

DYEING OF POLYESTER FABRIC USING A NATURAL DEEP EUTECTIC SOLVENT

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(ORIGINAL SCIENTIFIC PAPER)

UDC: 667.494.674:677.027

DOI: 10.5937/savteh2402045N

Considering large water quantities that are consumed for the process of dyeing different textile materials, there is a constant expansion focused on designing more sustainable dyeing methods. Disperse dyes used to dye polyester are not soluble in water, so toxic dyeing auxiliaries (carriers and dispersants) are often used to dissolve them. In current work, a betaine-based natural deep eutectic solvent (NADES) was used in a dual manner: to ensure reduction of generated wastewater and eliminate the need for environmental-unfriendly auxiliaries such as dispersants and carriers. Dyeing experiments were carried out with different dye concentrations in the 1-5% range. Regarding the conventional method, dyeing was carried out at 100 °C for 45 min with the addition of carrier, dispersing agent, and acetic acid. In contrast, NADES-based method involved fabric dyeing in a mixture of NADES and distilled water in ratio of 70:30. The pH value of 4 was maintained identical for both methods. Dyeing efficiency was analyzed according to color strength, fastness to washing and light. NADES-based method demonstrated better overall performance, without affecting both polyester fabric's tensile strength and elongation at break. Based on the obtained results, betaine-based NADES can be used as a "green" medium for polyester dyeing.

Keywords: polyester, dyeing, natural deep eutectic solvent, disperse dye

Introduction

Polyester fibers, composed of linear chain polymers containing ester groups, have a broad range of applicability, including the textile industry [1, 2]. These fibers are typically rod-shaped, homogeneous, and extremely crystalline. Dyeing of polyester fibers is often performed at high temperatures (~120-130 °C) because of their hydrophobic nature [3, 4]. The most commonly used dyes for polyester dyeing are disperse dyes. However, these types of dyes are characterized by poor water solubility since they lack water-soluble groups and usually exist as pre-polymers with non-ionic characteristics [5, 6].

To increase disperse dyes hydrosolubility and improve their stability by preventing particle aggregation, auxiliaries (such as dispersants and carriers) must be added to the dye solution. However, some auxiliaries are highly toxic and problematic regarding the environment [7, 8]. Although dispersants improve the dyeing process, they are difficult to remove once the process has been finalized, generating a great amount of wastewater. Therefore, it is necessary to improve current techniques for fiber dyeing, to reduce the overall negative impact on the environment [9-12].

Several new dyeing techniques have been developed, such as ultrasonic, supercritical CO₂, plasma processing, and non-aqueous solvent dyeing; all of which use a certain solvent as a dyeing medium instead of water. Since water can be replaced by different non-aqueous solvents, the formation of wastewater is reduced [13]. Although disperse dyes dissolve more readily in conventional organic solvents than in water, these solvents are extremely poisonous, flammable, and volatile due to their lower boiling points and even lead to textile deterioration, limiting their use in fabric dyeing. The benefits of natural deep eutectic solvents (NADESs) as non-toxic compounds, which include higher boiling points, low cost, biodegradability, and the ability to dissolve a broader range of both organic and inorganic compounds, can make them environmentally-friendlier substitutes for conventional volatile organic solvents, including the process of dyeing various natural and synthetic fibers [14]. The physicochemical characteristics of NADESs may be tailored by modifying the molar ratio of the components; some of them are cheap, natural, widely available, and safe for handling [15].

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The manuscript received: 07. 07. 2024.
Paper accepted: 20. 11. 2024.

Literature data have demonstrated the beneficial effect of different NADESs for the dyeing fabrics. Dyeing of polyester with Disperse Blue 56 in NADES composed of choline chloride, urea, and glycerin (1:1:1) proved to be better than conventional dyeing in an aqueous solution, as well as sublimation fastness. Also, polyester fibers dyed in NADES were stiffer than those undyed and dyed in an aqueous solution, while wash and light fastnesses are almost similar [16].

