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Advanced Evaporation and Filtration Techniques for Reclaiming High – Purity Salts from Reactive Dying Wastewater

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ABSTRACT: Cotton reactive dyeing continues to rely heavily on the use of large quantities of soluble inorganic salts such as sodium chloride and sodium sulphate to achieve optimum dyefibre fixation. Even with the emergence of low-salt dyeing technologies, commercial dyeing processes still generate effluents containing high total dissolved solids (TDS), accompanied by intense coloration and undesirable auxiliary chemicals. Discharge of such effluents poses significant environmental burdens, making recovery and reuse of salt a critical necessity for achieving sustainable Zero Liquid Discharge (ZLD) systems.

This study investigates the recovery, purification, and reuse of salt from dyehouse effluents through a series of membrane separation techniques followed by concentration and crystallization using multiple-effect evaporators, pusher centrifuge, and agitated thin-film dryer (ATFD) systems. Special emphasis is placed on removing extraneous components—particularly hardness-forming ions (Ca^{2+}, Mg^{2+}), residual reducing agents (e.g., sodium hydrosulphite), and other impurities—that adversely affect shade reproducibility in subsequent dyeing cycles. Purification of recovered salt was conducted through chelation and filtration techniques to improve its suitability for reuse. Comparative analysis reveals that salt regenerated via the ATFD method exhibits optimal purity, stable physicochemical properties, and highly consistent dyeing performance.

The ATFD system also provides operational advantages such as single-pass evaporation, low operating pressure, minimal film thickness, and high turndown ratio. The study concludes that salt recovery and reuse not only support ZLD compliance but also reduce environmental load and carbon footprint by substantially decreasing the need for fresh salt. Further research is recommended to enhance purification efficiency for salts recovered via pusher centrifuge and other methods.

KEYWORDS: Reactive dyeing, Salt recovery, Zero Liquid Discharge (ZLD), Total Dissolved Solids (TDS), Membrane separation, ATFD, Pusher centrifuge, Effluent treatment, Textile wastewater, Sustainable.

I. INTRODUCTION

Cotton reactive dyeing is one of the most preferred coloration methods in the textile industry because it provides excellent wash fastness, brilliant and vibrant shades, and high versatility in application. Despite these advantages, the process depends heavily on inorganic saltsprimarily sodium chloride (NaCl) and sodium sulphate (Na_2SO_4)to promote dye exhaustion and fixation by overcoming the electrostatic repulsion between negatively charged cotton fibres and anionic reactive dye molecules. The quantity of salt required is substantial, typically ranging from 50 to 120 g/L for light shades and from 80 to 160 g/L for medium and dark shades.

As a result, the dyeing process generates effluents with extremely high total dissolved solids (TDS often exceeding 10,000 ppm), deep and persistent coloration from unfixed dye molecules, and a mixture of metallic ions and organic auxiliaries such as surfactants, wetting agents, levelling agents, and sequestering chemicals. These contaminants make the effluent highly detrimental to the environment; if discharged untreated, they contribute to soil salinity, groundwater contamination, reduced agricultural productivity, and disruption of aquatic ecosystems. To address these concerns and meet increasingly strict environmental regulations, the textile industry is moving toward the implementation of Zero Liquid Discharge (ZLD) systems, which aim to eliminate wastewater discharge entirely by recovering and reusing water, salts, and other valuable components.

The need for salt recovery within a Zero Liquid Discharge (ZLD) framework arises from both environmental imperatives and economic pressures faced by the textile industry. Reactive dyeing processes generate large volumes of high-TDS effluent due to the extensive use of sodium chloride and sodium sulphate, and this saline wastewater cannot be directly discharged into natural water bodies without causing severe ecological damage, including soil salinization, groundwater contamination, and disruption of aquatic life. ZLD systems are designed not only to eliminate liquid effluent discharge but also to maximize the reuse of water and chemicals within the dyeing process.

However, without efficient salt recovery, the ZLD system becomes financially burdensome because purchasing fresh salt for every dyeing cycle significantly increases operating costs, and disposing of untreated salt-rich concentrate remains environmentally unsafe. Recovering salt from dyehouse effluent helps close the material loop by converting waste salt back into a usable resource, thereby reducing dependency on fresh salt extraction and lowering the overall chemical footprint of the process. Moreover, effective salt recovery enhances the sustainability of ZLD operations by reducing solid waste generation from evaporator crystallizers and improving overall process efficiency. Thus, salt recovery is a critical component of modern ZLD frameworks, enabling textile dyeing units to achieve both regulatory compliance and long-term environmental and economic sustainability

II. LITERATURE REVIEW

Reactive dyes are unique among dye classes because they form true covalent bonds with cellulose fibres. Through nucleophilic substitution or addition mechanisms, the reactive groups on the dye molecule chemically link with the hydroxyl groups of cellulose, creating a stable, permanent attachment. This covalent bonding is the key factor that sets reactive dyes apart from direct, vat, and other dye types, which rely primarily on weaker physical or secondary interactions. As a result, fabrics dyed with reactive dyes exhibit exceptional wash fastness, superior colour retention, and long-term stability, making them highly suitable for high-performance and durable cotton textiles.

