JEE Journal of Ecological Engineering

Journal of Ecological Engineering 2024, 25(11), 83–99
https://doi.org/10.12911/22998993/192676
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Received: 2024.08.23
Accepted: 2024.09.23
Published: 2024.10.01

Effects of Dosage and Stirring Speed Variations in the Use of Bittern as a Natural Coagulant to Remove Biological Oxygen Demand, Chemical Oxygen Demand, Total Suspended Solids and Dye Concentrations from Batik Industry Wastewater

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ABSTRACT

This study aimed to determine the effect of bittern coagulant dosage and rapid stirring speed on reducing the concentrations of biological oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), and dye absorbance in batik industry wastewater, as well as to identify the optimum coagulant dosage and stirring speed. Wastewater samples were collected from a batik industry in the batik center of Sidoarjo, East Java, Indonesia. Dosage variations of 5%, 10%, 15%, and 20% were tested alongside rapid stirring speeds of 100 rpm, 130 rpm, and 160 rpm. The study was conducted on a laboratory scale using the jar test method. Initial wastewater characteristics showed BOD, COD, TSS, and dye absorbance concentrations of 185.68 ± 29.34 mg/L, 10.091 ± 363.24 mg/L, 2.231.33 ± 155.55 mg/L, and 0.212 ± 0.02, respectively. Statistical analysis using the Pearson correlation test and Two-Way ANOVA revealed that variations in coagulant dosage and stirring speed significantly impacted the reduction percentages of BOD, COD, TSS, and dye absorbance. The optimal coagulant dosage was found to be 5%, and the optimal stirring speed was 100 rpm, with reduction percentages for BOD, COD, TSS, and dye absorbance being 80.32%, 65.86%, 92.35%, and 70.77%, respectively.

Keywords: bittern, BOD, coagulation, COD, flocculation, TSS.

INTRODUCTION

The textile industry is one of the largest manufacturing sectors, both in Indonesia and globally. Within Indonesia, one of the most significant segments is the batik industry (Tangahu et al., 2019). The demand and sales of batik cloth have surged since it gained international recognition, leading to the expansion of batik industry centers across various regions in Indonesia. This growth, however, has resulted in an increased volume of wastewater production (Fitriani et al., 2023b; Nimatuzahroh et al., 2022). Batik industry wastewater is characterized by its complex pollutants and high color intensity (Daud et al., 2022; Putra and Airun, 2021). According to Regulation of the Minister of

Environment and Forestry Number P.16/MENLHK/SETJEN/KUM.1/4.2019, which concerns wastewater quality standards for the textile industry, the maximum allowable concentrations for BOD, COD, and TSS are 60 mg/L, 150 mg/L, and 50 mg/L, respectively (Fitriani et al., 2023b; Imron et al., 2023). Therefore, it is essential to treat the batik industry wastewater to reduce its pollutant load. One effective method for this is coagulation-flocculation (Kurniawan et al., 2023b, 2020).

Coagulation is a wastewater treatment process that destabilizes colloidal particles, while during flocculation, these destabilized particles form larger aggregates called flocs (Ahmad et al., 2022a; Owodunni et al., 2023). This process requires a coagulant to facilitate particle sedimentation (Fitriani et al., 2023a, 2020). In Indonesia, one promising natural coagulant is bittern, the residual water from salt production, which is typically a waste byproduct (BinAhmed et al., 2015; Lee et al., 2003; Soedjono et al., 2021). Previous studies have demonstrated the effectiveness of bittern in reducing various pollutants. The use of bittern for coagulation-flocculation process can achieve an 85% reduction in BOD concentration (Diwani and Rafie, 2002), an 80% reduction in dye concentration (Albuquerque et al., 2013), and reductions in COD and TSS concentrations by up to 71% and 97%, respectively (Ayoub et al., 2011).

The factors influencing the coagulation-flocculation process include coagulant dosage and rapid stirring speed (Kurniawan et al., 2023b, 2022b). A low coagulant dosage can result in insufficient floc nuclei, reducing particle collisions and hindering floc formation (Igwegbe et al., 2021b, 2021a). Conversely, an excessively high coagulant dosage can prevent proper floc formation and may even increase turbidity (Ramli et al., 2023). Rapid stirring speed is crucial, especially for floc formation, as the appropriate speed ensures the coagulant disperses quickly and evenly throughout the wastewater. This optimal condition enhances the likelihood of collisions between particles and the coagulant (Kurniawan et al., 2022a; Nimatuzahroh et al., 2022). To date, there have been many studies utilizing alternative sources of coagulant to treat wastewater. However, the use of bittern as a coagulant to treat dye containing wastewater from batik industry is still rarely analyzed.

On the basis of the explanation above, this study was undertaken to investigate the use of bittern coagulant in the coagulation-flocculation process for reducing concentrations of BOD, COD, TSS, and dye absorbance in batik industry wastewater. The study examined the effects of different bittern coagulant dosages and rapid stirring speeds on the treatment efficacy. Specifically, coagulant dosages of 5%, 10%, 15%, and 20% were tested. Additionally, the rapid stirring speeds were varied at 100 rpm, 130 rpm, and 160 rpm to determine the optimal conditions for maximizing pollutant reduction.

MATERIALS AND METHODS

Tools and chemicals

The tools used in this study consisted of a JLT4 Velp Scientifica jar test flocculator, a 500 mL AGC IWAKI beaker, a thermometer, a Senz pH pen, a refractometer, an Eutech TN-100 turbidimeter, a B-One BOD 110L incubator, a Winkler bottle, a VOSSO aerator, a Hettich-EBA 20 centrifuge, a 96 well U microplate, a Thermo Scientific UV-VIS Spectrophotometer, a desiccator (Duran), a cuvette, a digestion vessel (Iwaki CTE33), a heater with a heating block (VELP Scientifica), a Buchner funnel, a vacuum pump (Value), an oven (Memmert) and SEM EVO® MA 10 which is also equipped with a quantax energy dispersive X-ray (EDX) Detector from Bruker for microanalysis.

The materials used in this study were batik industry wastewater of ± 9 L and residual seawater from salt production (bittern) of ± 5 L as a coagulant. The materials needed for COD analysis are aquademin, potassium bichromate (K_2CrO_7), concentrated sulfuric acid (H_2SO_4), mercury sulfate ($HgSO_4$), $Ag_2(SO_4)$ powder or crystals, and potassium hydrogen phthalate ($HOOC-C_6H_4COOK$). The materials needed for BOD analysis are distilled water, potassium dihydrogen phosphate (KH_2PO_4), disodium hydrogen phosphate heptahydrate ($Na_2HPO_4 \cdot 7H_2O$), ammonium chloride (NH_4Cl), magnesium sulfate ($MgSO_4 \cdot 7H_2O$), anhydrous calcium chloride ($CaCl_2$), ferric chloride ($FeCl_3 \cdot 6H_2O$), microbial seeds from wastewater, sulfuric acid (H_2SO_4), NaOH, sodium sulfite (Na_2SO_3), glacial acetic acid (CH_3COOH), potassium iodide (KI), and starch indicator solution. The materials needed for TSS analysis are 0.45 µm pore size filter paper with a diameter of 55 mm, label paper, and 5 L of distilled water.

