Enhancement of Coffee Processing Mill Effluent Treatment by Coagulation-Flocculation: A Comparative Study of Commercial Coagulants and the Effect of pH

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ABSTRACT

The effectiveness of chemical coagulation-flocculation for treating coffee processing mill effluent (CPME) was evaluated in this study using various commercial coagulants, such as ferric chloride, aluminium sulphate (alum), polyaluminum chloride, ferrous sulphate, chitosan, alum-lime, and ferric chloride-lime. Untreated CPME initially had a pH of 4.5, which increased to between 5 and 9 after pH adjustment with 0.25 M sodium hydroxide. Alum demonstrated the best performance, achieving 98% turbidity removal, 30% COD reduction, 85% NH_3-N removal, and complete iron removal at an optimal dosage of 1000 mg/L. The effect of pH on coagulation-flocculation effectiveness was also investigated, with alum being most effective at pH 7–8. While turbidity and iron were effectively removed using alum, its performance in reducing COD and color was limited. Additionally, NH_3-N removal efficiency varied with alum dosage, but residual nitrogen was consistently present in the effluent. As none of the treated samples met the Environmental Quality (Industrial Effluent) Regulation 2009 Standard B discharge limits, particularly for COD and color, post-treatment is necessary. Adsorption is suggested as a viable post-treatment technique to improve CPME treatment quality and ensure compliance with regulatory standards. Integrating coagulation-flocculation with adsorption may offer a more effective and environmentally friendly approach for CPME treatment.

Keywords: Coffee effluent; coagulation-flocculation; commercial coagulants; alum; color removal

INTRODUCTION

Nowadays, coffee is the most popular beverage globally, with coffee beans being the second largest traded commodity after petroleum. Coffee production increased by about 1.5% from 2015 (151.6 million 60-kg bags) to 2016 (153.9 million 60-kg bags) (International Coffee Organization, 2017). Meanwhile, production further increased to more than 170 million 60-kg bags in 2018 (Hejna, 2021). This trend is expected to continue rising over the years to meet global demand. Originating from the *Rubiaceae* family, Coffea is the genus of plants that produce coffee. The most common coffee types in Malaysia are *Coffea liberica* (Liberica) and *Coffea canephora* (Robusta), with Liberica accounting for about 90% of total consumption. (Nor & Wahab, 2016). Additionally, according to Omar et al (2022), coffee consumption in Malaysia increased from 635,000 bags in 2020 to 800,000 bags in 2021. This indicates a rapid increase in coffee demand due to a shift in lifestyle among Malaysians, who now prefer coffee as their main beverage over tea. Coffee beans are typically processed in three ways:wet (washed), dry (natural), and honey (a blend of wet and dry) (Hejna 2021). The wet method is most widely employed in coffee processing plants because it provides coffee with

a superior aroma and flavour than the dry method (Dadi et al. 2018). To produce 1 kg of green coffee beans, approximately 5-15 L of water is consumed (Hue & Bittenbender, 2006), resulting in a significant volume of coffee processing mill effluent (CPME) throughout the de-pulping, fermenting, and washing processes (Said et al. 2020). According to Said et al. (2023), each kilogramme of coffee cherries generates 20-45 kg of CPME.

CPME contains high levels of organic contaminants such pectin, proteins, and sugars (Dadi et al. 2018), as well as low pH levels (3-5) (Abu Hasan et al. 2022). CPME also contains flavonoids, which change the color from green to black but have no effect on chemical oxygen demand (COD) or biological oxygen demand (BOD). Furthermore, the presence of chlorogenic acids renders it unsuitable for plants since it is phytotoxic, reducing seed germination and plant growth (Janissen and Huynh, 2018). If the effluent is discharged into bodies of water without treatment, it can create public health issues such as eye, ear, and skin irritation (Said et al. 2023), breathing difficulties, stomach pain, and dizziness (Haddis and Devi 2008). As a result, all industries must comply with industrial waste standards by creating waste treatment facilities. Malaysia's standard for industrial effluent discharge is stated in the Environmental Quality (Industrial Effluent) Regulation 2009.

