



REPUBLIC OF TURKEY
ADANA ALPARSLAN TRKES SCIENCE AND TECHNOLOGY UNIVERSITY

INSTITUTE OF GRADUATE STUDIES
DEPARTMENT OF NANOTECHNOLOGY AND ENGINEERING SCIENCES

REUSE OF WASTEWATER
IN YARN DYEING PROCESSES

MURAT CAN YAPA
MASTER OF SCIENCE

ADANA 2023



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SUPERVISOR
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submitted by **Murat Can YAPÇA** in partial fulfilment of the requirements for the degree of **Master of Science in Department of Nanotechnology and Engineering Sciences, Adana Alparslan Türkeş Science and Technology University** by,



I hereby declare that all information in this thesis has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all information that is not original to this work.

Murat Can YAPÇA

ABSTRACT

REUSE OF WASTEWATER IN YARN DYEING PROCESSES

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Department of Nanotechnology and Engineering Sciences

Supervisor: Assoc. Prof. Dr. Ahmet Refah TORUN

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In the textile dyeing industry where water consumption is high, the aim is to reduce carbon and water footprint by treating wastewater with appropriate recovery methods and using it in new dyeings. The textile dyeing industry is widespread in our country and water consumption is high. Therefore, reusing wastewater for certain types of yarn will significantly reduce water consumption. The dyeing of products in textile dyeing factories can consist of 3-15 baths depending on the dyeing process, and clean water is used in all of these baths. Therefore, wastewater is generated at every stage. These wastewaters are sent to the wastewater pool to not be reused. When the dyeing processes of different yarn types such as viscose, cotton, acrylic, polyester, and nylon are examined, they are dyed with different numbers of baths. In addition, the dye ratios of the given dyeing agents are different from each other. For example, when the water remaining in the bath is examined after dyeing acrylic, clear water is seen, while the situation is the opposite for viscose/cotton. At this stage, wastewater samples generated in each dyeing process can be analyzed with appropriate methods and reused for dyeing certain yarn types. By applying treatment processes according to the content of wastewater, British Textile Technology Group and Goodman and Porter textile wastewater reuse criteria can be met or wastewater that has been treated at a maximum level can be integrated into the dyeing process or post-dyeing washing baths with nearly 100% performance. It is estimated that 20% of the world's clean water resources are used in the textile industry and up to 100 liters of water are required to produce one kilogram of textile product. In this direction, by applying environmentally friendly methods to reduce high water consumption in textile dyeing factories and aiming to reuse wastewater, the daily water consumption in textile dyeing factories is aimed to be minimized. In this thesis, research has been conducted on acrylic (cationic dyeing) and cotton (reactive dyeing), which are relatively easier to dye than other fiber types. For a facility with a daily water consumption of 100 tons,

the aim is to discharge 30% of the water to the recycling pool and to use all of the discharged water again in dyeing processes.

Key Words: Climate Change, Textile Dyeing, Waste Water, Recycling, Carbon Footprint



ÖZET

İPLİK BOYA PROSESLERİNDE ATIK SUYUN YENİDEN KULLANILMASI

Murat Can YAPÇA

Nanoteknoloji ve Mühendislik Bilimleri Anabilim Dalı

Danışman: Doç. Dr. Ahmet Refah TORUN

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Su tüketiminin yüksek olduğu tekstil boya endüstrisinde, atık suyun uygun geri kazanım yöntemleriyle arıtılarak yeni boyamalarda kullanılması ve böylece karbon ayak izi ve su ayak izinin azaltılması hedeflenmektedir. Ülkemizde tekstil boya sanayi çok geniş bir alana yayılmaktadır ve su tüketimi fazladır. Bu nedenle, belirli iplik türleri için atık suyun yeniden kullanılması, su tüketimini önemli ölçüde azaltacaktır. Tekstil boyahanelerinde ürünlerin boyanması, boyama prosesine göre 3-15 banyodan oluşabilmekte ve bu banyoların tamamında temiz su kullanılmaktadır. Dolayısıyla her aşamada atık su oluşmaktadır. Bu atık sular, tekrar kullanılmamak üzere atık su havuzuna gönderilmektedir. Viskon, pamuk, akrilik, polyester ve naylon gibi boya prosesleri incelendiğinde farklı sayıda banyolar ile boyanmaktadır. Ayrıca verilen boyarmaddelerin boya oranları da birbirinden farklıdır. Örneğin; akriliğin boyanması sonucu banyoda kalan su incelendiğinde berrak bir su görülürken viskon/pamuk için durum tam tersidir. Bu aşamada, her boya prosesinde oluşan atık su numuneleri uygun yöntemlerle analiz edilebilir ve belirli iplik türlerinin boyanmasında tekrar kullanılabilir. Atık suyun içeriğine göre arıtma prosesleri uygulanarak British Textile Technology Group ve Goodman and Porter tekstil atık suları geri kullanım kriterlerine getirilmesi ya da maksimum seviyede arıtılabilmiş olan atık suyun boyama veya boya sonrası yıkama banyolarında %100'e yakın performans ile boyama prosesine entegre edilmesi gerçekleştirilebilir. Dünyadaki temiz su kaynağının %20'sinin tekstil endüstrisinde kullanıldığı ve bir kilogram tekstil ürünü üretmek için 100 litreye kadar su gerektiği düşünülmekte olup, bu doğrultuda tekstil boyahanelerindeki yüksek su tüketiminin azaltılması için çevre dostu yöntemlerin uygulanmasıyla atık suların tekrar kullanımı hedeflenerek, tekstil boyahanesinde kullanılan günlük su tüketiminin minimuma indirilmesi amaçlanmaktadır. Bu tez yazısında, diğer elyaf türlerine oranla nispeten daha kolay boyanabilen akrilik (katyonik boyama) ve pamuk (reaktif boyama) üzerinde

arařtırmalar yapılmıřtır. Günde 100 ton su tüketime olan bir tesis için suyun %30'unun geri dönüşüm havuzuna deřarj edilmesi ve deřarj edilen suyun tamamının tekrar boyamalarda kullanılması hedeflenmektedir.

Anahtar Kelimeler: İklim Deęiřiklięi, Tekstil Boyama, Atık Su, Geri Kazanım, Karbon Ayakizi



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1. INTRODUCTION

Our country has extensive water resources, controlled use of resources is necessary for sustainable transfer of water to future generations. According to information from the United Nations, clean water sources are rapidly depleting due to reduced precipitation caused by global warming, excessive evaporation, rapid consumption, and pollution. Therefore, it is now necessary to implement more effective measures for more efficient use of water worldwide. Unfortunately, many dyeing factories in our country are not ready for this change. Therefore, the recycling of wastewater and its reuse in processes in dyeing factories with higher water consumption has become necessary for environmentally friendly production and is of great importance. In addition, a global climate crisis is currently being experienced and its negative effects are increasing every day. Countries are publishing action plans to reduce the effects of the global climate crisis. The European Union implemented the Green Deal in 2019 as one of the most important steps to achieve the 2050 carbon neutral targets to reduce the global climate crisis. Following this, as of July 16, 2021, the Presidency of the Republic of Turkey published the Green Deal Action Plan, stating the necessity of carrying out our competitiveness with developed countries in an environmentally friendly framework.

The depletion of clean water sources in many regions of the world and in our country is particularly adversely affecting the textile industry. Textile companies consume approximately 20% of the world's clean water sources. In light of this data, the most efficient action plan for the use of our clean water sources must be implemented.

The unfixed dye rates discharged during the dyeing process and after washing in the acrylic and cotton dyeing process will be examined in this thesis. After separating the discharge water according to the analysis results, the amount of chemicals remaining in the wastewater will be analyzed, and the wastewater that can be treated will be examined to be reused for dyeing. All experiments conducted during the research were applied in yarn dyeing format.

Acrylic is a type of fiber dyed with cationic dyes. The process begins with a chemical called retarder in an acidic environment. Retarder is a colorless chemical that acts as a fake dye. When the pH is adjusted to appropriate conditions, the retarder added to the environment adheres to

the yarn surface. Thus, when dye enters the bath, the dye does not stick to the yarn and errors such as abrasion and color difference do not occur. After a certain temperature, the retarder begins to break down, and dye molecules begin to adhere to the areas where the retarder breaks down. As the temperature increases in a controlled manner, dye molecules continue to adhere to the yarn surface and the dyeing process occurs. When cationic dyeings are examined, bright colors are obtained. In addition, in these dyeings, the dye is attracted to the negatively charged yarn surface, which increases the color fastness. During the dyeing process, excess dye is not absorbed by the yarn and is discharged with the wastewater. In addition, some chemicals used during the dyeing process may also be discharged with wastewater. Therefore, the discharged wastewater should be analyzed and treated, and the wastewater that can be reused for dyeing should be separated.

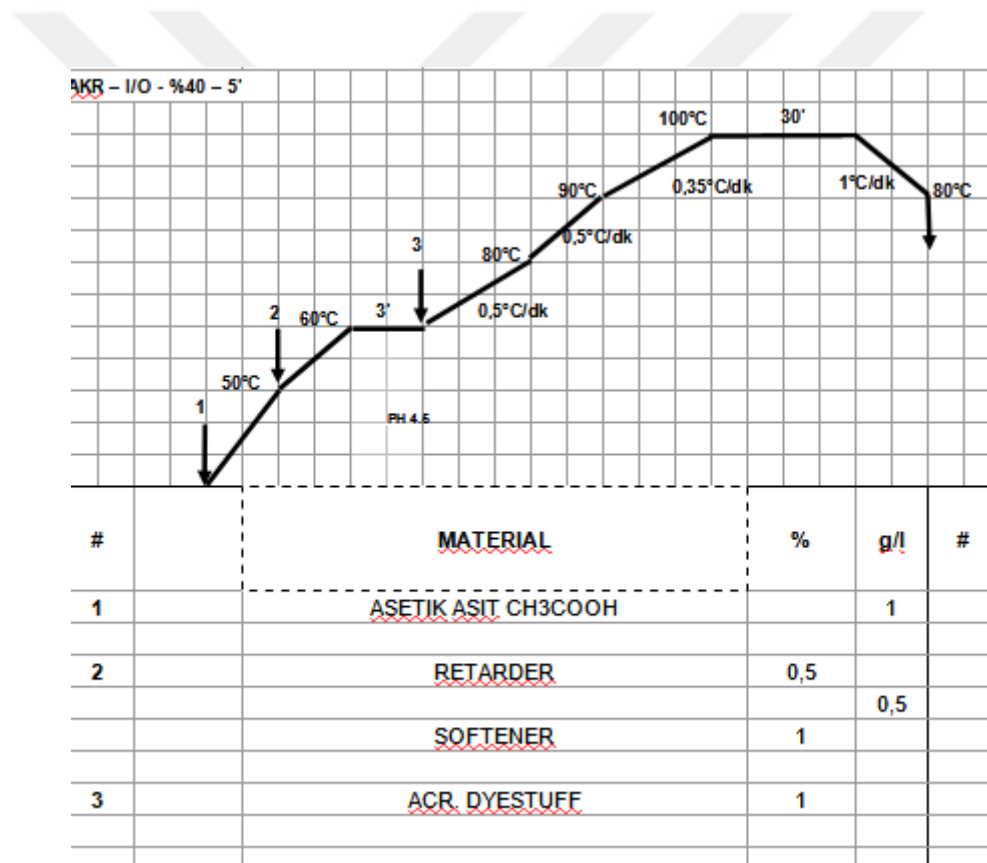


Figure 1. Acrylic Dyeing Processes

Viscose and cotton are fiber types that are dyed with reactive dyes through entirely chemical reactions, unlike acrylic. In the process that begins in a neutral environment, the yarn is prepared for dyeing with various chemicals, and after the dye is added to the bath, the pH of the environment is controlled to ensure the dye binds to the fiber. Approximately 30% of the dye added to the environment remains in the bath as unfixed dye, which is why the number of

washing baths is higher in the reactive dyeing process. The simplest form of the standard cotton yarn dyeing process is as follows:

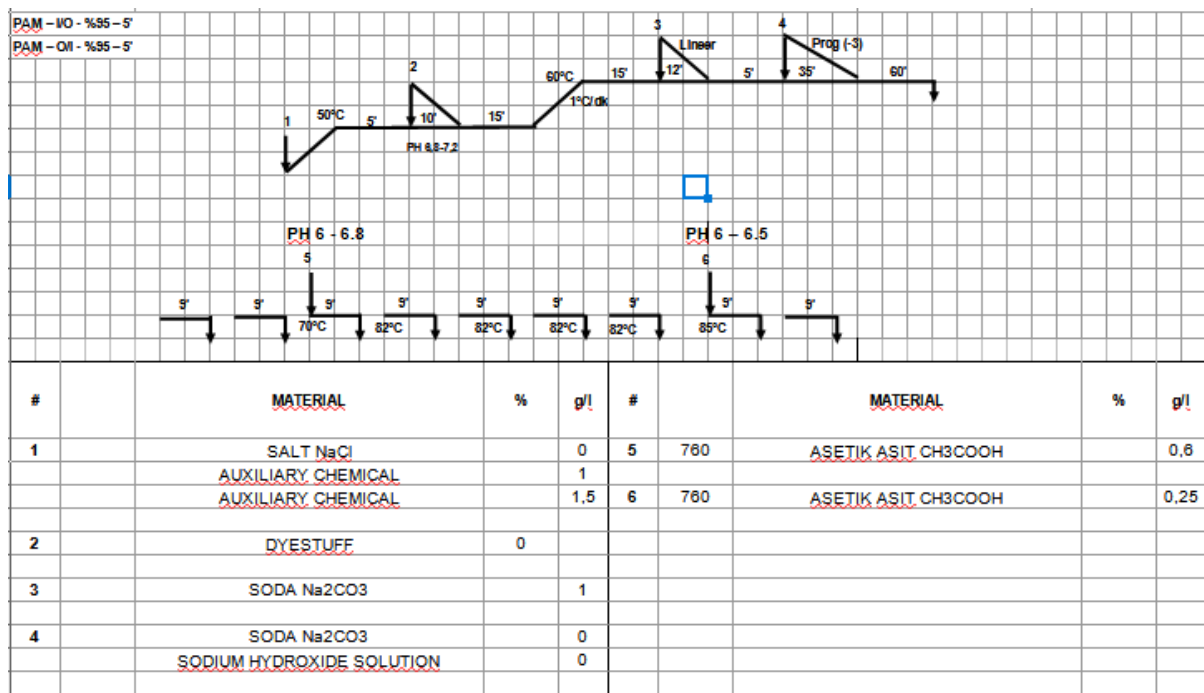


Figure 2. Cotton Dyeing Processes

1.1. Previous Works

The introduction highlights the global issue of freshwater scarcity and the increasing reliance on desalination as a solution. Membrane distillation (MD) is identified as a promising desalination method due to its low energy requirements and operational costs. Vacuum membrane distillation (VMD), a type of MD, is introduced as a process where a feed solution is brought into contact with a microporous membrane, and vacuum is applied on the opposite side to create a driving force for mass transfer. The study focuses on investigating the performance of VMD for desalination. The effect of feed flow rate, feed temperature, and salt concentration on permeation flux is examined. It is found that higher feed flow rates enhance mixing and decrease temperature polarization, resulting in increased permeation flux. Similarly, higher feed temperatures lead to greater desalination production rates. The desalting degree, representing the percentage of salt rejected by the membrane, is shown to be consistently high, reaching 99.99% throughout the experiments. Fouling potential and membrane durability are also assessed. It is observed that fouling of the membrane is negligible during the six-month experimental period, and any minor reduction in permeate flux is easily

removed through water washing. The study suggests further investigation into long-term fouling effects. The energy requirement of VMD is discussed, emphasizing its low operational costs and potential use of renewable energy sources. In conclusion, the study demonstrates the potential of VMD for desalination purposes. It shows that VMD can achieve high desalting degrees, produce almost distilled water, and maintain stable flux rates. The findings indicate that VMD could be a viable option for desalinating high-salt concentration solutions. (Bhausahab et al. 2010)

The introduction highlights the issue of freshwater scarcity and the need for industries, such as textile dyeing, to recycle and reuse wastewater. Reverse osmosis (RO) is identified as an effective technology for treating wastewater. The study focuses on using RO-treated water for dyeing cotton fabric and compares the performance and colorfastness of fabric dyed with RO clean water and ground water. The laboratory dyeing procedure involves immersing cotton fabric samples in dyeing pots containing color solutions and either ground water or RO clean water. The samples are dyed, rinsed, neutralized, soaped, and washed. Shade variation, color fastness to wash, and color fastness to rubbing are evaluated using appropriate methods. Spectrophotometric evaluation is also conducted. The results show that the shade differences between ground water-dyed and RO clean water-dyed fabric samples are insignificant visually. The color fastness to wash is rated between 4 and 5 for both types of water, indicating no significant differences in color staining on various fiber types. The study concludes that reusing RO-treated water for dyeing purposes is feasible and yields satisfactory results in terms of shade variation and dyeing performance. In conclusion, the study demonstrates the potential of reusing RO-treated water for textile dyeing, providing a way to minimize waste generation and reduce the consumption of natural resources. The findings suggest further experiments with larger quantities of fabric to validate the results on a larger scale (Foisal, 2016).

Industrial textile processing involves pretreatment, dyeing, printing, and finishing, which consume a lot of energy, water, and produce chemical pollution. Cotton is the most commonly dyed textile fiber, and the most commonly used reactive dyes produce heavily colored effluents with high salt and BOD/COD values. A four-step process was proposed to recover water and mineral salts while leaving spent dyes in the reject stream. The process involved cartridge filtration, acidification, nanofiltration, and reverse osmosis. Experiments were conducted on the process variables to recover 99% of sodium chloride, and recycled brines were used in new dyeing operations with no difference in the results, validating the process's innovative feature of recycling water and mineral salts (Moulin, 2006).

This work discusses the environmental issues associated with the textile industry and how wastewater generated by this industry can be treated using physico-chemical methods, such as chemical coagulation and electrocoagulation. The study evaluates the efficiency of a combined process of chemical coagulation and electrocoagulation on synthetically produced dyes C.I. Reactive Yellow 145 and C.I. Reactive Red 194 to reduce water consumption. The results show that this process is very efficient in decolorizing dye wastewater, with a reduction of up to 99.1% and 96.15%. The study concludes that the reuse of treated wash-off wastewater will improve the overall water efficiency of the textile industry (Ali, 2019).

