**COTTON DYEING WITH REACTIVE DYES IN SEA WATER**

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ABSTRACT

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| **Introduction:** Using sea water as a textile dyeing medium is an innovative and environmentally friendly approach that addresses the challenges associated with conventional fresh water dyeing methods.  **Matrials and Methods:** Dyes with one functional group were used in the work: Remazol red B and Ostazin blue H-BR. The reflectance of the samples was measured at wavelengths 400-700 nm and the CIELab color coordinates were determined. Based on the reflection value at the wavelength of maximum adsorption for each sample the color intensity (K/S) was calculated according to the Kubelka-Munk equation.  **Results and Discussion:** Color lightness L\* is the vertical coordinate of the three-dimensional CIELab system and shows the similarity of chromatic color to achromatic colors. The higher the value of L\*, the more similar a chromatic color is to white - achromatic color. For both reactive dyes used, the highest lightness is achieved by samples dyed with real sea water without NaCl, and the difference in lightness is greater on samples dyed in a darker shade. The color intensities of the samples dyed with 1% Remazol red B in sea water have minimally changed in relation to the intensity of the reference sample dyed in distilled water. Fabric dyed in simulated and real sea water with 50% less NaCl concentration has a minimally higher color intensity compared to the sample dyed in distilled water. Dyeing with 3% reactive dye Ostazin blue H-BR in sea water results in reduced color intensity, which is more pronounced than dyeing with 1% dye. In the case of a system with real sea water the reduction in intensity is about 12%.  **Conclusion:** Based on the results obtained by reflection spectrophotometry, it can be concluded that it is possible to dye cotton fabric with selected monofunctional reactive dyes in light shades with half the amount of NaCl compared to standard dyeing. |

