Reusability of pre-treatment water obtained via textile wastewater by coagulation and filtration methods in reactive and disperse dyeing

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I. INTRODUCTION

Brundtland, known as the World Commission on Environment and Development, defined sustainability as "development that meets the needs of the present without compromising the ability of future generations to meet their own needs" [1, 2]. The new concept of sustainability, referred to as the Triple Bottom Line, is an approach that includes social, environmental, and economic performances [3]. This triple bottom line is related to human welfare (basic needs, personal development, balanced society), environmental welfare (healthy environment, climate, energy, and natural resources) and economic welfare (future preparedness and economy) [4]. The textile industry is an environmentally damaging process and consumes large amounts of natural resources (water, oil, soil) [5]. The consumption of natural resources all over the world and the increase in waste costs cause the topics of recycling or reuse to come to the forefront [6].

Chemicals used in the textile industry constitute approximately 25% of the chemicals produced in the world [7]. Many of the chemicals used in the textile processes are known to have harmful effects and are discharged into the environment by evaporating or dissolving in water after applications [8]. Textile is recognized as the second most water consuming and water polluting sector [9]. In a traditional dyeing and finishing process, 200 tons of water is polluted for 1 ton of fabric [10].

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ABSTRACT

Besides high-water consumption in the textile industry, color, quantity, and variety of chemicals in discharged wastewater draws attention both in terms of environmental and economic aspects. Therefore, efficient, and reliable treatment techniques are needed. In this context, coagulation is a very important step in the treatment of wastewater. In this study, water recovery from textile wastewater taken from different discharge points was investigated by using coagulation and filtration methods. Iron (III) chloride hexahydrate (FeCl₃·6H₂O) was used as a coagulant. The optimum conditions were determined by studying the effect of different coagulant dosages and pH values. The filtration process was performed using 12-25 µm and <2 µm porous filters. Laboratory-scale reactive and disperse dyeing processes have been carried out to demonstrate the reusability of wastewater. At the end of the study, pollution removal efficiencies were determined as 91.1% for suspended solids, 64.7% for color and 72.1% for chemical oxygen demand. The findings of this study showed that coagulation and filtration will be useful processes for the enterprise in the pre-treatment of textile wastewater. It has been determined that the obtained pre-treatment waters can be used in disperse and reactive dyeing. Despite the pollution load in the pre-treatment water, it has been determined that dyeing errors do not occur, especially in reactive dyeing. This study revealed the effect of the pollution load in the pre-treatment water obtained by recovery methods from the wastewater discharged from textile dyehouse processes on the dyeing processes. In this study, it was aimed to emphasize that in order to reduce the water footprint, wastewater discharged from chemical processes can be classified and recycled and that pre-treatment water can be reused despite the pollution it contains.
The environmental effects of textile industries are related to high water consumption as well as the variety, quantity and color of chemicals in wastewater. Wastewater from finishing and dyeing processes in the textile industry is usually high in terms of both color, and organic content [11]. Polluters from textile processes vary greatly and depend on the chemicals used [12]. The textile industries use significant amounts of water and chemicals for the wet processing of textiles. These chemicals are widely used in bleaching, desizing, scouring, dyeing, printing, and finishing [13]. Wastewater from the textile industry is known to have a large amount of suspended solids (SS), a high fluctuating pH, a high temperature and a strong colorfulness as well as a high chemical oxygen demand (COD) [11]. High levels of COD and substances containing color make the treatment of wastewater very difficult [14]. Industrial polluters change frequently the physicochemical characteristics like acidity, temperature, salinity, or turbidity of water bodies at discharge points, causing ecosystem changes [12].

Dyes used in textile industries have a toxic character, are not easily degrade, quite stable and are not removed from wastewater by conventional methods. The toxic wastes usually amass at tropic level causing a harmful biological impact and a higher ratio of waterborne illness because of the long-time persistence and non-degradable nature of wastewater [12]. For color removal from wastewater, there are various treatment methods such as coagulation/flocculation, precipitation, oxidation, adsorption, biological process, ionizing/gamma radiation [11, 12]. However, particularly when applied to for treating large waste streams, many of these methods are cost-prohibitive [11].

The disposal of textile industrial wastes may cause huge costs [12]. However, the interest in the recovery of processed water in the textile industry is increasing. The treated wastewater must meet the quality standards before reuse in textile processes and efficient and reliable treatment techniques must be available. Wastewater recycling can be economically possible because of reduced discharge fees and reduced water intake costs [15]. The coagulation-flocculation process is a very important step in wastewater treatment and is widely used because of the cost-effectiveness and simplicity of the process. The activity of the coagulation-flocculation process potently influences the whole treatment performance. Therefore, the productivity increase of the coagulation step appears to be a key aspect of the development of the whole treatment performance [16].