Choline chloride:urea (1:1) NADES-assisted dyeing of polyester fibers with Disperse Orange 30, Disperse Blue 56, and Disperse Red 82 was also superior in terms of being efficient at 100 °C, *i.e.* at a temperature value lower than those applied for the conventional dyeing (120-130°C) [17]. Choline chloride:ethylene glycol (1:2) NADES significantly improved the dyeing of polyester fabrics with disperse dyes (C.I. Disperse Blue 79 and C.I. Disperse Red 343), excluding the need for a dispersant. Both dyes completely dissolved in NADES and showed excellent stability. As a result, the color strength and rubbing fastness were clearly enhanced, even at a lower temperature [18].

Zheng et al. [19] converted polyethylene terephthalate (PET) fabrics from hydrophobic to hydrophilic by using a choline chloride:oxalic acid (1:1) NADES. Besides the increased water absorption capacity, the NADES-treated PET fabric expressed improved anti-static and dyeing properties, compared to the raw (untreated) PET fabric. The best results for dyeing polyester fabric with anthraquinone dyes were obtained after 80 min with choline chloride:glycerol (1:3) and choline chloride:urea (1:4) NADESs, with an optimal temperature of 130 °C and a dye concentration of 0.05 mg/mL [20]. NADESs have not executed the change regarding the structure and stability of fibers, but increased color intensity [20]. Silk dyeing with Acid Blue 281 in choline chloride:urea NADES as a swelling agent was performed at 100 °C. NADES-method dyed silk fabric showed the desired color strength at 60 °C for 37 min, with the maximum strength observed at 70 °C after 45 min [21]. Choline chloride-based NADES with different hydrogen bond donors (oxalic, citric, and malic acid) were used as a water-free medium enabled successful dyeing of wool fabric at 80 °C with smaller amount of C.I. Acid Red 249. Besides higher K/S value, the dyed fabric showed similar color fastness values to those dyed via conventional dyeing in aqueous solution [22].

Choline chloride:lactic acid (1:2) NADES proved to be an adequate water-free medium for multifiber fabrics' dyeing with non-toxic crude gel prodigiosin, produced by non-pathogenic *Serratia plymuthica*.

At optimal dyeing conditions (60 °C, 1 h, pH 8.3) this NADES-method was suitable for obtaining high K/S values and improved ΔE values, especially for nylon and wool fabrics. Moreover, nylon dyed with 3% L-cysteine at pH 8.3 expressed superior wash fastness, while the use of NADES contributed to exceptional

light fastness [23].

Although various quaternary ammonium salts are employed as constituent for NADESs preparation, choline chloride (vitamin B4) is still the most commonly used due to several advantages: it is cheap, biodegradable, non-toxic and possesses strong affinity for formation of intermolecular interactions [24, 25]. As widely available compound, it is mostly used as an additive in animal feeding formulations [24]. However, in recent years, betaine has been shown to be the most promising substitution for choline chloride. Betaine is an organic salt containing positively charged quaternary nitrogen and anionic carboxylic acid group, possessing distinctive features. It is a non-toxic, readily biodegradable osmolyte playing fundamental roles in cellular metabolic processes [25]. In comparison to choline chloride, found on the market only as a synthetic product, betaine is obtained from renewable sources, since it is a by-product of sugar production [25, 26]. Moreover, due to its higher biodegradability, degree of sustainability and lower toxicity, betaine is justifiably receiving more attraction in the research community [26].

In this paper, the process of polyester fiber dyeing with C.I. Disperse Violet 26 was improved by including betaine-based NADES. The process was performed at 100 °C for 45 min at different dye concentrations (1-5%). The aim was to use NADES to eliminate the need for dyeing auxiliaries such as carriers and dispersants, as well as to reduce water consumption during the dyeing process.

Materials and Methods

Materials

The 100% polyester fabric with 75 g/m² mass per unit area was procured from Yumco (Vranje, Serbia).