*Keywords: cotton, dyeing, reactive dyes, sea water, salt, color intensity*

1. INTRODUCTION

The textile industry is a sector that is completely dependent on water in the entire production chain, i.e. there is no textile production without the use of water. The technology of dyeing and final chemical finishing of textiles has created a huge pollution problem because it is one of the most chemically intensive industries in the world and the number 1 polluter of clean water (after agriculture) (Kant, 2012). Dyeing of cotton is particularly water intensive, as about 125 liters of water can be consumed per kilogram of cotton fiber for dyeing and finishing (Uddin et al. 2023). This water is returned to nature as toxic waste, which contains residual dyes and dangerous chemicals. It is estimated that over 10000 different dyes and pigments are used in the textile dyeing industry and over 1,6x106 tons of synthetic dyes are produced world wide annually (Burkinshaw & Salihu, 2019). US$1 billion worth of dye is lost to waste water annually due to inefficient dyeing and finishing processes (Miah, 2022). In waste water textile dyeing have been identified 72 toxic substances, 30 of which cannot be removed. Salts account for nearly 50% of the pollution and contribute most to high values ​​of total dissolved solids (TDS), biochemical oxygen demand (BOD) and chemical oxygen demand (COD) (Lara et al. 2022). Today, 2,3 billion people live on the planet with a lack of drinking water, of which 733 million live in areas that do not have drinking water from the so-called ″improved″ sources (sources protected from contamination) (Ozturk, 2022). It is estimated that around 4000 people die world wide every day from diseases caused by consuming polluted water (Orsag et al. 2020). For these reasons, it is necessary in all sectors to undertake comprehensive measures for the protection of fresh water resources as part of sustainable development, which especially applies to the textile industry. Given the fact that the textile dyeing process is recognized as one of the environmentally unfriendly industrial processes, it is of utmost importance to understand the critical points of the dyeing process in order to find an alternative, environmentally acceptable method. Recently, sea water has been studied as an alternative source of water in textile dyeing in order to save a large amount of fresh water. Sea water is a solution composed of one phase consisting of two components, pure water and a set of dissolved salts, the amount of which is expressed by salinity. The main ions present in seawater are chloride (Cl-), sodium (Na+), sulfate (SO42-), magnesium (Mg2+), calcium (Ca2+) and potassium (K+). On average, seawater has a salinity of about 3,5% (35 g/dm3) (Rajaa et al. 2020, [Devandand](https://www.researchgate.net/scientific-contributions/G-Devanand-2156281727?_sg%5B0%5D=hA0EIbaULrL1htZAyzlglkWzS7Eh_GXGyJj8LovBDf-RhegcU-c6WFcyqPl2HT5wv4J1IdE.B9TIFYiojK8j3a2cWqEQM4CZP5dpWaO0Rc9bshnWBKq_H9DMu4kujbHMOXLlPO4Gxze4N0tQtii_NGC5OGPnsw&_sg%5B1%5D=L4DQY205BNUDnFrzD4j1-1yUHWEnvmSI--aGW9zoLTkIu43zWFqBjaTpWDr83BZAYU_VaSk.lDLb3H_6oSL4PoTm3SrfUp2ri9yAwO594zwzJST3xaJD5prrJyKTlES0o9nb1twSThNIp3IEGRoYwfRBEB0G3A&_tp=eyJjb250ZXh0Ijp7ImZpcnN0UGFnZSI6InB1YmxpY2F0aW9uIiwicGFnZSI6InB1YmxpY2F0aW9uIiwicG9zaXRpb24iOiJwYWdlSGVhZGVyIn19) & [Parthiban](https://www.researchgate.net/scientific-contributions/M-Parthiban-2156292974?_sg%5B0%5D=hA0EIbaULrL1htZAyzlglkWzS7Eh_GXGyJj8LovBDf-RhegcU-c6WFcyqPl2HT5wv4J1IdE.B9TIFYiojK8j3a2cWqEQM4CZP5dpWaO0Rc9bshnWBKq_H9DMu4kujbHMOXLlPO4Gxze4N0tQtii_NGC5OGPnsw&_sg%5B1%5D=L4DQY205BNUDnFrzD4j1-1yUHWEnvmSI--aGW9zoLTkIu43zWFqBjaTpWDr83BZAYU_VaSk.lDLb3H_6oSL4PoTm3SrfUp2ri9yAwO594zwzJST3xaJD5prrJyKTlES0o9nb1twSThNIp3IEGRoYwfRBEB0G3A&_tp=eyJjb250ZXh0Ijp7ImZpcnN0UGFnZSI6InB1YmxpY2F0aW9uIiwicGFnZSI6InB1YmxpY2F0aW9uIiwicG9zaXRpb24iOiJwYWdlSGVhZGVyIn19) 2019). The natural presence of salt in sea water could significantly reduce the need for additional chemical salts, thereby reducing the negative environmental impact and costs associated with the dyeing process. This particularly applies to the reactive dye/cotton dyeing system, which is characterized by the presence of large amounts of inorganic salts in the dye bath. Reactive dyes today account for about 60% of the world consumption of dyes for cellulosic fibers and the absorption of these dyes in the presence of inorganic salts (NaCl or Na2SO4) is attributed to reduced solubility and aggregation of the dyes via coplanar association (Burkinshaw & Salihu, 2019). The mentioned phenomena affect that the color distribution between the fiber phase and the solution phase moves to the side of the fiber. Zerin et al. dyed cotton fabric with reactive dyes in concentrations of 1,2% and 2,2% in ground water and sea water (Zerin et al. 2017). Samples dyed in sea water has a lower color intensity of compared to samples dyed in ground water by about 10%. Fastness to washing and friction had identical values. In a similar experiment, the group of authors examined the possible application of sea water from the bay of Bengal for dyeing cotton with reactive scarlet dye, in a concentration of 2% and 3%, on the mass of the material. Samples dyed with ground water had 33% and 21% higher color intensity, respectively, due to the greater tendency of cotton to swell in ground water (Solaiman et al. 2019). Ferreira et al. recently used sea water as an alternative for dyeing cotton with reactive dyes (Souza Ferreira et al. 2021). They used colors of different functional groups and concentrations of 0,1% to 5% by weight of the material. In the bath with sea water, salt was added only in the three highest concentrations. Samples dyed in a light shade (0,1%) had a higher intensity when dyed in sea water. In other concentrations, a higher color intensity was achieved in distilled water, which led the authors to the conclusion that it is necessary to improve the process of using sea water for better results. Recently, sea water has been used for dyeing cotton and polyester fabrics (Karim et al. 2021). The use of sea water in the dyeing of these fabrics contributed to a greater intensity of color on cotton by 15% and polyester by 3%. The aim of this work is to study the possibility of using sea water as a process medium for dyeing cotton with monofunctional reactive dyes. The assessment of the usability of sea water as a suitable dyeing medium was carried out based on the comparative quality of cotton dyeing in distilled water, simulated sea water and real sea water.