Reactive groups play a crucial role in determining the fixation behaviour and overall performance of reactive dyes. Studies indicate that Mon chlorotriazine, Di chlorotriazine, vinyl sulfone, and hetero-bifunctional dyes exhibit distinct reactivity profiles based on the chemical nature of their functional groups. Highly reactive systems, such as Di chlorotriazine, achieve rapid fixation with cellulose but are more prone to hydrolysis, which can reduce dye utilization. In contrast, medium-reactive groups, including Mon chlorotriazine and vinyl sulfone, provide improved levelling and greater reproducibility during dyeing. Hetero-bifunctional dyes, combining two reactive moieties, balance reactivity and stability to enhance fixation efficiency and overall colour performance.

Cotton fibres acquire a negative surface charge when dispersed in an aqueous medium, and reactive dye molecules typically carry a similar negative charge. As documented in the literature, this like-charge interaction creates substantial electrostatic repulsion, which markedly reduces dye exhaustion during the dyeing process. To overcome this limitation, electrolytes such as sodium chloride or sodium sulphate are introduced to suppress the ionic repulsion by screening the negative charges on both fibre and dye. This salt-mediated reduction in electrostatic barriers enables more effective dye adsorption and promotes uniform fibre–dye interaction.

Effluents from textile dyeing operations frequently contain suspended fibres, unfixed dyes, inorganic particulates, and various forms of sludge. The presence of these materials substantially increases turbidity and can destabilize downstream treatment systems, particularly membrane-based processes such as ultrafiltration and reverse osmosis, by causing fouling, scaling, and reduced flux. To ensure efficient operation and prolong membrane lifespan, pre-treatment steps are often necessary. Techniques such as coagulation, flocculation, sedimentation, and pre-filtration are commonly employed to remove suspended solids and aggregates, thereby reducing turbidity, minimizing fouling potential, and improving the overall efficiency and reliability of the subsequent membrane treatment stage.

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III. RESEARCH METHODOLOGY

Pre-treatment is a critical phase in water and wastewater treatment designed to remove large particles, suspended solids, and selected dissolved substances prior to the main treatment processes. Initially, pH adjustment was carried out to correct the acidity or alkalinity of the effluent and optimize downstream reactions such as coagulation and disinfection. This was achieved by adding suitable acids like sulfuric acid or bases such as lime or sodium hydroxide to maintain the desired pH range. Following this, coagulation–flocculation was performed using chemicals such as aluminiumsulphate or ferric chloride, which neutralized particle surface charges and promoted the formation of larger flocs. Gentle mixing enhanced particle collisions and floc growth, improving settling efficiency.

Filtration was then carried out using media like sand, gravel, or activated carbon to remove remaining suspended solids. This step improved water clarity, reduced turbidity, and protected downstream systems. Membrane separation was subsequently applied as an advanced treatment stage to remove dissolved and fine impurities. Ultrafiltration removed colloids, bacteria, and high-molecular-weight compounds, while reverse osmosis operated under high pressure to reject dissolved salts and contaminants. The process produced purified permeate suitable for reuse and a concentrated brine stream for further processing.

The concentrated brine generated from reverse osmosis was treated through evaporation and crystallization to recover valuable salts. A Multiple-Effect Evaporator system was used to efficiently remove water in successive stages, utilizing vapor from one effect to heat the next and enhance energy efficiency. As concentration increased, the solution reached saturation, leading to crystallization of salts. The resulting slurry was processed in a pusher centrifuge, where centrifugal force enabled continuous separation of salt crystals from the mother liquor with reduced moisture content. The separated solids were then dried using an Agitated Thin-Film Dryer, where material was spread over a heated rotating surface to facilitate rapid moisture removal.

This ensured efficient heat transfer and minimized thermal degradation, producing dry and free-flowing salt crystals. Further purification was carried out using chelating agents such as EDTA or NTA to remove heavy metals, followed by activated carbon treatment to eliminate organic impurities and colorants. Microfiltration removed fine suspended particles, and ion exchange reduced hardness by replacing calcium and magnesium ions with sodium ions. The purified salts were characterized using analytical techniques such as titration, conductivity measurement, and moisture analysis. Finally, dyeing trials on cotton fabrics demonstrated that the recovered salts performed comparably to conventional industrial salts, confirming their suitability as sustainable alternatives in textile processing.