Jar testing method

The study employed a 4 × 3 factorial completely randomized design (FCRD) due to the presence of multiple interrelated factors. These factors included the coagulant dose and the fast stirring speed. The coagulant dose had four levels: 5%, 10%, 15%, and 20%, expressed as the percent volume per volume (%V/V) of bittern coagulant in the wastewater. The fast stirring speed had three levels: 100 rpm, 130 rpm, and 160 rpm. Consequently, there were twelve treatment combinations (4 × 3) between the coagulant dose and fast stirring speed, each replicated three times.

The wastewater sample, which had received varying dosages of bittern coagulant in a beaker, was first stirred using a jar test flocculator at different fast stirring speeds. This was followed by slow stirring at 30 rpm for 15 minutes (Ebeling et al., 2004). After stirring, the sample was allowed to sit for 45 minutes to separate into two layers, filtrate and sediment (Ebeling and Ogden, 2004). The supernatant (filtrate) was then analyzed for COD concentration using the closed reflux spectrophotometric method, BOD concentration using the Winkler method, TSS concentration using the gravimetric method, and dye absorbance using the spectrophotometric method.

Data analysis

Chemical oxygen demand (COD) analysis

COD calculation used the following equation:

$$\mathrm{COD} = C \times f \tag{1}$$

where: C – COD concentration (mg/L), f – dilution factor.

Biological oxygen demand (BOD) analysis

BOD calculation used the following equation.

$$Normality\ of\ sodium\ thiosulfate = \frac{20 \times 0.025}{a} \tag{2}$$

where: a – volume of thiosulfate required for titration, DO – dissolved oxygen (mg/L).

$$Dissolved\ oxygen = \frac{a \times N \times 8000}{V} \tag{3}$$

where: DO – dissolved oxygen (mg/L), a – volume of sodium thiosulfate used for titration (mL), N – normality of the sodium thiosulfate solution used (ek/L), V – Winkler bottle volume (mL).

$$BOD_5 = \frac{[(X0\text{-}X5)\text{-}(B0\text{-}B5)](1\text{-}P)}{P} \tag{4}$$

where: BOD_5 – biological oxygen demand (mg O_2/L), X_0 – dissolved oxygen of the sample at t = 0 days (mg O_2/L), X_5 – dissolved oxygen of the sample at t = 5 days (mg O_2/L), B_0 – dissolved oxygen of the control at t = 0 days (mg O_2/L), B_0 – dissolved oxygen of the control at t = 5 days (mg O_2/L), P – dilution factor.

Total suspended solids (TSS) analysis

TSS calculation used the following equation.

$$TSS = \frac{(b-a) \times 1000 \times 1000}{V} \tag{5}$$

where: TSS – total suspended solids concentration (mg/L), b – weight of filter paper and filtrate after drying (g), a – weight of empty filter paper after drying (g), V – sample volume (mL), 1000 – g to mg conversion factor, 1000 – mL to L conversion factor

Dye absorbance analysis

The optimum wavelength measurement was carried out in the visible light wavelength range of 400–750 nm (Ahmed et al., 2018). The analysis was carried out by determining the optimum wavelength of the batik industry wastewater sample, then measuring the absorbance of the sample using the optimum wavelength obtained previously. The measurement was carried out using a UV-VIS spectrophotometer.

Removal efficiency and standard deviation calculation

The calculation of removal efficiency and standard deviation (STD) can be seen in the following equation.

$$Removal\ efficiency\ (\%) = \frac{C_0 - C_1}{C_0} \times 100\% \tag{6}$$

where: C_0 – Initial concentration (mg/L), C_1 – Final concentration (mg/L).

$$s = \sqrt{\frac{n\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} xi\right)^2}{n(n-1)}} \tag{7}$$

where: s – standard deviation, x_i – x-value i, n – total number of data population.

Statistical analysis

Statistical analysis was performed using the Shapiro-Wilk normality test and the Levene test for homogeneity. Data were considered normally distributed and homogeneous if the p-value was greater than 0.05, allowing for further analysis using correlation analysis. For normally distributed data, the parametric Product Moment Correlation (PMC) test, also known as the Pearson Correlation test, was used. For non-normally distributed data, the non-parametric Spearman Rank Correlation test was applied (Antia, 2020). The next step involved conducting a Two-Way Analysis of Variance (ANOVA), which requires data to be both normally distributed and homogeneous. If these conditions were not met, the non-parametric Friedman's Two-Way Analysis test was used instead. Finally, the data were analyzed using Duncan's test to determine significant differences between variations.

RESULTS AND DISCUSSION

Bittern coagulant characteristics

The bittern used in this study was taken from a salt pond located in Tambak Cemandi Village, Sedati District, Sidoarjo Regency, East Java, Indonesia. The sampling was carried out in October. The characteristics of seawater coagulants from salt production residue (bittern) in this study are shown in Table 1.

The measured salinity value of the bittern coagulant was 49.3‰. The results of the initial characteristic tests for other parameters are as follows: temperature of 33 °C; pH of 6.9; turbidity of 49.2 NTU; magnesium ion concentration (Mg^{2+}) of 20.550 mg/L; and chloride ion concentration (Cl^{-}) of 232.142 mg/L.

Batik industry wastewater characteristics

The wastewater of the batik industry used in this study was taken from industry X located in Jetis Batik Village, Sidoarjo Regency, East Java, Indonesia. The characteristics of the batik industry wastewater in this study are shown in Table 2.

The initial characteristics of batik wastewater indicate that the concentrations of BOD, COD, and TSS parameters exceed the quality standards stated in the Regulation of the Minister of Environment and Forestry Number P.16/MENLHK/SETJEN/KUM.1/4.2019 concerning Wastewater Quality Standards for the textile industry. Therefore, processing is needed to reduce the concentrations of these parameters, so that they would not cause pollution (Fitriani et al., 2023c).