The textile and ready-made garments (RMG) industry is vital to the economy of Bangladesh, contributing to employment and national exports. However, the industrial growth of the sector has posed environmental challenges, particularly related to water consumption and wastewater generation. Textile industries consume a significant amount of water, with approximately 110 liters needed to dye 1 kg of cotton fabric. The wastewater generated contains dyes, chemicals, surfactants, and traces of metals, which pose risks to human and aquatic life when discharged into water bodies. To address these challenges, researchers and industrialists have explored wastewater reuse as a cost-effective and environmentally friendly solution. Various treatment methods have been proposed, such as membrane treatment, adsorption, coagulation, filtration, biological and enzymatic techniques, Fenton process, sonochemical process, electrochemical process, ion exchange, and supercritical water oxidation. However, many of these methods involve additional operational and maintenance costs, which may not be easily acceptable to factory owners. The present research paper focuses on the direct reuse of textile wastewater without any treatment process, specifically for the scouring-bleaching of cotton goods. The study analyzed different wastewater streams from textile dyeing processes, including rinsing water, to identify those that could be reused without treatment. Among the collected wastewater samples, the study found that before dyeing rinsing (BDR) water showed the best performance in terms of scouring-bleaching and dyeing. It exhibited satisfactory whiteness, shade matching, and color fastness properties. Other wastewater types, such as after scoured-bleached rinse (ASR) and acid water (AW), also showed potential for reuse but had slight differences compared to BDR. The study concludes that by segregating and identifying suitable wastewater streams, textile industries can reuse wastewater without the need for additional treatment, reducing pollution levels and freshwater consumption. The research provides a cost-effective water recycling method and highlights the importance of waste minimization for sustainable development. The findings suggest that industrialists should investigate the quality of their own

rinsing water and consider its reuse as process water, reducing the reliance on fresh water and minimizing waste generation. Overall, the study emphasizes the need for a balance between economic growth and environmental protection, promoting sustainable development in the textile industry (Shaid et al. 2013).

Membrane technology, specifically nanofiltration and reverse osmosis, has been explored as an attractive option for treating textile effluents and producing reusable permeate. Combining membrane processes with conventional methods such as coagulation-flocculation and biological treatments has been studied to improve effluent quality and reduce fouling. The reuse of textile wastewater, particularly from polyester dyeing, is a significant challenge due to the large amounts of water involved. This study focuses on treating and reusing wastewater from a polyester dyeing textile mill using homogenization-decantation treatment and polyvinylidene difluoride (PVDF) ultrafiltration membranes. The permeate obtained from the treatment process is reused in new polyester dyeing processes, and the fabrics dyed with reused water are evaluated. The homogenization-decantation treatment helps create a more uniform feed for the membrane process, improving its efficiency. The combined treatment achieves substantial removal of COD and color, with 66% and 30% removal rates, respectively. The membrane treatment proves to be scalable at a semi-industrial scale without fouling issues. The results indicate promising potential for permeate reuse in the textile industry, as evidenced by minimal color differences when compared to fabrics dyed with softened tap water (Buscio et al. 2015).

This study focuses on a case study of a pilot-scale wastewater reclamation facility in a textile processing industry in China. The textile industry in China consumes large amounts of water, exacerbating water scarcity problems. The wastewater generated by textile processing is characterized by high levels of color, suspended solids, and chemical oxygen demand. Conventional treatment methods have limitations in removing color from the wastewater. Therefore, the study implemented a three-stage treatment process, including anaerobic-aerobic treatment, biological aerated filter, and membrane technology. The results showed that the system achieved high removal efficiencies for COD, color, and turbidity, meeting the water quality requirements for textile dyeing and finishing processes. The study also assessed the economic viability of the wastewater reclamation system, showing that it could lead to cost savings compared to other water supply sources. Overall, the study demonstrates the potential of wastewater reclamation as a sustainable solution for water scarcity and pollution in the textile industry (Lu et al. 2010).

This study provides valuable insights into the oxidation of tannery wastewater to enhance its biodegradability. However, there are a few areas that could benefit from further discussion or improvement:

Limited scope: The study focuses specifically on the oxidation of biologically pre-treated separated streams of tannery wastewater. It would be beneficial to expand the scope to include a broader range of tannery wastewater samples from different sources or locations. This would provide a more comprehensive understanding of the effectiveness of oxidation treatment across various tannery wastewater compositions. **Lack of mechanistic understanding:** While the study reports the positive effects of oxidation on biodegradability, it lacks detailed insights into the mechanisms underlying the structural changes and improved biodegradability. Further research and analysis are needed to elucidate the specific reactions and transformations occurring during the oxidation process. **Limited assessment of treatment efficiency:** The study primarily focuses on the removal of COD and UV-absorbance as indicators of treatment efficiency. It would be beneficial to assess other important parameters, such as the removal of specific organic compounds or the reduction of inorganic salts, to provide a more comprehensive evaluation of the treatment performance. **Long-term stability and cost-effectiveness:** The study provides limited information on the long-term stability of the oxidation process and its cost-effectiveness compared to other treatment options. Assessing the operational stability and evaluating the economic feasibility, including factors such as ozone generation and energy consumption, would provide a more complete understanding of the practical viability of the proposed oxidation treatment. **Environmental impact assessment:** The study does not address the potential environmental impacts associated with ozone generation and the disposal of oxidation by-products. Considering the potential formation of harmful by-products or the energy requirements of the ozone generation process, a thorough environmental impact assessment would be valuable to ensure the overall sustainability of the treatment approach.

Addressing these points would strengthen the study's findings and provide a more comprehensive analysis of the oxidation treatment of tannery wastewater, including its effectiveness, mechanisms, long-term stability, cost-effectiveness, and environmental implications (Jochimsen and Jekel, 1997).

This work provides valuable information on the challenges associated with reactive dyes in the textile industry and proposes a technique using swept-potential electrochemical detection to identify and quantify dye components in complex textile wastewaters. However, there are a

few points worth considering: **Limited scope:** The study focuses on the detection and quantification of Reactive Blue 19 and its derivatives. While this is useful for understanding the behavior of this particular dye, it would be beneficial to investigate a broader range of reactive dyes to assess the generalizability of the findings. **Lack of discussion on treatment methods:** The study primarily focuses on the identification and quantification of dye components but does not discuss potential treatment methods for removing or degrading these dyes. Including a discussion on effective treatment techniques would provide more practical insights for addressing dye pollution in textile wastewaters. **Stability and variability of dye components:** The study acknowledges that the estimated concentrations of dye components in the textile mill effluent may have changed over time, highlighting the instability and variability of dye components in complex wastewater matrices. Further research on the stability and transformation of reactive dyes in textile wastewaters would enhance our understanding of their behavior and aid in the development of more effective treatment strategies. **Environmental implications:** The study does not address the environmental implications of reactive dyes and their derivatives in wastewater. It would be valuable to discuss the potential ecological and human health impacts associated with the presence of these dyes in the environment, as well as the need for sustainable and environmentally friendly approaches to dye pollution prevention and treatment. **Reproducibility and validation:** The study does not provide information on the reproducibility or validation of the proposed technique. Including data on the precision and accuracy of the swept-potential electrochemical detection method would strengthen the reliability of the results and their applicability to other wastewater samples.

Considering these points would provide a more comprehensive analysis of the challenges posed by reactive dyes in textile wastewater and offer insights into potential solutions for their detection and removal. Additionally, addressing the environmental implications and providing validation data would enhance the practical relevance and reliability of the study (Camp and Sturrock, 1990).

The passage highlights the challenges associated with wastewater generated in the textile wet processing industry and the potential use of membrane treatment systems to address these challenges. Here are some comments and observations: **Comprehensive overview:** The passage provides a comprehensive overview of the issues related to wastewater generation in the textile industry, including the high water and chemical consumption, environmental impacts, and water scarcity concerns. It also acknowledges the need for effective wastewater treatment methods. **Importance of membrane systems:** The passage emphasizes the role of membrane

systems in pollution prevention, water recycling, and closing the water loop in both urban and industrial settings. Membrane treatment is highlighted as a means to reduce fresh water intake, optimize costs, and control pollution. Segregation of wastewater streams: The study identifies different wastewater streams in textile wet processing, such as pre-treatment, dyeing and first washing, and post-washing wastewater. This segregation allows for targeted treatment approaches tailored to the specific characteristics of each stream. Membrane filtration results: The study presents results of membrane filtration experiments using different membranes, including UP005, NF200, and NF90. The performance of these membranes in terms of rejection percentages for various parameters, such as TSS, COD, color, and conductivity, is discussed. Reclaimed water quality: The study evaluates the quality of reclaimed water obtained through membrane treatment by comparing it with the quality of water typically used in textile dyeing processes. The reclaimed water is found to meet the required color variation limits for dark shades but may require further treatment for lighter shades. Flux performance: The passage mentions the decline in flux (permeate flow rate) over time for the different membranes. It notes that NF200 showed better flux performance than UP005 and NF90 membranes, although NF90 membrane exhibited superior removal of color, turbidity, conductivity, TSS, and COD. Lab-scale dyeing process: The study includes a lab-scale dyeing process using the reclaimed water and compares it with dyeing using standard water. The results suggest that the reclaimed water obtained from NF90 membrane filtration meets the required color variation limits for light shades, indicating its potential for reuse in textile wet processing. Operation and maintenance costs: The passage mentions that the operation and maintenance costs of membrane treatment systems were estimated for the best treatment scenario. However, detailed cost analysis and comparison with other treatment methods are not provided. Overall, the passage provides useful insights into the challenges of textile wastewater treatment and the potential of membrane systems for addressing these challenges. Further research and analysis would be beneficial to assess the economic feasibility and scalability of the proposed treatment methods (Nadeem et al. 2019).

The text describes a study conducted on the treatment of textile wastewater, specifically dyeing discharge, using advanced oxidation processes (AOPs) as a solution for water management in the textile industry. The aim was to develop a method for treating the highly contaminated dyeing discharge and reusing it as a source of "ready to use" brine, thereby minimizing environmental damage. The study focused on the influence of the wastewater matrix, including the presence of salts (such as NaCl) and textile auxiliaries (such as alkalines and surface-active

agents), on the effectiveness of AOPs. The AOPs tested included ozone (O₃) alone, ozone combined with hydrogen peroxide (H₂O₂), ozone combined with ultraviolet (UV) light, and ozone combined with both UV light and hydrogen peroxide. The decolorization and mineralization of the wastewater were evaluated during the treatment. The results showed that ozonation effectively decolorized the dyeing discharge, regardless of whether it was a single dye solution, a dye mixture, or simulated industrial wastewater. The wastewater matrix, including the presence of salts and textile auxiliaries, did not significantly affect the decolorization stage of the ozonation process. However, the matrix did influence the overall absorbed ozone dose, with higher pollutant contents requiring a higher dose. The study also found that the combination of ozone and hydrogen peroxide (O₃/H₂O₂) was effective in achieving higher levels of mineralization, with a reduction in chemical oxygen demand (COD) of up to 90%. However, the use of ozone combined with UV light or ozone combined with UV light and hydrogen peroxide (O₃/UV and O₃/UV/H₂O₂) was not recommended due to the high costs associated with these processes. Based on the findings, the study suggests that ozonation is a suitable option for decolorizing textile wastewater, especially when the reuse of the treated wastewater as brine is desired. For greater mineralization, the O₃/H₂O₂ process is recommended. The study emphasizes the importance of considering the real conditions of textile wastewaters, including the presence of multiple dyes and textile auxiliaries, in research and industrial applications to ensure reliable results and cost-effective solutions (Bilińska, Gmurek, and Ledakowicz, 2017).

The text highlights the high water consumption and energy requirements of various textile processes and emphasizes the need for water and wastewater management solutions. According to the information presented, global industrial water consumption is projected to increase significantly, and the textile industry is one of the sectors with high water consumption. The limited availability of water resources necessitates the use of treated wastewater to reduce raw water consumption and minimize the amount of discharged wastewater. This is especially important for industries with heavy water consumption such as food, textile, steel, pulp, paper, and chemistry. Best Available Techniques (BAT) have been identified as a means to reduce raw material usage and water consumption in many countries, particularly in the textile sector. The European yearly average fresh water consumption in the textile industry is stated to be approximately 600 million m³, with 108 million m³ of this amount requiring treatment as textile wastewater containing various pollutants. The text mentions that the textile industry consists of different unit operations, such as desizing, printing, dyeing, scouring, washing,

rinsing, bleaching, mercerizing, carbonization, finishing, and dyeing processes. These processes require significant amounts of water and generate large quantities of wastewater. The specific water consumption in the textile industry can vary depending on the process and fabric type but is generally around 2.5-932 L per kg of product. In Turkey, the textile industry is reported to be the second industry with the highest water consumption, accounting for 15% of industrial water consumption. The consumption of water in Turkey's textile industry was projected to increase from 410 million m³ in 2000 to 22 billion m³ by 2030. Besides water consumption, the text also mentions the considerable energy consumption in the textile industry, primarily for heating the baths used in wet processing. Therefore, the recovery of hot wastewater is important for energy efficiency and cost reduction. The composition of textile wastewater is stated to be different from that of other industrial sectors, and it contains various pollutants such as dyes, detergents, sulphides, solvents, heavy metals, and inorganic salts. The treatment and recovery of textile wastewater have been the focus of numerous studies, with membrane technologies, particularly ceramic membranes, being recommended as effective options for wastewater reuse and chemical recovery. The text also notes that while the Textile Best Available Technique (BAT) Reference document provides general recommendations for textile wastewater treatment and reuse, studies on hot wastewater recovery and reusability, specifically in textile processes, are limited. Therefore, the study described in the text aimed to investigate the applicability of ceramic membrane ultrafiltration for the recovery of hot wastewater in the textile industry. The study focused on a textile factory in Turkey and selected potential hot wastewater recovery points based on wastewater amount, temperature, and accessibility. Cross flow ceramic membrane filtration experiments were conducted using different membrane cut-offs to evaluate the reusability of the wastewater in the processes and calculate water-related costs. Overall, the text provides an overview of the challenges related to water consumption and wastewater generation in the textile industry and highlights the importance of water and wastewater management strategies, such as the use of treated wastewater and membrane filtration, to address these challenges (Dilaver et al. 2018)

The provided text discusses the issue of wastewater generation in various industries, such as textile, refineries, chemical, plastic, and food-processing plants. These industries produce wastewaters with a notable content of organics and strong color. Traditional methods of wastewater treatment, such as adsorption, biological oxidation, and coagulation, have limitations when applied individually due to the complexity and variability of industrial effluents. To address the need for more effective and efficient wastewater treatment,

researchers have been exploring advanced oxidation processes, including electrochemical techniques. Electrochemical treatment offers advantages such as versatility, energy efficiency, automation, and cost-effectiveness. In this process, the main reagent is the electron, which degrades organic pollutants without generating secondary pollutants or sludge. Different anode materials, such as ruthenium, lead, and tin oxide-coated titanium, have been tested in electrochemical treatment for organic pollutant degradation. The choice of anode material influences the oxidation rate, and ruthenium oxide-coated titanium has shown better performance in terms of chemical oxygen demand (COD) reduction. The instantaneous current efficiency (ICE) and percentage COD reduction increase with an increase in current density up to a certain point, beyond which they start to decrease. The reaction rate constant for organic pollutant degradation is influenced by the current density, and its increase becomes insignificant beyond a certain value. The text also discusses the analysis of UV spectra and chromatograms of raw and treated effluents, showing the reduction of organic carbon and the elimination of peaks corresponding to organic compounds in the treated effluent. Furthermore, the study explores the potential reuse of electrochemically treated wastewater for fabric dyeing applications. The treated wastewater quality is compared to that of groundwater, and it is found to be suitable for dyeing processes. The dye uptake of the fabric using the treated wastewater is measured, and the results indicate that the dyeing quality is comparable to conventional dyeing processes. In conclusion, the electrochemical treatment of wastewater offers a promising solution for the simultaneous removal of color and organic pollutants. It has the potential to minimize water consumption in textile industries and can provide treated wastewater that meets the quality requirements for reuse in dyeing processes (Mohan, Balasubramanian and Basha, 2007).

The provided text discusses the use of natural dyes in textile coloration and the application of spectroscopy techniques to analyze and evaluate the dyeing process. Natural dyes, derived from plants, are known for their potential environmental friendliness and biodegradability. Madder, a plant root, is one of the oldest natural dyes, containing anthraquinones such as alizarin and purpurin. Spectroscopy, including transmittance, absorbance, and reflectance measurements, is commonly used in the dyeing industry to analyze dyes in solution and determine their absorbance spectra, maximum absorbance wavelength, and absorptivity. Derivative spectrophotometry and colorimetry are also utilized for quantitative analysis and characterization of color stimuli. Principal component analysis (PCA) is a statistical method used to extract relevant information from complex data sets. It generates new variables, called

principal components, which are linear combinations of the original variables. PCA can help identify the directions of maximum variance in the data and simplify data analysis. In the experimental section, the materials and methods used for the study are described. Iranian woolen yarn was scoured and mordanted using potash-alum. The madder natural dye was extracted, and its absorbance spectra were measured using a spectrophotometer. The woolen yarn was dyed with different concentrations of madder dye, and absorbance spectra were measured again to assess the dye absorption. The exhausted dye solution was reused for further dyeing, both with and without reconstruction. Reflectance spectra were measured, and the dye concentration was calculated using Kubelka-Munk equation. The results indicate that the absorbance spectra of the initial dye solution differ from those of the exhausted dye solution. Statistical techniques, such as correlation analysis and scatter diagrams, were used to assess the similarity between the spectra. The colorimetric parameters of the dyed samples were measured, and their color fastness properties were evaluated. The results showed that the quality of samples dyed with the reconstructed dyebath was comparable to those dyed in the initial stage. The study demonstrates that reusing exhausted dyebath can lead to significant cost savings in terms of water and madder usage. The analysis and evaluation of dyeing processes using spectroscopy techniques provide valuable insights into the behavior of natural dyes and the potential for their reuse (Shams-Nateri, 2011).

