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Flocculation and Coagulation Process of Dye Wastewaters Cleaning Using Neutral Metal Salt and Multi-Layer Filtration

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Abstract: Handloom weaving has been a foundation of traditional textile production across India, supporting the livelihoods of rural artisans. The sector is mostly decentralized. The dyeing operations associated with this industry generate substantial volumes of wastewater, contributing to environmental pollution and water scarceness. This study aims to develop a cost-effective and eco-friendly decentralized wastewater treatment system personalized to the needs of handloom clusters. The research focuses on the treatment of sulphur and reactive dye wastewater, emphasizing colour removal and chemical oxygen demand (COD) reduction using a combination of coagulation/flocculation (CF) followed by multilayer membrane filtration. The study systematically evaluates the performance of neutral metal salts, including Magnesium chloride ($MgCl_2$), Aluminum sulphate ($Al_2(SO_4)_3$), Ferric chloride ($FeCl_2$), and Calcium carbonate ($CaCO_3$), to determine the most effective coagulant for optimal dye removal and pH neutralization. The proposed treatment approach aims to enable the safe reuse of treated water in the dyeing process, reducing freshwater dependency and promoting sustainability in the handloom sector. Findings indicate that integrating efficient coagulation-flocculation mechanisms with gravity-based filtration systems can provide a scalable and decentralized wastewater treatment solution for rural handloom clusters. The study underscores the potential of low-cost, environmentally sustainable water management practices, offering a viable alternative to conventional wastewater disposal methods in Impoverished regions.

Keywords: wastewater treatment, handloom industry, dye effluent, coagulation-flocculation, metal salts, COD reduction, colour removal, sustainability.

I. INTRODUCTION

The textile industry is a major contributor to global pollution, generating approximately 92 million tonnes of waste annually. Several factors drive these alarming figures, including overconsumption, manufacturing waste, lack of awareness, and greenwashing practices, among others (Madhav et al., 2018). Since the industrial revolution, the use of chemical dyes has been prevalent, and the unsupervised, untreated discharge of dye-laden wastewater into natural water bodies has led to serious health risks, sanitation challenges, and harm to aquatic ecosystems (Lu et al., 2015). Over time, the use of chemical dyes has also permeated the handloom sector. Although its environmental impact is smaller than that of the industrial sector, the handloom sector's disorganized structure and continued reliance on chemical dyes present significant concerns (Kant, 2012). Unlike the organized textile industry, which often allocates resources for wastewater treatment, the handloom sector lacks the infrastructure and funding to manage these pollutants. This has led to the degradation of groundwater quality and poses severe health risks to artisans, who often work without protective gear (Islam et al., 2022). Awareness about the environmental impact of chemical dye disposal among artisans in handloom clusters is often limited. Many artisans focus primarily on the immediate demands of their craft and livelihood, and as a result, sustainability practices, such as safe disposal of chemical dyes, may not be a priority (Gupta et al., 2017). Although some are aware of the harmful effects these dyes can have on local water bodies and soil, the lack of accessible waste management systems and affordable alternatives discourages the adoption of eco-friendly methods. In most cases, artisans dispose of the used chemical dye baths—containing caustic soda (NaOH), hydrogen peroxide(H_2O_2), and other chemicals—by directly discharging them onto open land or nearby water sources (Azizullah et al., 2011). This practice, though common, leads to soil degradation and water contamination, harming the surrounding environment and agricultural land. Without proper waste treatment facilities, these hazardous chemicals often accumulate in the ecosystem, posing long-term risks to both human health and biodiversity (Mishra et al., 2018). The reality of dye disposal reflects a complex interplay of awareness, indifference, and helplessness. Some dyers do understand the harmful consequences of using chemical dyes and the damage caused by irresponsible disposal. However, despite their awareness, they lack the resources or infrastructure to implement better disposal methods. Without government or organizational intervention, they remain trapped in a cycle of harmful practices out of necessity. While some on other hand lack the knowledge of dye disposal and some do not prioritize proper dye disposal, reflecting the need for both education and tangible support for change (Pereira et al., 2012).