The effect of bittern coagulant dosage and rapid stirring speed on biological oxygen demand (BOD) concentration in batik industry wastewater

BOD measurement was carried out using the Winkler method based on Indonesian National Standard SNI 6989.72:2009. The graph of the

Table 1. Bittern coagulant characteristics

No.	Parameter	Unit	Value
1.	pH	–	6.9
2.	Temperature	°C	33
3.	Color	–	Clear yellowish
4.	Salinity	‰	49.3
5.	Turbidity	NTU	49.2
6.	Magnesium (Mg^{2+})	mg/L	20.550
7.	Chloride (Cl^{-})	mg/L	232.142

Table 2. Batik industry wastewater characteristics

Sampling number	Parameter						
	pH	Temperature (°C)	Color	COD (mg/L)	BOD (mg/L)	TSS (mg/L)	Dye absorbance
1	10.2	27.5	Dark red	9.925	211.19	2.126	0.220
2	11.7	28.0		10.508	153.66	2.158	0.190
3	10.6	26.5		9.841	192.08	2.410	0.226

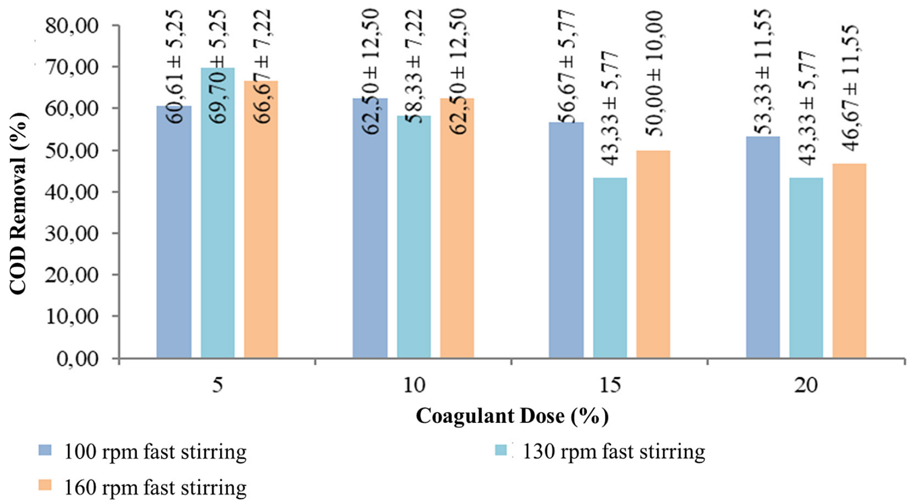


Figure 1. Average percentage reduction in BOD concentration based on bittern coagulant dose (%)

percentage decrease in BOD concentration based on the bittern coagulant dose along with its standard deviation is shown in Figure 1. On the basis of Figure 1, the percentage of BOD reduction varies with different coagulant doses at the same stirring speed, ranging from 38% to 75%. At a 10% coagulant dose with a stirring speed of 100 rpm, the reduction percentage increased to 62.50 ± 12.50%. However, with 15% and 20% coagulant doses, there was a decrease in the percentage of BOD reduction. The graph shows that at stirring speeds of 130 rpm and 160 rpm, the reduction percentage tends to decrease with increasing coagulant doses. This occurs because the waste reaches a saturated condition, where the bittern coagulant can no longer form floc clumps, causing the floc to become brittle and break easily (Alnawajha et al., 2022; Owodunni et al., 2023). Excessive coagulant addition leads to overabsorption of cations by colloidal particles in the batik industry wastewater, resulting in positively charged colloidal particles that repel each other, causing floc deflocculation (Kurniawan et al., 2022b; Owodunni et al., 2023). Rapid stirring speed significantly affects the effectiveness of the coagulation-flocculation process. Both excessive and insufficient stirring speeds can reduce the efficiency of solids removal. Once the floc reaches a saturated condition, increasing the stirring speed will not increase its size. Instead, higher stirring speeds decrease coagulation effectiveness, as the flocs break down into smaller particles that are difficult to settle (Ahmad et al., 2022b; Alnawajha et al., 2024). Figure 2 illustrates the percentage decrease in BOD concentration based on rapid stirring speed, along with its standard deviation.

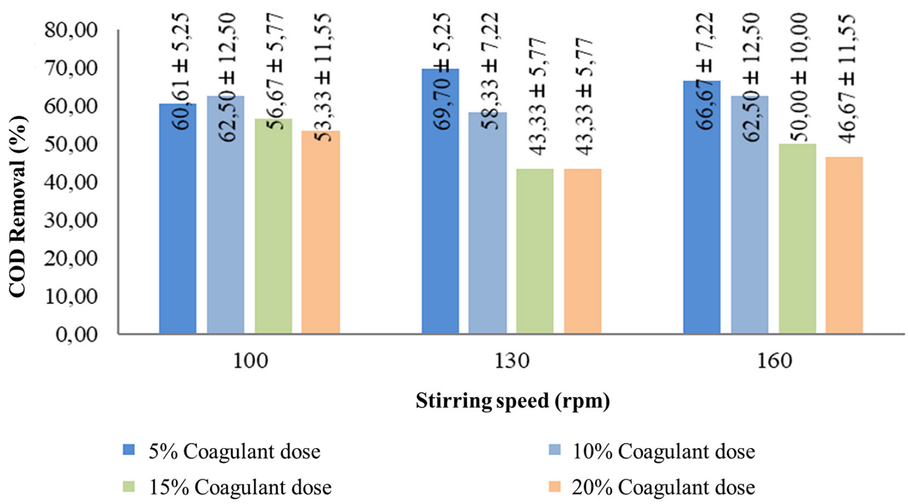


Figure 2. Average percentage reduction in BOD concentration based on fast stirring speed (rpm)

With a coagulant dose of 5%, the percentage reduction in BOD concentration increases as the fast stirring speed increases from 100 rpm to 130 rpm, but then decreases at a stirring speed of 160 rpm. This reduction at 160 rpm can be attributed to the excessive stirring speed, which causes the flocs to break down into smaller particles that are difficult to settle. At stirring speeds of 130 rpm and 160 rpm, the percentage reduction in BOD concentration tends to decrease as the coagulant dose increases. According to the percentage data of BOD reduction, the normality test and Levene's test for homogeneity yielded p-values of 0.346 and 0.709, respectively, indicating that the data are normally distributed and homogeneous (p-value > 0.05). The correlation test results showed a correlation coefficient of -0.650 for the coagulant dose and -0.067 for the fast stirring speed. The p-value ≠ 0.000 indicates that H_{01} is rejected while H_{a1} is accepted. This means there is a moderate negative correlation between coagulant dose and BOD reduction percentage, and a weak negative correlation between fast stirring speed and BOD reduction percentage (AL Falahi et al., 2021; Nasir et al., 2023). These values suggest that increasing the bittern coagulant dose and the stirring speed results in a lower percentage of BOD reduction. The detailed correlation test results are presented in Table 3. On the basis of the results of the Two-Way ANOVA statistical test, a p-value of 0.000 was obtained, indicating a significant difference (p-value < 0.05). DMRT was performed at a significance level (α) of 0.05. The DMRT results showed no significant difference between the addition of 5% and 10% doses. Therefore, a 5% dose is considered more effective, as it requires a smaller volume of bittern coagulant. The percentage of BOD reduction did not show a significant difference concerning the fast stirring speed. However, the DMRT test data revealed that the highest average percentage reduction was achieved at a fast stirring speed of 100 rpm.

