JPIChE 51 (2) 2023: 75-84

PAKISTAN INSTITUTE OF CHEMICAL ENGINEERS Since 1969

Journal of Pakistan Institute of Chemical Engineers

journal homepage: www.piche.org.pk/journal

DOI: https://doi.org/10.54693/piche.05128

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Effective Removal of Rhodamine 6G (Basic Red-1) Reactive Dye from Textile Industry Effluent Using Waxy Corn Starch

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Submitted: 17/01/2024, Accepted: 30/04/2024, Online: 02/05/2024

Abstract

The textile industry generates effluents containing toxic dyes, which have detrimental effects on the environment, including air, soil, and water contamination. These dyes also pose risks to human health and disrupt aquatic ecosystems. Developing ecological and cost-effective methods to address this issue is a major challenge. This study focuses on utilizing waxy cornstarch, which exhibits unique properties such as high biodegradability, environmental compatibility, low sludge volume, small particle size, and high surface area (1.27 m^2/g), for removing the reactive dye Rhodamine 6G (Basic Red-1) from textile wastewater. The composition of waxy cornstarch was analyzed, including the determination of protein content using the Kjeldahl apparatus, as well as moisture content, ash content, and fine fiber content through experimental methods. The rate of dye removal from wastewater was assessed using a UV-spectrophotometer. The experimental design was selected, and trials were conducted using a glass testing apparatus. Dye removal rates were investigated at different dosages from 10 mg/L to 50 mg/L, temperatures ranges 25 to 45°C, and pH ranges from 4 to 10. The highest dye removal efficiency of 83.6% was achieved at a pH of 5, a temperature of 40 °C, and a coagulant dosage of 40 mg/l. These findings highlight the potential of waxy cornstarch as a natural coagulant for the effective removal of Rhodamine 6G dye from textile wastewater. It was investigated that the higher temperature break the bonding forces that's why a way clear to made a bond with dye molecule Rhodamine 6G.

Keywords: Bio-polymer, Coagulation, Dye removal, Natural coagulant, Reactive dye, Waxy Corn Starch, Wastewater

1. Introduction:

The cheaper and environmentally friendly technologies for removal of dyes from industrial effluent are a key challenge. The different dyes when exposed to humans can cause serious diseases like asthma, respiratory diseases, and many skin diseases [1]. Many textile dyes produced genotoxicity, mutagenicity, and Immunoglobulin [2, 3]. Many pollutants of Textile industrial wastewater are harmful. Due to exit of this kind of wastewater into the atmosphere and aquatic bodies affects hostile changes in the state of the environment. Also, the buildup of dye stops the infiltration of sunlight; and interrupts the aquatic ecosystem [4]. Many Coagulants, like aluminum-based ones, cause serious Alzheimer's disease, a physical disease condition that damages the human brain, initially identified by the German neurologist Alois Alzheimer. It also includes progressive brain damage, communication problems, and memory loss '[5]. The treatment of effluent containing dyes with eco-friendly material

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is of considerable position because it is a more environmental friendly approach and produces less sludge.

The chemical industry, particularly the textile sector, produces liquid waste that contains various colors due to factors such as the material's molecular weight, composition, and intricate structure. As a result, the dyes in this waste exhibit a low level of biodegradability [6]. According to the current literature, numerous traditional methods and techniques, including physical, biological, and chemical processes, remove colors. In addition, some innovative techniques like chemical oxidation or advanced oxidation processes are also being implemented [7].

Chemical oxidants, such as (H_2O_2), ozone (O_3), and permanganate (MnO_4), were capable of altering the functional group and chemical composition of compounds, including colors [8]. The method described was effective in removing soluble colors, but it may not be as efficient for removing insoluble pigment vat dyes, which typically exhibit slower reactivity and require longer reaction times [9]. The efficacy of dye removal using ozone as an oxidizer was also affected by pH levels. In the case of Reactive Yellow 84 hydrolyzed dye, the efficiency of ozonization decreases as pH is lowered [10]. The process of chemical coagulation and flocculation entails introducing certain chemicals into the substance undergoing treatment to modify the physical state of impurities that are dissolved or suspended. This results in their agglomeration and eventual removal through sedimentation. Coagulation, in particular, was highly effective in eliminating suspended and colloidal impurities that may be difficult or slow to settle. Therefore, it is frequently employed as a preliminary treatment in various water treatment procedures [11]. Nevertheless, the use of natural coagulants was being increasingly emphasized due to their eco-friendliness. Potato starch was a superior choice because it operates effectively even in cold water and at lower concentrations than mineral salts. Consequently, it generates less sludge compared to the chemical coagulant alum. Additionally, biopolymers like starch sludge can be degraded easily and efficiently by microorganisms [12], as demonstrated in studies of sludge from a kaolinite suspension system [13] and a milk processing plant [14]. The resulting wastewater promotes plant growth and proves beneficial to the soil while remaining non-harmful to the plants.