Chemicals

The chosen disperse dye (C.I. Disperse Violet 26, chemical structure provided in Figure 1) was purchased from Hoechst (Frankfurt, Germany). Betaine hydrochloride (Tokyo Chemical Industry, Tokyo, Japan), glycerine (Meilab, Belgrade, Serbia) and urea (Centrohem, Stara Pazova, Serbia) were employed for NADES preparation. The chemicals used for dyeing were: acetic acid (Zorka Pharma-Hemija DOO, Šabac, Serbia), Dilatin DBI (Sandoz, India Ltd.) as a carrier, CHT-Dispergator XHT-S (CHT GmbH, Tübingen, Germany) as a dispersing agent and distilled water. Sodium hydroxide and sodium hydrosulfite were bought from Centrohem (Stara Pazova, Serbia), while non-ionic detergent Sarabid DLO was from CHT GmbH (Tübingen, Germany).

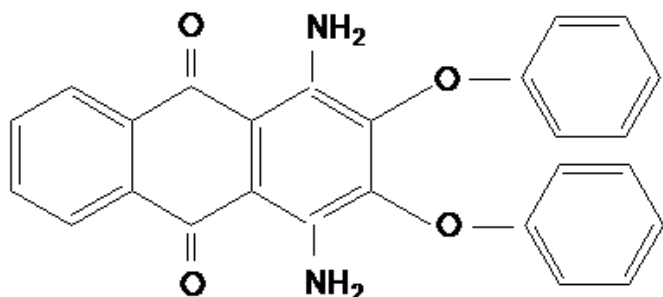


Figure 1. Chemical structure of C.I. Disperse Violet 26

Preparation of NADES

Equimolar amounts of betaine hydrochloride, glycerine, and urea were mixed in a beaker. The mixture was continuously stirred for 20 min at 35 ± 5 °C in a magnetic stirrer at 1100 rpm until a homogeneous and transparent liquid was obtained. The NADES was stored in a desiccator with CaCl_2 .

Pre-dyeing process

Pre-dyeing processing of polyester fabric: washing in detergent Sarabid LDR (CHT GmbH, Tübingen, Germany) in concentration 2 g/l for 30 min, at 60 °C and ratio 1:30.

The dyeing processes

Both conventional and NADES-assisted dyeing processes were conducted at the following identical conditions: temperature of 100 °C, duration of 45 min, bath ratio of 1:50, pH value of 4 at different dye concentrations (1, 2, 3, 4, and 5%, w/w).

Conventional dyeing method: dyeing auxiliaries: 20% acetic acid, 2% carrier, 2% dispersing agent.

NADES-assisted dyeing method: NADES/distilled water ratio of 70:30 without any dyeing auxiliaries.

Post-dyeing process

Post-dyeing processing of polyester fabric consisted of the following steps: reduction cleaning (with 2% sodium hydroxide and 2% sodium hydrosulfite at 60 °C for 15 min), rinsing with cold water, soaping (with 2% non-ionic detergent at room temperature) and drying in a dryer at 100 °C.

Physical properties of polyester

The tensile strength and elongation at break characteristics were measured on a MesdanLab Strength Tester dynamometer in accordance with the ISO 13934/1 standard. Tensile strength and elongation at break values were measured for undyed, conventional and NADES dyed polyester fabric.

Fastness testing of dyed polyester fabric

EN ISO 10–B02 standard was used for the evaluation of color fastness to light of dyed polyester. SRPS EN ISO 105–C06 standard was carried out to determine color fastness to washing of dyed polyester fabric.

Color strength of dyed polyester fabric

Relative color strength (K/S) and CIELab coordinates (L^* , a^* , b^*) of dyed fabrics were measured using a spectrophotometer (Datacolor Spectraflash SF600X, New York, USA). The K/S values of dyed samples of fabrics were calculated using the Kubelka-Munk equation [27]:

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

where K is the absorption coefficient, S is the scattering coefficient, and R is the decimal portion of the dyed fabric's reflection.

Whiteness index of fabric

The standard method to estimate the whiteness was performed according to ISO 105-J02 is: Instrumental assessment of relative whiteness. The CIE whiteness index (WI) under the CIE 1964 standard observer (10°) and D65 illuminant was calculated by equation 2 [28]:

$$WI = Y_{10} + 800 (x_{n,10} - x_{10}) + 1700 (y_{n,10} - y_{10}) \dots \dots \dots (2)$$

where, Y_{10} is Y-value, x_{10} , y_{10} are chromaticity coordinates of the sample fabric and $x_{n,10}$, $y_{n,10}$ are the coordinates of the reference white (for D65 illuminant 10° standard observer, $x_{n,10}=0.3310$, $y_{n,10}=0.3310$), respectively.