Coagulation is one of the most important physicochemical processes used in wastewater treatment and aims to overcome the forces that stabilize the suspended particles. The coagulation process targets the colloidal particles with a diameter of $10^{-7}$ to $10^{-14}$ cm in wastewater. Coagulation occurs in successive stages that allow the particles to collide and the agglomeration to grow. This leads to the destabilization of the particles and charge neutralization. In most wastewater treatment processes, the separation of colloidal particles from the aqueous phase containing both dissolved and suspended particles is an important step. The coagulation processes are used in particular to remove suspended solids from the wastewater. These particles differ significantly in terms of shape, particle size, composition charge, density, and source. The understanding of the interaction between these factors is important for the correct management of coagulation processes and the choice of coagulants [17]. However, the main disadvantage of coagulation/flocculation process is the sludge produced in very large quantities in textile wastewater treatment [18]. Disposal of the sludge formed after precipitation is a great challenge. During the sludge disposal process, pollutants may leach into water bodies and soil [19]. Today, sludge management is one of the main problems that need to be solved in terms of technical and ecological applications. Sludge disposal process is one of the main cost elements in treatment plants. Disposal of sewage
sludge can generally be done by dewatering the sludge, reducing its volume, and transporting it to storage units [20].

In the coagulation–flocculation process, the whole treatment can be divided into two successive processes. The first one is the coagulation process which occurs destabilization of a solution or colloidal suspension. The aim of coagulation is to overcome the factors supported by a stable system. It is usually achieved by the use of iron or aluminum salts as a coagulant agent [16]. Especially the use of FeCl₃ as coagulant has an important place in the removal of dyes, metal ions, phosphorus, as well as oil and grease from wastewater [18]. The second one is the flocculation process which states the precipitation of destabilized particles. In this context, they come together to form large agglomerates separated more easily by gravity [16]. The process efficiency depends on the wastewater characteristics, pH, temperature, type and dosage of the coagulant, intensity, and duration of mixing [18].

The interest in the recovery of process water in the textile industry is increasing day by day. Besides, treated wastewater must meet quality standards before being reused again [15]. The coagulation method is seen as a very important step in wastewater treatment. It is also widely used because of its practicality and cost-effectiveness in applications [16]. There are many effective methods used in the treatment of textile wastewater such as ultrafiltration, nanofiltration, reverse osmosis, electrochemical processes, electrocoagulation, ozonation, adsorption, photocatalysis and advanced oxidation processes. Operate or periodic maintenance of these methods are difficult [18, 21]. When different treatment techniques are compared, the coagulation process has been reported to be simpler and cheaper, as well as requiring less supervision [19].

The coagulation/flocculation method is a technique that has been frequently used for many years as the main treatment or pre-treatment stage in textile wastewater treatment [22-25]. This method has the great ability to remove organic, inorganic, and colloidal substances [26]. It has an excellent ability to change the color of textile waste and reduce the total suspended solids load [18]. Textile industries that use large quantities of water aim to reduce the water cost by reusing treated wastewater [15]. In this study, it was aimed to determine the character of the real textile wastewater, which contains a significant type of pollution and is discharged from the existing processes in the enterprise depending on the dyehouse processes, whose chemical content changes every year in the textile industry, and to emphasize the importance of the effectiveness of the coagulation and filtration methods frequently used in pre-treatment. The overall aim of this study is to remove pollution in textile wastewater by using the coagulation and filtration methods and find out the reusability of pre-treated water in disperse and reactive dyeing processes in the same industry.

II. EXPERIMENTAL METHOD

2.1 Materials

In this study, wastewater samples were taken from two discharge points of Kinteks Weaving and Dyeing Industry Inc. in Türkiye. The wastewater from the washing process (1st discharge point) combines with the wastewater from the other processes (dyeing and textile finishing processes), and then these are discharged at the last discharge point (2nd discharge point). 2nd discharge point contains all process wastewater as pre-treatment, dyeing, and washing. All coagulation experiments were performed using a beaker, and magnetic stirrer at room
Temperature. FeCl₃·6H₂O was used as a coagulant agent. FeCl₃ is an acidic, dark brown chemical, used as a coagulant in drinking water, and wastewater treatment systems. It is preferred over other coagulant materials because of its more affordable price. HCl was used for adjusting pH in the coagulation process. FeCl₃·6H₂O and HCl were purchased from Merck. The filtration process was performed by grade 589/1 (pore size: 12-25 μm) and 589/3 (pore size: < 2 μm) filter papers.

In the reactive dyeing process, commercial dyes coded EVER.RED ED 4B, EVER.BLUE L-ED and EVER.RED ED-S were used for burgundy color dyeing. In the reactive dyeing process, commercial dyes coded EVER.YELL LX, EVER.RED LX and EVER.BLUE CLX were used for pink color dyeing. In the reactive dyeing process, commercial dyes coded EVER.RED LX, EVER.BLUE CLX and EVER.BLUE ED-G were used for blue color dyeing. In the disperse dyeing process, commercial dyes with codes SET.YELL BR 2RFL, SET.RED P2G5 and SET.N.BLUE PBL were used for burgundy color dyeing. In the disperse dyeing process, commercial dyes with codes SET.YELL BR 2RFL, SET.RED P2G5 and SET.N.BLUE PBL were used for pink color dyeing. In the disperse dyeing process, commercial dyes with codes SET.YELL CE5G, SET.RED CEBR and SET.BLUE CE3R were used for beige color dyeing.

