactive dye concentrations rose from 2% to 4%, indicating that the dyed nylon 6 electrospun nanofibrous mats darkened. However, the L^* values of electrospun nanofibrous mats dyed by US method were slightly lower than those of the electrospun nanofibrous mats dyed by CN method at same dye concentration, indicating that the US dyed electrospun nanofibrous mats were darker than convetionally dyed. Whereas in both cases, negative b^* values of all the dyed samples near to the center of the b^* scale show slightly dull blue colour, and the negative a^* value was due to greenness in the dyed samples. The dullness in the samples is also evident from the lower C^* values.

Colourfastness Properties

In Table 2, results for colourfastness to light showed that the dyed sample using the US technique received ratings that were one grade higher than those for the dyed sample using the CN technique. However, the overall light fastness for both methods is moderate. In general, both the CN and US techniques received extremely good scores for colourfastness to washing (colour change and stains on multifibre). Such colourimetric and fastness properties demonstrate that the dyed nylon 6 electrospun nanofibrous mats have the potential to be considered for advanced apparels.

Table 1. **Colourimetric values of dyed nylon 6 electrospun nanofibrous mats by both methods (with different dye concentrations).**

CI Reactive Black 5 Dye (%)	L^*		a^*		b^*		C^*		h^o	
	CN	*US*	*CN*	*US*	*CN*	*US*	*CN*	*US*	*CN*	*US*
2	67.77	64.60	-7.57	-8.21	-14.79	-18.87	16.61	20.58	242.91	246.50
2.5	65.89	63.82	-8.26	-8.56	-17.15	-18.97	19.03	20.81	244.30	245.71
3	63.04	63.34	-7.81	-8.89	-15.78	-18.17	17.61	20.23	243.67	243.91
3.5	63.63	62.68	-8.24	-8.76	-17.51	-19.36	19.35	21.25	244.80	245.65
4	62.78	59.90	-8.25	-8.26	-18.15	-18.21	19.94	19.99	245.57	245.59

Table 2. **Colourfastness properties of nylon 6 electrospun nanofibrous mat dyed with CI Reactive Black 5 dye.**

Dyeing method	Washing fastness							Light fastness Blue wool reference
	Change in colour	Staining on Multifibre [a]						
		CT	CO	PA	PES	PAN	WO	
CN	4/5	4/5	5	3/4	5	5	4/5	4
US	5	4/5	5	4/5	5	5	5	5

[a] CT, cellulose triacetate; CO, cotton; PA, polyamide; PES, polyester; PAN, polyacrylonitrile; WO, wool.

Saving the Thermal Energy, Dyeing Time and Amount of Dye in US Method

In this investigation, the US approach demonstrated potential reductions in terms of heat energy, dyeing time, and dye buildup. Table 3 illustrates the thermal energy usage for dyeing nylon 6 electrospun nanofibrous mats (estimated as indicated in the experimental section), dyeing time and the colour buildup on to the substrate in terms of the *K/S* value. The constants in this experimentation were mass of electrospun nanofibrous mats of 1 g, dyeing liquor ratio of 15:1, and the dye concentration of 1% on mass of substrate. In CN method, colour yield of 1.04 was found at 70 °C. The alike shade with slightly more yield was comprehended, i.e. colour yield of 1.05, at 40 °C in the US method. This demonstrated the saving of 1200 *kcal* (200%) thermal energy by US method.

The table also shows how much time can be saved while dyeing nylon 6 electrospun nanofibrous mats. At constant dyeing temperature of 80 °C, a colour yield of 1.42 at 70 min for CN method and more colour yield, i.e. 1.54, was obtained at 60 min by the US method. This displays a time savings of 10 min (66.7%) throughout the dying procedure.

Moreover, the table results showed that using the US approach, practically similar colour yield values could be obtained with a lower dye concentration. The colour yield of around 1.42 was obtained with 4% dye concentration in the CN method, whereas a similar colour yield was realized, i.e. 1.4, with 3% dye concentration in the US method. This demonstrates the saving of around 25% in the dye concentration needed during dyeing process. The discharge of less polluted effluent will ultimately result in a significant reduction in dyeing costs and associated environmental advantages.