Results and Discussion

Figure 2 shows conventionally dyed samples of polyester fabric with different dye concentrations.

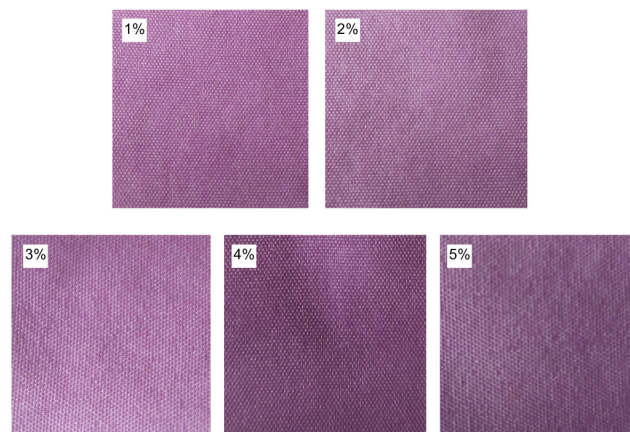


Figure 2. Conventionally dyed sample of polyester fabric with different dye concentrations

NADES-dyed samples at different dye concentrations are shown in Figure 3.

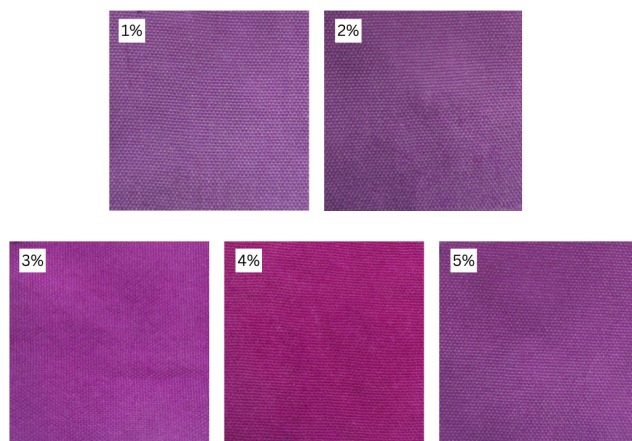


Figure 3. NADES-dyed samples of polyester fabric with different dye concentrations

The results of tensile strength and elongation at break of undyed, conventionally and NADES polyester are listed in Table 1. The tensile strength and elongation at break values (table 1) for all dyed samples of polyester were lower than those for undyed samples (834 and 492 N and 42.5% and 28.1% respectively); the lowest value for the tensile strength parameter was observed for the NADES dyed sample (828 and 486 N), while the lowest value for the elongation at break parameter was observed for NADES dyed polyester (40.1 and 27.2).

Table 1. Tensile strength and elongation at break of the undyed, conventionally and NADES-method dyed samples of polyester fabric

Tensile strength (N)				Elongation at break (%)			
Undyed		Conventionally		NADES		Undyed	
warp	weft	warp	weft	warp	weft	warp	weft
834	492	831	489	828	486	42.5	28.1

The whiteness index of fabric is given in table 2.

Table 2. Whiteness index of polyester fabric

Whiteness index, WI	
Light source	Value
D65 10	75.37

During dyeing, hydrogen bonds are formed between the dye molecules and ester and carboxyl groups in the polyester fabric. Due to the strong hydrophobic character and compact structure of the fibers, the dyeing process is slow. To increase the velocity of the dye diffusion into the fibers, it is necessary to either modify the existing dyeing procedure or rearrange/modify the fibers' surface. Color strength curves (K/S) and CIELab parameters determine the effect of polyester dyeing; the textile samples with the highest reflectance have the lowest K/S parameter values and vice versa [29, 30].

The molecular size of NADESs can affect both the dispersion efficiency of the dyes/pigments and their ability to penetrate the polyester fiber more efficiently, ena-

bling uniform dyeing. Solvents with smaller molecules have better penetration capacity, while those with larger molecules can affect adhesion of the dye to the fibers [18]. Urea, glycerin, and betaine hydrochloride have molecules of different sizes and properties, so their interaction with dye and polyester will be specific and unique. For example, glycerin has a higher molecular weight than urea but is less dispersed in water, while betaine hydrochloride has a specific chemical structure which affects its interactions with dye and fabric [16, 18].