In the reactive dyeing process, 100% cotton fabric was used (105 g/m² Weft: Ne 30/1 OE, Warp: Ne 30/1 OE). In the disperse dyeing process, 100% polyester fabric was used (112 g/m², Weft: Ne 30/1 OE, Warp: Ne 30/1 OE). Dyeing processes were performed by a MATHIS branded laboratory scale device. The fastness measurements of the fabrics were performed with Minolta CM-3600D spectrophotometer. Reactive and disperse dyeings were carried out in the laboratory of Kinteks company, based on the company’s dyeing recipes and processes. All chemicals and recipes used in the dyeings were supplied from the laboratory of Kinteks company.

2.2 Method

Wastewater samples were taken in accordance with TS ISO 5667-10 standard and collected in sampling bottles. In the first step, characterization of the wastewater samples was made according to standard methods. The analyzed parameters (suspended solids (SS), chemical oxygen demand (COD) and color) were demonstrated in Figure 1. The SS, COD and color measurements were performed according to SM 2540 D:2005 / gravimetric method, SM 5220 B:2005 / open reflux method and SM 2120 C:2005 / spectrophotometric method, respectively. In the second step, coagulation and filtration methods were applied to wastewater, and then pre-treatment waters were obtained. The beakers containing wastewater were placed on the magnetic stirrer and then the coagulant (FeCl₃·6H₂O) was added. The pH of the wastewater was adjusted using 0.1 M HCl. The wastewater samples were stirred at 400 rpm for 2 minutes and then stirred at 100 rpm for 20 minutes. After the coagulation process, precipitation of pollution agglomerated was expected for 40 minutes. In the optimization study, the wastewater was first filtered from 12-25 μm and then <2 μm pore size filter papers after the precipitation.

In the optimization of the coagulation process, to determine the optimum coagulant dosage and the optimum pH value of the 1st discharge point were studied in the range of 520-600 mg/l, and 6.5-8.0, taking samples at different times, respectively. The pH value of the wastewater was adjusted to 7.0 value. And then 520, 560, and 600 mg/l coagulant were used to determine the optimum coagulant amount. The pH values of wastewater were adjusted to 6.5, 7.0, 7.5, and 8.0 to determine the optimum pH value using optimum coagulant dosage. For
determined the optimum coagulant dosage and the optimum pH value of the 2nd discharge point were studied in the range of 640-720 mg/l, and 6.0-9.0, respectively. The pH value of the wastewater was adjusted to 7.0 value. And then 640, 680, and 720 mg/l coagulant were used to determine the optimum coagulant amount. The pH values of wastewater were adjusted to 6.0, 7.0, 8.0, and 9.0 to determine the optimum pH value using optimum coagulant dosage. Optimum coagulation was performed using the optimum coagulant dosage and pH value for both discharge points. Finally, the wastewater was filtered to obtain the pre-treatment water.

![Figure 1. Characteristics of wastewater from different discharge points (Washing wastewater: 1st discharge point, Wastewater from the last discharge point: 2nd discharge point)](image)

In the last step, the reusability of pre-treatment water was investigated in laboratory scale reactive and disperse dyeing process. In the reactive dyeing recipe, cotton fabric weight was taken as 5 g. The dyeing recipes were adjusted in the liquor ratio of 1:10. Reactive dyeing studies were performed by adjusting the pH value in the range of 10.50-11.00. The reactive dyeing process was carried out at a constant temperature of 60 ºC for 60 minutes. The dyeing diagram is shown in Figure 2. After the dyeing process, neutralization and washing processes were carried out and the dyeing process was terminated. In the neutralization process, the fabrics were treated with 1000 ml of acid solution adjusted to pH 5.5-6.0 with acetic acid in a glass beaker for 2 minutes and the fabrics were rinsed with water. In the washing process, the fabrics were treated with 1 g/l washing soap solution in a glass beaker at 95 ºC for 5 minutes. And then the fabrics were dried at 110 ºC in the drying-oven.

![Figure 2. Reactive dyeing process](image)
In the disperse dyeing recipe, polyester fabric weight was taken as 5 g and the recipes were adjusted in the liquor ratio of 1:10. Disperse dyeing studies were performed by adjusting the pH value in the range of 4.50-5.00. The disperse dyeing process was performed at a constant temperature of 130 °C for 45 minutes and terminated by washing and rinsing of fabrics. The dyeing diagram is shown in Figure 3. After the dyeing process, washing process was carried out and the dyeing process was terminated. In the washing process, the fabrics were treated with 4 g/l washing soap solution in a glass beaker at 95 °C for 5 minutes. And then the fabrics were dried at 110 °C in the drying-oven.