Table 3. **Comparative values of dyeing temperature, dyeing time, colour buildup (K/S values) and thermal energy consumption for US and CN dyeings.**

S. No.	Dyeing Temp. (*°C*)	Dyeing Time (*min*)	Dye Conc. (*%*)	*K/S* CN method	*K/S* US method	Thermal Energy consump. (*kcal*)
1	40	60	1	0.86	1.05	600
2	50	60	1	1.03	1.14	1000
3	60	60	1	1.03	1.18	1400
4	70	60	1	1.04	1.23	1800
5	80	60	1	1.35	1.54	2200
6	80	30	1	0.74	1.16	-
7	80	40	1	0.94	1.28	-
8	80	50	1	1.07	1.31	-
9	80	60	1	1.35	1.54	-
10	80	70	1	1.42	1.72	-
11	80	30	2	0.92	1.28	-
12	80	30	2.5	1.12	1.37	-
13	80	30	3	1.31	1.40	-
14	80	30	3.5	1.32	1.50	-
15	80	30	4	1.42	1.72	-

Analysis of the Dyeing Effluent

Table 4 displays the characteristics of the dyeing effluent used in this investigation (pH and TDS). The results disclosed that the effluent is slightly alkaline in pH. However, the TDS content of the dyeing effluent was considerably decreased (i.e. around 28%) for the US method mainly due to the use of reduced amount of the dye in comparison to the conventional method. Due to the issues with the release of reactive dye effluent due to high TDS concentrations, this finding implies that the US technique might be recommended. This result indicates that the US dyeing approach might be favored considering the issues related to the discharge of reactive dye effluent due to high TDS concentrations.

Table 4. **Analysis of reactive dyeing effluent.**

Dyeing method	pH	TDS (mg/L)
US	7.9	520
CN	7.9	720

Surface Morphology of Undyed and Dyed Nylon 6 Electrospun Nanofibrous Mat

The SEM images of nylon 6 electrospun nanofibrous mats before and after dyeing (using both the CN and US methods) with reactive dyes under ideal circumstances are shown in Fig. 5(a-c). The Fig. 5a's regular and bead-free surface morphology suggests that the nylon 6 polymer concentration, binary solvent system selection, and electrospinning process parameters used were all at their best. In the remaining pictures, the dyed nylon 6 electrospun nanofibrous mats had a slightly enlarged morphology in contrast to the undyed nylon 6 electrospun nanofibrous mat but nevertheless had a stable structure regardless of the application of thermal energy or US energy.

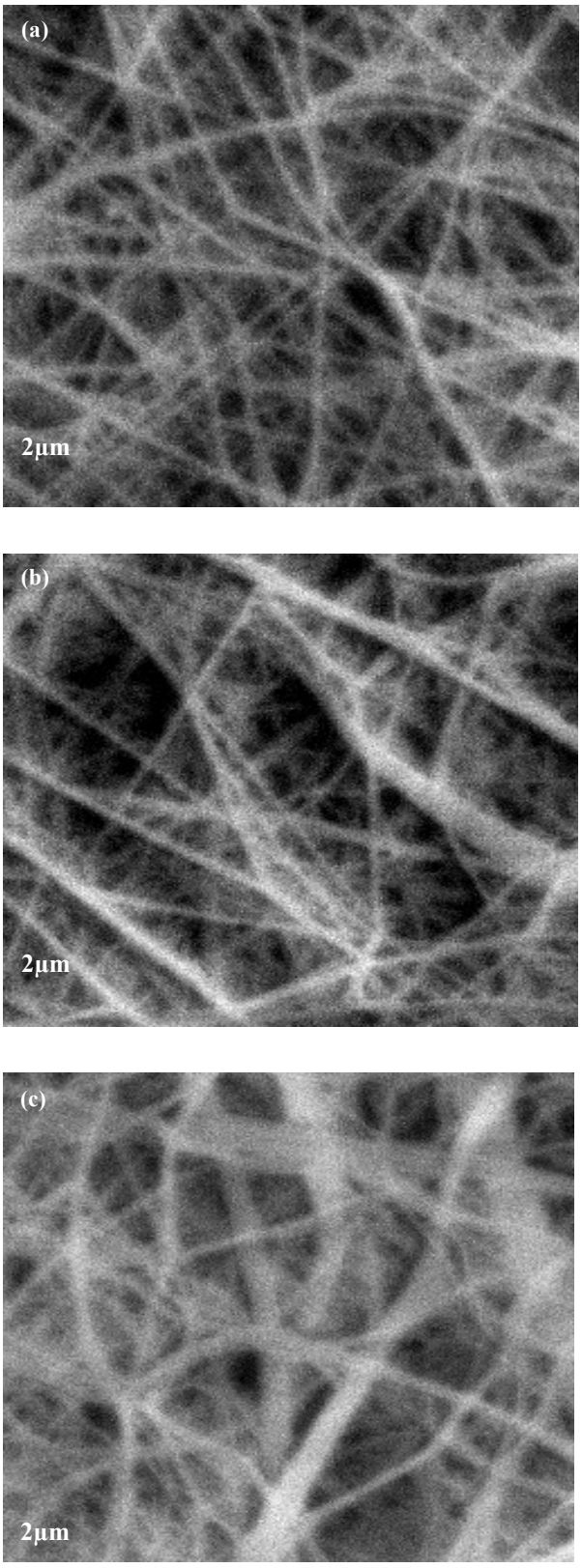

Figure 5. **SEM images of nylon 6 electrospun nanofibrous mats: (a) undyed, (b) CN dyed, and (c) US dyed**