The effluent contains color and total dissolved solids (TDS), making it difficult to biodegrade. The study focuses on three textile dyeing units in Tirupur, India, using different treatment techniques for water and salt recovery and reuse, with technical and economic analyses. The methodology involves selecting three units based on factors like production capacity, treatment systems, and membrane processes. The primary treatment involves physico-chemical treatment (PCT) to remove color and solids. Unit III incorporates a biological treatment system for significant COD reduction. The performance of pretreatment units varies across the units. Adequate pretreatment is crucial for efficient RO performance. Unit II shows better performance due to the right combination of pretreatment methods. The performance of membrane processes in terms of TDS removal and water recovery is generally high, but some units experience RO membrane deterioration. Unit III achieves the highest TDS removal efficiency, while Unit II suffers from low TDS removal. Cost analysis indicates varying profits for the units. Unit I achieves the highest profit due to lower operational costs. The revenue from water recovery is more significant than that from salt recovery. In conclusion, the RO process is technically and economically viable for water recovery and reuse in textile dyeing. NF permeate is suitable for dyebath water reuse, and the recovery of sodium chloride using NF is

more economical than sodium sulfate recovery using MEE. Lowering chemical usage through better operation and maintenance practices improves economic feasibility (Vishnu, Palanisamy and Joseph, 2008).

Tanneries produce large quantities of wastewater containing various pollutants, making their treatment challenging. Electrochemical methods, particularly electrocoagulation, have shown promise for treating various types of waste water. The study used a laboratory-scale reactor consisting of an undivided electrocoagulation cell with aluminum and mild steel electrodes. The reactor was made of organic glass and had four anodes and three cathodes. The tannery wastewater samples used in the study were collected from the first sedimentation tank of a tannery company in Anhui Province, China. Electrocoagulation is a complex process involving sacrificial metal anodes (aluminum or iron) that produce coagulating agents and electrolytic gases, such as hydrogen, at the cathode. The metal ions form coagulated species and metal hydroxides that destabilize and aggregate suspended particles or precipitate and adsorb dissolved contaminants. The study found that the electrocoagulation process effectively removed pollutants from tannery wastewater. The removal efficiency of chemical oxygen demand (CODCr), total organic carbon (TOC), sulfide, colority, and ammonia (NH₃-N) improved with increasing electrocoagulation time and current density. The pH value increased during the electrocoagulation process when aluminum electrodes were used, while it increased gradually when mild steel electrodes were employed. The study concluded that electrocoagulation could be an effective method for treating tannery wastewater. It could remove various pollutants, and the two-stage electrocoagulation process was particularly effective as a pretreatment step for biological processes (Jing-wei et al. 2007).

The increasing costs of water, energy, and wastewater treatment, as well as changing environmental regulations, have driven the need for water recycling in the textile industry. Dyeing processes result in wastewater containing various pollutants, including dyes, which are challenging to remove, particularly reactive dyes used for cotton fabrics. Various treatment methods have been proposed, but oxidative treatments, such as ozone oxidation, appear promising due to their degradation of pollutants and color removal without producing sludge. The study aims to investigate the feasibility of using ozone technology for recycling reactive dye effluents for dyeing cotton fabrics. The experimental setup consists of an ozone generator, ozonation chamber, and ozone destructor. The dyeing process involves recycling dye baths, treating the effluents with ozone until complete color removal, and reusing the treated effluents for further dyeing. The results indicate that as the number of recycling processes increases, the

time required for complete decoloration increases, and the COD is reduced. Ozone treatment leads to a decrease in pH due to the formation of acidic by-products during oxidation. The study also evaluates the effect of recycling on the quality of dyeing, including color reproduction, lightness difference, and levelness of dyeing. It is found that recycling can be carried out for a certain number of cycles before reaching unacceptable color difference values. However, the levelness of dyed fabrics remains excellent or good, even in cases where color difference values are higher. In conclusion, ozone oxidation proves to be a promising method for purifying textile wastewater for reuse, reducing both environmental and economic costs. It allows for the recycling of effluents for multiple dyeing cycles while maintaining good levelness in dyed fabrics (Senthilkumar and Muthukumar, 2007).

The study focuses on the development of a low-cost and effective adsorption material for removing reactive dyes from wastewater generated during the dyeing and finishing processes of textile fabrics. The process of dyeing with water-based media leads to environmental issues, including water pollution due to the discharge of chemical additives and unfixed dyes. The researchers developed a nano-cellulose hybrid containing polyhedral oligomeric silsesquioxane (R-POSS) as a functional and reactive reagent. The R-POSS monomer possesses multi-N-methylol groups and can readily crosslink with cellulose, forming a network structure containing nano-sized inorganic particles. The hybrid material was prepared by crosslinking cellulose fabric with R-POSS, resulting in nano-sized cubic cores and numerous organic functional groups ($-C-N-$) that provide significant adsorption capacity for reactive dyes. The effects of temperature and time on the adsorption process were also studied. The adsorption of the dyes increased initially with time and then reached equilibrium after a certain period. Higher temperatures resulted in lower amounts of adsorbed dyes, indicating increased desorption rates at higher temperatures. In conclusion, the nano-cellulose hybrid containing R-POSS proved to be a promising adsorption material for removing reactive dyes from wastewater. Its high adsorption capacity, low-cost nature, and ease of reuse make it a potential solution for tackling water pollution caused by textile dyeing processes. The study contributes to the development of eco-friendly and sustainable methods for wastewater treatment in the textile industry (Xie, Zhao and He, 2011).

The researchers prepared chitosan nanoparticles using the ionic gelation method with sodium tripolyphosphate (STPP). The nanoparticles exhibited a narrow size distribution with a mean diameter of 86 nm and a zeta potential of +28 to +48 mV, indicating good adsorption potential

for negatively charged dyes like RR120. The study investigated the effect of chitosan and STPP concentrations on dye removal and found that a chitosan to STPP mass ratio of around 3:1 resulted in maximum dye removal efficiency. Furthermore, the nanodispersion outperformed dissolved chitosan in removing the dye. The researchers also studied the influence of pH on dye removal and observed an optimum pH range of 4-5, beyond which the efficiency decreased. At lower pH values, the efficiency declined due to gradual protonation of the sulfonate groups in the dye molecule. Temperature had an impact on the adsorption process, with an optimal temperature of around 30°C, above which the efficiency decreased due to increased solubility of both the dye and the adsorbents. The adsorption isotherm data was analyzed using the Langmuir model, which revealed a high adsorption capacity of around 910 mg/g for the chitosan nanodispersion and 51 mg/g for dissolved chitosan. The adsorption kinetics were best described by the pseudo-second order model, indicating a faster adsorption rate for the nanodispersion compared to dissolved chitosan. In conclusion, the study demonstrates that chitosan nanoparticles are highly effective for removing anionic dyes from wastewater, offering a promising solution for water decontamination. The unique properties of chitosan make it a versatile and environmentally friendly adsorbent for various applications in wastewater treatment (Momenzadeh et al. 2011)

The study explores the use of Jute fibers as an adsorbent for the removal of reactive dye from wastewater. It acknowledges the environmental damage and health risks associated with dye pollution in water bodies and emphasizes the need for effective treatment methods. Adsorption is considered a promising approach due to its effectiveness and minimal production of unwanted by-products. Jute fibers, known for their low cost, durability, and eco-friendly properties, are chosen as the adsorbent material. To enhance their adsorption capacity, the fibers are pretreated with alkali and then grafted with acrylic acid using gamma radiation. Chitosan, a natural polymer, is immobilized on the grafted Jute fibers to further improve dye adsorption. The grafting conditions, such as monomer concentration, dose, solvent, and inhibitor, are optimized to achieve the desired grafting yield. The treated Jute fibers are characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA). These techniques provide insights into the structural and morphological changes of the fibers after the grafting and immobilization processes. The adsorption experiments demonstrate that the amount of dye adsorbed by the treated Jute fibers increases with lower pH, longer contact time, higher temperature, and higher initial dye concentration. These findings align with previous

research on dye adsorption. The adsorption isotherm models, including Langmuir and Freundlich, are used to analyze the adsorption data. The Langmuir model provides a better fit to the experimental data, indicating a monolayer adsorption process. The study also explores the regeneration potential of the treated Jute fibers. It is found that the adsorbed dye can be desorbed by using strong acid, such as HCl, for full regeneration. However, the regeneration process leads to a gradual decrease in the dye adsorption efficiency of the fibers over multiple cycles. While the study presents valuable insights into the use of treated Jute fibers for dye adsorption, some limitations and areas for improvement can be identified. Firstly, the study could benefit from providing more detailed information on the optimization process for grafting conditions and the rationale behind the choices made. Secondly, the characterization techniques used could be further complemented by additional analyses, such as surface area measurements, to provide a comprehensive understanding of the structural changes in the treated fibers. Lastly, the regeneration process could be optimized to enhance the fibers' reusability and mitigate the decrease in dye adsorption efficiency over time. In conclusion, the study highlights the potential of treated Jute fibers as an effective adsorbent for the removal of reactive dye from wastewater. The grafting and immobilization processes, along with the optimization of adsorption parameters, demonstrate improved dye adsorption capacity. However, further research and optimization are needed to address the identified limitations and enhance the regeneration potential of the treated fibers for prolonged and sustainable use in dye removal applications (Hassan, 2015).

The article is challenge of treating industrial liquid waste (ILW) generated by the textile industry, which contains high levels of pollutants including organic and inorganic materials, heavy metals, and colorants. The article discusses the limitations of traditional chemical and biological treatment methods due to factors such as chemical reagent consumption, sludge generation, space requirements, and treatment duration. As an alternative, the study focuses on electrochemical processes, particularly electrocoagulation (EC), as a promising solution. The electrocoagulation process involves passing an electric current through the ILW, leading to the formation of coagulants from metal ions at the anode. The study examines the influence of operational variables such as pH, current density, and treatment time on the efficiency of color, turbidity, and chemical oxygen demand (COD) removal. Through experimental analysis and statistical modeling, optimal conditions for EC treatment are determined. For ILW originating from dyeing linen, the study finds that EC treatment achieves significant removal efficiencies: 80% for color, 65% for turbidity, and 24% for COD. The results are dependent on factors like

pH, with pH 9 being optimal for color removal. Turbidity and COD removal are influenced by pH as well as current density. Additionally, the study evaluates the potential for reusing the treated ILW in the dyeing process. By using the treated water in dyeing wool fabrics, it is demonstrated that the treated wastewater does not negatively impact dye fixation or fabric color. The research emphasizes the economic and environmental benefits of recycling ILW within the textile industry, reducing water consumption, and lowering wastewater volume.

In conclusion, the article highlights that electrocoagulation offers an effective and economically viable method for treating industrial liquid waste from the textile industry. The study demonstrates the importance of optimizing operational variables to achieve efficient pollutant removal and emphasizes the potential for reusing treated wastewater in industrial processes (Núñez et al. 2019).

The research is imperative of water security, a key Sustainable Development Goal set by the UN. The textile industry's dyeing processes pose a substantial environmental challenge due to their high water consumption and the release of effluents containing residual dyes and chemicals. Reactive dyes, commonly used for their wash-resistant shades on cotton, contribute significantly to this issue. The study focuses on the application of UV/H₂O₂ (ultraviolet light and hydrogen peroxide) treatment to decolorize wastewater generated from reactive dyeing processes, enabling its reuse for subsequent dyeing cycles. The investigation explored the effect of pH on the UV/H₂O₂ treatment's efficiency, considering three pH levels (4, 7, and 11). Acidic conditions (pH 4) were found to be most effective in decolorizing the wastewater, with pH 7 also showing promising results. The removal of color was attributed to the formation of hydroxyl radicals (HO•), which degrade the dye molecules. The study established the potential for wastewater reuse, as pH adjustments were required to counter the alkaline pH resulting from the dyeing process. Additionally, the study evaluated the quality of cotton fabrics dyed using decolorized wastewater in terms of coloristic properties and fastness to light and washing. The results indicated that the quality of dyed fabrics remained acceptable for textile use, with minimal color differences (ΔE^*) observed. The fastness properties were not compromised by reusing the decolorized wastewater, further validating its viability for reuse. In conclusion, the research successfully demonstrates the feasibility of employing UV/H₂O₂ treatment to efficiently decolorize reactive dye wastewater, allowing for its reuse in subsequent dyeing cycles. The study underscores the significance of pH adjustments and the importance of maintaining the electrolyte concentration. The findings contribute to sustainable water

management strategies in the textile industry, aligning with the UN's water security goals (Bezerra et al. 2021).



2. DYEING PRINCIPLES

The dye substances used in the textile dyeing industry are not composed of a single component. To achieve the desired shade of color, dye production involves mixing different shades of dyes. A dye from the same batch may not have the same strength for each production time. Additionally, similar shades of dyes can have different mixtures or variations in the red-yellow direction for different levels of darkness. Dyes are typically dissolved in soft water. If hard water is used, certain types of dyes may react with the ions in the hard water, causing precipitation and hindering the desired color formation. In addition to soft water, water temperature and auxiliary chemicals are important for the dissolution of some dyes. The selection of the appropriate dye for creating a specific color can vary depending on the desired color, colorfastness values, and cost considerations. Durable dye groups and selections of dyes that do not undergo chemical reactions with each other are important considering the intensity and tone of the color and the washing processes after dyeing. The shape of the product to be dyed also plays a significant role in selecting the appropriate dye. Different dye selections at different temperatures can be made for the same yarn dyed in bobbin or hank form to achieve the same color.

2.1. Polyamide (PA) and Wool (WO)

Polyamide is chemically related to wool. Amino groups are integral components of both fibres. Polyamide can be dyed with the same dyes as wool. Basic problem with blends of wool and polyamide is to achieve solid shades on both fibres.

Parameters influencing tone-in-tone dyeing of WO/PA

- Dye concentration
- Pale to medium shade depth
- PA is dyed generally deeper than WO
- Dye uptake on PA has to be retarded by a blocking agent

Medium to deep shade depth often tone-in-tone dyeing. Deep shade depth WO is often dyed darker than PA. PA part has to be covered up with dyes having high affinity for PA.

Besides the dye selection based on end use requirements like wet and light fastness properties, each individual dye has a specific affinity to wool and polyamide. This means that some dyes dye wool darker and some dye polyamide darker. For dye combinations it is very important that the individual dyes distribute similar between wool and polyamide and that they react similar to the same amount of blocking agent to give good solidity.

PA 6 has a higher dye affinity compared to PA 66 and needs therefore more blocking agent to achieve solid shades in wool / polyamide blends. Besides this differences based on chemical composition, the fibre fineness (dtex), fibre surface / cross section (round, trilobal, ect.), degree of delustering, texturing, and pretreatment (heat setting) influences the color depth on polyamide.

Wool quality like provenience, fibre diameter, pretreatment, influences the uptake and shade depth on wool. If the wool has been treated by a shrink-resist-process the dye affinity is increased appreciably. In most of these cases wool is then dyed darker compared to polyamide.

Blocking agents consist of colorless anionic compounds having high affinity to polyamide. They saturate the polyamide fibre and reduce therefore dye affinity to the fibre and the dye is pushed more to the wool. The amount of blocking agent has to be adjusted so that an even distribution can be achieved between the fibres. The necessary amount of blocking agent depends on the used dyes, dye concentration, fibre type and blend ratio, and has to be determined in preliminary lab trials simulating as close as possible bulk conditions.

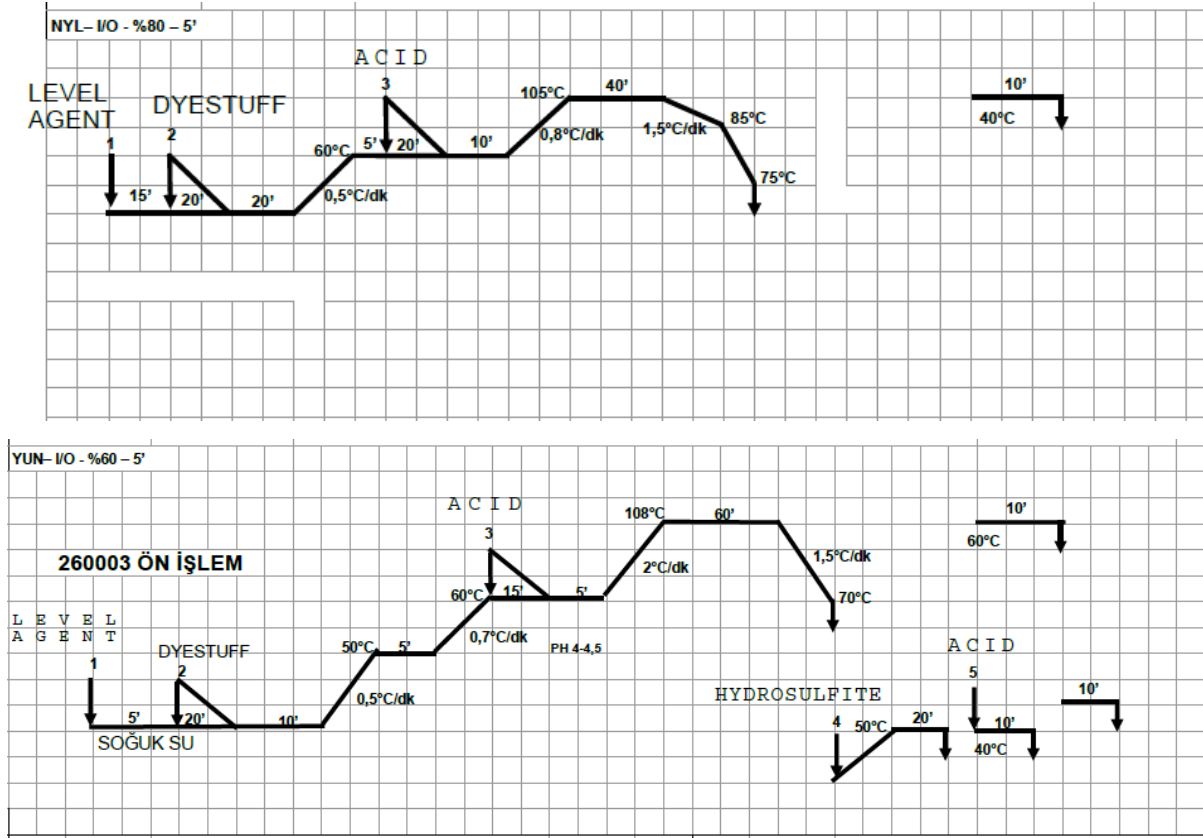


Figure 3. Polyamide Dyeing Processes

2.2. Reactive Dyeing

Reactive dyes are important coloring agents used in the textile industry, which create permanent coloration by forming chemical bonds with fibers. These dyes were developed in the mid-20th century and represented a revolutionary advancement in textile dyeing. The history of reactive dyes began with advancements in synthetic chemistry in the late 19th century. In 1856, William H. Perkin synthesized an organic compound called aniline, which led to the production of mauveine, a purple dye. This discovery was a fundamental step in the production of synthetic dyes. Later, in the early 20th century, German chemist Paul Friedländer conducted research on the development of dyes that could form permanent bonds with fibers during the textile dyeing process. In 1908, Friedländer discovered reactive dyes that could be chemically bonded to textile fibers. However, these early reactive dyes had limited use and were not commercially successful. The modern form of reactive dyes was developed in the 1950s by Swiss chemists Jean Jacques and Jean François Tronchet. During this period, various reactive dyes were synthesized under their leadership and successfully used in the textile dyeing industry. The development of reactive dyes was revolutionary in terms of creating permanent coloring by forming chemical bonds with fibers and offering a wide range of colors.