Environmental protection has become an important global concern therefore industries are considering eco-friendly and low-cost technologies. As per our research sludge produced will contain biodegradable material and purified wastewater will not be hazardous and toxic for the environment because of the removal of harmful dye from it [15]. Further experiments can be carried out to treat this waste biologically after coagulation if satisfactory results are achieved then this will give very high removal efficiency. Research shows that natural coagulants are not harmful to microorganisms. Literature confirmed that this coagulation and flocculation process has successfully combined with filtration and efficient & satisfactory results are achieved so it can be combined with filtration. Sludge contains natural polymeric material that can be used as a soil conditioner or can be disposed off in an eco-friendly manner. Below are the commonly used classifications of natural coagulants for treatment purposes.

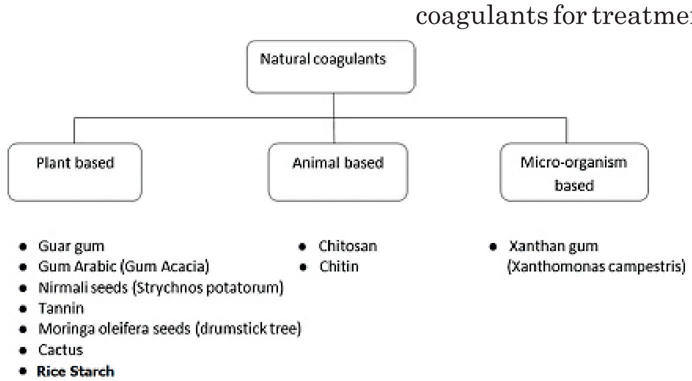

Figure-1: Classification of natural coagulants

Nirmali seeds give Polysaccharide (Trichinas potatorum) shown to effectively remove 80% of kaolin solution turbidity [15]. Moreover, it can remove 99.5% of water turbidity with a dosage of only 400mg/L. Further increasing the dosage of the seed extract after reaching maximum adsorption can result in increased dye removal due to the absence of available adsorption sites on the seed extract surface. A good biofuel can be made from M. oleifera seeds [16].

Natural coagulants provide numerous adsorption sites due to their high molecular weight and long chain structure. In addition to charge neutralization, adsorption, and inter-particle bridging occur when polysaccharide chains sorb particulates. A standard biochemical test was conducted to identify bio flocculant-producing bacteria by analyzing the 16S rRNA gene sequence. Bacteria were isolated from activated sludge samples taken from effluent plants. The purified bioflocculant solution produced by these bacteria was used for removing dyes from wastewater, resulting in dye removal efficiency of up to 97.04% under diverse nutritive conditions, pH, and temperature. The amount and efficiency of dye removal are prejudiced by numerous factors, including the type and nature of the dye, temperature, pH, and flocculant concentration. Bacteria-produced flocculants are biodegradable, sustainable, and provide a cleaner and more affordable alternative for dye removal from wastewater. Although electrochemical treatment is a highly effective method for controlling water contamination, the removal of methyl red azo dye from textile wastewater remains a challenge. Electrooxidation (EO) and biotreatment (BT) are conventional methods for textile wastewater treatment, and their combination is highly recommended. EO can remove 70% of COD and 81% of color, and when followed by BT, COD and color can be further reduced by 92% and 95%, respectively. Electrochemical processes generally require lower temperatures than their non-electrochemical equivalents, and no additional chemicals are required. This process is suitable for treating effluent from industries [17]. Methyl violet is a harmful pollutant in aquatic ecosystems, toxic to animals, causing respiratory and digestive tract irritation. In a study, palm kernel carbon was used as a biological absorbent to remove methyl violet from textile industry sewage. Optimal conditions for adsorption were determined, achieving high removal percentages (98.85% for modified adsorbent, 91.08% for unmodified) [18].