This demonstrates that the electrospun nanofibrous mats were resistant to the wet and heat treatments used throughout the dyeing process. Whereas, the dyed fibres look swollen, indicating that the dye molecules are properly fixed in the fibres. The slight swelling of the fibres may be attributed to the molecular absorption and interaction with the polymer matrix. The conventional dyeing method (Fig. 5b) resulted in an average fibre diameter increase of around 7-10%, whereas the ultrasonic dyeing method (Fig. 5c) exhibited comparatively lower diameter increase of around 4-6%. This suggests that ultrasonic cavitation may enhance dye penetration into the fibre structure without excessive fibre swelling, potentially leading to more uniform dye diffusion.

FT-IR Analysis of Undyed and Dyed Nylon 6 Electrospun Nanofibrous Mats

Figure 6 shows the FT-IR spectra of nylon 6 electrospun nanofibrous mats that have been dyed and undyed using the US batchwise process. The 1645 cm^{-1} and 1541 cm^{-1} absorption peaks are associated with the Amide I and II groups, respectively [23]. The NH-stretching at 3304 cm^{-1}, the CH_2 stretching peak at 2915 cm^{-1}, and the vibrations of nylon-6's chemical structure at 2848 cm^{-1} were additional absorption peaks [24]. Whereas, in the dyed electrospun nanofibrous mat, extension in the peak slightly at 2325 cm^{-1}, that appears because of CO_2 [25], indicates hydrogen or chemical bonding between the dye molecule and the fibre polymer. Moreover, the new absorption peak located at 3420 cm^{-1} is associated with O-H stretching vibrations [26], which could be associated with the absorbed moisture or hydroxyl group in the dye. However, similar peaks, especially at 1645 cm^{-1}, suggest that the basic nylon 6 structure remains intact after dyeing.

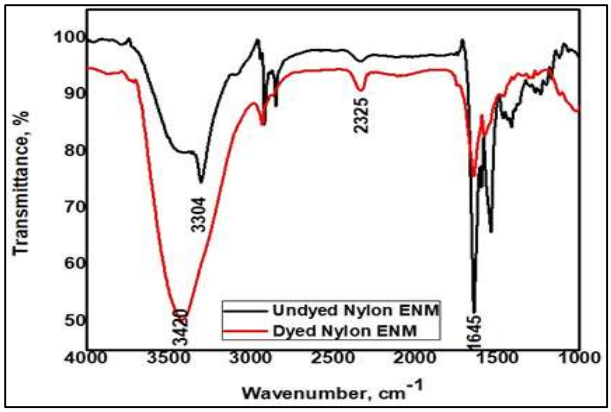

Figure 6. **FT-IR spectra of nylon 6 electrospun nanofibrous mats (a) undyed (b) dyed with CI Reactive Black 5**

Conclusion

It is concluded that the US dyeing of nylon 6 electrospun nanofibrous with reactive dyes can be successfully carried out, offering a sustainable and energy efficient alternative to conventional dyeing methods. Successful coloration was assured by enhanced colour yield (*K/S*: 1.72 vs. 1.42), improved colourfastness, and the morphological stability confirmed by the SEM and FT-IR analyses. Additionally, the US dyeing method demonstrated significant process improvements, including 25% reduction in dye consumption, 1200 *kcal* of thermal energy saving, more than 65% reduction in dyeing time, and 28% reduction in the effluent TDS content, making it an eco-friendly coloration approach. These findings suggest that US dyed nanofibers can be used for functional and high performance textile applications and can be further explored for scalability.

Acknowledgement

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Conflict of Interest

We declare that we have no conflict, financial, competing or any other interests against the research work presented in this manuscript.