The effect of bittern coagulant dosage and rapid stirring speed on chemical oxygen demand (COD) concentration in batik industry wastewater

COD measurement was carried out using the closed reflux spectrophotometric method based on SNI 6989.2:2009. The wavelength used was 600 nm. The percentage graph of COD concentration reduction based on the bittern coagulant dose along with its standard deviation is shown in Figure 3.

Figure 3 illustrates that the average percentage reduction in COD concentration varies with different dosages of bittern coagulant, ranging from 42.84% to 83.46%. The lowest reduction percentage, 42.84%, is observed at a 20% dose with a fast stirring speed of 160 rpm, while the highest reduction percentage, 83.46%, occurs at a 5% dose with a fast stirring speed of 160 rpm.

On the basis of the research results shown in Figure 3, it is evident that as the coagulant dose increases, the percentage of COD concentration removal decreases. This decrease can be attributed to the restabilization process of colloidal particles caused by excessive doses. During this process, the generally negatively charged colloidal particles become positively charged, leading to repulsive forces between them. As a result, larger flocs cannot form, increasing the levels of suspended substances and, consequently, higher COD levels in the sample (Ahmad et al., 2022a; Kurniawan et al., 2021).

Table 3. Results of pearson correlation statistical test of BOD reduction percentage

Parameter		Coagulant dose	Fast stirring speed	%Removal BOD
Coagulant dose	Pearson correlation	1	0.000	-0.650**
	Sig. (2-tailed)		1.000	0.000
	N	36	36	36
Fast stirring speed	Pearson correlation	0.000	1	-0.067
	Sig. (2-tailed)	1.000		0.699
	N	36	36	36
%Removal BOD	Pearson correlation	-0.650**	-0.067	1
	Sig. (2-tailed)	0.000	0.699	
	N	36	36	36

Note: **correlation is significant at the 0.01 level (2-tailed)

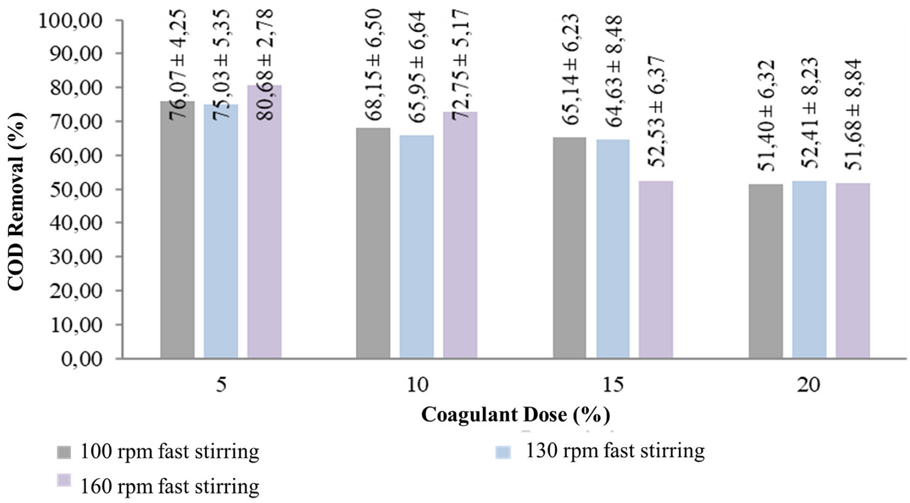


Figure 3. Average percentage reduction in COD concentration based on bittern coagulant dose (%)

Fast stirring speed significantly affects the reduction of suspended solids concentration, which in turn influences the high COD concentration in batik industry wastewater using the coagulation-flocculation method. According to (Alnawajha et al., 2022), particle impact speed is proportional to acceleration, and acceleration is related to the shear force in water, which can affect the formation of microfloc. Therefore, excessively fast stirring can result in suboptimal collisions between pollutant particles and coagulants due to the large shear force in the water, which hinders the formation of microfloc. Figure 4 presents the graph of the percentage decrease in COD concentration based on fast stirring speed, along with its standard deviation.

The decrease in COD concentration based on varying stirring speeds is influenced by the addition of coagulant dose to the sample. At doses of 15% and 20%, the percentage of COD removal decreases as the stirring speed increases. This occurs because excessively high stirring speeds result in suboptimal collisions between colloidal particles and coagulants, hindering proper microfloc formation and leading to imperfect sedimentation. Consequently, colloidal particles are not effectively removed, preventing a decrease in COD concentration (Alnawajha et al., 2024, 2023). According to Kurniawan et al. (2022b), excess cations can disrupt the colloidal stability process. Aktas et al. (2012) stated that stirring speeds exceeding the optimum no longer increase floc size, as the floc reaches a saturated condition. When the stirring speed surpasses the optimum level, flocs break down into smaller particles that are difficult to settle.

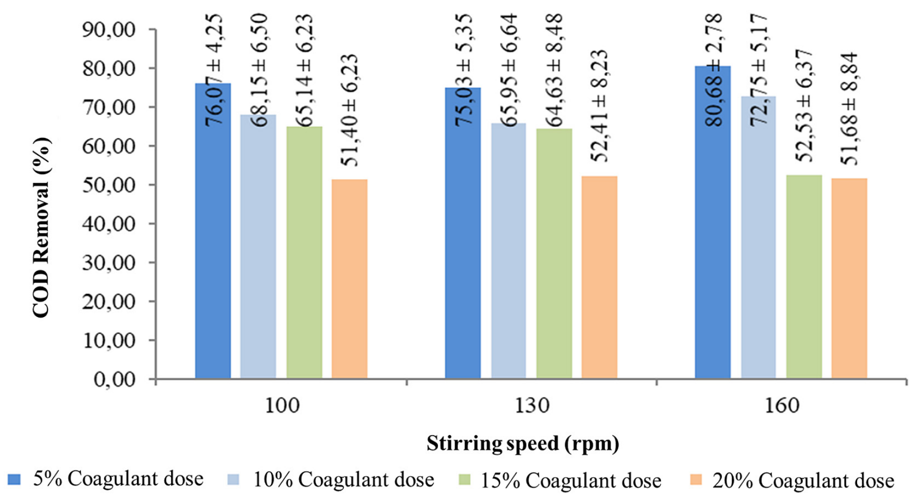


Figure 4. Average percentage reduction in COD concentration based on fast stirring speed (rpm)

On the basis of the COD reduction percentage data, the normality test and Levene's homogeneity test yielded p-values of 0.211 and 0.808, respectively, indicating that the data are normally distributed and homogeneous (p-value > 0.05). The correlation test results showed a correlation coefficient of -0.830 for the coagulant dose and -0.028 for the fast stirring speed. Since the p-value ≠ 0.000, H01 is rejected and Ha1 is accepted. According to Tangahu et al. (2018) and Wan Jusoh et al. (2023), the correlation between variations in coagulant dose and fast stirring speed with the percentage reduction in COD concentration shows a moderate negative correlation for coagulant dose and a weak negative correlation for stirring speed. This negative correlation suggests that higher doses of bittern coagulant and increased stirring speeds result in lower percentages of COD reduction. The detailed results of the correlation test are presented in Table 4.