![Figure 3. Disperse dyeing process](image)

Gray scale staining degree of reactive and disperse dyed fabrics were measured using a Minolta CM-3600D bench-top spectrophotometer. The color measurements of the fabrics were performed by the same device. The fastness tests of fabrics were carried out according to ISO 105 standard.

### III. RESULTS AND DISCUSSION

#### 3.1. Effect of Coagulant Dose

The effect of the coagulant addition on the pollution removal efficiency was determined by studying 3 different concentrations for the 1st discharge point and the 2nd discharge point. The optimum coagulant dose was determined as 560 mg/l and 680 mg/l for 1st and 2nd discharge points, respectively. When the turbidity of the wastewater was examined, the pollution removal efficiency was observed to decrease in less and higher dosages than the optimum dosages. Optimum coagulant dose added wastewater was filtered from grade 589/1 (pore size: 12-25 µm) and 589/3 (pore size: < 2 µm) filter papers, respectively and then iron analyzes were performed.

#### 3.2 Effect of pH Value on The Removal Efficiency

The effect of pH value on pollution removal efficiency was determined by studying 4 different pH values for the 1st discharge point and the 2nd discharge point. The optimum pH values were determined as 7.0. When the turbidity of the wastewater in the images was examined, the pollution removal efficiency was observed to decrease in lower and higher values than the optimum pH values.
3.3 Optimum Coagulation and Filtration

The optimum coagulation studies were performed in the presence of optimum values. The optimum values of discharge points are shown in Table 1. Many studies were carried out for wastewater samples taken from different discharge points at different times. The optimum values determined for the 2nd discharge point were determined as limit values for a treatment plant to be established. The optimum pH value and coagulant dosage were determined in laboratory-scale studies by observing the formation of clusters and then the color and turbidity of the pre-treatment water.

<table>
<thead>
<tr>
<th>Optimum Parameters</th>
<th>1st discharge point</th>
<th>2nd discharge point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coagulant dosage</td>
<td>560 mg/l</td>
<td>680 mg/l</td>
</tr>
<tr>
<td>pH</td>
<td>7.0</td>
<td>7.0</td>
</tr>
</tbody>
</table>

The wastewater was filtered through filter papers. It was observed that the clarity of the wastewater increased after filtration. The experimental images of the coagulation and filtration processes are shown in Figure 4.

In order to determine the efficiency of the pre-treatment methods, firstly, measurements were made on the samples taken from the last discharge point (2nd discharge point) with the highest pollution load. Then, the measurement results of the pre-treatment water obtained were compared with the measurement results of both process water and wastewater. The removal efficiencies of 2nd discharge point and the characteristics of the process water are shown in Table 2. Pollution removal efficiencies of pre-treatment water for 2nd discharge point were determined as 91.1% for SS, 64.7% for color, 72.1% for COD.

In the experimental studies, it was observed that the pH values decreased with the addition of coagulants. This decrease is related to the acid character of Fe^{3+} metal. H^+ ions are released in the hydrolysis reaction by the
addition of iron salt to water [27]. The pH value of the pre-treatment water after optimum coagulation and filtration was decreased from 10.42 to 4.54.

| Table 2. The removal efficiencies of 2nd discharge point and process water characteristics |
|---------------------------------|-----------------|-----------------|-----------------|
| SS (mg/l) | Color (Pt-Co) | COD (mg/l) | pH |
| 2nd discharge point | 113 | 721 | 786 | 10.42 |
| Pretreatment water | 10.10 | 254.63 | 219.12 | 4.54 |
| Removal efficiency (%) | 91.1 | 64.7 | 72.1 |
| Process water | < 5 | < 18.5 | 9.08 | 7.36 |

3.4 Effect of Pre-treatment Water Usage on Reactive Dyeing

In this study, whether pre-treatment water used in reactive dyeing recipes has a suitable character was examined. The reactive dyeing recipes of burgundy, pink, and blue colors were demonstrated in Table 3, Table 4, and Table 5, respectively. After the dyeing process, the sample fabrics were washed and dried. Images of the reactive dyed fabrics are demonstrated in Figure 5. The measurement results of cotton fabrics dyed with burgundy, pink, and blue colors were demonstrated in Table 6, Table 7, and Table 8, respectively. Dye recipes containing process water were taken as standard. Rubbing staining, multifiber staining (washing fastness), and color measurement values of the cotton fabrics were found as same or very close to the values of dyed fabrics as standard. In this context, it was determined that the washing pre-treatment water has a characteristic that can be used in reactive dyeing processes.