The core objective of the research was based on the characterization of effluent/textile waste water, characterization of waxy corn starch to measure protein content by using the Kjeldahl Apparatus to achieve maximum reactive dye removal efficiency by optimizing parameters through the Jar Test Apparatus. Also find optimum coagulant dosage, pH, temperature conditions, and mixing time to compare the results with other coagulants.

2. Materials And Methods:

The parameters of waxy corn starch were determined through experimentation, with a focus on its moisture content. To measure this, a vacuum oven was used to remove moisture from a five-gram sample of the starch for a specified amount of time and temperature, while maintaining a steady state vacuum of 150 mm of Mercury. The weight loss of the sample during this process was considered as the moisture content. Specifically, the sample was heated at a constant temperature of 120 ± 0.5 ?C for 4 hours, and the moisture content was calculated using a specific formula.

Moisture, % = (Loss in Weight, g /Sample Weight, g) × 100

Dry Substance, % = 100 - Moisture, %

2.1 Fiber content of waxy corn starch: To separate the fiber from a waxy corn starch sample, a specific mesh screen is used to screen it, and the resulting fiber is then treated to remove any starch or grit bound to it. This treated fiber is then collected, washed, and dried, after which its weight is measured to determine the actual quantity. The purpose of treating the fiber is to eliminate any starch or grit present, which can interfere with the calculation of the pure fiber quantity. A mesh screen of 0.044 mm (0.0017 inches) known as Brass Sieve No. 325, according to the USA Standard Test is utilized for this purpose. To determine the quantity of fiber or starch grit value, 50 grams of starch are poured onto the mesh screen, and the residue is washed with a slow stream of water. The remaining residue is then rinsed into a weighed evaporating china dish, and it is then dried in a vacuum oven at 100 ?C for an hour, after which it is cooled and weighed. To obtain the pure fiber quantity, the residue is rinsed into a 250 ml flask Erlenmeyer with about 50 ml of cleaned water, followed by the addition of 30 ml calcium chloride solution. This mixture is boiled for 5 minutes on a hotplate, followed by an additional 15 minutes of boiling. After cooling, the material is washed through the mesh screen with a stream of water, and the residue is rinsed into a weighed evaporating china dish and evaporated on a steam bath. The formula for determining fine fiber in waxy corn starch is also provided [40].

$$\text{Pure Fiber (ppm)} = F\,F = \text{Residual Wt,g} \times 1{,}000{,}000/(50\text{ g sample} \times \text{d.s.,\%}/100) \quad (1)$$

$$\text{Pure Fiber (ppm)} = FF = (\text{Fiber ppm}/1000) \times 584 \quad (2)$$

2.2 Protein content of waxy corn starch used in experimentation: From Fisher Scientific (USA) The Kjeldahl gadget was acquired under catalog number 13-120-50. The Kjeldahl procedure entails the breakdown or oxidation of nitrogen-rich materials. The digestion solution is alkalized with sodium hydroxide and ammonia, then distilled into H_2SO_4. The quantity of ammonia obtained is determined quantitatively through back titration using standard base, and the protein content is then calculated accordingly. This technique is particularly effective for analyzing starch.

2.3 Process description:

In this study, a Whatman filter paper is used to enclose a five-gram sample of waxy corn starch in a Kjeldahl flask with 10 grams of digestion mixture. Slowly, 50 grams of sulfuric acid is added to the mixture along the walls of the flask, and the mixture is heated using an electrical heater until a light greenish color appears. The fumes are extracted using an exhaust system. After the digestion process is complete and the light greenish color appears, the heaters are turned off, and the mixture is allowed to cool. A receiving flask is prepared for the distillation process from the modified Kjeldahl flask. To prepare the receiving flask, 150 ml of distilled water is added to a volumetric flask, along with 3 to 4 drops of methyl red indicator and 50 ml of 0.1N H_2SO_4. The Kjeldahl flask is then cooled from the outside using water, and distilled water is added to fill it up to 400 ml. A few drops of phenolphthalein are added, and a spatula is used to add a pinch of zinc dust. Finally, The receiving flask is then titrated, and the protein content of the sample is determined using a standard formula.

$$\text{Total Nitrogen\%} = \frac{[(\text{ml of acid*N of acid}) - (\text{ml of NaOH*N of NaOH})] \times 14.007*100\%}{\text{Weight of sample (grams)*1000 mg}} \quad (3)$$

2.4 Ash content of waxy corn starch:

An Electric Muffle Furnace (CARBOLITE) with automatic temperature control (thermocouple pyrometer system) is capable of maintaining a constant temperature of 1000˚C. The waxy corn starch sample is placed in the furnace and heated for a specific duration until the sample is completely oxidized. The remaining residue is weighed and expressed as the ash content. To perform this, 5 grams of waxy corn starch sample is taken in a weighed empty china dish and heated at a temperature of 600 ˚C for 2 hours until complete oxidation.