IV. RESULTS AND DISCUSSION

The comparison of salt quality obtained from different concentration and drying techniques Pusher Centrifuge, Multiple-Effect Evaporator (MEE), and Agitated Thin-Film Dryer (ATFD) highlights significant differences in purity, hardness, and contaminant levels. Salt produced by the Pusher Centrifuge exhibits moderate purity, with relatively high levels of hardness, organic impurities, and visible colour contamination. This is primarily because the centrifuge efficiently separates crystals from mother liquor but does not remove fine soluble impurities or trace organics. Salt obtained from the MEE shows high purity, with medium hardness and reduced organic content, and only slight color contamination; the multiple stages of evaporation allow partial removal of dissolved impurities while concentrating the brine. In contrast, salt processed through the ATFD achieves very high purity, minimal hardness, low organic content, and negligible color contamination.

The thin-film drying process under controlled heat ensures the formation of clean, free-flowing crystals and enhances the removal of residual moisture and impurities. These results indicate that ATFD-treated salts are superior for applications requiring high-quality, low-contaminant salts, while Pusher Centrifuge salts may require additional purification for industrial or food-grade use.

ATFD (Agitated Thin-Film Dryer) salt exhibits superior physical and functional characteristics compared to salts obtained by other concentration and drying methods. One of the key advantages is minimal caking, which results from the controlled thin-film drying process that prevents excessive crystal agglomeration and allows formation of free-flowing granules. This enhances handling, storage, and transportation without the need for additional anti-caking agents. The moisture content of ATFD salt is very low, typically below 1–2%, due to the high heat transfer efficiency of the thin-film dryer and the continuous removal of water as a thin layer. Low residual moisture not only improves shelf life but also reduces microbial growth and minimizes the risk of dissolution-related issues during storage.

Additionally, ATFD salt demonstrates excellent solubility characteristics, dissolving rapidly and uniformly in aqueous solutions, which is particularly important for industrial and textile applications where consistent ion availability is critical. Overall, the integration of AI techniques in IDS frameworks in 2024 significantly improves detection capabilities, adaptability, and interpretability. However, balancing detection accuracy, computational efficiency, and real-time responsiveness continues to be a focal research area.

V. CONCLUSION

Several recommendations for improving salt recovery and purification processes emerge from the study. First, for salts obtained via the Pusher Centrifuge, the implementation of advanced multi-stage purification, combining activated carbon treatment and ion exchange, is advised to effectively remove residual organics, colorants, and hardness-causing ions, thereby enhancing purity and dyeing performance. Second, the adoption of online monitoring systems for critical parameters such as hardness, purity, and moisture content is recommended to ensure consistent salt quality and facilitate real-time adjustments during processing.

Third, the development of low-energy Agitated Thin-Film Dryer (ATFD) systems could improve sustainability by reducing operational energy consumption while maintaining the high-quality, low-moisture characteristics of the salts. Fourth, further research is needed on the recovery of mixed salts from complex industrial effluents and understanding their behaviour in different textile processes, which would expand the applicability of recovered salts beyond simple sodium chloride or sulphate streams.

Finally, the exploration of emerging technologies such as membrane crystallization offers a promising alternative for energy-efficient salt recovery with precise control over crystal size and composition, potentially complementing or replacing traditional evaporation and drying techniques. Collectively, these recommendations aim to enhance process efficiency, sustainability, and industrial applicability of recovered salts.

VI. FUTURE WORK

1. Develop advanced multi-stage purification techniques combining activated carbon and ion exchange for higher salt purity.
2. Implement real-time online monitoring systems for parameters such as hardness, purity, and moisture content.
3. Optimize the performance of pusher centrifuges to improve solid–liquid separation efficiency and reduce residual moisture.
4. Design and develop low-energy Agitated Thin-Film Dryer (ATFD) systems to enhance energy efficiency.
5. Investigate recovery and purification of mixed salts from complex industrial effluents.
6. Study the behaviour and performance of recovered salts in various textile dyeing and finishing processes.
7. Explore membrane crystallization as an energy-efficient alternative for salt recovery and controlled crystal formation.
8. Improve membrane technologies (UF/RO) to reduce fouling and increase operational lifespan.
9. Conduct scale-up studies for industrial implementation of the integrated salt recovery process.
10. Perform life cycle assessment (LCA) and cost analysis to evaluate economic and environmental sustainability.

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