| Table 3. Reactive dyeing recipe for burgundy color |
|---------------------------------|-----------------|-----------------|
| Color : Burgundy | | |
| **Chemicals** | **Concentration** |
| **Dyes** | | |
| EVER.RED ED 4B | 0.2788% |
| EVER.BLUE L-ED | 0.2618% |
| EVER.RED ED-S | 2.5245% |
| Salt | 80 g/l |
| Soda Ash | 20 g/l |

| Table 4. Reactive dyeing recipe for pink color |
|---------------------------------|-----------------|-----------------|
| Color : Pink | | |
| **Chemicals** | **Concentration** |
| **Dyes** | | |
| EVER.YELL LX | 0.0025% |
| EVER.RED LX | 0.0077% |
| EVER.BLUE CLX | 0.00113% |
| Salt | 80 g/l |
| Soda Ash | 20 g/l |

| Table 5. Reactive dyeing recipe for blue color |
|---------------------------------|-----------------|-----------------|
| Color : Blue | | |
| **Chemicals** | **Concentration** |
| **Dyes** | | |
| EVER.RED LX | 0.0844% |
| EVER.BLUE CLX | 0.1881% |
| EVER.BLUE ED-G | 0.6116% |
| Salt | 80 g/l |
| Soda Ash | 20 g/l |
**Figure 5.** Dyeing images of cotton fabrics (1: Process water, 2: Pre-treatment water)

<table>
<thead>
<tr>
<th>Measurement parameters</th>
<th>Burgundy</th>
<th>Process water (Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing Fastness Test</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Wet</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Dry</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Color Measurement</td>
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<td></td>
</tr>
<tr>
<td>Light source: D65-10°</td>
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<tr>
<td>CIELab values</td>
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<td>0.144</td>
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<td>-0.210</td>
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</tr>
<tr>
<td>$\Delta E^* \leq 1.00$</td>
<td>0.394</td>
<td>$\leq 1.00$</td>
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<tr>
<td>Total Color Deviation</td>
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<tr>
<td>Washing Fastness Test</td>
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<td></td>
</tr>
<tr>
<td>Pre-treatment water</td>
<td>5</td>
<td>4-5</td>
</tr>
<tr>
<td>Process water</td>
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<table>
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<tr>
<th>Measurement parameters</th>
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<th>Process water (Standard)</th>
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<td></td>
</tr>
<tr>
<td>Wet</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Dry</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Color Measurement</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Light source: D65-10°</td>
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<tr>
<td>CIELab values</td>
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<td>$\Delta E^* \leq 1.00$</td>
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<td>$\leq 1.00$</td>
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<td>Total Color Deviation</td>
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<tr>
<td>Washing Fastness Test</td>
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<tr>
<td>Pre-treatment water</td>
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</tr>
<tr>
<td>Process water</td>
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Table 8. Fabric measurement results of blue colored reactive dyeing

<table>
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<tr>
<th>Measurement parameters</th>
<th>Blue Pre-treatment water</th>
<th>Blue Process water (Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing Fastness Test</td>
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</tr>
<tr>
<td>Wet</td>
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<td>4</td>
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<tr>
<td>Dry</td>
<td>4-5</td>
<td>4-5</td>
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<tr>
<td>Color Measurement</td>
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<td></td>
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<tr>
<td>Light source: D65-10º</td>
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</tr>
<tr>
<td>CIELab values</td>
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<td>a*</td>
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<tr>
<td></td>
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<td>-0.036</td>
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<tr>
<td>Total Color Deviation</td>
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<tr>
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<tr>
<td>Acetate</td>
<td>5</td>
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</tr>
</tbody>
</table>

3.5 Effect of Pre-treatment Waters on Disperse Dyeing

In this study, whether pre-treatment water used in disperse dyeing recipes has a suitable character was examined. The disperse dyeing recipes of burgundy, pink, and beige colors were demonstrated in Table 9, Table 10, and Table 11, respectively. After the dyeing process, the sample fabrics were washed and dried. Images of the disperse dyed fabrics are shown in Figure 6. The measurement results of polyester fabrics dyed with burgundy, pink, and beige colors were shown in Table 12, Table 13, and Table 14, respectively. Dye recipes containing process water were taken as standard. According to the results, rubbing staining (rubbing fastness), multifiber staining (washing fastness), and color measurement values of the fabrics dyed in different colors were determined as the same or very close to the standard dyeing fabric values. In this study, it was determined that the pre-treatment water has an effective characteristic in all disperse dyeing processes containing different colors.