Figures 4 and 5 show the K/S parameters of conventionally and NADES dyed polyester at different dye concentrations in the range of wavelengths of visible light λ_{max} = 400 - 700 nm. Regarding the influence of concentration, the maximum dye concentration of 5% provides the darkest coloration and the highest K/S values for both fabric samples, dyed by either the conventional and NADES-method. However, the values for color strength are significantly different, *i.e.* the K/S values were higher for NADES-dyed samples. K/S values suggest polyester dyeing in darker shades when NADES was employed as a "green" medium indicating a great absorptivity of Disperse Violet 26. Increased sorption and uniform dyeing by NADES-modified polyester fabric is the result of chemical and morphological changes in the surface layers of fibers. Also, polyester fibers with the same aggregation structure can accommodate multiple small-dimension dye molecules and show a higher K/S value.

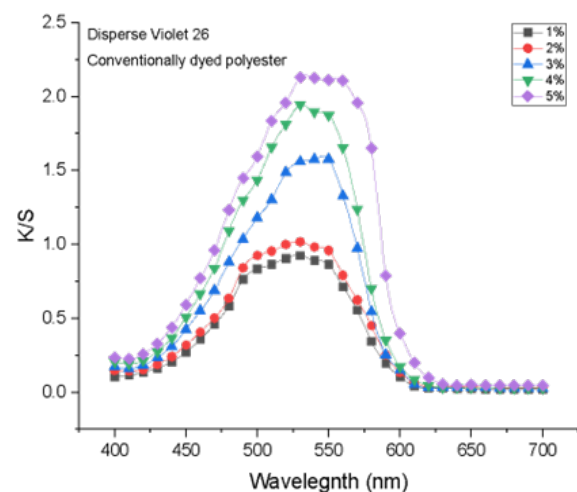


Figure 4. K/S values of conventionally dyed polyester at different dye concentrations

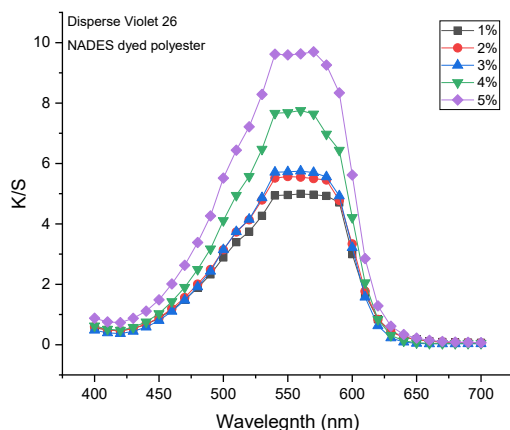


Figure 5. K/S values of NADES dyed polyester at different dye concentrations

Table 3 shows a comparison K/S values of conventionally and NADES dyed sample at different dye concentrations. It is noticeable that at all initial dye concentrations, NADES dyed samples have higher K/S values.

Table 3. Comparison of K/S values for conventionally and NADES dyed samples at different dye concentrations

Dye concentration, %	Conventionally	NADES
1	0.89	4.93
2	0.98	5.51
3	1.58	5.70
4	1.89	7.66
5	2.12	9.62

Table 4 shows CIELab parameters for undyed, conventionally and NADES dyed polyester fabric sample at different dye concentrations and light sources. Parameter L^* has the highest value at light source A10, i.e. 91.45 for an undyed sample compared to all dyed samples. This means that the smaller L^* is given the darker coloring of sample. The lowest value of L^* at the light source F02 10, i.e. 33.83 is noticeable in the NADES dyed sample at a dye concentration of 5%, which gives the darkest coloring of the polyester fabric. At the same dye concentration, the conventionally dyed sample at the light source F02 10 has a value of 58.88. The highest values of L^* in conventionally dyed samples are noticeable at a concentration of 1%, i.e. 67.51 and light source A 10. In NADES dyed samples, the highest values of L^* were observed at the same concentration and the same light source A 10 = 46.66.