The development of reactive dyes brought about a significant transformation in the textile dyeing industry. These dyes provide long-lasting coloration to textile products due to their superior colorfastness and strong bonding with fibers compared to other dyeing agents. Additionally, reactive dyes offer an environmentally friendly approach as they are used in water-based dyeing systems. Today, reactive dyes are one of the most commonly used coloring agents in the textile industry. Continuous efforts are being made to develop new reactive dyes and conduct research on dyeing processes. Reactive dyeing techniques are constantly being improved to optimize the interaction between dyes and fibers, enhance color intensity, and improve environmental sustainability. Reactive dyes are dyeing agents for textiles that originated in the 19th century with advancements in synthetic chemistry and made a significant leap in the mid-20th century. These dyes are special compounds that react with fibers to provide permanent and diverse coloring in the textile industry. Nowadays, the development of reactive dyeing processes and reactive dyes plays a crucial role in the textile industry.

Reactive dyeing method is a widely used textile dyeing technique worldwide. This technique is implemented in numerous countries within the textile industry. Here are some examples: Switzerland: The modern form of reactive dyeing was developed in Switzerland in the 1950s by Jean Jacques and Jean François Tronchet. During this period, Switzerland played a pioneering role in synthesizing and commercially utilizing reactive dyes. Dyeing companies in Switzerland made significant progress in applying reactive dyes to textile products, enhancing colorfastness and permanence. These advancements led Switzerland to become a leading country in reactive dyeing technology. Germany: Germany is also known as a leading country in reactive dyeing technology. In the early 20th century, German chemist Paul Friedländer discovered dyes that could react with textile fibers in a reactive manner. Friedländer's work laid the foundation for reactive dyeing technology. The chemical industry in Germany made great strides in synthesizing and developing reactive dyes, playing a significant role in reactive dyeing technology.

India: India is recognized as a major producer and exporter in the textile industry. Reactive dyeing technology has contributed to India's significant position in the textile sector. Textile companies in India apply reactive dyeing methods to color various fibers and achieve long-lasting and vibrant colors in textile products. India is an important center for the implementation and development of reactive dyeing technology. China: China is a leading country in textile production and export. Reactive dyeing methods are widely used in the textile

industry in China. Dyeing facilities in China apply reactive dyes to textile products to achieve permanent coloration. Due to its size and importance in the textile industry, China is among the countries where reactive dyeing technology is extensively utilized. Other Countries: Reactive dyeing methods are used in many countries worldwide. These countries include Turkey, Pakistan, Bangladesh, Brazil, Thailand, and Italy, which are significant in terms of textile production and trade. These countries have embraced reactive dyeing technology and use it to achieve permanent and vibrant colors in their textile industries. Reactive dyeing is a dyeing technique used in the textile industry that involves chemical reactions at a molecular level to dye the fibers. This method relies on reactive dyes, which are special compounds that create permanent chemical bonds on the surface of cellulose fibers (such as cotton and linen) during the dyeing process. Reactive dyes are typically organic compounds containing halogen or aqueous solutions that become activated at alkaline pH values. Reactive dyeing offers several advantages compared to other dyeing methods. These include high colorfastness, a wide range of colors, good wash durability, environmental friendliness, and the ability to allow fibers to breathe. However, the reactive dyeing process can be quite complex, requiring careful control of numerous parameters at each stage.

Chemically, the reactive dyeing process is based on the reaction of the dye molecules' reactive groups with textile fibers. These reactions can vary depending on the chemical structure of the dye and the structure of the fibers. Generally, the dyeing process involves the hydrolysis of the dye molecule under alkaline conditions, the reaction of the dye molecule's reactive groups with the hydroxyl groups of the fibers, and the fixation step to stabilize the dye. Reactive dyeing is an effective dyeing method widely used in the textile industry. It provides permanent and vibrant colors to textile products with high colorfastness and durability. Additionally, it is considered an environmentally friendly option as it generates low waste and reduces water consumption. Cellulosic fibers, such as cotton and linen, are suitable for the reactive dyeing process. These fibers contain hydroxyl (-OH) groups on their surface, which allows them to interact with the reactive groups of the dyes. The reactivity of cellulosic fibers is related to the presence and location of hydroxyl groups. The cellulose molecule is a long polymer chain composed of glucose units. Each glucose unit has three hydroxyl groups (at the C2, C3, and C6 positions), which are the sites where reactive dyes can bind. During the reactive dyeing process, the hydroxyl groups of cellulosic fibers react with the reactive groups of the dye molecules. These bonds ensure a strong attachment of the dye molecules to the textile fibers, contributing to high colorfastness and durability of the dye. Factors that enhance the reactivity

of cellulosic fibers include fiber structure, accessibility and density of hydroxyl groups. Crystalline regions of cellulose may limit the interaction of hydroxyl groups with reactive dyes, while amorphous regions have higher reactivity. Various methods can be employed to increase the reactivity of cellulosic fibers for the reactive dyeing process. The pretreatment step helps in removing dirt and oils from the fibers, facilitating the interaction of hydroxyl groups with reactive dyes. In this step, alkaline environments can enhance the reactivity of hydroxyl groups. Additionally, certain chemicals can be used to increase the surface activity of cellulosic fibers and release hydroxyl groups. During the reactive dyeing process, the activation of the dye can also affect the reactivity of cellulosic fibers. Dyes are typically activated under alkaline conditions, releasing their reactive groups. This facilitates the interaction of dye molecules with hydroxyl groups and ensures the dye's permanence on the fibers. The reactivity of cellulosic fibers is essential for the effectiveness and quality of dyeing processes in the textile industry. The natural structure of cellulose and the reactive nature of hydroxyl groups allow for strong chemical bonding with reactive dyes, resulting in high colorfastness, good wash durability, and environmentally friendly dyeing options. The reactivity of cellulosic fibers can be optimized by adjusting dyeing process parameters and the selection of dyes, to achieve desired color intensity, durability, and permanence. Reactive dyes are widely used dyeing agents in the textile industry, as they form permanent chemical bonds with fibers, enabling the dyeing of textile materials. The chemical structure of these dyes includes various functional groups, which significantly influence colorfastness, dyeability, and chemical reactivity. Reactive dye molecules typically consist of water-soluble groups, chromophore groups, bridge groups, reactive groups, and other functional groups. Water-soluble groups facilitate the interaction and solubility of dye molecules with water. These groups have hydrophilic properties, enabling the dye molecules to disperse homogeneously in aqueous environments. For example, the sulfonic acid group enhances the water solubility of dye molecules. Chromophore groups are responsible for the dye molecules' coloring properties. These groups contain conjugated double bond systems or aromatic rings that allow for electronic transitions. This enables the dye molecules to absorb and reflect light at specific wavelengths, resulting in color formation. For example, the azo ($-N=N-$) group provides colorfulness to dye molecules. Bridge groups are responsible for enabling chemical bonding between dye molecules and textile fibers. These groups react with the reactive functional groups of dye molecules and the reactive functional groups present in the textile fibers. As a result, dye molecules are covalently bonded to textile fibers, enhancing dye permanence. Bifunctional or trifunctional reactive groups are commonly used to allow stronger bonding between dye molecules and textile fibers. Reactive groups

enable the chemical reaction between dye molecules and textile fibers. These groups can interact with hydroxyl groups, amino groups, or other active functional groups present in the textile fibers. Reactive groups facilitate the formation of covalent bonds between dye molecules and textile fibers, enhancing dye durability against external factors such as washing or friction. For example, reactive ester groups, widely used among reactive dyes, create permanent bonds by reacting with textile fibers.

These various groups within reactive dyes contribute to their effectiveness in achieving permanent and vibrant colors on textile materials. The reactivity of cellulosic fibers, the natural structure of cellulose, and the reactive properties of hydroxyl groups allow for strong chemical bonding between dye molecules and fibers. This results in high colorfastness, good wash durability, and environmentally friendly dyeing options. The reactivity of cellulosic fibers can be optimized by adjusting dyeing process parameters and selecting the appropriate reactive dyes, ensuring desired color intensity, durability, and permanence.

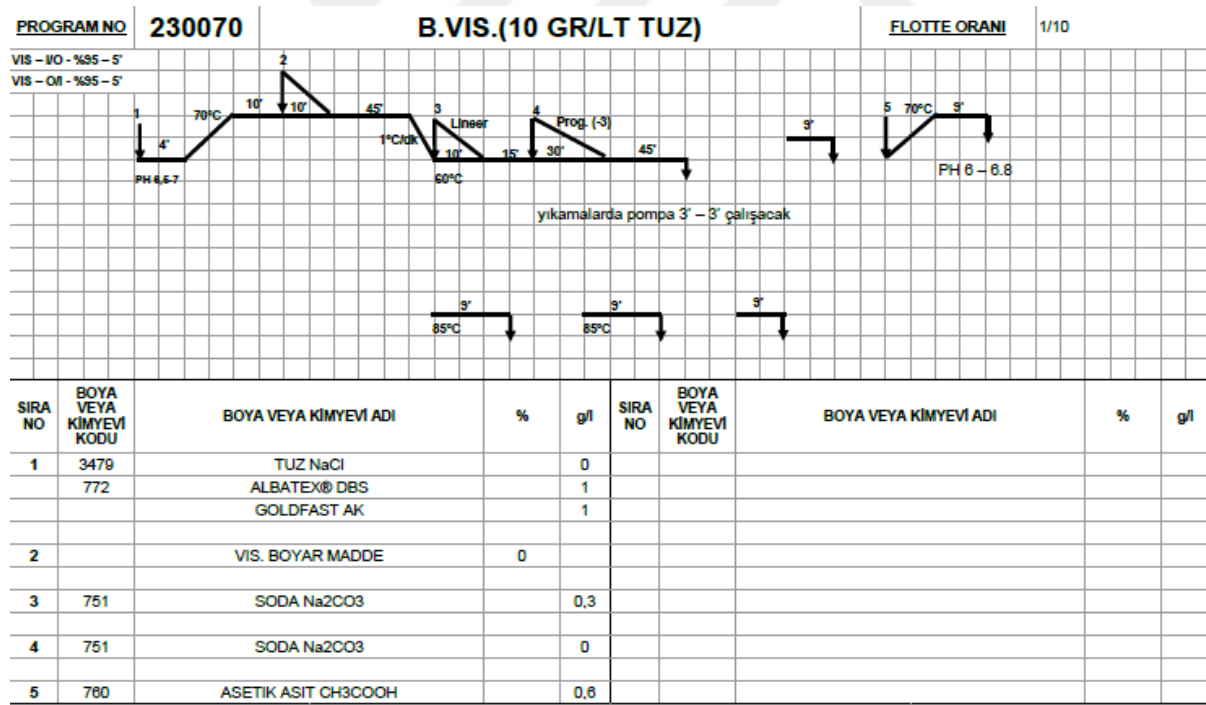


Figure 4. Viscose Dyeing Processes Chart For Light Colors

In the above image, the chart shows the dyeing process in a vertical package dyeing machine, specifically for light shades of 100% viscose dyeing. As seen in the image, the circulation pump is set to operate in both directions for 5 minutes. After water is added to the dyeing machine,

salt and auxiliary chemicals are added in cold water at a pH range of 6.6-7 for a preparation time of 4 minutes. The machine heats the water to a maximum temperature of 70°C using maximum acceleration, and the dye solution is dosed into the machine within 10 minutes with a linear dosage at this temperature. While the flotte circulates continuously inside-out and outside-in through the fabric packages, the temperature of the water is lowered again to the desired value in order to prepare the alkaline environment for the reactions between the dye and the viscose fibers.

As seen from the chart, this process utilizes a two-stage soda dosage. In the first stage, a very small amount of soda is dosed linearly for a duration of 10 minutes. The remaining amount of soda, calculated based on the total percentage of the dye, is progressively dosed in the second stage. The main idea behind the progressive dosage is to ensure a slow transition of the soda into the alkaline environment by initially dosing a small amount, aiming to distribute the dye uniformly throughout the entire region of the fiber.

Total Dosing Qty.			450,00				
Curve	8,00%	Dosing Qty.	5 min. Dosing Qty.	5 min. Dosing %	10 min. Dosing Qty.	10 min. Dosing %	
1,000	0,88%	3,972	23,3	5%	13%	24%	
1,080	0,95%	4,290					
1,166	1,03%	4,633					
1,260	1,11%	5,004					
1,360	1,20%	5,404					
1,469	1,30%	5,837	34,2	8%	28%		
1,587	1,40%	6,304					
1,714	1,51%	6,808					
1,851	1,63%	7,353					
1,999	1,76%	7,941					
2,159	1,91%	8,576	50,3	11%		76%	
2,332	2,06%	9,262					
2,518	2,22%	10,003					
2,720	2,40%	10,803					
2,937	2,59%	11,668					
3,172	2,80%	12,601	73,9	16%	60%		
3,426	3,02%	13,609					
3,700	3,27%	14,698					
3,996	3,53%	15,874					
4,316	3,81%	17,143					
4,661	4,11%	18,515	108,6	24%		76%	
5,034	4,44%	19,996					
5,437	4,80%	21,596					
5,871	5,18%	23,323					
6,341	5,60%	25,189					
6,848	6,05%	27,205	159,6	35%	60%		
7,396	6,53%	29,381					
7,988	7,05%	31,731					
8,627	7,62%	34,270					
9,317	8,22%	37,011					
113,28	100,00%						

Figure 5.Progressive Dosing Calculation

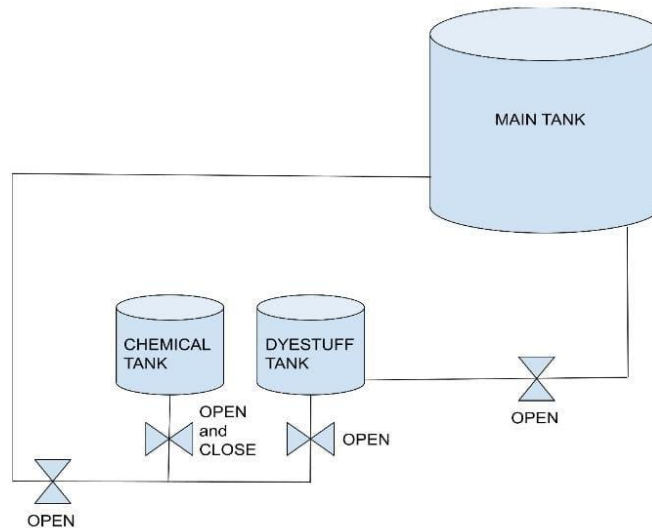


Figure 6. Progressive Dosing Diagram

As depicted in the above diagrams, during the progressive dosage, there is a circulation between the dye solution tank and the main tank, with a valve continuously open between them. During this circulation, the valve of the chemical tank, which contains the soda solution, doses a small amount of soda into the pipe where the circulation occurs by performing quick open-close actions initially. The speed of the pneumatic valve's open-close motion and the duration of the valve being open increase as the total dosage time approaches its end. As a result, the soda being sent to the main tank becomes more diluted through continuous circulation with the dyed water, and the momentum of the flotte entering the alkaline environment is significantly slowed down. Therefore, the chemical reaction progresses slowly, allowing the dye to establish a healthy and uniform bond with the fibers.

After the dye bath is kept for a certain period and then emptied, the washing steps are initiated. Since the dye and the fibers are now bonded to each other, meaning that the coloring process in the yarn is complete, the process continues by transitioning to an acidic environment to fully finish the dyeing and remove any unfixed dye from the bath. The number of washing cycles may vary depending on the shade of color and the amount of dye used.