2. material and methods

In the experiment, fabric samples of 100% cotton, surface weight 220 g/m2, were dyed. The fabric is industrially prepared for dyeing. The samples for dyeing had a mass of 3 g. The volume of the bath was 160 cm3. Chemicals used in the work: Remazol red B, reactive dye with vinyl sulfone group - VS (DyStar - Germany, C.I. Reactive Red 22, C.I. 14824), chromogen structure: monoazo; MR 590,51, figur 1a), Ostazin blue H-BR, reactive dye with monochloro triazine reactive group - MCT (Chemapol - Czech Republic, chromogen structure: anthraquinone, C.I. Reactive Blue 5, C.I 61205:l, MR 840,11, figure 1b), NaCl - salt, agent for increasing exhaustion dye, Na2CO3 - salt, agent for regulating the pH of the solution. Dyeing was performed by Ahiba apparatus (TYP G7B) in glass cuvettes with vertical movement of the material. The dye concentration was 1 and 3% by weight of the material, NaCl concentration 0-80 g/dm3 and Na2CO3 concentration 10 and 20 g/dm3. The formulations of the dyeing solution and the designations of the samples are shown in Table 1. The pH of the bath was 10,5 - 11. The dyeing was performed in distilled, simulated sea water and real sea water. Dyeing was performed using the all-in method. The initial dyeing temperature was 30oC and within 30 minutes a final temperature of 60oC was reached for the VS color and 90oC for the MCT color, where it dyed for 70 minutes. After dyeing and multiple rinsing, treatment with a soapy solution at 95oC for 15 minutes was performed. After the final washing, the samples were dried at room temperature. Table 1 contains the designations of the samples depending on the formulation of the dye bath.

**Table 1. Sample designations and dye bath composition**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Designations of samples** | **Water** | **Conc. dye %** | **NaCl, g/dm3** | **Na2CO3 g/dm3** |
| **R1; B1** | **DW** | **1** | **50** | **10** |
| R2; B2 | SSW | 1 | 25 | 10 |
| **R3; B3** | **DW** | **3** | **80** | **20** |
| R4; B4 | SSW | 3 | 50 | 20 |
| R5; B5 | RSW | 1 | 0 | 10 |
| R6; B6 | RSW | 1 | 25 | 10 |
| R7; B7 | RSW | 3 | 0 | 20 |
| R8; B8 | RSW | 3 | 50 | 20 |

**R-** Remazol red B; **B-** Ostazin blue H-BR, DW- distilled water; SSW – simulated sea water; RSW – real sea water (Aegean Sea)

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**Fig. 1. Structural formulas Remazol red B (a) and Ostazin blue H-BR (b)**

The composition of the simulated sea water is shown in Table 2 (Broadbent et al. 2018).

**Table 2. Composition of simulated sea water**

|  |  |  |
| --- | --- | --- |
| **Salt** | **Concentration g/dm3** | **Molarity M** |
| NaCl | 24,72 | 0,425 |
| KCl | 0,67 | 0,009 |
| CaCl2 | 1,03 | 0,0093 |
| MgSO4⋅7H2O | 6,29 | 0,0255 |
| MgCl2⋅6H2O | 4,66 | 0,023 |
| NaHCO3 | 0,18 | 0,002 |

Using a Spectraflash SF600X reflectance spectrophotometer (Datacolor - USA), the reflectance of the samples was measured at wavelengths of 400-700 nm and the CIELab color coordinates were determined. Based on the reflectance value at the wavelength of maximum adsorption for each sample, the color intensity (K/S) was calculated according to the Kubelka-Munk equation:

 ...............(1)

the wavelength of maximum absorption for Remazol red B is 520 nm and Ostazin blue H-BR is 610 nm.

Based on the color intensity before and after the soap treatment, the percentage of fixed dye, F(%), was determined using the following equation:

 .............. (2)

where the subscript *T* refers to the fabric treated in a soap solution, and the subscript *0* to the untreated fabric, immediately after dyeing.

Color uniformity *σ(λ)* was calculated by measuring the K/S values ​​of 20 random spots at the wavelength of maximum absorption λ and using equations 3 and 4 (Ristić et al.):

 .............. (3)

 ............................... (4)

Where is *σ(λ)* the color uniformity of each random spot *K/S* value from (*K/S)λ*, λ is the wavelength of dye maximum absorption, *n* is the number of tested spots, and *(K/S)i,λ* is the *K/S* value of each random spot. Color uniformity expressed by *σ(λ)* improves as the value of *σ(λ)* decreases.

On the dyed samples, color fastness to washing at 60oC was determined using the ISO 105-C06 standard and to friction using the ISO-105-X12 standard.