II. TYPICAL DYE PROCESS IN THE CLUSTER

1. **Preparing the Dye Bath:** Dyes (vat, sulphur, acid, or reactive) are dissolved in water at the right temperature and concentration as per the dye's requirements. For vat and sulphur dyes, caustic soda is added to create an alkaline environment needed to reduce the dye, making it soluble in water.
2. **Addition of Hydrogen Peroxide:** Hydrogen peroxide is often used in combination with caustic soda to oxidize and fix the colour in the yarn (especially in vat and sulphur dyeing). It helps the dye develop its final shade after application to the yarn.
3. **Dyeing Process:** The yarn is submerged in the dye bath and agitated for uniform dye penetration.
4. **After-Treatment:** The yarn is washed thoroughly 4 to 5 times to remove excess dye and chemical residues.
5. **Approx water consumption:** For 1.5 kg of yarn dyeing, approx. 25-40 litres of water is consumed. After treatment process further requires 50 litres. For each 1.5 kgs of yarn dyeing 90 litters of water is required (Mikucioniene et al., 2024).

Fig 1: Image of dye disposal at the backyard of the artisan house

III. MATERIALS AND METHOD

The research employed the following materials and equipment to replicate dye wastewater treatment processes commonly observed in handloom clusters and evaluate their efficacy. Magnesium Chloride [$MgCl_2$], Used as a coagulant to facilitate the removal of suspended particles and reduce chemical oxygen demand (COD). Alum [$Al_2(SO_4)_3$], Employed as a traditional coagulant to evaluate its efficiency in removing colour and reducing COD. Ferric Chloride [$FeCl_2$], tested for its coagulation properties particularly in addressing high dye concentrations whereas Calcium Carbonate [$CaCO_3$], is used in combination with other coagulants to aid in pH adjustment and enhance treatment efficiency (De Paulaa et al., 2014). All the chemicals are procured from the Hanumath Scientifics, Bhubaneswar.

Water Treatment and Filtration Systems, Membrane Filter: A semi-permeable membrane was used to remove fine particulates and dissolved contaminants from the treated water, ensuring a higher level of purification and Charcoal Filtration System consisted of six treatment chambers designed to sequentially remove residual contaminants, adsorb organic impurities, and further neutralize the treated water. An outlet chamber was included to collect the final treated water, ready for reuse in dyeing operations (Marcucci et al., 2003).

IV. EQUIPMENT

Analytical Equipment and Supplies pH Meter is used to monitor and adjust the pH of the wastewater during and after treatment. Beakers and Flasks for preparing solutions and conducting coagulation experiments. Magnetic stirring Apparatus id used to ensure thorough mixing of coagulants with wastewater and activated Charcoal packed into the filtration chambers for its high adsorption capacity to remove colour and residual chemicals. A Gravel and stones in filtration system and measuring Cylinders and Pipettes: For precise measurement of chemical quantities and water samples.

V. METHODOLOGY

Dyewater samples were replicated in the lab as done in clusters for research work and treated with magnesium chloride [$MgCl_2$], alum [$Al_2(SO_4)_3$], and ferric chloride[$FeCl_2$] and calcium carbonate [$CaCO_3$] to identify the most effective treatment method. After which the treated water is passed down through a membrane filter and charcoal filtration system. The charcoal filtration system consists of Six chambers are for the treatment itself, and one outlet chamber is designated to receive the treated water. The primary goal is to neutralize wastewater pH and enable its reuse in the dyeing process, ensuring sustainability in the handloom sector.

Initially dye wastewater samples were prepared using vat dye and sulphur dye. One litre of each dye solution was prepared and subsequently divided into four sections of 250 ml each, placed in separate beakers. The experiment involved six samples for testing i.e. 2.6gms of each coagulant, alum in 250ml in vat dye, ferric chloride in vat dye, calcium carbonate in vat dye, alum in sulphur dye, ferric chloride in sulphur dye, calcium carbonate in sulphur dye is used. Before adding the coagulants, the initial pH of each dye solution was measured. The coagulants were then introduced into the respective 250 ml dye wastewater samples. Each sample was subjected to magnetic stirring at a constant speed for 10 minutes to ensure uniform mixing of the coagulant. Subsequently, the samples were allowed to settle under static conditions for sedimentation kept it 24 hrs at room temperature.