Table 9. Disperse dyeing recipe for burgundy color

<table>
<thead>
<tr>
<th>Color : Burgundy</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemicals</td>
<td></td>
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<tr>
<td>Dyes</td>
<td></td>
</tr>
<tr>
<td>SET.YELL BR 2RFL</td>
<td>0.838%</td>
</tr>
<tr>
<td>SET.RED P2G5</td>
<td>1.9%</td>
</tr>
<tr>
<td>SET.N.BLUE PBL</td>
<td>0.288%</td>
</tr>
<tr>
<td>Dispersing agent</td>
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</tr>
<tr>
<td>Acetic acid</td>
<td>0.43 g/l</td>
</tr>
</tbody>
</table>

Table 10. Disperse dyeing recipe for pink color

<table>
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<th>Color : Pink</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemicals</td>
<td></td>
</tr>
<tr>
<td>Dyes</td>
<td></td>
</tr>
<tr>
<td>SET.YELL BR 2RFL</td>
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</tr>
<tr>
<td>SET.RED P2G5</td>
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<td>SET.N.BLUE PBL</td>
<td>0.00876%</td>
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<tr>
<td>Dispersing agent</td>
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<tr>
<td>Acetic acid</td>
<td>0.43 g/l</td>
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Table 11. Disperse dyeing recipe for beige color

<table>
<thead>
<tr>
<th>Chemicals</th>
<th>Color : Beige</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dyes</td>
<td>SET.YELL CE5G</td>
<td>0.0184%</td>
</tr>
<tr>
<td></td>
<td>SET.RED CEBR</td>
<td>0.0084%</td>
</tr>
<tr>
<td></td>
<td>SET.BLUE CE3R</td>
<td>0.00474%</td>
</tr>
<tr>
<td>Dispersing agent</td>
<td>1 g/l</td>
<td></td>
</tr>
<tr>
<td>Acetic acid</td>
<td>0.43 g/l</td>
<td></td>
</tr>
</tbody>
</table>

Figure 6. Dyeing images of polyester fabrics (1: Process water, 2: Pre-treatment water)

Table 12. Fabric measurement results of burgundy colored disperse dyeing

<table>
<thead>
<tr>
<th>Measurement parameters</th>
<th>Burgundy</th>
<th>Process water (Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing Fastness Test</td>
<td>Wet</td>
<td>4-5</td>
</tr>
<tr>
<td></td>
<td>Dry</td>
<td>4-5</td>
</tr>
<tr>
<td>Color Measurement</td>
<td>L*</td>
<td>a*</td>
</tr>
<tr>
<td>Light source:</td>
<td>CIELab values</td>
<td></td>
</tr>
<tr>
<td>D65-10º</td>
<td>-0.763</td>
<td>0.102</td>
</tr>
<tr>
<td>Acceptable limit:</td>
<td>ΔE*≤1.00</td>
<td></td>
</tr>
<tr>
<td>Total Color Deviation</td>
<td></td>
<td>0.797≤1.00</td>
</tr>
<tr>
<td>Washing Fastness Test</td>
<td>Pre-treatment water</td>
<td>3-4</td>
</tr>
<tr>
<td></td>
<td>Process water</td>
<td>3-4</td>
</tr>
<tr>
<td>Wool</td>
<td>Acrylic</td>
<td>Polyester</td>
</tr>
<tr>
<td>3-4</td>
<td>4-5</td>
<td>5</td>
</tr>
<tr>
<td>Acrylic</td>
<td>Polyester</td>
<td>Polyamide</td>
</tr>
<tr>
<td>4-5</td>
<td>4-5</td>
<td>5</td>
</tr>
<tr>
<td>Polyester</td>
<td>Polyamide</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td>4-5</td>
<td></td>
</tr>
<tr>
<td>Polyamide</td>
<td>Cotton</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cotton</td>
<td>Acetate</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 13. Fabric measurement results of pink colored disperse dyeing

<table>
<thead>
<tr>
<th>Measurement parameters</th>
<th>Pink</th>
<th>Process water (Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing Fastness Test</td>
<td>Pre-treatment water</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Process water</td>
<td>4-5</td>
</tr>
<tr>
<td>Color Measurement</td>
<td>L*</td>
<td>a*</td>
</tr>
<tr>
<td>Light source:</td>
<td>CIELab values</td>
<td></td>
</tr>
<tr>
<td>D65-10º</td>
<td>0.010</td>
<td>-0.241</td>
</tr>
<tr>
<td>Acceptable limit:</td>
<td>ΔE*≤1.00</td>
<td></td>
</tr>
<tr>
<td>Total Color Deviation</td>
<td></td>
<td>0.539≤1.00</td>
</tr>
<tr>
<td>Washing Fastness Test</td>
<td>Pre-treatment water</td>
<td>4-5</td>
</tr>
<tr>
<td></td>
<td>Process water</td>
<td>4-5</td>
</tr>
<tr>
<td>Wool</td>
<td>Acrylic</td>
<td>Polyester</td>
</tr>
<tr>
<td>4-5</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Acrylic</td>
<td>Polyester</td>
<td>Polyamide</td>
</tr>
<tr>
<td>4-5</td>
<td>4-5</td>
<td>5</td>
</tr>
<tr>
<td>Polyester</td>
<td>Polyamide</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td>4-5</td>
<td></td>
</tr>
<tr>
<td>Polyamide</td>
<td>Cotton</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cotton</td>
<td>Acetate</td>
<td></td>
</tr>
<tr>
<td>4-5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 14. Fabric measurement results of beige colored disperse dyeing