The Two-Way ANOVA statistical test yielded a p-value of 0.000 for the variation in coagulant doses, indicating a significant difference (p-value < 0.05). The DMRT was performed at a significance level (α) of 0.05. The DMRT results showed that the highest average reduction in COD concentration was achieved with a 5% coagulant dose. Thus, a 5% dose is considered effective, as it requires a smaller volume of bittern coagulant. For variations in fast stirring speed, no significant difference was observed in COD concentration reduction. However, the DMRT test data indicated that a stirring speed of 100 rpm produced the highest average reduction in COD concentration.

Effect of bittern coagulant dosage and rapid stirring speed on total suspended solids (TSS) concentration in batik industry wastewater

TSS measurement was conducted using gravimetric method based on SNI 06-6989.3:2004.

Table 4. Results of pearson correlation statistical test of COD reduction percentage

Parameter		Coagulant dose	Fast stirring speed	%Removal COD
Coagulant dose	Pearson correlation	1	0.000	-0.830**
	Sig. (2-tailed)		1.000	0.000
	N	36	36	36
Fast stirring speed	Pearson correlation	0.000	1	-0.028
	Sig. (2-tailed)	1.000		0.871
	N	36	36	36
%Removal COD	Pearson correlation	-0.830**	-0.028	1
	Sig. (2-tailed)	0.000	0.871	
	N	36	36	36

Note: **Correlation is significant at the 0.01 level (2-tailed)

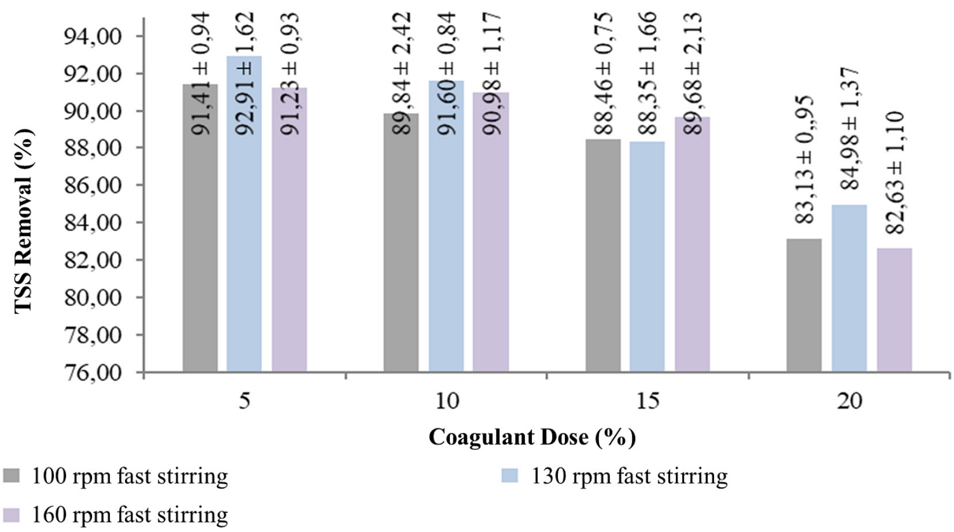


Figure 5. Average percentage decrease in TSS concentration based on bittern coagulant dose (%)

The percentage graph of TSS concentration reduction based on bittern coagulant dose along with its standard deviation is shown in Figure 5.

Figure 5 shows that the average percentage reduction in TSS concentration varies with different bittern coagulant doses, ranging from 81.53% to 94.53%. The lowest reduction, 81.53%, is observed with a 20% coagulant dose at a fast stirring speed of 160 rpm. In contrast, the highest reduction, 94.53%, is achieved with a 5% coagulant dose at a stirring speed of 130 rpm.

Figure 5 shows that higher coagulant doses result in a smaller percentage of TSS concentration removal. This occurs because excessive coagulant addition does not enhance sediment formation but rather disrupts it, leading to increased turbidity in the wastewater. When the coagulant and flocculant are in excess, they can cause deflocculation or restabilization of colloids, as the colloid particles become electrically charged and repel each other, causing previously formed flocs to break apart (Ahmad et al., 2021b, 2021a). Moreover, if the coagulant dose exceeds the optimal level, it will not form effective flocs but will instead increase the number of particles in the sample, negatively impacting the TSS value.

Fast stirring speed is a crucial factor affecting the reduction of TSS concentration in batik industrial wastewater using the coagulation-flocculation method. The fast stirring speed influences the frequency of collisions between particles, which affects particle destabilization and the formation of microflocs (Ahmad et al., 2021b, 2021a). Excessive stirring speeds can cause the microflocs to break apart and become unstable. Conversely, stirring speeds that are too slow result in suboptimal particle collisions, reducing the effectiveness of the coagulant in binding contaminant particles in the wastewater (Lin et al., 2021). Figure 6 displays the percentage decrease in TSS concentration based on fast stirring speed, along with its standard deviation.

Figure 6 indicates that the percentage decrease in TSS concentration increased at a stirring speed of 130 rpm but then decreased at a stirring speed of 160 rpm. This decrease at higher stirring speeds can occur because excessive stirring can reduce coagulation effectiveness by causing microflocs to break down into smaller particles that are difficult to settle (Alnawajha et al., 2024, 2022). These findings align with research by Asward et al. (2019), which indicates that increasing stirring speed leads to a decrease in TSS removal efficiency.

The percentage data for TSS reduction were subjected to statistical testing. The normality test and Levene's homogeneity test yielded p-values of 0.708 and 0.437, respectively, indicating that the data are normally distributed and homogeneous (p-value > 0.05). The correlation test results showed a correlation coefficient of -0.861 for coagulant dose and 0.049 for fast stirring speed. Since the p-value ≠ 0.000, H_{01} is rejected and H_{a1} is accepted. According to Fitriani et al. (2023), the correlation between variations in coagulant dose and fast stirring speed with the percentage reduction in TSS concentration shows a moderate negative correlation for coagulant dose and a weak positive correlation for stirring speed. The detailed results of the correlation test are presented in Table 5. The Two-Way ANOVA statistical test yielded a p-value of 0.000