2.5 Reactive Dye:

The Reactive dye used in the study, Basic Red-1, was acquired from "Sigma-Merck" (Germany) and transported to the laboratory. It is Rhodamine 6G, with a molecular formula of $C_{28}H_{31}ClN_2O_3$ and a molecular weight of 479 g/mol. It has two hydrogen bond donors and five hydrogen bond acceptors. Waxy cornstarch, on the other hand, was obtained from a local market nearby.

2.6 Stock Solution Preparation:

A beaker was used to prepare the stock solution by adding 0.01g of Basic reactive red 1 dye and 1g of sodium carbonate, and then water was added to increase the volume of the solution to 1000 ml. Treatment: Initially, we collected five equal volume samples of 20 ml each of the stock solution in separate beakers with 250 ml capacity. In each beaker, we added varying quantities of waxy corn starch, namely, 0.01 g, 0.02 g, 0.03 g, 0.04 g, and 0.05 g. These samples were allowed to settle for 24 hours and were then filtered using "Whatman filter paper." Subsequently, the liquid filtrate from each beaker was subjected to "Ultraviolet Absorption Spectroscopy" to identify the best adsorbent quantity for removing the dye from the solution.

The experiment was carried out using a method and procedure agreed upon by Blackburn [19], which helped determine the optimal temperature, adsorbent quantity, and pH level required for dye treatment.

3. Rseults And Discussions:

The table-1 & 2 below presented the results of the composition analysis of effluent and waxy corn starch after the experiments. It was necessary to analyze the coagulant as the components present in waxy corn starch can impact the final outcomes when it is utilized as a natural coagulant for dye removal. Four parameters, namely temperature, pH, concentration, and time, are considered in the removal of reactive dye using waxy corn starch. These parameters are important factors that can influence the effectiveness of the dye removal process. That influenced on the adsorption process, surface charge, ionization state, and overall efficiency of the dye removal system.

Table-1: Characteristics of effluents

pH	6
Temperature	42°C
COD	705 mg/L
BOD	427 mg/L
TSS	High (1236 mg/L)
Color	Reddish
TDS	2469 mg/L
Salinity	256 µS/cm
Heavy Metals:	
a) Chromium	1.86 mg/L
b) Copper	2.66 mg/L
c) Ferrous	1.16 mg/L
d) Cadmium	0.001 mg/L

Table-2: Composition of waxy corn starch

Waxy starch Component	**Percentage (%)**
Waxy Moistures	11.5 %
Waxy Fine fibers	0.089% or 890 ppm
Waxy Proteins	0.92 %
Waxy Ash	0.31 %
Waxy Starch	87.179 %

A complete factorial design was used for conducting all the experiments, thereby studying the key interaction and impact of coagulation dose, temperature, and pH on the dye removal process. The study examined the impact of temperature, pH, and waxy corn starch dosage on the efficiency of waxy corn starch color removal during the flocculation process. To evaluate the effectiveness of waxy corn starch in removing color, wastewater adsorption was considered a crucial parameter in the experiments. The results of these experiments are presented below.

3.1 Effect of Temperature:

Unlike cellulose and amylose, which have linear structures, amylopectin contains branching structures with approximately 1 out of every 20 residues attached to branching points (- (1 → 6) - branching). These branching structures provide additional sites for dye molecules to interact with during the adsorption process.