Probable Chemical Reaction of green vat dye with magnesium chloride. Hydrolysis (if alkaline conditions exist)**:** In alkaline medium, Mg^{2+} can react with hydroxide ions to form Mg $(OH)_2$, which further aids in dye coagulation (Tchobanoglous et al., 2003).

$$MgCl_2+2NaOH\rightarrow Mg\ (OH)_2\downarrow+2NaCl \qquad (1)$$

The formed $Mg(OH)_2$ acts as a flocculant, trapping dye molecules and settling them out and the chemical reaction between ferrous chloride ($FeCl_2$) and black sulphur dye in a dye disposal process primarily involves oxidation-reduction and precipitation mechanisms. Oxidation of Fe^{2+} to Fe^{3+}, in the presence of oxygen or an oxidizing agent, Fe^{2+} is oxidized to Fe^{3+}, which forms insoluble ferric hydroxide ($Fe(OH)_3$) in alkaline conditions.

$$4FeCl_2+O_2+6H_2O\rightarrow 4Fe(OH)_3+8HCl \qquad (2)$$

Black sulphur dyes typically contain sulphide (-S-) or thiol (-SH) functional groups that react with Fe^{2+} or Fe^{3+}, leading to precipitation of insoluble iron-sulphide complexes (Zollinger, 2003).

$$FeCl_2+S-Dye\rightarrow FeS(precipitate)+Dyefragments \qquad (3)$$

Alternatively, in the presence of oxygen

$$Fe(OH)_3+S-Dye\rightarrow Fe-DyeComplex(insoluble)+H_2O \qquad (4)$$

Final Precipitation will be the iron-dye complex, FeS, and Fe $(OH)_3$ precipitate, allowing for their removal via filtration or sedimentation (Hunger, 2003) [14]. The reaction between alum $Al2(SO_4)_3$ and a green vat dye in a dye disposal process typically involves the precipitation of dye molecules through charge neutralization and flocculation (Broadbent, 2001). The general reaction can be written as:

$$Al_2(SO_4)_3+6H2O\rightarrow 2Al(OH)_3\downarrow+3H_2SO_4 \qquad (5)$$

The freshly formed $Al(OH)_3$ acts as a coagulant, adsorbing and trapping the dye molecules, leading to their precipitation. If the green vat dye is represented as Dye (assuming it is an anionic dye), the reaction can be

$$Al(OH)_3+Dye-\rightarrow[Al(OH)_3-Dye] \qquad (6)$$

VI. COAGULATION FLOCCULATION TEST

The coagulants were added to the waste water simultaneously, and the experiment was carried out. Post-treatment, the effectiveness of each coagulant was evaluated based on three key parameters: pH of the treated solution, floc structure, and extracted dye colour. These parameters served as indicators of the coagulant's ability to reduce impurities, enhance sedimentation, and recover the dye effectively. The following table below shows different parameters obtained after floc sedimentation (El-Gohary and Tawfik, 2009).

TABLE 1: PARAMETERS OBSERVED AFTER THE PROCESS

Coagulant	**Dye type**	**Initial pH**	**pH after extraction**	**Dye extracted**	**Extracted dye colour**	**Flocs left**	**Flocs structure**
Magnesium chloride, **$MgCl_2$**	Green Vat dye	11.98	6.09	170ml	Yellow	80ml	Dense, compact, and settled down
Ferric chloride, **$FeCl_2$**	Black sulphur dye	11.8	5.5	150ml	Orange	100ml	Settled down
Aluminum sulphate, **$Al_2(SO_4)_3$**	Green Vat dye	11.98	7.03	170ml	Colour less with little bit tint of dye.	80ml	Flaky flocs
Aluminum sulphate, **$Al_2(SO_4)_3$**	Black sulphur dye	11.8	6.95	100ml	More or less colour less with little bit tint of dye.	70ml	Cloudy flocs
Calcium carbonate, **$CaCO_3$**	Green Vat dye	11.98	---	250ml	Fully dissolved turning the colour of dye into white	---	Dissolved
Calcium carbonate, **$CaCO_3$**	Black sulphur dye	11.8	---	250ml	Fully dissolved turning the colour of dye into white	---	Dissolved