<table>
<thead>
<tr>
<th>Measurement parameters</th>
<th>Beige</th>
<th>Pre-treatment water</th>
<th>Process water (Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubbing Fastness Test</td>
<td>Wet</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Dry</td>
<td>4-5</td>
<td>5</td>
</tr>
<tr>
<td>Color Measurement</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Light source:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CIELab values</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L*</td>
<td>0.207</td>
<td>0.290</td>
<td>0.232</td>
</tr>
<tr>
<td>a*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>b*</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acceptable limit:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ΔE* ≤1.00</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total Color Deviation</td>
<td></td>
<td>0.425 ≤ 1.00</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Washing Fastness Test</th>
<th>Pre-treatment water</th>
<th>Process water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wool</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Acrylic</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Polyester</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Polyamide</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Cotton</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Acetate</td>
<td>4-5</td>
<td>4-5</td>
</tr>
</tbody>
</table>

3.6 Cost analysis of the treatment plant

In the studies carried out in 2020, it was determined that the coagulation method is a very effective pretreatment in the treatment of textile wastewater. After the studies, pre-treatment waters were obtained by repeated chemical precipitation processes of wastewater taken from two different discharge points at different time periods, considering the process intensities in the enterprise. After the disperse and reactive dyeings made with the pre-treatment water, it has been determined that the wastewater in the enterprise does not cause a disadvantage. After dyeing, color measurements were made at different points on the sample fabrics. According to the color measurement tests, it was determined that a homogeneous dyeing was achieved in both disperse and reactive dyeing processes. According to the rubbing fastness, washing fastness and color measurement tests, it was determined that the waste water of the enterprise could be recovered by designing a treatment plant.

A cost analysis has been made for the treatment plant investment. In the studies carried out, it was determined that a treatment plant suitable for the enterprise conditions should be designed with a capacity of 1500 m³/day. The treatment plant is designed as 3 sections.

In the first section, the enterprise wastewaters will be sent to the balancing pool by passing through a grid and a drum sieve to retention of the solid particles, respectively. The wastewater in the balancing pool will be sent first to the neutralization unit and then to the aeration pool. The water coming out of the aeration pool will be sent to the biological precipitation pool and then to the chemical treatment unit. The chemical treatment system is divided into 3 sections: neutralization unit, coagulation unit and flocculation unit. In the chemical treatment unit, the sedimentation of the pollutants in the wastewater will be ensured. The sludge settled on the bottom of the settling basin and the flocs that can accumulate on the surface will be transferred to the sludge pool by the pump and surface scraper, respectively. The treated water on the surface will be transferred to the filtration pool. After the dewatering process is done in the sludge dewatering unit, the wastewater from the system will be sent to the balancing pool. The water from the settling basin will be sent to the 3rd section, the filtration unit. The water coming out of the sand and carbon filter will be sent to the reverse osmosis unit. The water leaving the reverse osmosis unit is expected to have zero conductivity. At the end of these processes, it is predicted that 750-900 m³/day water can be recovered from the system, which is planned to be operated with 50-60% efficiency.
The conductivity of the process water used in the enterprise is in the range of 1000-1500 µS/cm. In the laboratory studies, it was determined that the conductivity values of the waste waters taken from two different discharge points after treatment were in the range of 2000-3000 µS/cm and did not cause dyeing errors. Therefore, it is predicted that the water coming out of the sand and carbon filter will be given to the clean water tank to obtain a water character with a conductivity value range of 1500-2000 µS/cm and to recover 900-1000 m³/day of water.

Estimated the enterprise costs for the designed wastewater treatment plant;

1. Chemical cost    : 660 TL/day
2. Electricity cost    : 540 TL/day
3. Chemical cost of filtration system : 528 TL/day
4. Personnel cost    : 450 TL/day
5. Maintenance and repair costs  : 100 TL/day
6. Sludge cost    : 300 TL/day

**Total estimated daily cost**  : 2578 TL/day

**m³ cost of water earned**  : 2.5 TL – 3.0 TL/m³

The total cost per m³ of process and waste water has been determined as approximately 3.0 TL and is almost the same as the m³ cost of treatment water.

**IV. CONCLUSION**

Water usage increases day by day due to population-dependent needs. Rapid depletion of water resources and discharge from different areas of usage to nature pose a serious threat to human lives and other living creatures.

It is estimated that 4 trillion liters of wastewater are discharged daily in the world. The water consumption in the textile industry in Turkey is between 20 and 230 m³ for 1 tonne of fabric. In addition to this water consumption in textiles, the textile industry also increases wastewater pollution with the abundance of chemical use (paint, auxiliary chemicals, etc.) [28].

It is clear that water usage and discharge in the textile industry cannot be ignored. Therefore, this study, it was aimed to reduce costs by the reuse of pre-treatment waters obtained from the treatment of textile wastewater.