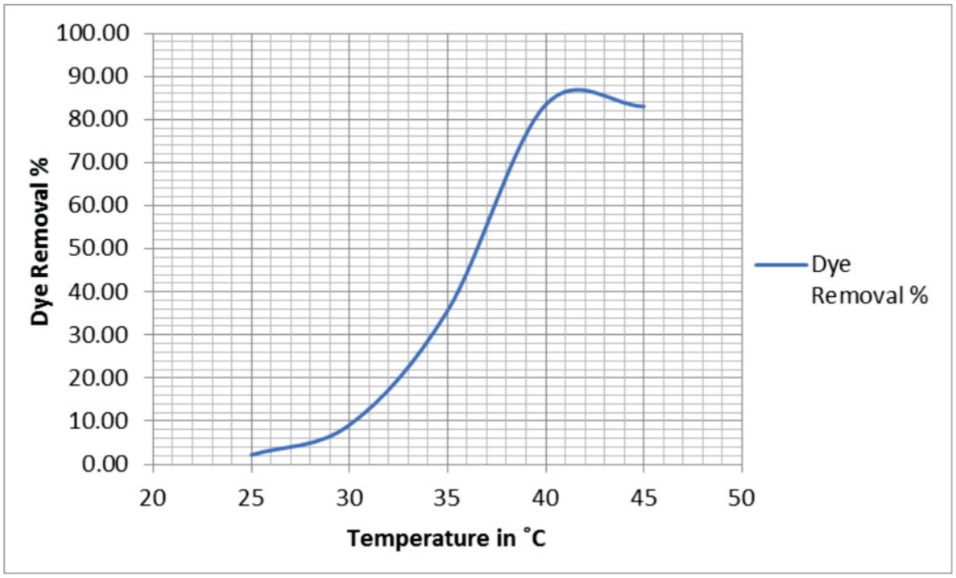

Figure-2: Effect of temperature on Dye Removal

Waxy corn starch, which is used as a coagulant in this context, facilitates dye removal. The hydrophobic nature of waxy corn starch allows it to come into contact with the dye molecules, promoting their adsorption. Additionally, waxy corn starch maintains a negative charge, which is beneficial for dye removal due to electrostatic forces. The negative charge of the starch attracts and binds with positively charged dye molecules, aiding in their removal.

Overall, elevated temperatures contribute to the removal of dyes by promoting the interaction between dye molecules and the starch components, particularly amylopectin. The hydrophobic nature and negative charge of waxy corn starch further facilitate dye removal through contact and electrostatic forces, respectively.

3.2 Effect of pH on color removal: The impact of pH has been examined as it affects not only the stabilization of the suspension but also the surface charge of the coagulant. The optimal removal was observed at low pH, where electrostatic force dominates in conjunction with a low dosage value.

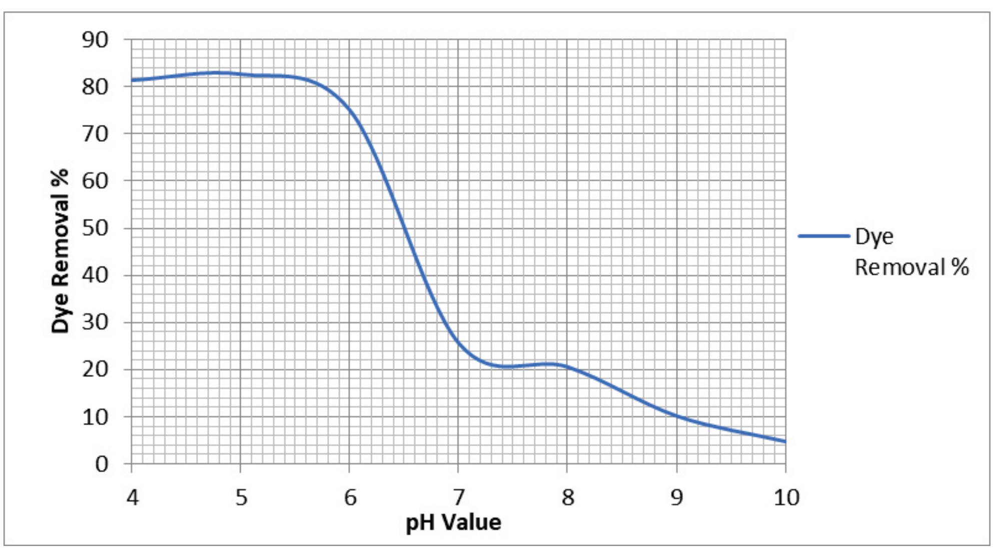

Figure-3: Effect of pH on Dye removal

A decrease in pH as shown in figure-3 leads to an increase in donor sites for hydrogen bonding, resulting in the creation of a positive charge on the surface of waxy corn starch. This allows for efficient removal of dye via electrostatic forces between the negatively charged hydrophobic sites containing dye and the waxy corn starch. At high temperatures, however, the negative charge is dominated by the degradation of intermolecular and intramolecular hydrogen bonds between starch chains. Nevertheless, combining low doses with acidic pH provides a viable method for effective color removal.