Fig 2: Sulphur treated

Fig 3: Vat dye treated with $FeCl_2$ with alum

VII. GRAVITY BASED MULTILAYER FILTRATION

An effective method for improving water quality by removing odour and taste, often caused by the presence of dissolved chemicals. Substances such as hydrogen sulphide and chlorine, which contribute to undesirable odours and flavours, can be mitigated through this process. Activated carbon, particularly in the form of granular activated carbon (GAC), operates by adsorbing these chemicals onto its porous surface as water passes through the filter (Esteki et al., 2024). This adsorption mechanism facilitates the removal of a wide range of organic and inorganic contaminants, enhancing the sensory and chemical properties of the treated water. This study employed a laboratory-based experimental design to evaluate the efficiency of various coagulation agents and a low-cost activated carbon filtration system for the treatment of synthetic dye wastewater (Gunes and Ayol, 2021). The methodology was structured in two phases: chemical coagulation and post-treatment filtration. The treated water is passed through a simple filtration system consisting of charcoal, gravel, sand, to remove the odour and colour from treated dye water. A low-cost charcoal filtration system offers affordable and efficient solution for treating wastewater, particularly in rural and small-scale settings. Designed using readily available materials, the system consists of layers to enhance filtration efficiency (Fersi et al., 2021).

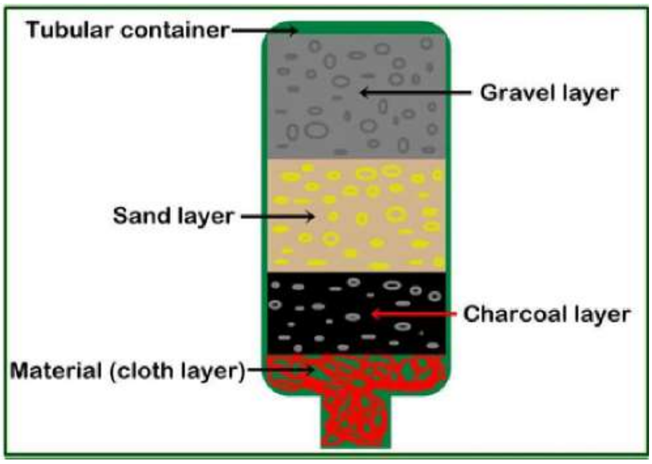

Fig 4: Improvised Charcoal Filter

The base layer is composed of coarse gravel, which traps larger debris and sediments. Above this, a layer of granular activated charcoal adsorbs organic and inorganic contaminants, effectively removing Odors, colours, and dissolved chemicals such as chlorine and hydrogen sulphide. A fine sand layer further refines the water by filtering out smaller particles and enhancing clarity (Yuanli et al., 2019). These layers are housed within a simple structure made from clay pots or PVC pipes, creating a compact and scalable setup. The treated water exits through an outlet chamber, ready for reuse in non-potable applications like dyeing in handloom clusters. This filtration system is not only cost-effective but also sustainable, offering communities a practical method to address water pollution while supporting environmentally friendly practices (Hassan et al., 2020).

VIII. RESULTS AND DISCUSSION

The pH of each of the samples are measure using supplied pH meter. The pH values can be seen from the table 2. Sample obtained from the $Al_2(SO_4)_3$ pH is much closer to the neutral water pH due to the precipitation of dye molecules through charge neutralization and flocculation. Magnesium chloride ($MgCl_2$): pH 6.2 (Slightly acidic), Ferric chloride ($FeCl_2$): pH 5.5 (More acidic), Aluminum sulphate ($Al_2(SO_4)_3$): Two samples with pH 7.03 and 6.95 (Near neutral), Calcium carbonate ($CaCO_3$): No recorded pH (Possibly neutralized or dissolved), Ferric chloride ($FeCl_2$) led to the lowest pH (5.5), making the water more acidic. Aluminum sulphate ($Al_2(SO_4)_3$) resulted in a near-neutral pH (~7.0), indicating good buffering. Magnesium chloride ($MgCl_2$) maintained a mildly acidic environment (6.2). Calcium carbonate ($CaCO_3$) dissolved or did not significantly alter the pH.