3. results and discussion

The reflection spectrophotometer of the newer generation was used to measure the reflectance values ​​of samples dyed with 1 and 3% dye, in the range of wavelengths 400-700 nm. Reflection diagrams show the ratio of reflected and absorbed light energy and the area of ​​maximum absorption. Samples dyed with 1% Remazol red B (figure 2a) have higher reflection energy compared to samples dyed with 3% dye (figure 2b). It is noticeable that the differences in reflection between the samples dyed with 1% dye are smaller, i.e. the reflection energy of the samples is very similar throughout the visible light spectrum. Based on this, it can be assumed that the fluctuations in the shade of color are minimal on the samples dyed in sea water compared to the reference (standard) sample dyed in distilled water. Samples dyed with 3% Remazol red B have greater differences in reflection energy, and it is noted that the sample dyed with distilled water (P3) has the lowest reflection value. A higher reflectance value of a sample indicates a lighter tone. All samples have a minimum reflectance value at a wavelength of 520 nm. Similar tendencies are observed for the reflection diagrams of samples dyeing with Ostazin blue H-BR (Figure 3). The registered differences in reflectance between samples dyed with 1% dye are smaller compared to the differences in reflectance of samples dyed with 3%. In samples dyeing with 3% Ostazin blue H-BR, the reflection lines of each sample are clearly demarcated and noticeable. In the entire spectrum of visible light, the sample marked B7 has the highest reflectance value, i.e. sample dyed in real sea water without NaCl addition. All samples have a minimum reflectance value at the wavelength of 610 nm.

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**Fig. 2. Reflections of cotton fabric samples dyed with Remazol red B, a-1%, b- 3%**

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**Fig. 3. Reflections of cotton fabric samples dyed with Ostazin blue H-BR, a-1%, b- 3%**

Color lightness L\* is the vertical coordinate of the three-dimensional CIELab system and shows the similarity of chromatic color to achromatic colors. The higher the value of L\*, the more similar a chromatic color is to white - achromatic color. Compared to the sample dyed in distilled water with a standard recipe - sample R1 (c=1%), the samples dyed with Remazol red B in sea water - samples R2, R5 and R6, have higher L\* coordinate values ​​by 1-4% (Figure 4a). On samples dyed 3% Remazol red B, samples dyed in sea water have a lightness higher by 8-17% (Figure 4b). Samples dyed in a light shade with 1% Ostazin blue H-BR in sea water have a higher lightness value by 2-7%, and samples dyed with 3% have a higher brightness value by 6-18% compared to the corresponding samples dyed in distilled water (Figure 5). For both reactive dyes used, the highest brightness is achieved by samples dyed with real sea water without NaCl, and the difference in brightness is greater on samples dyed in a darker shade.

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**Fig. 4. Lightness of color L of samples stained with Remazol red B a-1%, b- 3%**

|  |  |
| --- | --- |
| **(a)** | **(b)** |

**Fig. 5. Lightness of color L of samples stained with Ostazin blue H-BR a-1%, b- 3%**

The color intensities of the samples (K/S) determined at the wavelength of maximum absorption are shown in table 3. In the same table are the percentages of fixed color (F) for each dyeing system. The color intensities of the samples dyed with 1% Remazol red B in sea water (R2, R5 and R6) have minimally changed in relation to the intensity of the reference sample dyed in distilled water (R1). Fabric dyed in simulated and real sea water with 50% less NaCl concentration has a minimally higher color intensity compared to the sample dyed in distilled water. The addition of NaCl in the bath manifests it self as greater dye exhaustion, i.e. as a greater color intensity of fabric. Based on the color intensity of the samples, it can be concluded that the formulation of the simulated sea water was well chosen. Samples dyed with 3% Remazol red B have greater changes in color intensity due to changes in technological and chemical dyeing parameters, since all samples had a lower color intensity of 20% to 50% compared to the color intensity of the standard sample. Sample R7 dyed in real sea water only with Na2CO3 to regulate the alkaline environment and without the addition of NaCl has a dramatically lower intensity compared to the standard sample. Samples dyed in the presence of a reduced amount of NaCl (🠗38%), in simulated and real sea water, also had a lower intensity in an identical amount (🠗24%). From the obtained results, it can be concluded that the system with 3% Remazol red B dye is more sensitive to the reduced amount of NaCl compared to the system with 1% dye. This is in accordance with the general theory of the influence of salt on the equilibrium exaustion of anionic dyes, as well as the fact that significantly higher sea water hardness and the presence of other chemicals have a more pronounced negative impact in a bath with a higher concentration of reactive dye.

On samples of cotton fabric dyed with Ostazin blue H-BR, the color intensity of the depends on the formulation of the dye bath. Samples dyed with 1% dye in sea water with 25 g/dm3 NaCl (samples B2, B6) had minimal fluctuations compared to the sample dyed with 50 g/dm3 in distilled water (sample B1, standard). The sample dyed without the addition of NaCl (B5) has an almost 30% lower color intensity compared to the standard. Dyeing with 3% reactive dye Ostazin blue H-BR in sea water results in reduced color intensity, which is more pronounced than dyeing with 1% dye. In the case of a system with real sea water (sample B8), the reduction in intensity is about 12%. When it is taken into account that the concentration of NaCl was reduced from 80 to 50 g/dm3 (🠗38%), and that the dyeing was performed at a high bath ratio, then the drop in intensity is not dramatic. In the dyeing bath without added NaCl (sample B7), a dramatic drop in the utilization of the selected reactive dye was registered, as the intensity of the dye was reduced by 52%.