The destabilization of particles can be explained clearly by the charge neutralization mechanism. Adsorption of dyes on polysaccharides involves a combination of hydrogen bonds, electrostatic forces, and van der Waal interactions, with electrostatic forces having a significant effect on low pH conditions, according to Blackburn [19]. The efficiency of the process must be improved by facilitating the mechanism of charge neutralization (acidic pH) and reducing the concentration of waxy corn starch under conditions that support charged neutralization (acidic pH). Biopolymers with high molecular weight, such as waxy corn starch, may participate in hydrogen bonding through their active hydroxyl groups, which could lead to polymer self-coiling and reduced availability of active functional groups to capture the dye.

3.3 Effect of Dosage on Coagulation: The impact of the dosage has been investigated in figure-4 to avoid under or overuse of the flocculant, which may result in reduced performance of the process. Hence, finding the optimal dose is crucial to enhance the treatment efficiency while minimizing the cost. The efficiency of dye removal can be improved with a higher dose of biopolymer due to the greater charge density of the polymer, leading to an increase in neutral polymer adsorption.

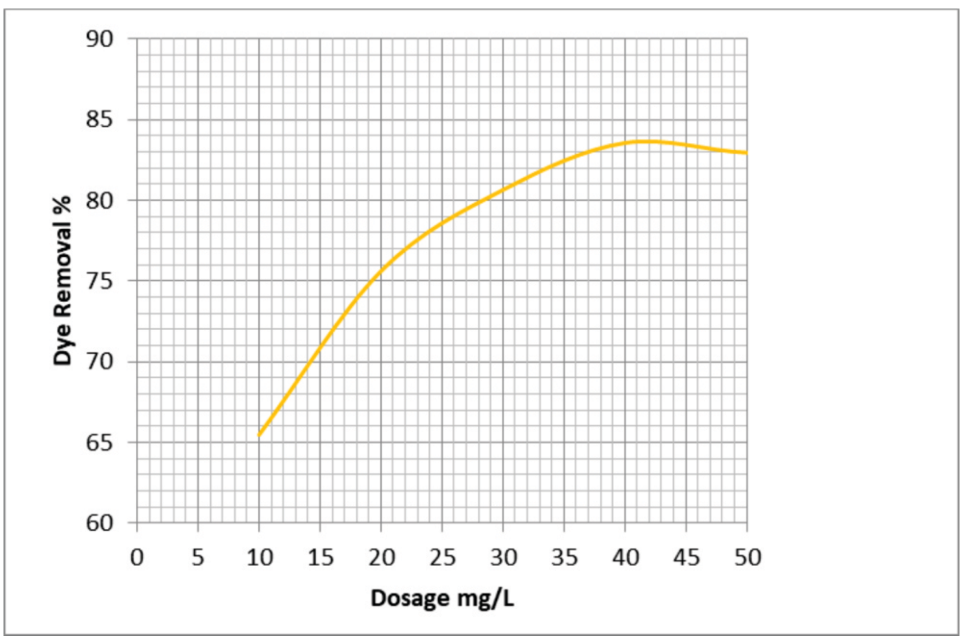

Figure-4: Effect of coagulant dosage on dye removal

The investigation determined that an optimal dose of 40 mg/L of waxy corn starch was sufficient for effective dye removal. Using a dosage beyond this optimal point resulted in the re-stabilization of colloids, which can reduce the effectiveness of the treatment. The reason behind the increase in dye removal with an increase in dosage can be explained by the increased surface area of the absorbent, i.e., waxy corn starch.

3.4 Effect of Mixing Time on Coagulation: As shown in figure-5, when the mixing time increases, there is a corresponding increase in the efficiency of dye removal. This means that the longer the mixing time, the more effective the process becomes in removing the dye from the system. It suggests that increasing the mixing time enhances the efficiency of dye removal. However, there is a limit to this improvement, as after 300 minutes of mixing, the removal efficiency plateaus, and no further substantial increase is observed.