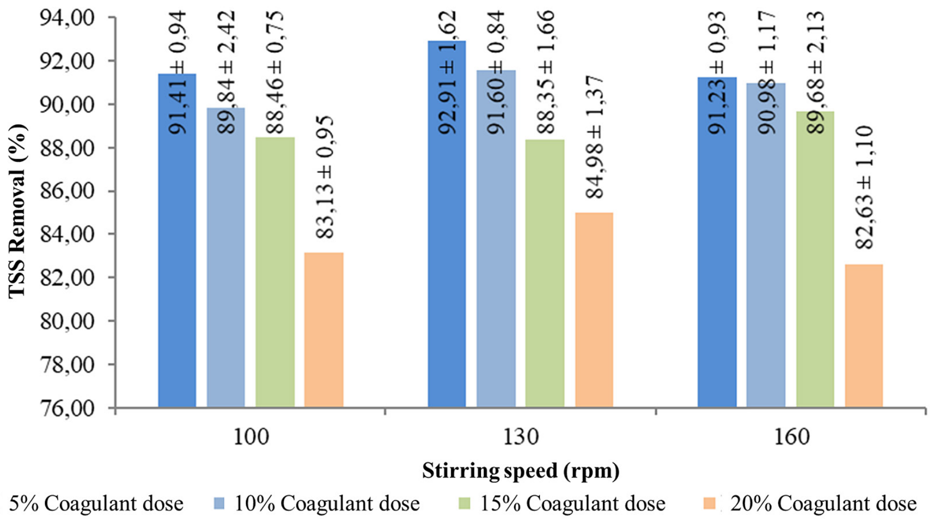


Figure 6. Average percentage reduction in COD concentration based on fast stirring speed (rpm)

Table 5. Results of Pearson correlation statistical test of percentage of TSS reduction

		Coagulant dose	Fast stirring speed	%Removal TSS
Coagulant dose	Pearson correlation	1	.000	-.861**
	Sig. (2-tailed)		1.000	.000
	N	36	36	36
Fast stirring speed	Pearson correlation	.000	1	.049
	Sig. (2-tailed)	1.000		.871
	N	36	36	36
%Removal TSS	Pearson correlation	-.861**	.049	1
	Sig. (2-tailed)	.000	.871	
	N	36	36	36

Note: **Correlation is significant at the 0.01 level (2-tailed)

for the variation in coagulant doses, indicating a significant difference (p-value < 0.05). The DMRT was conducted at a significance level (α) of 0.05. The DMRT results revealed no significant difference between the 5% and 10% coagulant doses. However, the highest average reduction in TSS concentration was observed at a 5% dose, making it the most effective as it requires a smaller volume of bittern coagulant. In contrast, no significant difference was found in TSS concentration reduction with variations in fast stirring speed.

The effect of bittern coagulant dosage and rapid stirring speed on the absorbance of dye in batik industry wastewater

Measurement of dye parameters was carried out using the Spectrophotometry method. The optimum wavelength obtained based on preliminary tests was 400 nm, so that the complementary color of the sample was yellow-green (Underwood and Day, 2003). The graph of the percentage decrease in dye absorbance based on the bittern coagulant dose along with its standard deviation is shown in Figure 7.

The dye used in the batik industry wastewater studied here is naphthol ($C_{10}H_8O$). Under alkaline waste conditions, the hydroxyl group in naphthol dye becomes destabilized. The hydroxyl ions in this dye react with magnesium ions to form a precipitate (Romano et al., 2023). Magnesium hydroxide ($Mg(OH)_2$) precipitate, characterized by its large adsorptive surface area and positively charged surface, facilitates a reduction in dye absorbance in batik waste through charge neutralization and an adsorptive coagulation mechanism. Consequently, bittern is considered an effective coagulant for removing dye absorbance due to its high magnesium ion (Mg^{2+}) content (Albuquerque, 2013).

The results of this study exhibit variability, with the highest percentage decrease in dye

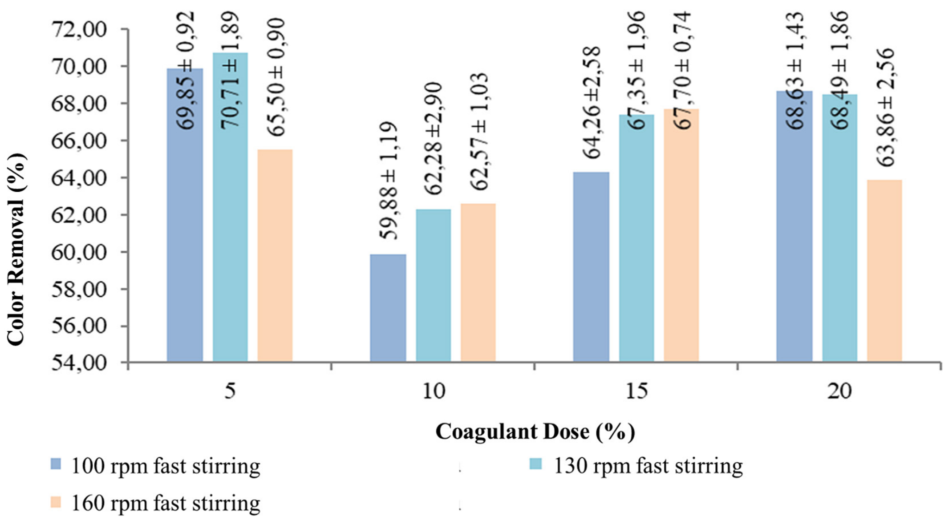


Figure 7. Average percentage decrease in dye absorbance based on bittern coagulant dose (%)

absorbance achieved using a 5% dose at a fast stirring speed of 130 rpm. The data indicate that increasing the dose does not consistently lead to a greater reduction in dye absorbance. A significant decrease in dye absorbance at a 10% dose is observed due to the sample pH being below the optimal range for magnesium hydroxide ($Mg(OH)_2$) precipitate formation, which inhibits precipitate formation (Romano et al., 2023). Figure 8 presents the graph of the percentage decrease in dye absorbance based on fast stirring speed, along with its standard deviation.

Figure 8 demonstrates that the results for the three variations in fast stirring speed at each dose show minimal differences. The analysis reveals fluctuations in dye absorbance values with changes in stirring speed. A decrease in the percentage reduction of dye absorbance may occur due to insufficient stirring, which prevents even dispersion of the coagulant in the batik industry wastewater, thereby reducing the effectiveness of coagulation-flocculation. Conversely, excessive stirring can also lead to decreased reduction percentages as it causes flocs to break down again, resulting in increased turbidity in the waste (Chik et al., 2024; Kurniawan et al., 2023a).