Also, there are differences in the values of a^* and b^* , as well as differences in hue (H^*) and chroma (C^*). Coordinates a^* and b^* have positive values for undyed, as well as for all dyed samples. The highest value of a^* is noticeable in the NADES sample which is dyed with 5% of disperse dye (light source D65 10), i.e. 43.45, while the smallest a^* value for dyed samples is visible in the conventional dyed sample with 2% dye, i.e. 12.94 at light source F02 10. A positive a^* value indicates a red shade

of color, i.e. the coloring is more red and less green.

The positive and highest value of b^* is noticeable in the undyed sample 2.31 (light source F02 10). This indicates that the coloring is more yellow than blue. For all dyed samples, the b^* value is negative, which means that the color is more blue and less yellow. The lowest b^* value is noticeable in the conventionally dyed sample at a dye concentration of 1% (light source A 10) -14.87, while the highest value is visible in the sample dyed in NADES at a dye concentration of 5% -39.40 (light source F02 10).

Hue H^* of the undyed sample at light source F0 10 is 76.63, while all dyed samples have similar numerical values for all three light sources. Chroma C^* is 2.62 in the undyed sample, while the highest values are observed in the dyed samples in NADES, which can be related to the influence of the natural deep eutectic solvent on the color shade of the polyester fabric. The lowest value of C^* is noticeable in the sample with 1% disperse dye, i.e. 25.18 (illuminant D65 10), while the highest value was observed in the sample dyed with 5% of the dye in NADES 51.43 (light source D65 10).

Table 4. CIELab system parameters for undyed, conventionally and NADES dyed polyester fabric

Light source	Undyed sample				
	L^*	a^*	b^*	C^*	H^*
D65 10	91.23	0.73	2.02	2.14	70.11
A 10	91.45	1.42	2.20	2.62	57.22
F02 10	91.32	0.55	2.31	2.38	76.63
Conventionally dyed sample, 1%					
D65 10	66.47	20.14	-18.26	25.18	317.81
A 10	67.51	19.09	-14.87	22.20	322.07
F02 10	65.05	14.39	-21.95	24.25	303.25
NADES dyed sample, 1%					
D65 10	44.39	35.19	-27.23	44.49	322.26
A 10	46.66	32.19	-20.42	38.12	327.61
F02 10	42.07	25.08	-33.76	42.06	306.61
Conventionally dyed sample, 2%					
D65 10	65.46	18.24	-19.11	27.01	316.83
A 10	67.38	17.45	-15.09	24.43	321.08
F02 10	65.08	12.94	-22.67	26.38	302.05
NADES dyed sample, 2%					
D65 10	43.42	43.89	-28.61	52.39	326.91
A 10	46.83	41.00	-21.43	45.37	334.64
F02 10	40.49	31.54	-36.44	48.19	310.88
Conventionally dyed sample, 3%					
D65 10	63.98	21.41	-20.22	29.45	316.64
A 10	65.01	20.16	-16.72	26.19	320.33
F02 10	62.35	15.33	-24.40	28.81	302.13
NADES dyed sample, 3%					
D65 10	43.17	35.53	-29.76	45.09	322.00
A 10	45.44	32.39	-21.96	38.58	327.00
F02 10	40.79	25.45	-37.34	42.75	306.54
Conventionally dyed sample, 4%					
D65 10	63.92	19.49	-21.51	30.88	316.48
A 10	64.85	18.44	-17.33	26.98	320.27
F02 10	62.42	13.94	-25.33	29.33	301.98
NADES dyed sample, 4%					
D65 10	39.85	41.19	-30.00	55.08	327.00
A 10	43.51	42.82	-22.22	47.36	334.72
F02 10	36.61	33.26	-38.26	50.97	310.73
Conventionally dyed sample, 5%					
D65 10	60.57	24.61	-21.95	32.98	318.28
A 10	61.96	23.15	-17.78	29.19	322.47
F02 10	58.88	17.64	-26.59	31.91	303.56
NADES dyed sample, 5%					
D65 10	36.78	43.45	-31.51	51.43	327.66
A 10	40.31	40.39	-23.15	44.28	335.80
F02 10	33.83	31.14	-39.40	47.14	311.33

Table 5 represents the rating of wash and light fastness for all dyed polyester fabrics. Better light fastness results are noticeable with the samples dyed in NADES, with an almost maximum rating of 7. In addition, NADES-

assisted dyed sample have high wash fastness ratings and have good fastness, which means little color transfer on polyester and cotton fabric. Table 6 shows the photos of dyed samples and their fastness to washing and light.