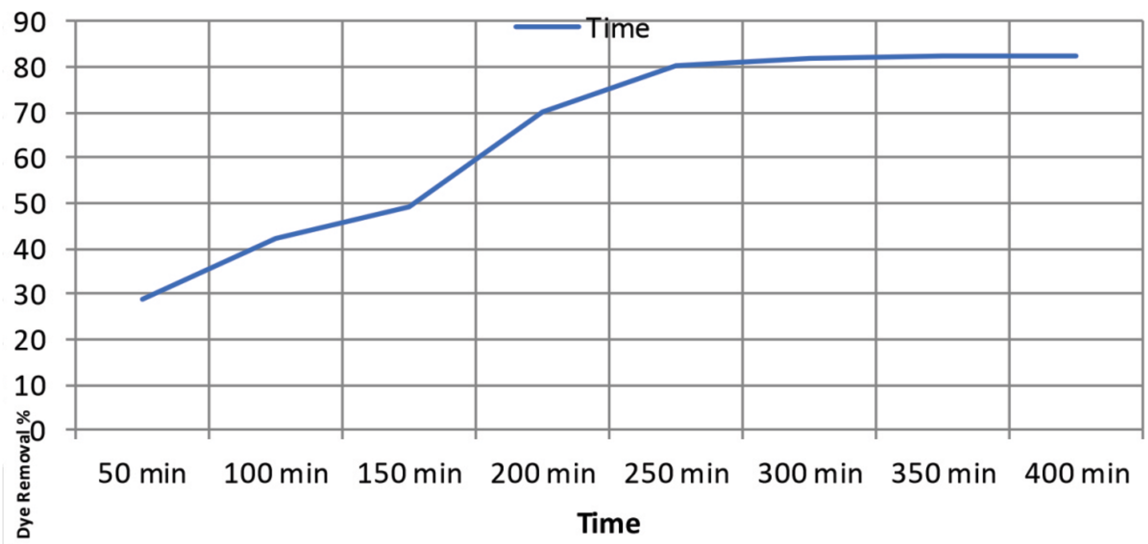

Figure-5: Effect of mixing time on dye removal

3.5 Comparison With Previous Research"

A brief comparison of our research is given in table-3 which shows the removal percentage when applied different parameters and compared with previous research data.

Table-3: Different coagulants with removal efficiency

Coagulant	Parameter	Removal Percentage	Reference
Corn starch	At 0.5mg/L and 4 pH the Kaolin, Staphylococcus aureus and Escherichia coli removed percent	98 %	[20]
Banana peel AC	Cd(II) removal at 20 mg/L, 4 pH, 25 ˚C	98.35 %	[21]
Carica papaya seeds	methylene blue dye removal at 100 seeds/100 L	80%	[20]
Feronia limonia seeds	SW(clay) 100NTU Turbidity removal at 20gm/L dosage and 8 pH of synthetic water	77%	[22]
Phoenix dactylifera date seeds	SW (Bentonite) Turbidity removal of synthetic water of 100NTU at 30gm/L dosage and 8 pH	94% (at 120th min)	[22]
Tamarindus indica seeds	SW (clay) 100NTU at 20gm/L dosage and at 8 pH, turbidity removal of synthetic water.	80%	[22]
Waxy Corn Starch	40 mg/L at 5 pH and 40 ˚C	83.6%	Current study
Raw mango seed	Methylene blue dye 50 mg/L concentration	99.1%	[23]
Tea waste	Acid Orange-7 dye concentration of 20g/L	99%	[23]

4. Conclusions:

The use of waxy corn starch as a natural coagulant in wastewater treatment has shown to be environmentally friendly and effective in removing dye from wastewater. The highest dye removal was observed at a pH of 5, where charge neutralization occurred efficiently, improving the process's effectiveness. An optimum dose of 40 mg/L of waxy corn starch was found to be sufficient, as over-dosage resulted in the re-stabilization of colloids. Additionally, a lower temperature of 20 °C was found to be inadequate for dye removal, while the best results were obtained at higher temperatures, specifically at 40°C. This is because the higher temperature was able to break the intermolecular and intramolecular hydrogen bonds among the polymer chains, making it easier for the polymer to bond with the dye molecules. The sludge produced was found to contain biodegradable materials and could be further treated or disposed of safely.

Further research is necessary to identify the most effective coagulant or combination of coagulants that can yield positive outcomes even under varying pH ranges and other treatment-affecting factors. Additionally, investigating the effects of natural coagulants is crucial to establish the sustainable use of natural materials in industrial-scale wastewater treatment. Coagulants that are compatible with specific industrial waste characteristics and types must be explored to achieve maximum removal efficiency. While this natural polymer can serve as a coagulant, it can also be utilized as a coagulant aid to improve process efficiency. Combining this process with other treatment methods may lead to higher satisfaction levels, and if satisfactory results are achieved, the combined process can be recommended.

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