According to the percentage data for dye absorbance reduction, the normality test and Levene's homogeneity test resulted in p-values of 0.509 and 0.181, respectively, indicating that the data are normally distributed and homogeneous (p-value > 0.05). The correlation test showed p-values of -0.006 for the coagulant dose and -0.086 for the fast stirring speed. Since the p-value ≠ 0.000, H_{01} is rejected and H_{a1} is accepted. According to Nimatuzahroh et al., (2022), the correlation between variations in fast stirring speed and the percentage reduction in dye absorbance reveals a weak negative correlation. This suggests that higher doses of bittern coagulant and faster

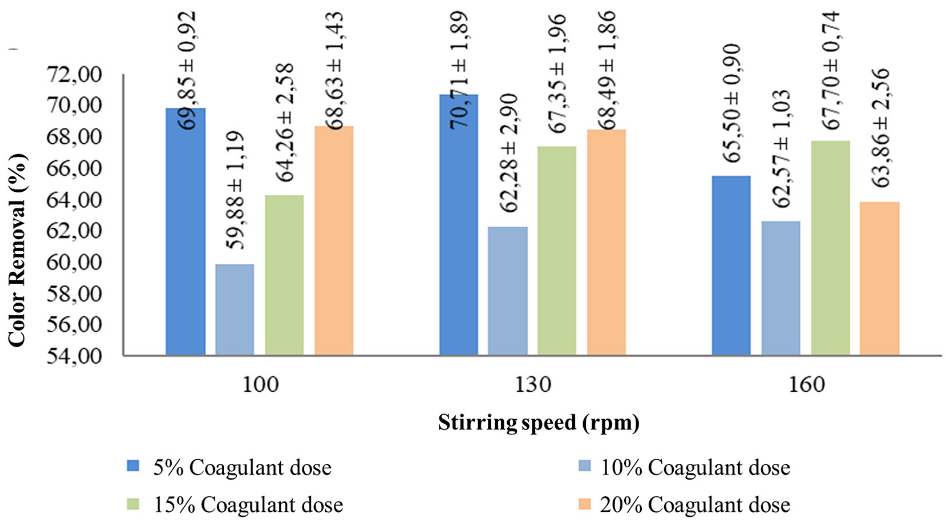


Figure 8. Average percentage decrease in dye absorbance based on rapid stirring speed (rpm)

Table 6. Results of Pearson correlation statistical test percentage reduction in dye absorbance

Parameter		Coagulant dose	Fast stirring speed	%Removal dye
Coagulant dose	Pearson correlation	1	0.000	-0.006
	Sig. (2-tailed)		1.000	0.970
	N	36	36	36
Fast stirring speed	Pearson correlation	0.000	1	-0.086
	Sig. (2-tailed)	1.000		0.619
	N	36	36	36
%Removal dye	Pearson correlation	-0.006	-0.086	1
	Sig. (2-tailed)	0.970	0.619	
	N	36	36	36

Note:**Correlation is significant at the 0.01 level (2-tailed) stirring speeds result in lower percentages of dye absorbance reduction. The detailed results of the correlation test are presented in Table 6.

The two-way ANOVA statistical test produced a p-value of 0.000, indicating a significant difference (p-value < 0.05). The DMRT was conducted at a significance level (α) of 0.05. The DMRT results revealed that a 5% coagulant dose combined with a fast stirring speed of 130 rpm significantly differed in dye removal percentage.

Coagulant dosage and rapid stirring speed that produce optimum percentage reduction in BOD, COD, TSS concentrations and dye absorbance in batik industry wastewater using bittern coagulant

On the basis of the analysis results, the average percentage reduction in BOD, COD, TSS, and dye absorbance concentrations can be seen in Table 7.

Table 7 presents the results of percentage reductions in BOD, COD, TSS, and dye absorbance concentrations achieved through coagulation-flocculation using various bittern coagulant doses. The highest average BOD removal, at 65.86%, was achieved with a 5% coagulant dose at a stirring speed of 130 rpm. The maximum COD concentration reduction, 83.46%, occurred with a 5% coagulant dose and a stirring speed of 160 rpm. The greatest TSS reduction, reaching 94.53%, was observed with a 5% coagulant dose at a stirring speed of 130 rpm. Dye absorbance was reduced by up to 72.60% using bittern coagulants, with the most effective reduction achieved with a 5% coagulant dose and a stirring speed of 130 rpm. Overall, the optimal results for all four parameters were obtained with a 5% bittern coagulant addition.

Determining optimal conditions involves several factors, including the percentage reduction of parameters, energy efficiency, and adherence to quality standards. According to the Regulation of the Minister of Environment and Forestry Number P.16/MENLHK/SETJEN/KUM.1/4.2019, which amends the Regulation of the Minister of Environment No. 5 of 2014 concerning wastewater quality standards for the textile industry, the thresholds are set at 60 mg/L for BOD, 150 mg/L for COD, and 50 mg/L for TSS for industries discharging less than 100 m^3/day. The color parameter threshold is 200 mg/L Pt-Co. Although the parameters have not fully met these quality standards, the final BOD concentration is close to the standard value. Statistical analysis indicates significant differences in the reduction percentages of BOD, COD, TSS, and dye concentrations based on coagulant dose variations, but no significant difference was observed with stirring speed variations. Thus, the stirring speed that consumes the least energy, 100 rpm, was selected.

On the basis of the considerations above, the optimal conditions identified in this study for bittern coagulant dosage and stirring speed are a 5% coagulant dose with a stirring speed of 100 rpm. Under these conditions (D1K1 treatment), the maximum reductions achieved were 65.86% for BOD concentration, 80.32% for COD concentration, 92.35% for TSS concentration, and 70.77% for dye absorbance. Referring to the performance

Table 7. Average percentage reduction in BOD, COD, TSS concentrations, and dye absorbance (%)

No	Code	BOD removal (%)	COD removal (%)	TSS removal (%)	Dye removal (%)
1	D1K1	60.61 ± 5.25	76.07 ± 4.25	91.41 ± 0.94	68.85 ± 0.92
2	D2K1	62.50 ± 12.50	68.15 ± 6.50	89.84 ± 2.42	59.88 ± 1.19
3	D3K1	56.67 ± 5.77	65.14 ± 6.23	88.46 ± 0.75	64.26 ± 2.58
4	D4K1	53.33 ± 11.55	51.40 ± 6.32	83.13 ± 0.95	68.63 ± 1.43
5	D1K2	69.70 ± 5.25	75.03 ± 5.35	92.91 ± 1.62	70.71 ± 1.89
6	D2K2	58.33 ± 7.22	65.95 ± 6.64	91.60 ± 0.84	62.28 ± 2.90
7	D3K2	43.33 ± 5.77	64.63 ± 8.48	88.35 ±1.66	67.35 ± 1.96
8	D4K2	43.33 ± 5.77	52.41 ± 8.23	84.98 ±1.37	68.49 ± 1.86
9	D1K3	66.67 ± 7.22	80.68 ± 2.78	91.23 ± 0.93	65.50 ± 0.90
10	D2K3	62.50 ± 12.50	72.75 ± 5.17	90.98 ±1.17	62.57 ± 1.03
11	D3K3	50.00 ± 10.00	52.53 ± 6.37	89.68 ± 2.13	67.70 ± 0.74
12	D4K3	46.67 ± 11.55	51.68 ± 8.84	82.63 ± 1.10	63.86 ± 2.56