Percentage of fixed dye, F, shows what proportion of the total amount of exhausted dye is fixed to the cotton cellulose by forming a covalent bond. The values percentage of fixed dye are shown in table 3. From the results shown, it can be considered that the change of the process medium in the studied dyeing systems had no effect on the covalent fixation of the dye, i.e. the reaction of the vinyl sulfonic or monochloro triazine group with the ionized hydroxyl group of cellulose.

**Table 3. Color intensity and degree of dye fixation**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Sample** | **K/S** | **F (%)** | **Sample** | **K/S** | **F (%)** |
| R1 | 1,03 | 82 | B1 | 0,76 | 78 |
| R2 | 1,04 | 81 | B2 | 0,74 | 79 |
| R3 | 3,00 | 78 | B3 | 0,54 | 70 |
| R4 | 2,28 | 85 | B4 | 0,78 | 68 |
| R5 | 0,85 | 80 | B5 | 2,50 | 78 |
| R6 | 1,07 | 78 | B6 | 1,98 | 77 |
| R7 | 1,47 | 83 | B7 | 1,20 | 74 |
| R8 | 2,29 | 77 | B8 | 2,16 | 69 |

Color uniformity *σ(λ)* and color fastness are shown in table 4. Analyzing the results for uniformity of coloring in table 4, it is noted that the changes in values ​​for uniformity of coloring are minimal, i.e. that all samples have a high uniformity. It can be concluded that the use of simulated or real sea water did not negatively affect the uniformity of dyeing of reactive dyes on cotton fabric. Fastness to washing and friction in terms of changing the shade of colored samples have high marks. The high fastness performance reflects the nature of the strong covalent bond between the dyes and the cotton cellulose.

**Table 4. Color uniformity and color fastness**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample** | ***σ(λ)*** | **Washing** | **Rubing**  **dry** | | **Rubing**  **wet** | |
| R1 | 0,130 | 4-5 | 4-5 | 4-5 | |
| R2 | 0,129 | 4-5 | 4-5 | 4-5 | |
| R3 | 0,128 | 4-5 | 4-5 | 4 | |
| R4 | 0,127 | 4-5 | 4 | 4 | |
| R5 | 0,128 | 4-5 | 4-5 | 4-5 | |
| R6 | 0,130 | 4-5 | 4-5 | 4 | |
| R7 | 0,129 | 4-5 | 4 | 4 | |
| R8 | 0,130 | 4-5 | 4-5 | 4 | |
| B1 | 0,186 | 4-5 | 4-5 | 4-5 | |
| B2 | 0,188 | 4-5 | 4-5 | 4 | |
| B3 | 0,190 | 4-5 | 4-5 | 4-5 | |
| B4 | 0,192 | 4-5 | 4-5 | 4 | |
| B5 | 0,187 | 4-5 | 4-5 | 4-5 | |
| B6 | 0,190 | 4-5 | 4 | 4 | |
| B7 | 0,190 | 4-5 | 4-5 | 4-5 | |
| B8 | 0,188 | 4-5 | 4-5 | 4-5 | |

4. Conclusion

The main characteristic of dyeing cotton with reactive dyes using the exhaustion method is the high consumption of water and chemicals, i.e. the large production of polluted water. The problem of missing fresh water encourages the study of different environmentally safe dyeing techniques. Recently, sea water has been studied as a suitable medium for dyeing, as it contains significant amounts of inorganic salts.

in this work, reactive dyes for cellulose fibers of different functional groups were used. dyeing results in distilled water were compared with dyeing results in simulated and real seawater. sea water from the aegean sea was used without filtering or other pretreatment. based on the results obtained by reflection spectrophotometry, it can be concluded that it is possible to dye cotton fabric with selected monofunctional reactive dyes in light shades with half the amount of nacl compared to standard dyeing. under these conditions, very similar intensities of coloring are achieved as with coloring in distilled water. for efficient dyeing in sea water in dark tones, at a reduced concentration of nacl, further process improvements are necessary. this implies primarily the preparation of water, by a suitable chemical method and filtering, in order to increase the swelling of the fiber and the cotton cellulose/reactive dye interaction. this will be the direction of our future research.

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