In this study, the treatment of washing wastewater (1st discharge point) and wastewater from the last discharge point (2nd discharge point) was carried out by coagulation and filtration methods. The important findings obtained in the study and suggestions for future studies are listed below.

- It was determined that the coagulation and filtration method was successful, especially in the pre-treatment of textile wastewater that did not contain dye. It was observed that the color removal
efficiency of the coagulation and filtration method was not sufficient in the pre-treatment of textile wastewater containing reactive and disperse dyes.

- Removal efficiencies of various studies based on coagulation and flocculation processes in the treatment of textile wastewater are given in Table 15. The removal efficiency of FeCl₃·6H₂O used in the treatment of the textile wastewater taken from the 2nd discharge point was compared with other studies in Table 15. According to the results in Table 15, it is seen that the character of textile wastewater, operating conditions and coagulant amounts significantly affect the removal efficiency.

### Table 15. The removal efficiency for FeCl₃ and FeCl₃·6H₂O used in this and other studies

<table>
<thead>
<tr>
<th>Coagulant agent</th>
<th>Coagulant dosage</th>
<th>Rapid mixing conditions</th>
<th>Slow mixing conditions</th>
<th>SS removal</th>
<th>Color removal</th>
<th>COD removal</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>FeCl₃ [21]</td>
<td>1000 mg/l</td>
<td>150 rpm 1 min.</td>
<td>30 rpm 20 min.</td>
<td>68.6%</td>
<td>84.7%</td>
<td>62.2%</td>
<td>9</td>
</tr>
<tr>
<td>FeCl₃ [28]</td>
<td>4000 mg/l</td>
<td>200 rpm 2 min.</td>
<td>40 rpm 15 min.</td>
<td>- -</td>
<td>54%</td>
<td>-</td>
<td>4</td>
</tr>
<tr>
<td>FeCl₃·6H₂O [20]</td>
<td>750 mg/l</td>
<td>150 rpm 1 min.</td>
<td>40 rpm 20 min.</td>
<td>96.44%</td>
<td>91.53%</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>FeCl₃·6H₂O (textile wastewater from 2nd discharge point)</td>
<td>680 mg/l</td>
<td>2 min 100 rpm</td>
<td>91.1% 20 min.</td>
<td>64.7%</td>
<td>72.1%</td>
<td>-</td>
<td>7</td>
</tr>
</tbody>
</table>

In the literature [18,29,30], the superiority of using FeCl₃ as a coagulant agent in textile wastewater treatment in terms of pollution removal efficiency is mentioned. Naghan et al. [30] examined the COD, SS and color removal efficiency from textile wastewater by using Al₂(SO₄)₃ and FeCl₃ as coagulant agents. The removal rate of COD, SS and color using Al₂(SO₄)₃ was obtained 36, 19 and 68.8%, respectively. The removal rate of COD, SS and color using FeCl₃ was obtained 72, 60 and 98% respectively. Studies have shown that COD, SS and color removal efficiency increases significantly when FeCl₃ is used instead of Al₂(SO₄)₃ as a coagulant. Therefore, FeCl₃·6H₂O was used as a coagulant in this study. However, the coagulant used reduces the pH value of the pre-treatment water, and when used in excess, it increases the pollution load of the pre-treatment water and causes color pollution. In addition, excessive use of coagulant causes staining errors, but no staining errors were observed in the study.

- According to the results, it was observed that the pollution that was minimized after the wastewater treatment did not make an attempt to disperse dyeing processes where different colors were used. These results reveal acceptable pollution character, and a reusable pre-treatment water in the dyeings.

- Textile wastewater is treated through many treatment steps. Each treatment step in treatment plants increases the cost. Therefore, in this study, the possibility of obtaining a low-cost pre-treatment water that does not cause errors in fabric dyeing as a result of the application of the coagulation step, which is one of the pre-treatment steps of the treatment process, was investigated. No dyeing errors could be detected, especially in the reactive dyeings made by recycling the wastewater taken from the
2nd discharge point, which has a high pollution load. This finding supports that pretreatment water recycled from pretreatment processes can be reused and costs can be reduced. In addition, it is anticipated that pre-treatment steps should be examined in terms of reducing water footprint and correct use of resources, and the findings will lead to future studies.

- By mixing the obtained pre-treatment water with process water in certain proportions, the character of the water can be improved and the cost of water usage can be reduced.
- Since the ion charge created by the pollution load in the obtained pre-treatment water does not cause any errors in reactive dyeing, the salt cost can be reduced by using pre-treatment water in salt-free reactive dyeing.
- It is not correct to say that the coagulation method is always effective in color removal of textile wastewater since color removal of wastewater from 2nd discharge point could not be fully realized. According to the results, it is foreseen that this method will facilitate the next step.
- The study is laboratory-based, it does not exactly correspond to the real-scale implementation, but it is an important data source and preliminary study for the real-scale application of the study.

ACKNOWLEDGEMENT

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REFERENCES