TABLE 2: pH VALUE AFTER SAMPLE PASSED FROM MULTILAYER FILTRATIONS.

Coagulant	pH after multilayer filtration
Magnesium chloride, $\mathbf{MgCl_2}$	6.2
Ferric chloride, $\mathbf{FeCl_2}$	5.5
Aluminum sulfate, $\mathbf{Al_2(SO_4)_3}$	7.03
Aluminum sulfate, $\mathbf{Al_2(SO_4)_3}$	6.95
Calcium carbonate, $\mathbf{CaCO_3}$	-
Calcium carbonate, $\mathbf{CaCO_3}$	-

Alum ($Al_2(SO_4)_3$) demonstrated superior coagulation efficiency, with negligeable visible impurities in the extracted dye. This performance was consistent across both vat and sulphur dyes. Fastest reaction was observed in case of Ferric Chloride ($FeCl_2$) with moderate efficiency, producing flocs that were dense. Calcium Carbonate ($CaCO_3$) shows poor performance in terms of dye water recovery and flocs formed were all dispersed in the dye solution. These findings highlight Alum's highest efficiency as a coagulant for treating dye wastewater.

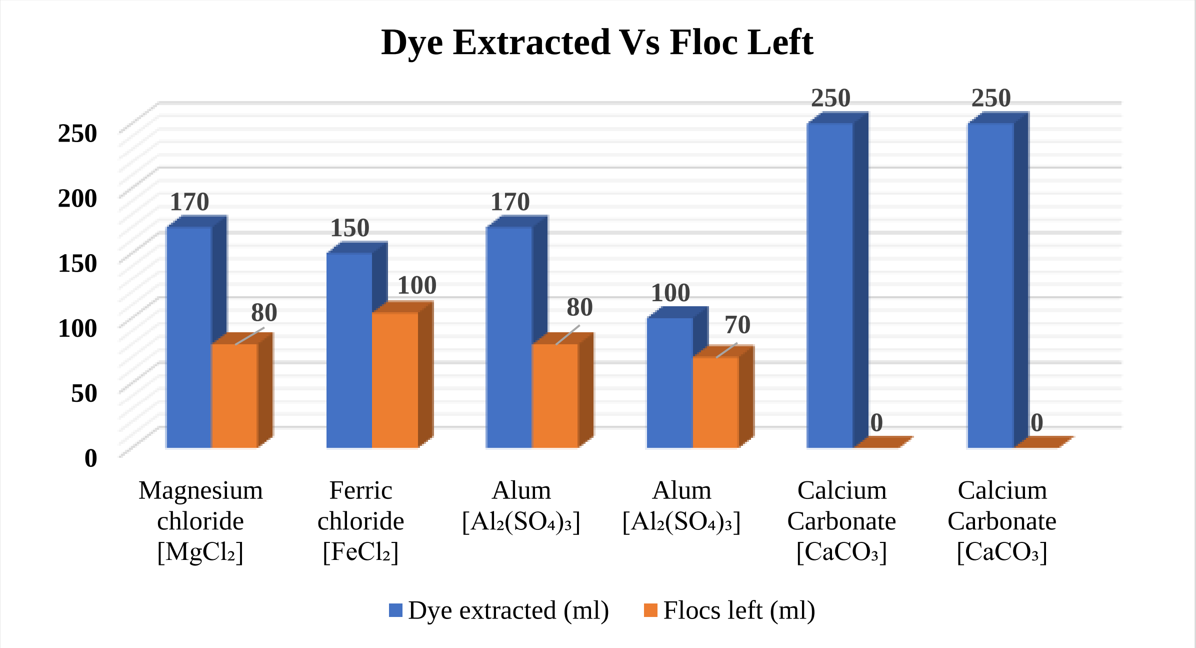

Fig 5: the amount of initial dye vs the flock weight obtained.