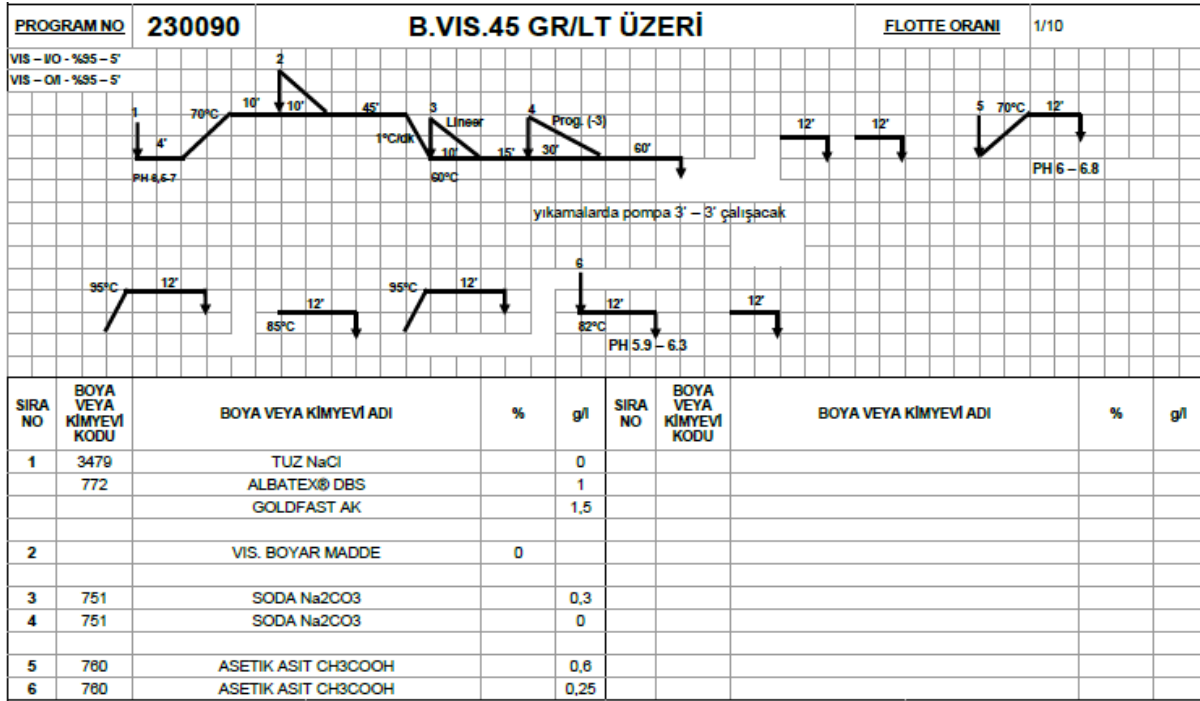


Figure 7. Viscose Dyeing Processes

In viscose dyeing, various reactive leveling agents can be used to distribute the dye solution more homogeneously and slow down the dyeing process, depending on the shade of color, amount of dye, and the intended market for the yarn. Reactive leveling agents are typically dosed with salt before the dyeing process to prepare the solution. Additionally, in some cases, as the color intensifies, a slightly more alkaline environment may be required, and in such situations, a small amount of sodium hydroxide can be added to the bath along with the soda ash. However, using sodium hydroxide has two disadvantages: excessive use can strip the dye from the fiber, and in cases where it is used, additional acid washes may be required in the post-dyeing washing process to restore the acidic environment. This is because a single acid wash may not be sufficient to return the environment to an acidic state.

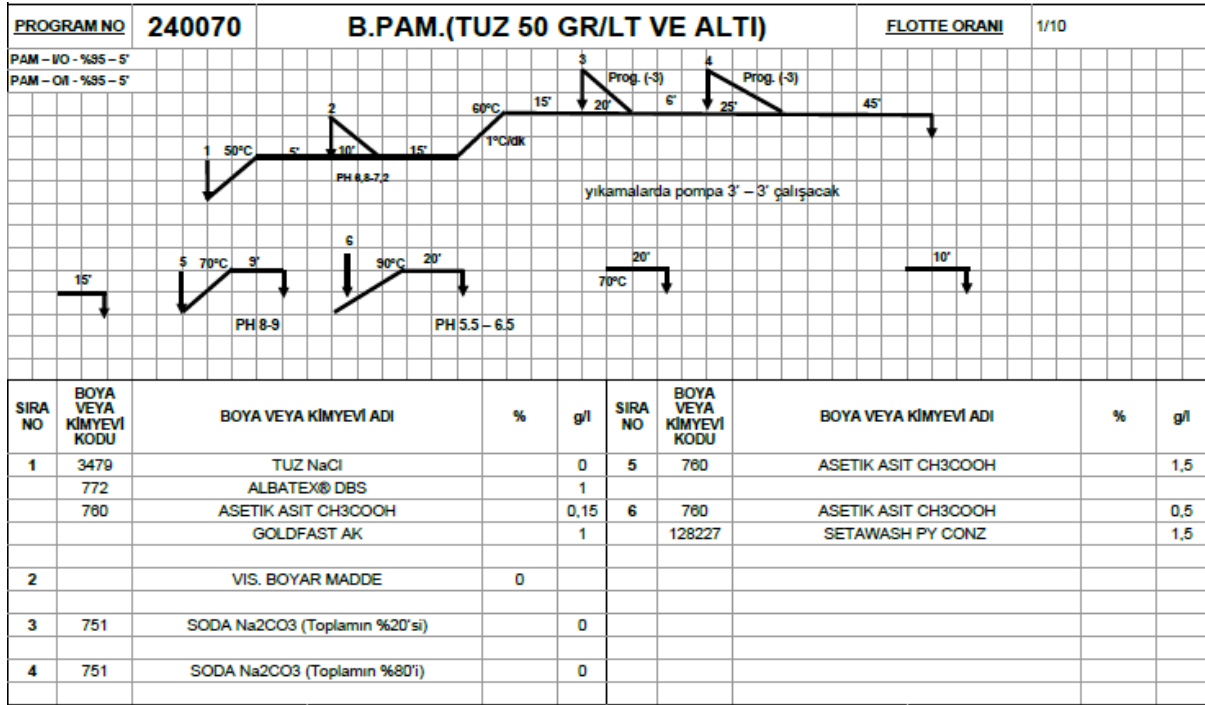


Figure 8. Cotton Dyeing Processes

The key difference between cotton and viscose dyeing lies in the fiber itself. Cotton, being a natural fiber, is slightly more challenging to bind the dye to compared to viscose. Its color fastness properties are generally inferior to viscose fiber. More chemicals are required during the chemical reaction to bind the dye to the cotton fiber, such as higher amounts of sodium chloride, soda ash, and sodium hydroxide. The graph above shows a cotton dyeing chart with medium-toned colors. Unlike the viscose dyeing chart, cotton dyeing is typically carried out at lower temperatures. In the textile industry in Turkey, many textile dyeing facilities prefer providing the dye in a hot environment for reactive dyeing. This is primarily due to color fastness and abrasion effects. Although most of the reactive dyeing process relies on chemical reactions, with pH conditions being crucial, cotton and viscose fibers are more receptive to dye binding in cold water. As cotton is a natural fiber, a lower temperature is preferred when dosing the dye solution to achieve the desired color fastness levels.

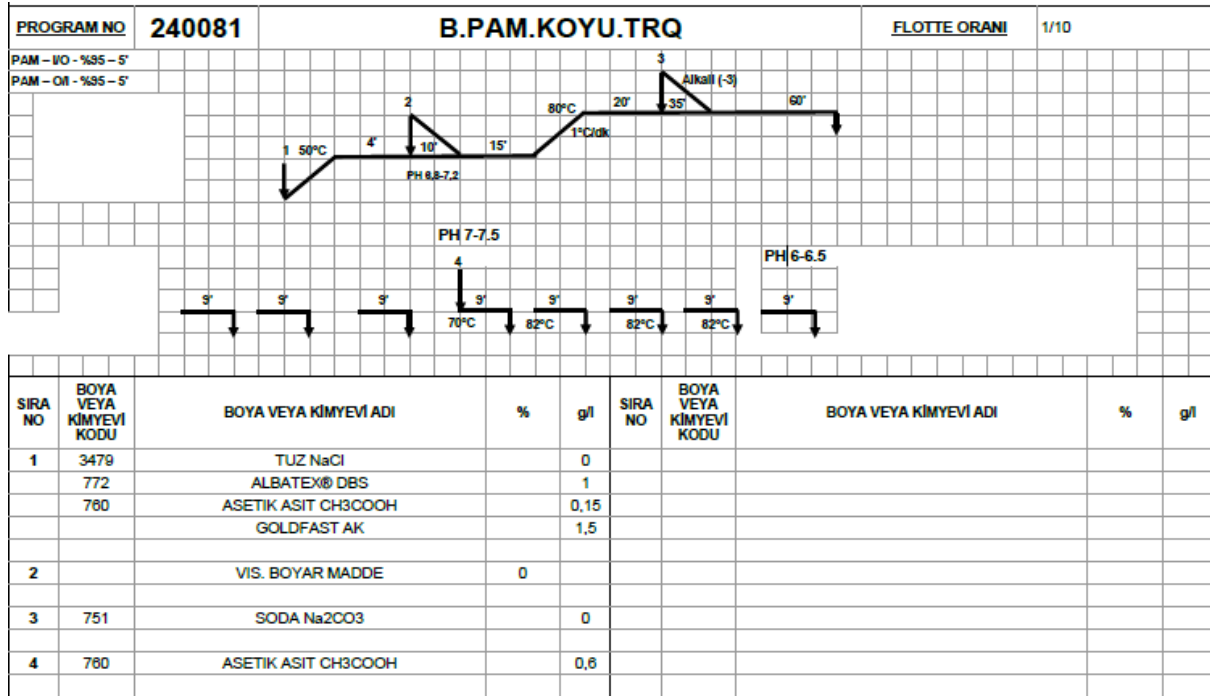


Figure 9. Turquoise Color Dyeing Processes

Turquoise, khaki, green tones, and similar colors are considered challenging to dye. The main reason for this is the risk of uneven distribution of the dye solution to the fiber, even when the dye is converted into a solvent. Consequently, the color fastness of these shades may be poorer compared to other colors, and undesirable outcomes can occur in the dyeing process. Moreover, the dyeing and solubility temperatures of the dyes used to achieve these colors can differ from standard dyes. In the turquoise color chart above, we can observe a cotton dyeing process that differs from a regular process. It involves dyeing at higher temperatures, transitioning to a higher temperature alkaline environment, and undergoing more extensive washing after dyeing.

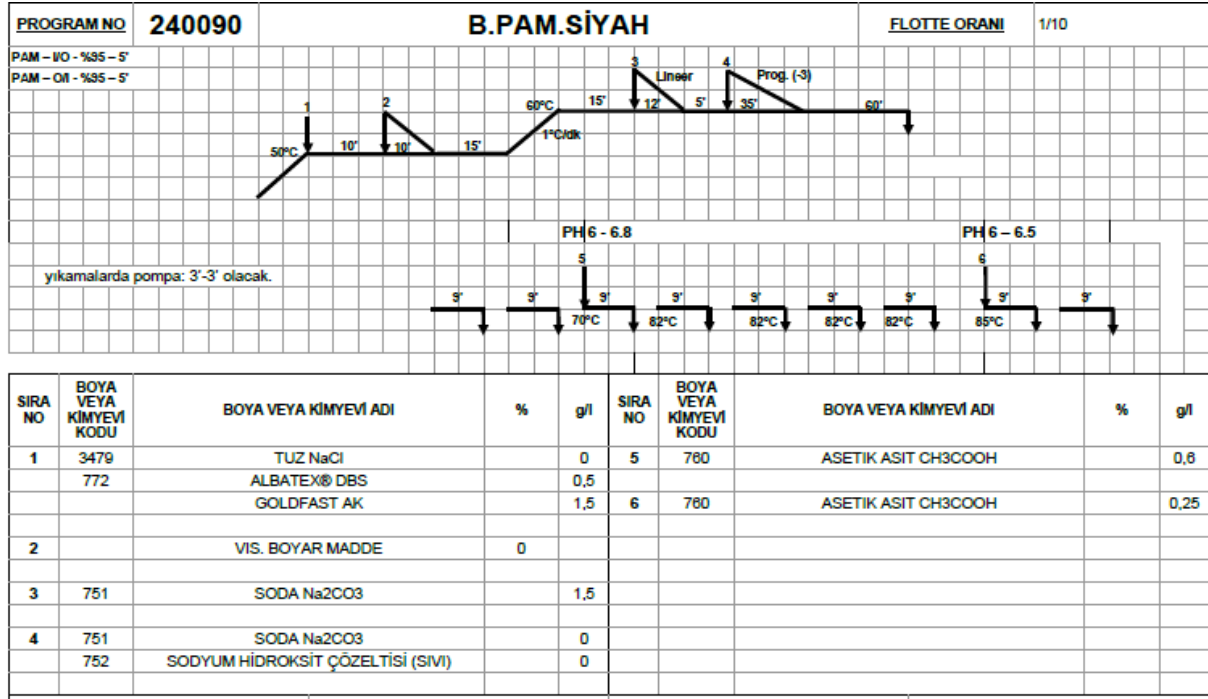


Figure 10. Black Color Dyeing Processes

3. DYEING AUXILIARY CHEMICALS

Wetting agent (Anionic, Aromatic Polyether Sulfonate - Levelling agent): A wetting agent is an auxiliary chemical used in reactive dyeing to reduce the surface tension of the fabric, allowing the dye to spread evenly on the fabric. Wetting agents have an anionic structure and are often referred to as aromatic polyether sulfonates. Wetting agents help increase the fabric's water absorption ability, aiding in the homogeneous and even distribution of the dye.

Ion scavenger (Anionic - Günerca): Ion scavengers are auxiliary chemicals used to neutralize or remove unwanted ions (such as metal ions) that may occur during reactive dyeing. Anionic ion scavengers act on negatively charged ions to render them ineffective. This helps prevent undesired color changes or other quality issues during the dyeing process.

Antifoaming agent (Nonionic, Polymer-based - Levelling agent): Antifoaming agents are auxiliary chemicals used to prevent fabric breakages and tears caused by mechanical forces during reactive dyeing. Nonionic antifoaming agents create a polymer layer on the fabric surface, protecting and enhancing the durability of the fabric fibers. This prevents damage to the fabric during the dyeing process.

Salt (Sodium sulfate - Levelling agent): Salt is an auxiliary chemical used in reactive dyeing to promote the dye's binding to the fabric. Sodium sulfate is a commonly used type of salt for this purpose. Salt interacts with the reactive groups of the dyes during the dyeing process, allowing them to attach to the fabric fibers and form a permanent bond. Additionally, salt helps control the spreading and diffusion of the dye during the dyeing process.

Soda ash (Sodium carbonate - Levelling agent): Soda ash is an auxiliary chemical used for pH regulation in reactive dyeing. Sodium carbonate is a commonly used type of soda ash. Soda ash is used to provide an alkaline environment during the dyeing process. The alkaline environment enables the activation of reactive dyes and their interaction with the fabric. Soda ash also helps optimize color formation and reactivation during the dyeing process.

Caustic soda (Sodium hydroxide - Levelling agent): Caustic soda is an auxiliary chemical used for pH regulation and the establishment of an alkaline environment in reactive dyeing. Sodium

hydroxide (NaOH) is a compound commonly used as caustic soda. Caustic soda increases the pH during the dyeing process, creating an alkaline environment and facilitating the interaction of the dye with the reactive groups. Caustic soda also helps ensure the optimal performance of reactive dyes during the dyeing process.



4. DYE STEP AND MACHINES USED IN THE DYEHOUSE

4.1. Package Dyeing

Bobbin dyeing refers to the process of dyeing yarn by winding it onto cylindrical bobbins. In the textile industry, both vertical and horizontal bobbin dyeing machines are used, but vertical machines are more commonly used. Although most textile books state that horizontal dyeing vessels are more advantageous than vertical vessels in terms of installation cost, low liquor ratio, and other factors, vertical dyeing vessels are actually more advantageous. This is because it is much easier to adjust the pressure of the circulating liquor from the carrier to the bobbin in vertical dyeing vessels. As a result, there is a lower risk of color variation between bobbins or differences in color intensity between the inner and outer parts of the bobbins. Therefore, vertical dyeing vessels offer a higher quality yarn dyeing capability compared to horizontal vessels. In vertical dyeing machines, which typically have a liquor ratio of approximately 1:8 to 1:12 depending on the product type, a cushion pressure of 1-3 bar is applied to ensure circulation of the liquor from the inside to the outside or from the outside to the inside of the bobbins, and the dyeing process takes place. The greatest advantage of bobbin dyeing is the ability to achieve high quantities of yarn dyeing with a low liquor ratio.

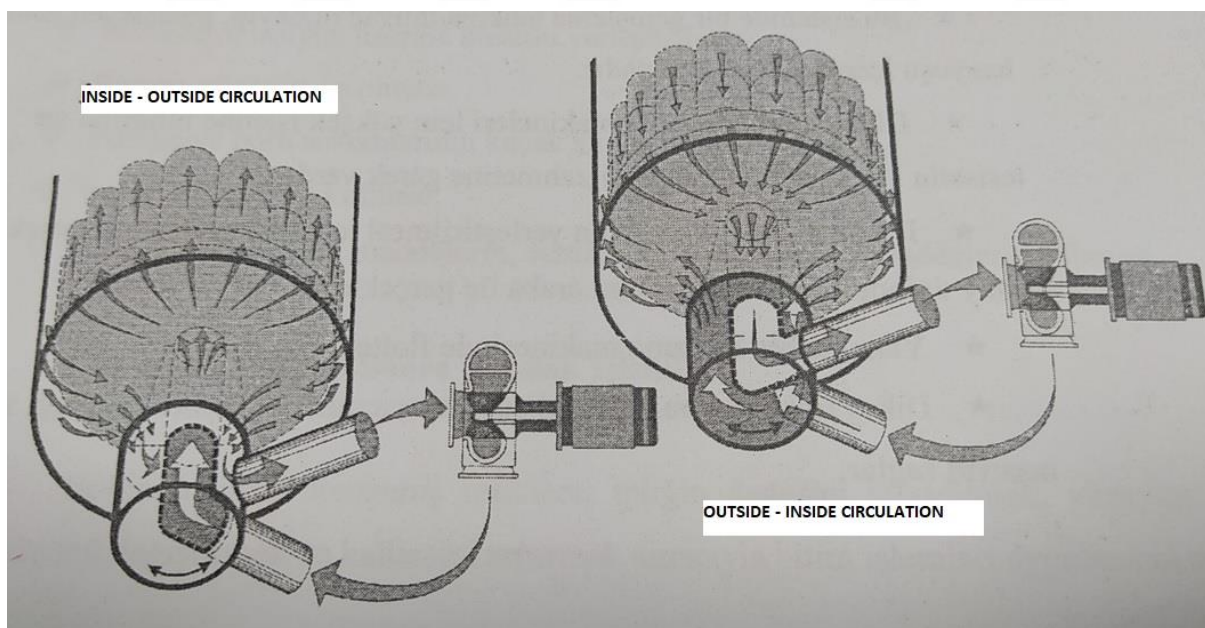


Figure 11. Circulation of Package Dyeing Machine (Bebekli, 2010)

In order to achieve successful bobbin dyeing, the density of the wound bobbins is crucial. Depending on the yarn quality, each winding must have a specific density and winding angle.