References

1. D. S. Katti, K. W. Robinson, F. K. Ko and T. Laurencin, *J. Biomed. Mater. Res.*, 70 (2004) 286. doi: 10.1002/jbm.b.30041
2. J. Lee, S. Balakrishnan, J. Cho, S.-H. Jeon and J.-M. Kim, *J. Mater. Chem.*, 21 (2011) 2648. doi: 10.1039/C0JM02287J
3. O. Mapazi, K. P. Matabola, R. M. Moutloali and C. J. Ngila, *Polym.*, 149 (2018) 106. doi: 10.1016/j.polymer.2018.06.028
4. F. Piccinno, R. Hischier, A. Saba, D. Mitrano, S. Seeger and C. Som, *J. Clean. Prod.*, 112 (2016) 1199. doi: 10.1016/j.jclepro.2015.06.105
5. A. Greiner and J. H. Wendorff, *Angew. Chem. Int. Ed.*, 46 (2007) 5670. doi: 10.1002/anie.200604646
6. L. Sumin, D. Kimura, A. Yokoyama, K. Lee, J. Park and I. Kim, *Text. Res. J.*, 79 (2009) 1085. doi: 10.1177/0040517508101
7. A. Khatri, S. Ali and A. Javeed, *Cellulose*, 30 (2023) 7955. doi: 10.1007/s10570-023-05396-1
8. L. Lara, I Cabral and J. Cunha, *Sustainability*, 14 (2022) 8353. doi: 10.3390/su14148353
9. A. Khatri, M. H. Peerzada, M. Mohsin and M. White, *J. Clean. Prod.*, 87 (2015) 50. doi: 10.1016/j.jclepro.2014.09.017
10. A. Q. Shah, T. G. Kazi, J. A. Baig, H. I. Afridi, G. A, Kandhro, S. Khan, N. F. Kolachi and S. K. Wadhwa, *Pak. J. Anal. Environ. Chem.*, 11 (2010) 12. http://www.pjaec.pk/index.php/pjaec/article/view/102
11. C. Udrescu, F. Ferrero and M. Periolatto, *Ultrason. Sonochem.*, 21 (2014) 1477. doi: 10.1016/j.ultsonch.2013.12.026
12. M. Khatri, F. Ahmed, A. W. Jatoi, R. B. Mahar, Z. Khatri and I. S. Kim, *Ultrason. Sonochem.*, 31 (2016) 350. doi: 10.1016/j.ultsonch.2016.01.020
13. S. Ali, M. A. Mughal, U. Shoukat, M. A. Baloch and S. H. Kim, *Carbohydr. Polym.*, 117 (2015) 271. doi: 10.1016/j.carbpol.2014.09.064
14. A.W. Jatoi, F. Ahmed, M. Khatri, A. Tanswari, Z. Khatri, H. Lee and I. S. Kim, *Ultrason Sonochem.,* 39 (2017) 34. doi: 10.1016/j.ultsonch.2017.04.010
15. A. Khatri, *Pad-steam Dyeing of Cotton with Reactive Using Biodegradable Alkaline Organic Salts*, Ph.D. Thesis, RMIT University Australia, 2011.
16. Z. Khatri, G. Mayakrishnan, Y. Hirata, K. Wei and I. S. Kim, *Carbohydr. Polym.*, 91 (2013) 434. doi: 10.1016/j.carbpol.2012.08.046
17. A. Sattar, A. Khatri, S. Ali and F. Ahmed, *Color. Technol.*, 140 (2024) 279. doi: 10.1111/cote.12713
18. S. M. Burkinshaw and A. E. Wills, *Dyes Pigments*, 34 (1997) 243. doi: 10.1016/S0143-7208(96)00074-5
19. M. Park, M. M. Rabbani, H. K. Shin, S.-J. Park and H. Y. Kim, *J. Ind. Eng. Chem.*, 39 (2016) 16. doi: 10.1016/j.jiec.2016.05.031
20. S. Ali, Z. Khatri, K. W. Oh, I.-S. Kim and S. H. Kim, *Macromol. Res.*, 22 (2014) 562. doi: 10.1007/s13233-014-2078-x
21. S. Ali, A. Khatri, A. Javeed and S. H. Kim, *J. Text. Inst.*, 111 (2020) 723. doi: 10.1080/00405000.2019.1661064
22. S. M. Burkinshaw and K. Gandhi, *Dyes Pigments*, 33 (1997) 259. doi: 10.1016/S0143-7208(96)00056-3

23. H. R. Pant, M. P. Bajgai, C. Yi, R. Nirmala, K. T. Nam, W.-I. Baek and H. Y. Kim, *Colloids Surf. A Physicochem. Eng. Asp.*, 370 (2010) 87. doi: 10.1016/j.colsurfa.2010.08.051
24. J. J. Ryan, R. Casalini, J. A. Orlicki and J. G. Lundin, *Polym. Adv. Technol.*, 31 (2020) 3039. doi: 10.1002pat.5028
25. M. Champeau, J.M. Thomassin, C. Jérôme and T. Tassaing, *J. Supercritic. Fluids*, 90 (2014) 44. doi: 10.1016/j.supflu.2014.03.006
26. I. Sriyanti, M. P. Agustini, J. Jauhari, S. Sukemi and Z. Nawawi, *J. Ilmiah Pendidik. Fis. Al-Biruni*, 9 (2020) 9. doi:10.24042/JIPFALBIRUNI.V9I1.5747