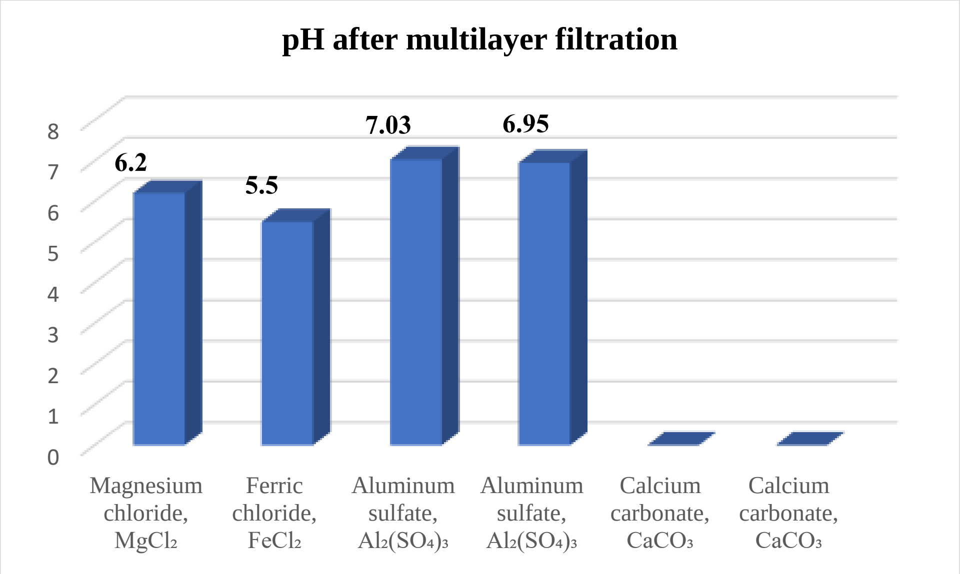

Fig 6: pH value after dye extraction

Exploring Alternatives for the Safe Disposal of Sludge Generated from Dye Water Flocculation:
Dye wastewater treatment processes, such as flocculation, produce large volumes of sludge as a byproduct. The safe and sustainable disposal of this sludge remains a pressing concern due to its environmental impact and potential for contamination. Recent efforts have focused on repurposing industrial waste into usable materials, fostering circular economy principles. Exploration and the feasibility of utilizing flocculation-derived sludge in textile applications, specifically in screen printing, block printing, and dyeing has done to minimize waste and promote resource recovery. The applications yielded unsatisfactory results due to the presence of fine concentrated particles within the sludge

- Screen Printing: Uneven application and poor adhesion resulted in inconsistent patterns.
- Block Printing: Particle aggregation disrupted smooth printing, causing blotchy designs.
- Dyeing: The sludge failed to provide uniform coloration, with visible residues on the fabric surface.

These challenges can be attributed to the nature of the sludge, which contains suspended impurities that resist uniform dispersion or binding. Furthermore, the small particle size impeded the integration of the sludge into textile mediums, leading to suboptimal outcomes.

Fig 7: Printing with alum sludge

CONCLUSION

The study demonstrates the effectiveness of coagulation as a primary method for treating dye wastewater, specifically vat and sulphur dyes. The results indicate that the choice of coagulant significantly influences the efficiency of floc formation, sedimentation, and dye recovery. Among the tested coagulants, Alum ($Al_2(SO_4)_3$) exhibited superior performance, producing dense and compact flocs with minimal impurities in the extracted dye for both types of wastewater. Ferric Chloride ($FeCl_2$) showed moderate efficacy, while Calcium Carbonate ($CaCO_3$) resulted in looser flocs and lower clarity in the recovered dye. The extracted dye water is treated in way that its pH comes to neutral. In case of alum the pH observed after coagulation was neutral, but in case of $FeCl_2$ the reaction was the fastest among all, but the pH observed was more towards acidic hence calcium carbonate is added in order to make the pH neutral. The study also highlights the importance of parameters such as pH, floc structure, and sedimentation behaviour in evaluating coagulant performance. These findings underscore the need for selecting appropriate coagulants customized to specific dye wastewater characteristics to optimize treatment efficiency. Further research could explore the integration of coagulation with advanced treatment methods, such as filtration or adsorption, to achieve more comprehensive wastewater management solutions.

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