Operational experience suggests that suitable bobbin densities are approximately 0.270 g/cm³ for viscose, 0.450 g/cm³ for polyester, and 0.500 g/cm³ for acrylic. However, bobbin density is not the only important factor; during winding, the movement of the yarn back and forth on the traveler, including the number of turns per stroke, where the next turn will start compared to the previous turn, and the angle between the yarns during each stroke, is also crucial. The applied winding techniques vary depending on the type of yarn, the target market for the product, and the quality requirements of the final product. A smooth and soft winding facilitates the dyeing process. To ensure that the liquor passes through the bobbins with equal pressure, it is necessary to have bobbins wound with equal densities from the bottom to the top of the carrier. If the liquor is evenly distributed with homogeneous pressure to all bobbins, no quality issues will occur during dyeing.

4.1.1. Bobbin Squeezing and Drying

In many facilities, squeezing is carried out using a centrifugal squeezing vessel in a circular shape. The bobbins are placed in a perforated basket within the vessel and rotated at a certain speed, allowing the water inside the bobbins to be expelled through the centrifugal force. Each fiber has different water retention capabilities, so the water expulsion process occurs at different speeds and durations. The most important aspect of bobbin squeezing is to ensure that the bobbins placed in the basket have equal weights. If a carrier has been left outside for a certain period of time, the weights of the top and bottom bobbins will become different. This is because the drained water will flow downward, causing a difference in weight between the bottom and top bobbins. Therefore, when placing the bobbins in the squeezing basket, it is advisable to progress from the top bobbins to the bottom bobbins in equal order, rather than individually placing each bobbin. The more homogeneously the water is expelled from the bobbins, the more effective the drying process will be.

For bobbin drying, radio frequency drying machines are commonly used in the industry. This is primarily due to their high drying capacity and lower cost compared to other drying methods. In a radio frequency drying machine, there are electrodes in the top and bottom parts, and when wet bobbins enter the drying chamber through a conveyor, they pass between these electrodes. The water between the electrodes creates an electric current in the chamber, causing the water inside the bobbins to move. Due to the friction between the water droplets, heat is generated, and after a while, the water turns into steam and is expelled through a chimney in the chamber. The emitted steam can be toxic, so regular emission measurements should be carried out. While

the machine is running, a significant amount of magnetic current is generated within the drying chamber. In fact, there is a fluorescent lamp in the chamber that does not have any electrical connections, and due to the magnetic current, the lamp stays continuously lit. Additionally, there is an oscillator chamber that contains two plates arranged at certain distances from each other. These plates move closer and farther apart, causing the power generated by the electrodes to increase or decrease.

Viscose, by nature, retains approximately 65% of its weight in water, while acrylic retains about 10%. Since viscose is not significantly affected by electrical methods during drying due to its nature, water expulsion, acrylic being a petroleum-based fiber, is highly susceptible to combustion. Therefore, the drying parameters should vary for each type of fiber. For example, under typical operating conditions, acrylic is dried with a power of 60 kW, viscose with 70 kW, and cotton with 40-60 kW. However, since the amount of water in acrylic fiber within a given area is much lower than that in viscose or cotton, the drying process can be completed more quickly, resulting in a shorter time spent in the chamber. This reduces the risk of combustion. During drying, the most important factor is to have equal amounts of water in the bobbins on the conveyor. If there are bobbins with different water amounts, the water content per unit area will increase, leading to higher kW consumption and increased vibration of the water. As a result, bobbins with a higher water content will not withstand the frictional force after a while, and the yarn may start to burn. Therefore, attention should be paid to the water weights of the bobbins. Additionally, it is advisable to install a metal detector at the entrance of the drying conveyor in yarn drying facilities to ensure safety, as the presence of a conductive substance within the fiber could lead to a fire.

4.2. Hank Dyeing

In the industry, hank dyeing is generally carried out using either pressurized closed hank dyeing vessels or non-pressurized open hank dyeing vessels. Hank dyeing vessels without cushion pressure, where yarn dyeing is performed, are referred to as "arm dyeing vessels" in the industry. In closed hank dyeing vessels, a cushion pressure of approximately 0.5-1 bar is applied inside the vessel, and a circulation pump circulates the liquor from the inside to the outside or from the outside to the inside of the vessel. The liquor continuously circulates from the bottom to the top of the hanks and from the top to the bottom, aiming to achieve a homogeneous dyeing. Several important points to consider include:

Ensuring that the hanks are evenly distributed on the carrier rods in terms of weight and density.

Placing a breakwater at the point where the circulation pump applies pressure to the liquor to prevent damage to the hanks.

Tying the hanks at specific intervals with a hank knot during the transfer process after dyeing to avoid excessive breakage.

In the system where dyeing is performed in arm dyeing vessels without cushion pressure, hanks are placed on the arms with equal densities. The arms are triangular in shape within the vessel, with one surface containing holes. The rotation speed and duration of the arms can be adjusted parametrically to allow the hanks to move while the dyeing process takes place using the liquor flowing out through the holes.

The main advantage of arm dyeing machines compared to closed hank dyeing vessels is the ability to dye with a low liquor ratio. This makes reactive dyeing processes easier and more repeatable. However, there are some limitations to consider. Due to the open nature of the vessel, yarn dyeing with recipes containing harmful chemicals is not suitable. Additionally, since there is no pressure inside the vessel, high-temperature dyeing, such as for polyester, cannot be performed as the temperature cannot exceed 100°C.

5. MATERIALS AND METHODS

For reactive dyeing trials, cotton will be used, and for cationic dyeing trials, acrylic fiber will be used. The main reason for choosing cotton is that it leaves more unfixed dye in the bath compared to viscose. If the discharge effluents are successful in re-dyeing cotton, similar results can be expected for viscose as well. The cotton trials will involve testing with black color. This is primarily because black is the most preferred color in the knitwear market worldwide. Many textile dye houses have the highest discharge volume from the effluents of black dyeing. Since a significant amount of about 30% of unfixed dye is released in the bath, a substantial amount of washing is required. If the discharged effluents from black dyeing can be reused for washing, the usage of clean water can be reduced by around 40%. In the case of acrylic fiber, the main concern regarding the reuse of discharged effluents for cationic dyeing lies in the presence of retarder in the bath. For example, while retarder is not used in black or dark colors, a high amount of retarder is used in light colors. Therefore, when light-colored dyes are discharged, the presence of retarder in the bath will be observed, and a decision will be made on whether it can be used for re-dyeing.

5.1. Cotton Dyeing Method

For the cotton dyeing experiment, five consecutive dyeings will be conducted. In each dyeing experiment, the initial bath containing the pre-treatment and dye solution will be clean water. For the five consecutive dyeing experiments, the following sequence of discharge waters will be used: In the first dyeing experiment, clean water will be used throughout. In the second dyeing experiment, the discharge water from the first dyeing experiment will be used in the five washing baths. In the third dyeing experiment, the discharge water from the first and second dyeing experiments will be used in the six washing baths. In the fourth dyeing experiment, the discharge water from the first, second, and third dyeing experiments will be used in the six washing baths. And in the fifth dyeing experiment, some of the discharge waters from the previous experiments will be used in the six washing baths. The sequence of the discharge waters to be used is provided in the table below.

1. Dyeing Experiment: Clean water
2. Dyeing Experiment: Discharge water from the first dyeing experiment (5 washing baths)

3. Dyeing Experiment: Discharge water from the first and second dyeing experiments (6 washing baths)
4. Dyeing Experiment: Discharge water from the first, second, and third dyeing experiments (6 washing baths)
5. Dyeing Experiment: Some of the discharge waters from the previous experiments (6 washing baths)

By following this approach, a combination of discharge waters and clean water will be used for the five dyeing experiments. This method helps reduce water consumption, minimizing environmental impact, and promotes more efficient use of water resources.

Table 1. Dyeing Trials

		1. DYEING TRIAL	2. DYEING TRIAL	3. DYEING TRIAL	4. DYEING TRIAL	5. DYEING TRIAL
1. BATCH	PRETREATMENT	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER
2. BATCH	DYEING	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER
3. BATCH	WASHING	FRESH WATER	1. DYEING 4. BATCH	1. DYEING 6. BATCH	1. DYEING 7. BATCH	1. DYEING 8. BATCH
4. BATCH	WASHING	FRESH WATER	1. DYEING 6. BATCH	1. DYEING 7. BATCH	1. DYEING 8. BATCH	1. DYEING 9. BATCH
5. BATCH	ACID WASHING	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER
6. BATCH	WASHING	FRESH WATER	1. DYEING 7. BATCH	1. DYEING 8. BATCH	1. DYEING 9. BATCH	1. DYEING 11. BATCH
7. BATCH	WASHING	FRESH WATER	1. DYEING 8. BATCH	1. DYEING 9. BATCH	1. DYEING 11. BATCH	2. DYEING 11. BATCH
8. BATCH	WASHING	FRESH WATER	1. DYEING 9. BATCH	1. DYEING 11. BATCH	2. DYEING 11. BATCH	3. DYEING 11. BATCH
9. BATCH	WASHING	FRESH WATER	1. DYEING 11. BATCH	2. DYEING 11. BATCH	3. DYEING 11. BATCH	4. DYEING 11. BATCH
10. BATCH	ACID WASHING	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER
11. BATCH	WASHING	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER
12. BATCH	SOFTENING	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER	FRESH WATER

During the cotton dyeing experiments, pH, conductivity (uS/cm), hardness (FR), P alkalinity (mg/L), M alkalinity (mg/L), iron (mg/L), and color (Pt-Co) analyses were conducted at different durations of the washing baths. These analyses were compared to the initial values and pre-drainage values of unfixed dye in the washing bath to examine if there were any differences. As a result of the examination, based on the samples taken from the third, fourth, sixth, seventh, eighth, ninth, and eleventh baths:

There were no major changes observed in pH, conductivity, hardness, and alkalinity values. However, when looking at the color analysis, it was observed that as the drainage time approached the final washing baths, there was an increase in the unfixed dye content in the bath.

Furthermore, there was a significant decrease in the amount of unfixed dye in the washing bath as it approached the end. According to the analysis results, it is believed that the unfixed dye content in the water significantly decreased from the sixth bath onwards, and the washing baths can be used for subsequent dyeing processes.

Table 2. First Dyeing Bath Analysis Results

BATCHS	WASHING MIN.	PH	CONDUCTIVITY (uS/cm)	HARDNESS (FR)	P ALKALINITY (mg/L)	M ALKALINITY (mg/L)	IRON (mg/L)	COLOR (Pt-Co)
3. BATCH	9°	11,08	35800	0,09	1250	2500	0	23600
	15°	11,08	36700	0,09	1250	2450	0	22200
4. BATCH	9°	10,84	9080	0,08	310	980	0	11880
	15°	10,87	9440	0,09	309	1008	0	10840
6. BATCH	9°	8,62	1235	0,08	6	250	0	3880
	15°	8,65	1283	0,09	6	260	0	3750
7. BATCH	9°	8,61	963	1	5	170	0	1018
	15°	8,62	974	1	5	175	0	1410
8. BATCH	9°	9,32	999	0,09	15	200	0	654
	15°	8,86	930	0,09	11	180	0	696
9. BATCH	9°	9,06	941	0,09	5	177	0	385
	15°	8,99	927	1	5	168	0	471
11. BATCH	9°	8,02	910	1	0	175	0	126
	15°	7,83	912	1	0	176	0	226

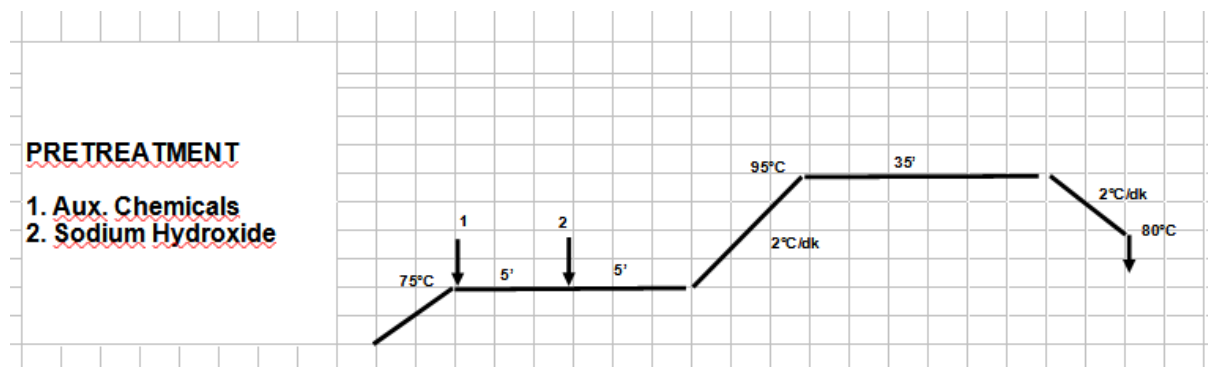


Figure 12. Pretreatment of Cotton Dyeing

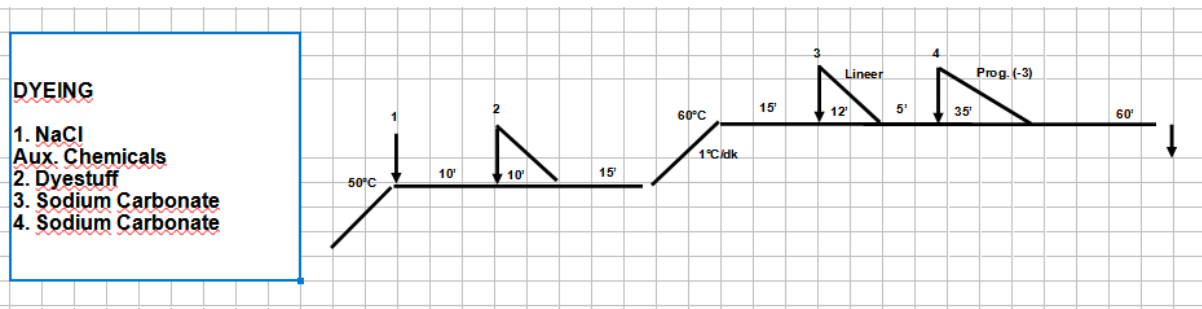


Figure 13. Dyeing Step of Cotton Dyeing for Black Color

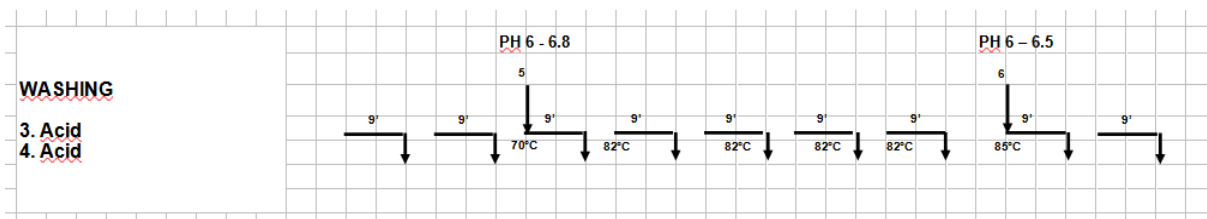


Figure 14. Washing Step of Cotton Dyeing for Black Collor

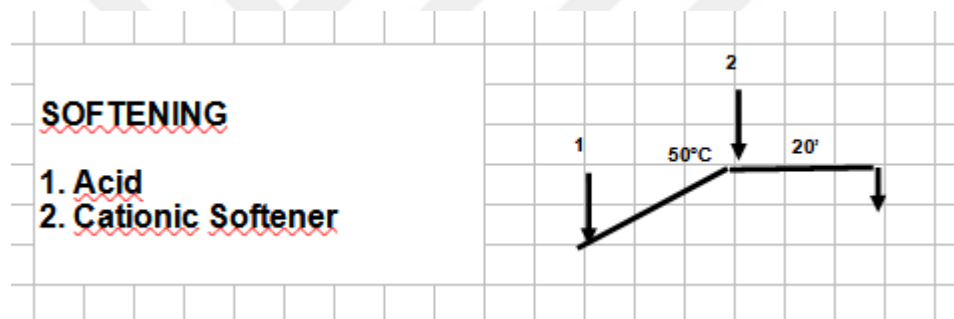


Figure 15. Softening Processes of Cotton Dyeing

 000000000592770	Reçete No : VFU907-VTL-B-110629 Dosya No : VFU907-VTL-B-110629 Prog.No : 240090
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Reçete Açıklama :

S. No	B/K	İ. No	Ürün Kodu	Br.Miktar	B.Ö.Br	Idx
10	K	21	TUZ NaCl	60,000000	GR/LT	☒
20	K	21	ALBATEX® DBS	0,250000	GR/LT	☒
30	B	23	SETAZOL GOLDEN YELLOW NG CONZ	0,443300	%	☒
40	B	23	SETAZOL RED NG CONZ	0,469300	%	☒
50	B	23	SETAZOL BLACK SDN	4,520000	%	☒
60	K	25	SODA Na ₂ CO ₃	1,500000	GR/LT	☒
70	K	27	SODA Na ₂ CO ₃	13,500000	GR/LT	☒
80	K	27	SODYUM HİDROKSİT ÇÖZELTİSİ (SIVI)	2,200000	GR/LT	☒
90	K	29	ASETİK ASİT CH ₃ COOH	0,600000	GR/LT	☒
100	K	31	ASETİK ASİT CH ₃ COOH	0,250000	GR/LT	☒

Figure 16. Dyeing Recipe



Figure 17. Cotton Dyeing Trials

5.2. Acrylic Dyeing Method

Although there are different types of acrylic dyeing processes, acrylic dyeings in yarn dyeing mills are often carried out in a single bath. This bath may contain various chemicals such as retarder, softener if needed, dye, and leveling agent. Acrylic dyeings are typically performed between 98-104°C. The biggest difference that sets acrylic dyeing apart from other fibers is that while many fibers shrink when exposed to hot water, acrylic exhibits elongation. Therefore, the critical point in acrylic dyeing is the hardness and shape of the dye package. In yarn dyeing mills, acrylic is usually dyed in conical-shaped packages. The main reason for this

is to prevent the yarn from flowing downward when the fiber starts elongating due to heat. In a conical-shaped package, even if the yarn elongates, it cannot flow downward due to gravity.