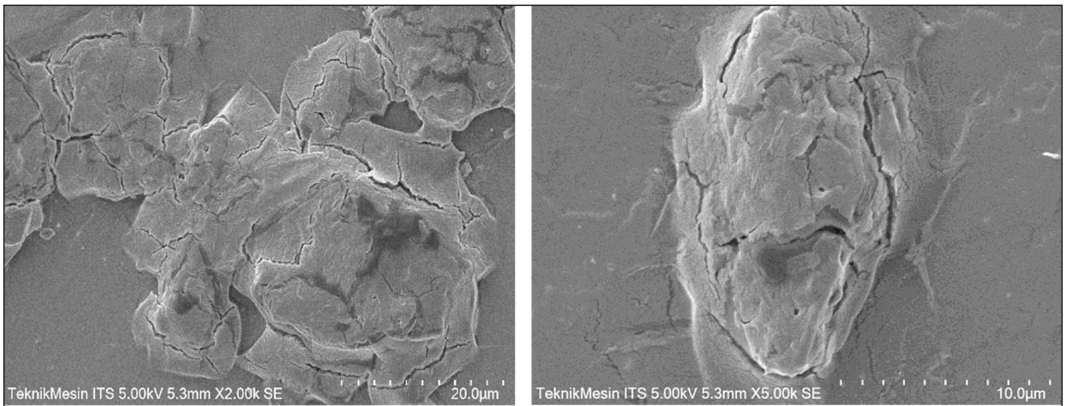
Note: explanation: a. code D (coagulant dose): D1 = 5%, D2 = 10%, D3 = 15%, D4 = 20%, b. code K (Fast stirring speed): K1 = 100 rpm, K2 = 130 rpm, K3 = 160 rpm.

of bittern as coagulant with exceptional results, further optimization regarding the treatment condition needs to be conducted to obtain a better removal. In addition to that, research to treat other types of wastewater can also be conducted to enrich the knowledge on the use of this bittern coagulant.

Characteristics of bittern coagulation and coagulation-flocculation sediment in batik industry wastewater based on scanning electron microscopy (SEM) and energy dispersive X-Ray spectroscopy (EDX) test results

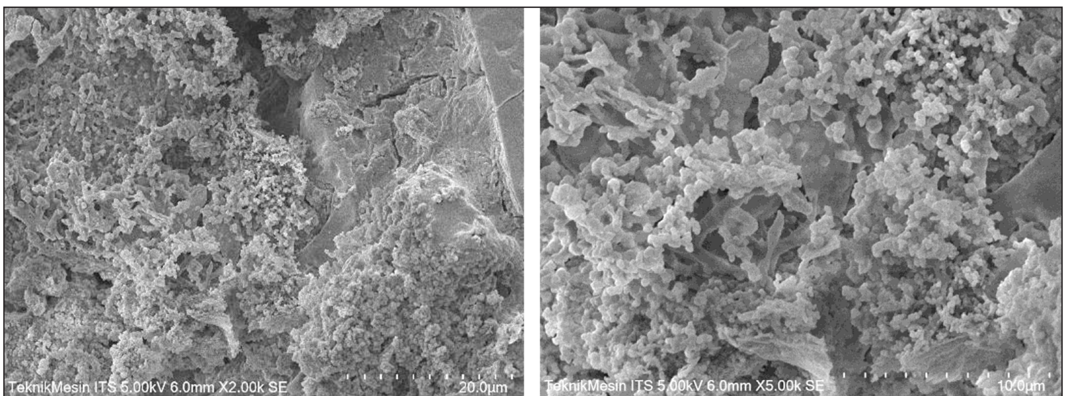
The SEM-EDX method for characterizing the mineral types in bittern and floc coagulants provides valuable information about the chemical compounds present in these substances by analyzing their elemental and oxide content. This technique also helps explore the physical and chemical properties of these compounds (Almansoory et al., 2021; Dampang et al., 2021). The analysis was conducted at magnifications of 2.000× and 5.000×. Morphological images of the bittern and sediment coagulants are shown in Figures 9 and 10.

On the basis of the SEM analysis shown in Figures 9 and 10, the bittern coagulant and the produced floc exhibit non-uniform shapes. The bittern coagulant appears as chunks with a relatively rough surface and numerous pores, and its size is noticeably larger than that of the floc at the same magnification. The research by Farmitha and Vaithyanathan (2015) indicated that at a magnification of 15,000×, the bittern sample has a particle size of 59 nm. In contrast, the floc from the coagulation-flocculation process using bittern coagulant tends to form irregular lumps with a relatively smooth surface. The bright colors in the images highlight elements with high atomic numbers, while the dark colors represent elements with lower atomic numbers. The next step in the analysis is energy dispersive X-Ray spectroscopy (EDX). The results of



(A) (B)

Figure 9. SEM micrographs of bittern coagulant. A) 2.000× magnification; B) 5.000× magnification



(A) (B)

Figure 10. SEM micrograph of the precipitate. A) 2.000× magnification; B) 5.000× magnification

Table 8. Composition of bittern coagulant and floc of batik industry wastewater

Element	Bittern coagulant (% mass)	Precipitate (% mass)
C	-	19.68
N	-	5.46
O	22.39	29.71
Na	22.94	10.14
Mg	8.91	6.25
Al	0.27	1.67
Si	0.32	0.48
S	5.86	5.80
Cl	37.53	19.40
K	1.29	1.42
Ca	0.49	–

the EDX spectrum analysis for the bittern and floc coagulants are detailed in Table 8.

Table 8 shows notable differences in the percentage weight of chemical compounds between the bittern coagulant and the resulting floc. The bittern coagulant is primarily composed of chloride, sodium, oxygen, and magnesium. In contrast, floc is dominated by oxygen, carbon, chloride, sodium, and magnesium. The comparison reveals that the floc contains carbon (C) and nitrogen (N) compounds, which were not detected in the bittern coagulant. This indicates that carbon and nitrogen may originate from the batik industry wastewater and become incorporated and precipitated during the coagulation-flocculation process.The comparison of component compositions reveals that the organic materials from the batik industry wastewater settle out as a result of the sedimentation process following the addition of bittern coagulant. The increase in the values of elements such as carbon (C), nitrogen (N), and oxygen (O) after the coagulation-flocculation process indicates that the bittern coagulant effectively removes organic contaminants through the combined processes of coagulation, flocculation, and sedimentation.

CONCLUSIONS

Treatment of dye containing wastewater (originated from batik industry) was successfully conducted by using bittern as coagulant. The study results indicated a correlation between the reductions in BOD, COD, TSS concentrations, and dye absorbance with variations in coagulant dosage and stirring speed. The optimal conditions for reducing these parameters in batik industry wastewater were found at bittern coagulant dose of 5% combined with a stirring speed of 100 rpm. Under these conditions, the percentage reductions achieved were 80.32% for BOD, 65.86% for COD, 92.35% for TSS, and 70.77% for dye absorbance. Higher coagulant dose and stirring speed gave a negative effect to the overall removal performance. Further optimization related to the treatment condition and trial for other types of wastewaters can be conducted to enrich the knowledge on the utilization of bittern as coagulant.

Acknowledgement

The authors would like to acknowledge Universitas Airlangga through the scheme of PKDN 1308/UN.3/LPPM/PT.01.03/2023 for funding this research.

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