Table 5. Fastness properties of conventionally and NADES dyed polyester fabrics

Method	Wash fastness at 40 °C			Light fastness
	Color change	Color transfer on white polyester fabric	Color transfer on white cotton fabric	
Con.	4	4	4	6-7
NADES	4-5	4	4-5	7

Table 6. Visual comparison of dyed samples and their fastness properties

Wash fastness				Light fastness
Conventionally dyed		NADES dyed		
Color transfer on white polyester fabric	Color transfer on white cotton fabric	Color transfer on white polyester fabric	Color transfer on white cotton fabric	
				

Conclusion

Betaine-based NADES, consisting of equimolar amount of betaine hydrochloride, urea, and glycerol, was employed as a “green” medium for polyester fabric’s dyeing. The application of NADES in polyester dyeing proved to be a superior method compared to the conventional method. At the maximum dye concentration, the highest K/S and CIELab parameters’ values for NADES-method dyed samples were observed, together with slightly better wash and light fastness scores. Moreover, the NADES-incorporating dyeing method failed to affect both tensile strength and elongation at break of the colored fabrics. The proposed dyeing process proved to be successful, eliminating the need for incorporating problematic dyeing auxiliaries such as carriers and dispersants, as well as reducing the generated wastewater quantity, thus ensuring a double benefit, *i.e.* from ecological and economic aspects.

Acknowledgments

This work was supported by the Republic of Serbia - Ministry of Science, Technological Development and Innovation (Programs for financing scientific research: 451-03-65/2024-03/200133 and 451-03-66/2024-03/200133 assigned to the University of Niš, Faculty of Technology, Leskovac).

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Izvod

**BOJENJE POLIESTARSKJE TKANINE POMOĆU PRIRODNOG
EUTEKTIČKOG RASTVARAČA**Milena M. Nikodijević[✉], Dragan Z. Troter[✉], Sandra S. Konstantinović[✉]

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UDK: 667.494.674:677.027

DOI: 10.5937/savteh2402045N

S obzirom na veliku potrošnju vode tokom bojenja različitih tekstilnih materijala, postoji stalna ekspanzija koja je usmerena na dizajniranje održivijih metoda bojenja. Disperzne boje koje se koriste za bojenje poliestera ne sadrže grupe koje su rastvorljive u vodi, tako da se za njihovo rastvaranje često koriste toksična pomoćna sredstva za bojenje (kerijeri i dispergatori). U ovom radu, prirodni eutektički rastvarač (NADES) na bazi betaina je korišćen na dvostruki način: da bi se obezbedilo smanjenje generisanih otpadnih voda i eliminisala potreba za pomoćnim sredstvima koja nije pogodna za životnu sredinu kao što su kerijeri i dispergatori. Eksperimenti bojenja su sprovedeni sa različitim koncentracijama boje u rasponu od 1-5%. Konvencionalna metoda bojenja je izvedena na 100 °C tokom 45 min uz dodatak kerijera, dispergatora i sirćetne kiseline. Suprotno tome, metoda zasnovana na NADES-u podrazumevala je bojenje u mešavini NADES-a i destilovane vode pri razmeri 70:30. pH vrednost od 4 korišćena je za obe metode. Efikasnost bojenja je analizirana prema jačini boje, postojanosti na pranje i svetlost. Metoda zasnovana na NADES-u je pokazala bolje ukupne performanse, bez uticaja na prekidnu silu i izduženje pri prekidu. Na osnovu dobijenih rezultata, NADES na bazi betaina može se koristiti kao "zeleni" medijum za bojenje poliestera.

Ključne reči: poliestar, bojenje, prirodni eutektički rastvarač, disperzna boja

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