Samples will be taken from various points within the baths of the acrylic dyeing experiments and analyzed in the laboratory. Samples will be taken from the flotte immediately before dyeing after chemical dosage, immediately after dye dosage, and just before draining the bath after the dyeing process is completed. These samples will be analyzed to determine the amount of retarder present. Chromatogram analysis will be conducted to examine whether there is any residue of retarder in the wastewater from the acrylic dye bath. Two consecutive dyeing experiments will be performed, and the wastewater from the first dyeing will be used in the bath of the second dyeing. There will be no changes in the chemical quantities in the second dyeing experiment.

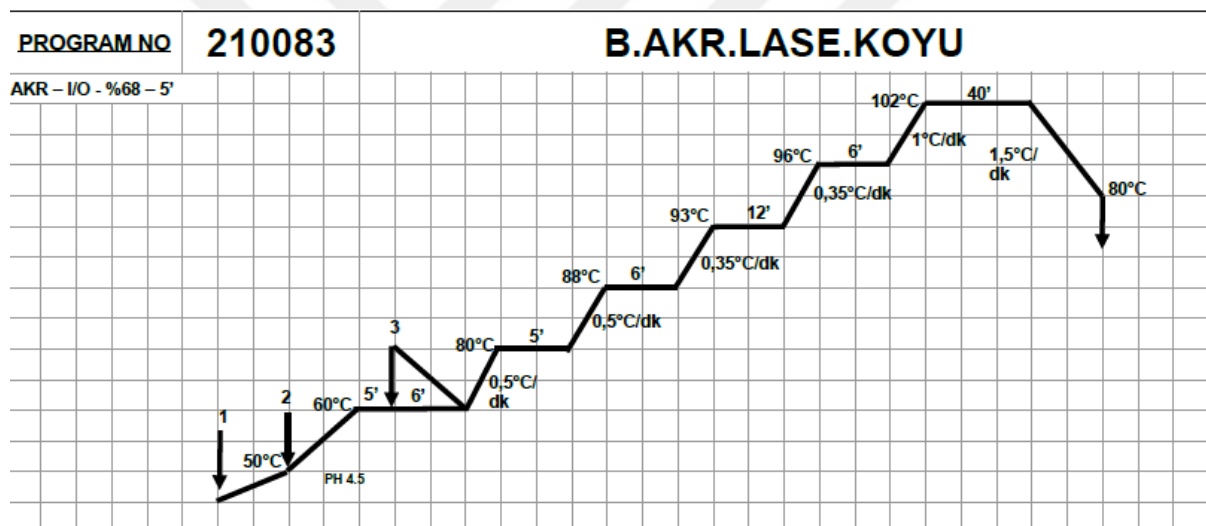


Figure 18. Acrylic Dyeing Processes

1. Acid
2. Retarder and Levelling Agent
3. Dyestuff

6. RESULTS AND DISCUSSIONS

During the experiments, the use of waste water was introduced for the first time in the second dyeing process, and the results were analyzed. When looking at the column indicating the unfixed dye ratio in the used waste waters, it can be observed that the unfixed dye ratio in the waste water increased by 169% in the third bath of the second dyeing compared to the previous amount.

In parallel with this, as can be seen in the tables:

The waste water obtained from the sixth bath of the first dyeing, when used in the fourth bath of the second dyeing, resulted in a 95% increase in the unfixed dye ratio.

The waste water obtained from the seventh bath of the first dyeing, when used in the sixth bath of the second dyeing, resulted in a 231% increase.

The waste water obtained from the eighth bath of the first dyeing, when used in the seventh bath of the second dyeing, resulted in a 250% increase.

The waste water obtained from the ninth bath of the first dyeing, when used in the eighth bath of the second dyeing, resulted in a 187% increase.

Although these increases may appear significant in terms of percentage, when looking at the numerical value of color measurement in Pt-Co units, it is still interpreted as being at usable levels for dyeing.

When the waste water obtained from the eleventh bath of the first dyeing was used in the ninth bath of the second dyeing, the water quantities were observed to be equal. However, in the test result, a negative percentage value is shown. Since most of the unfixed dye has already been removed from the fiber by this point in the process, the values appearing in this way were not considered abnormal.

Table 3. Second Dyeing Trial

		SECOND DYEING TRIAL	PH	CONDUCTIVITY (uS/cm)	HARDNESS (FR)	P ALKALINITY (mg/L)	M ALKALINITY (mg/L.T)	IRON (mg/L)	COLOR (Pt-Co)
1. BATCH	PRETREATMENT	FRESH WATER	-	-	-	-	-	-	-
2. BATCH	DYEING	FRESH WATER	7,41	-	-	-	-	-	-
3. BATCH	WASHING	1. DYEING 4. BATCH	11,28	38,3	0,9	1300	2600	0	32000
4. BATCH	WASHING	1. DYEING 6. BATCH	10,93	99,7	0,9	310	1021	0	7300
5. BATCH	ACID WASHING	FRESH WATER	6,58	-	-	-	-	-	-
6. BATCH	WASHING	1. DYEING 7. BATCH	8,08	1268	0,8	0	245	0	4670
7. BATCH	WASHING	1. DYEING 8. BATCH	8,81	979	1	7	180	0	2435
8. BATCH	WASHING	1. DYEING 9. BATCH	8,89	938	0,9	10	179	0	1350
9. BATCH	WASHING	1. DYEING 11. BATCH	8,62	923	0,9	6	178	0	68
10. BATCH	ACID WASHING	FRESH WATER	8,45	-	-	-	-	-	-
11. BATCH	WASHING	FRESH WATER	7,7	910	1	0	170	0	152
12. BATCH	SOFTENING	FRESH WATER	5,7	-	-	-	-	-	-

The percentage increases observed after using the waste water in subsequent dyeing processes are as follows:

The waste water from the sixth bath of the first dyeing process showed a 817% increase when used in the third bath of the second dyeing process.

The waste water from the seventh bath of the first dyeing process showed a 1049% increase when used in the fourth bath of the second dyeing process.

The waste water from the eighth bath of the first dyeing process showed a 616% increase when used in the sixth bath of the second dyeing process.

The waste water from the ninth bath of the first dyeing process showed a 484% increase when used in the seventh bath of the second dyeing process.

The waste water from the eleventh bath of the first dyeing process showed a 632% increase when used in the eighth bath of the second dyeing process.

The waste water from the eleventh bath of the second dyeing process showed a 361% increase when used in the ninth bath of the third dyeing process.

Table 4. Third Dyeing Trial

		THIRD DYEING TRIAL	PH	CONDUCTIVITY ($\mu\text{S/cm}$)	HARDNESS (FR)	P ALKALINITY (mg/L)	M ALKALINITY (mg/L)	IRON (mg/L)	COLOR (Pt-Co)
1. BATCH	PRETREATMENT	FRESH WATER	-	-	-	-	-	-	-
2. BATCH	DYEING	FRESH WATER	7,4	-	-	-	-	-	-
3. BATCH	WASHING	1. DYEING 6. BATCH	11,39	37800	1	1310	2530	0	34400
4. BATCH	WASHING	1. DYEING 7. BATCH	11,05	9530	0,9	325	1010	0	16200
5. BATCH	ACID WASHING	FRESH WATER	6,58	-	-	-	-	-	-
6. BATCH	WASHING	1. DYEING 8. BATCH	8,42	1306	0,9	6	260	0	4980
7. BATCH	WASHING	1. DYEING 9. BATCH	8,77	975	1	8	179	0	2750
8. BATCH	WASHING	1. DYEING 11. BATCH	8,76	929	1	8	178	0	1655
9. BATCH	WASHING	2. DYEING 11. BATCH	8,33	923	0,9	3	10	0	700
10. BATCH	ACID WASHING	FRESH WATER	6,45	-	-	-	-	-	-
11. BATCH	WASHING	FRESH WATER	7,58	879	1	0	160	0	126
12. BATCH	SOFTENING	FRESH WATER	5,5	-	-	-	-	-	-

The percentage increases observed after using the waste water in subsequent dyeing processes are as follows:

The waste water from the seventh bath of the first dyeing process showed a 2226% increase when used in the third bath of the fourth dyeing process.

The waste water from the eighth bath of the first dyeing process showed a 2845% increase when used in the fourth bath of the fourth dyeing process.

The waste water from the ninth bath of the first dyeing process showed a 1501% increase when used in the sixth bath of the fourth dyeing process.

The waste water from the eleventh bath of the first dyeing process showed a 1294% increase when used in the seventh bath of the fourth dyeing process.

The waste water from the eleventh bath of the second dyeing process showed a 1097% increase when used in the eighth bath of the fourth dyeing process.

The waste water from the eleventh bath of the third dyeing process showed a 463% increase when used in the ninth bath of the fourth dyeing process.

Table 5. Fourth Dyeing Trial

		FOURTH DYEING TRIAL	PH	CONDUCTIVITY ($\mu\text{S}/\text{cm}$)	HARDNESS (FR)	P ALKALINITY (mg/L)	M ALKALINITY (mg/L)	IRON (mg/L)	COLOR (Pt-Co)
1. BATCH	PRETREATMENT	FRESH WATER	-	-	-	-	-	-	-
2. BATCH	DYEING	FRESH WATER	7,37	-	-	-	-	-	-
3. BATCH	WASHING	1. DYEING 7. BATCH	11,5	35600	1	1380	2300	0	32800
4. BATCH	WASHING	1. DYEING 8. BATCH	9,76	8510	1	270	980	0	20500
5. BATCH	ACID WASHING	FRESH WATER	6,73	-	-	-	-	-	-
6. BATCH	WASHING	1. DYEING 9. BATCH	8,45	1251	0,9	4	240	0	7540
7. BATCH	WASHING	1. DYEING 11. BATCH	8,99	978	1	10	186	0	3150
8. BATCH	WASHING	2. DYEING 11. BATCH	9,05	919	1	12	175	0	1820
9. BATCH	WASHING	3. DYEING 11. BATCH	8,89	899	0,95	10	168	0	710
10. BATCH	ACID WASHING	FRESH WATER	6,4	-	-	-	-	-	-
11. BATCH	WASHING	FRESH WATER	7,69	883	1	0	165	0	133
12. BATCH	SOFTENING	FRESH WATER	5,6	-	-	-	-	-	-

The percentage increases observed after using the waste water in subsequent dyeing processes are as follows:

The waste water from the eighth bath of the first dyeing process showed a 4455% increase when used in the third bath of the fifth dyeing process.

The waste water from the ninth bath of the first dyeing process showed a 3807% increase when used in the fourth bath of the fifth dyeing process.

The waste water from the eleventh bath of the first dyeing process showed a 2502% increase when used in the sixth bath of the fifth dyeing process.

The waste water from the eleventh bath of the second dyeing process showed a 1296% increase when used in the seventh bath of the fifth dyeing process.

The waste water from the eleventh bath of the third dyeing process showed a 1567% increase when used in the eighth bath of the fifth dyeing process.

The waste water from the eleventh bath of the fourth dyeing process showed a 524% increase when used in the ninth bath of the fifth dyeing process.

Table 6. Fifth Dyeing Trial

		FIFTH DYEING TRIAL	PH	CONDUCTIVITY (uS/cm)	HARDNESS (FR)	P ALKALINITY (mg/L)	M ALKALINITY (mg/L)	IRON (mg/L)	COLOR (Pt-Co)
1. BATCH	PRETREATMENT	FRESH WATER	-	-	-	-	-	-	-
2. BATCH	DYEING	FRESH WATER	7,39	-	-	-	-	-	-
3. BATCH	WASHING	1. DYEING 8. BATCH	11,51	28000	1	1600	2200	0	31700
4. BATCH	WASHING	1. DYEING 9. BATCH	10,9	7210	1	390	858	0	18400
5. BATCH	ACID WASHING	FRESH WATER	6,73	-	-	-	-	-	-
6. BATCH	WASHING	1. DYEING 11. BATCH	8,3	1211	0,85	0	210	0	5880
7. BATCH	WASHING	2. DYEING 11. BATCH	8,81	958	1	7	180	0	3080
8. BATCH	WASHING	3. DYEING 11. BATCH	8,98	902	1	8	175	0	2100
9. BATCH	WASHING	4. DYEING 11. BATCH	8,71	899	0,9	7	172	0	830
10. BATCH	ACID WASHING	FRESH WATER	6,79	-	-	-	-	-	-
11. BATCH	WASHING	FRESH WATER	8,16	856	1	0	160	0	195
12. BATCH	SOFTENING	FRESH WATER	5,6	-	-	-	-	-	-

According to the results of the dyeing experiments, the samples taken from the yarn underwent durability tests and received passing grades. The wet durability scores ranged from 3 to 4 on the grey scale, while the dry durability scores ranged from 4 to 5. The washing durability tests showed that the dyed samples did not stain the microfiber cloth and also received passing grades.

After the durability tests, the dyed samples were knitted on a circular knitting machine and then compared to the reference color using a spectrophotometer. The dL values under D65 and TL84 illuminants were examined, and no significant color changes were observed. All dyeings remained below the threshold of 1.0 dL. Based on these findings, it was determined that approximately 40% of the waste water from reactive dyeings could be discharged into a separate tank instead of the waste water pool and reused in future reactive dyeing processes.

In the thesis study, black color, which is the most comprehensive color worldwide, was deliberately chosen. Considering the rapid depletion of resources globally, implementing such a practice even for black and very dark colors alone would be beneficial.

FASTNESS

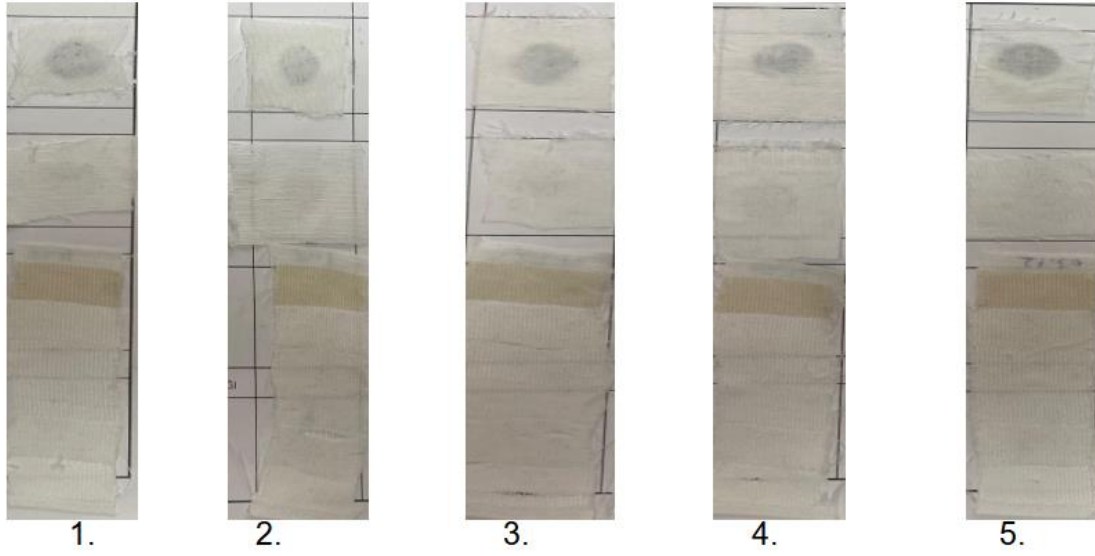


Figure 19. First and Second Trial Fastness Results

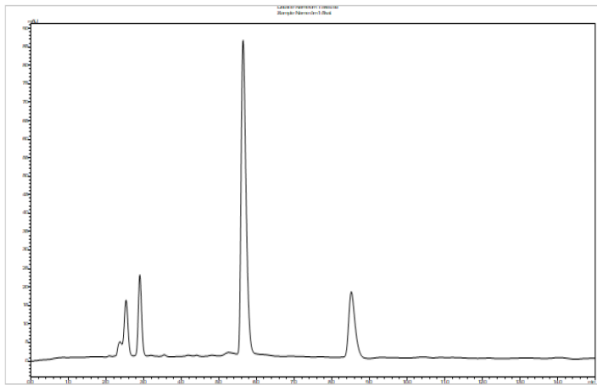
Standart İsmi:		L*	a*	b*	C*	h°		
4002/990010-İ.E:582325		14.14	1.25	-2.04	2.39	301.43		
		13.82	0.03	-2.66	2.66	270.54		
Numune Adı	DL*	Da*	Db*	DC*	DH*	DEcmc	P/F DEcmc	
29.11.21	0.24 L	0.01	-0.44 B	0.39 B	-0.21 B	0.62	Geçer	
TL84-10	0.18 L	-0.16 G	-0.56 B	0.56 B	-0.15 G	0.75		
30.11.21	0.17 L	0.00	-0.56 B	0.50 B	-0.27 B	0.75	Geçer	
TL84-10	0.08 L	-0.23 G	-0.73 B	0.74 B	-0.21 G	0.96		
01.12.21	0.46 L	0.01	-0.49 B	0.44 B	-0.23 B	0.78	Geçer	
TL84-10	0.37 L	-0.21 G	-0.65 B	0.65 B	-0.19 G	0.92		
02.12.21	0.50 L	-0.08 G	0.21 Y	-0.22 D	0.04 R	0.57	Geçer	
TL84-10	0.49 L	-0.21 G	0.21 Y	-0.21 D	-0.22 G	0.62		
03.12.21	0.32 L	-0.03 G	-0.43 B	0.36 B	-0.23 B	0.63	Geçer	
TL84-10	0.25 L	-0.13 G	-0.55 B	0.55 B	-0.12 G	0.75		

Figure 20. Spectrophotometer Color Comparison by Reference Color

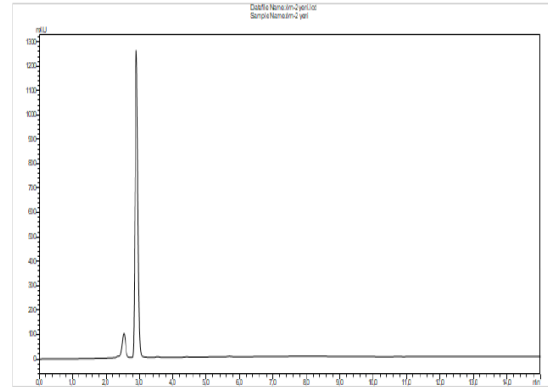
In the acrylic dyeing experiments, chromatogram results were examined. It was interpreted that the areas showing peaks on the graph indicated the presence of retarder and leveling agent. When looking at the test results of the first dyeing, a significant amount of retarder and leveling agent was detected in the bath after chemical and dye dosing. However, it was observed that almost all of the retarder and leveling agent were broken down and no residue remained in the bath after the dyeing process was completed.



RETARDER ANALİZ SONUCU



Şekil 1. 1 nolu örneğe ait kromatogram

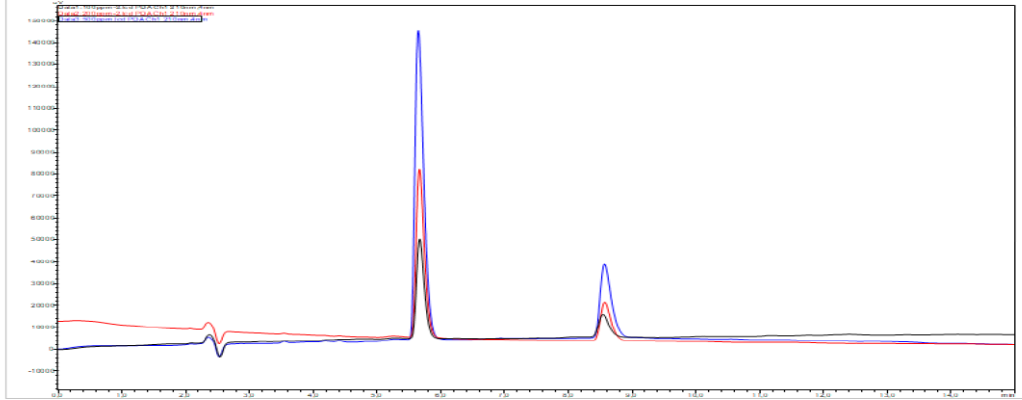


Şekil 2. 2 nolu örneğe ait kromatogram

ULUSOY TEKSTİL SAN. VE TİC. A.Ş.

Figure 21. First Trial Retarder Analysis Result

RETARDER ANALİZ SONUCU



Şekil 3. Standartların (100-200-500 ppm) kromatogramları

Numune	C12 (ppm)	Cx? (ppm)
1	1255,675	1068,93
2	saptanmadı	saptanmadı

ULUSOY TEKSTİL SAN. VE TİC. A.Ş.

Figure 22. First and Second Trial Retarder Analysis Result



Figure 23. First and Second Trial Fastness Results from Left to Right

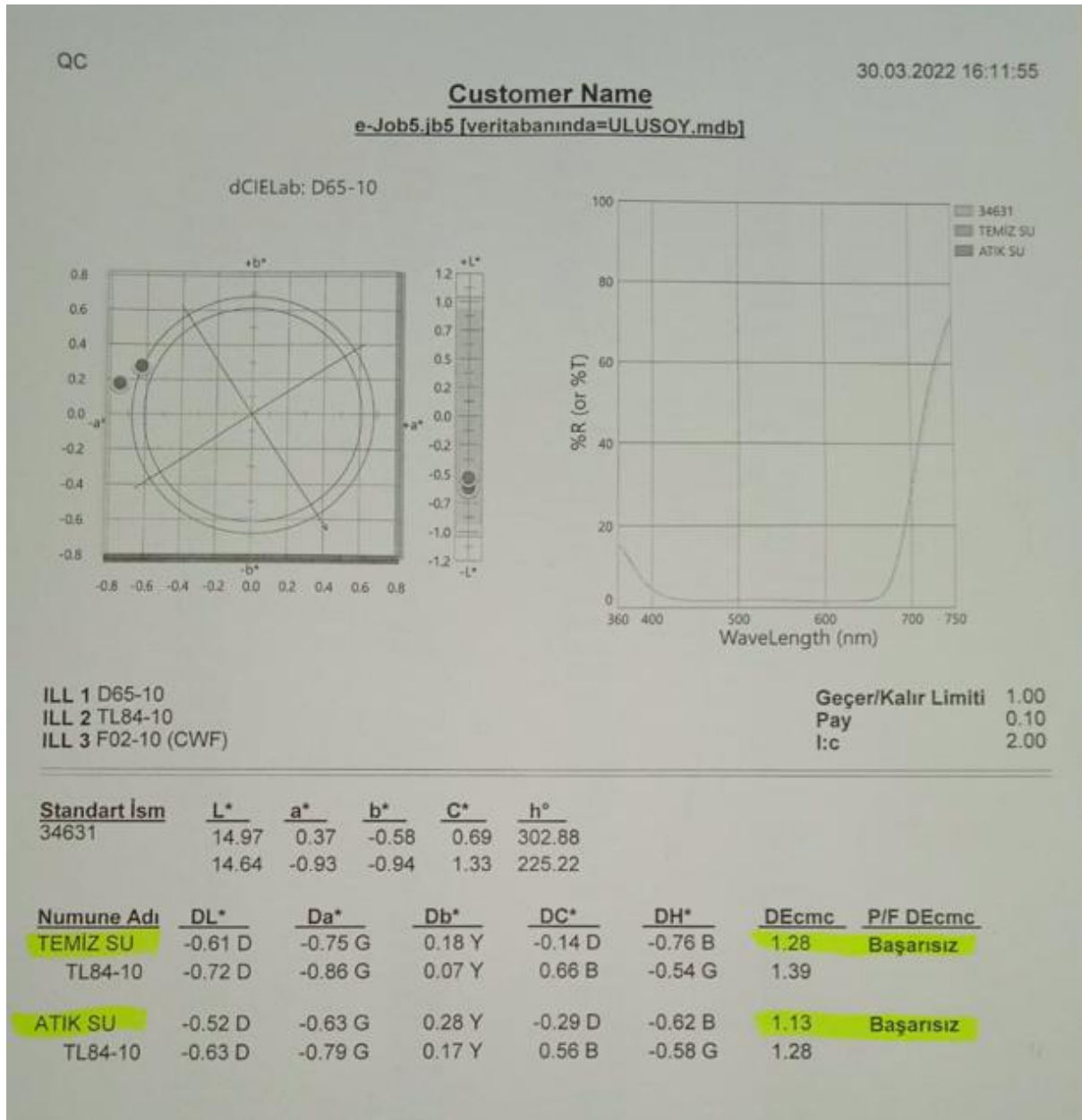


Figure 24. Spectrophotometer Color Comparison by Reference Color

However, in the second acrylic dyeing experiment, 100% of the water discharged from the first dyeing was used. Despite having excellent colorfastness values, when the color results were examined using a spectrophotometer, the dL values deviated from the reference color and remained in the conditional approval stage. This suggests that even if retarder and leveling agent were not detected in the bath during chromatogram analysis, the actual result of the

dyeing process indicates that the dye did not fully adhere to the fibers, leading to a deviation in color.

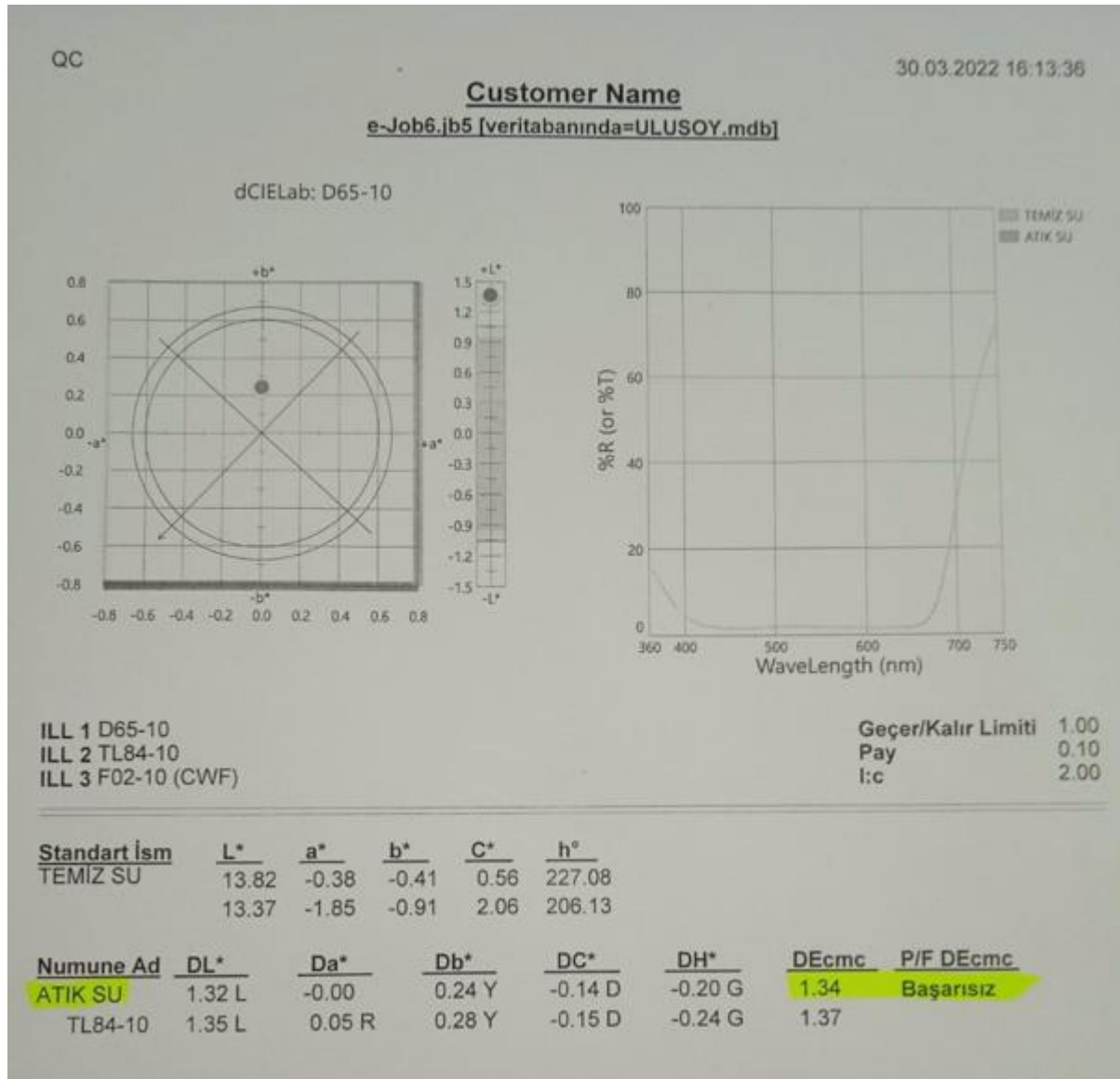


Figure 25. Spectrophotometer Color Comparison Between Trials

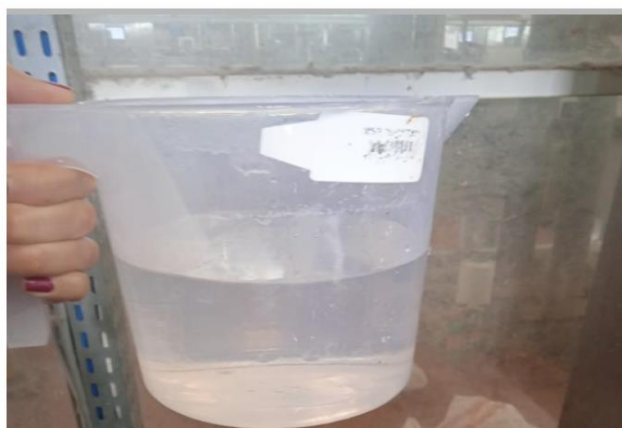


Figure 26. Wastewater After First Dyeing



7. CONCLUSION

It is estimated that approximately 20% of the clean water resources used in industrial settings are consumed by the textile sector. Even countries like Turkey, which have abundant water resources, need to use these resources wisely in order to sustainably transfer water to future generations. Recognizing the increasing importance of water, the European Union took a significant step in 2019 by implementing the Green Deal to reduce the water crisis and achieve the goal of "carbon neutrality" by 2050. Leading textile dyeing groups such as Inditex are researching products produced with technologies that reduce water consumption and testing the carbon footprint of the products they source from companies in terms of water consumption. Therefore, it is crucial for textile dyeing facilities to reuse wastewater in multiple processes and minimize water consumption.

To raise awareness and foster a sense of responsibility among employees regarding climate change, factories should undertake such projects and implement them. In addition to preserving natural resources, considering that the cost of reusing wastewater for a factory with a capacity of 20 tons/day of package dyeing is estimated to be around 60,000 USD, it is a project that will quickly amortize itself through the reduction of energy costs such as water and heating, as well as the reduction of unit dyeing costs. Moreover, a product with reduced carbon footprint and cost advantages has the potential to be preferred in the market. This, in turn, can motivate competitor companies to establish recycling systems, leading to a global improvement over the years.

For example, by establishing a cotton facility dedicated solely to black dyeing, which meets criteria such as Join Life and Care of Water and has a carbon footprint significantly below the market average, the unit dyeing and energy costs in a standard dyeing facility for black cotton processing, which is around 1.5 - 2 USD, can be reduced to the range of 0.5 - 1 USD. In an industry where there are facilities processing 30 tons/day of cotton, comprehensive reduction in water consumption can be achieved by implementing fast and simple solutions.

Overall, it is crucial for the textile industry to adopt practices that minimize water consumption and reduce their environmental footprint. By implementing sustainable dyeing processes,

raising awareness among employees, and investing in innovative technologies, the industry can contribute to the conservation of water resources and mitigate the impact of climate change.

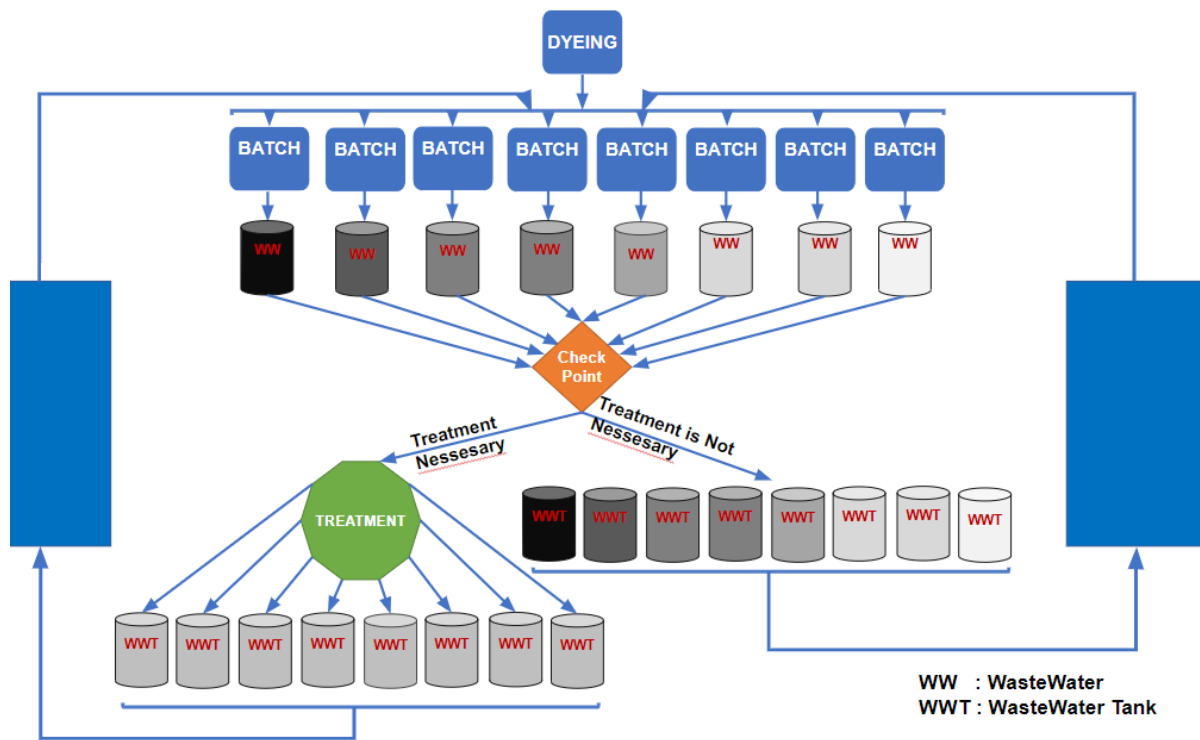


Figure 27. Treatment Diagram

The thesis study focused on directly using the wastewater obtained from cotton dyeing without undergoing any treatment process, by conducting specific analyses and directly dyeing sample dye vats. The dyeing experiments were conducted under the same standards as the industrial dyeing processes, without the need for laboratory dyeing, allowing for real results to be observed. Therefore, the thesis did not address wastewater treatment specifically. The study demonstrated that this wastewater could be used without any additional cost, as proven by laboratory analyses. If there is a need for treatment, simple treatment techniques can be employed to further enhance the process. For example, treatment membranes that convert hard water to soft water or perforated coils where second-quality decision coils are arranged can act as filters for the wastewater before it is discharged into the collection tank, thus establishing a cost-effective wastewater treatment facility. Additionally, alternative treatment methods, such as magnetic particle synthesis, which has not been previously utilized, can be explored, and different studies related to this can be observed. Magnetic particle synthesis, in its simplest form, utilizes a magnet to attract unfixed dyes and particles in the water, transferring the

purified clean water outside the system. After a while, the collected waste material takes on a sludge-like consistency and is sent to waste disposal facilities for disposal. This approach will reduce the amount of wastewater discharged by factories and provide cleaner water. By employing such wastewater treatment methods and others alike, the existing thesis study can be further improved.



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