Biological treatment is the most common method for treating industrial effluent. However, this method is not appropriate for CPME because it contains a complex structure of macromolecules such as tannins, lignin, phenolics, alkaloids, fatty acids, polysaccharides, and humic acid that are difficult to degrade and inhibit biological growth, (Ijanu et al. 2020; Shah et al. 2023). As a result, it is recommended that CPME treatment plants integrate various types of treatment, such as physical and chemical treatment. Physicochemical treatment, which can break down complex molecules in wastewater in a short period of time under controlled conditions (Ijanu et al. 2020), is expected to be more appropriate for CPME than biological treatment approaches. Furthermore, the treatment technology that will be implemented should be effective and easy to construct, especially for smallholder coffee processing mill.

In this study, CPME was investigated for treatment using the coagulation and flocculation process. Coagulation and flocculation are well established, cost-effective, and user-friendly methods for wastewater treatment (de Barros et al. 2020). Furthermore, this approach effectively removes natural organic matter, color, and turbidity (Haan et al. 2018; Khairuddin et al. 2024). Coagulation neutralizes charges, allowing colloidal particles to aggregate for easier separation from water (Abu Bakar et al. 2021). Flocculation, a slow stirring process, further enlarges these aggregates,

aiding in their settling and filtration. The choice of coagulants significantly affects coagulation-flocculation performance. Although coagulation-flocculation is widely used in industrial effluent treatment, most studies have focused on general industrial effluent rather than real coffee effluent (Ijanu et al. 2020; Shah et al. 2023). The complex composition of coffee wastewater, including tannins, lignin, and phenolics, presents unique challenges that existing studies have not adequately addressed. This gap limits comparisons of commercial coagulants and the identification of optimal treatment techniques. To address this gap, this study was conducted to assess the efficacy of various commercial coagulants in coagulation-flocculation and to determine the optimal process parameters for treating the CPME.

MATERIALS AND METHOD

CHARACTERISTICS OF COFFEE PROCESSING MILL EFFLUENT

CPME was collected from a coffee-processing mill in the northern part of Malaysia and stored at 4°C prior to use. The characteristics of the effluent were tabulated in Table 1 and compared with Standard B of the Environmental Quality (Industrial Effluent) Regulation 2009. The characteristics of coffee effluent significantly exceed discharge standards and require proper treatment.

TABLE 1. Characteristics of coffee processing mill effluent

Parameter	Unit	Value	Standard B
pH		4.54	5.5-9.0
Color	ADMI	1848	200
NH_3-N	mg/L	104	20
Phosphate	mg/L	10.2	-
COD	mg/L	6657	200
Turbidity	mg/L	51	-
Suspended solids	mg/L	0.184	100
Iron	mg/L	2.14	5.0

PERFORMANCE TEST

COAGULATION-FLOCCULATION

Seven commercial coagulants were evaluated, including ferric chloride, aluminium sulphate (alum), polyaluminum chloride, ferrous sulphate, chitosan, alum-lime, and ferric chloride-lime through Jar test experiment. Figure 1 depicts the flocculator used during the Jar test. The standard Jar

test was performed using 400 mL of coffee effluent at an initial pH 4.54. Coagulant dosages of 500 mg/L, 750 mg/L, and 1000 mg/L were applied for each type of coagulant.

Level of turbidity, COD, color, suspended solid, phosphate, iron, NH_3-N, and pH were determined before and after the coagulation-flocculations.

FIGURE 1. Flocculator used in the coffee processing mill effluent treatment

EFFECT OF PH

The effect of pH on the coagulation-flocculation process with the chosen coagulant was investigated by adding 400 mL of samples to beakers. The pH values investigated were 5, 6, 7, 8, and 9. The pH was adjusted at room temperature with 0.25 M sodium hydroxide (NaOH). The coagulant dosages of 500 mg/L, 750 mg/L, and 1000 mg/L were added, and the solution was vigorously stirred for 1 minute, followed by slow-speed stirring for an additional 30 minutes. The sample was allowed to settle for 30 minutes. Turbidity, COD, color, suspended solid, phosphate, iron, NH_3-N, and pH were measured before and after the Jar test.

ANALYTICAL METHOD

COD was determined using the reactor digestion method with a time-lapse heating reactor (HACH DRB200) and a spectrophotometer (HACH DR3900). NH_3-N and phosphate were analyzed using the Nessler and PhosVer 3 methods, respectively. Color was measured using the ADMI weighted ordinate method with the HACH DR3900 spectrophotometer. Suspended solids were determined using the photometric method with the HACH DR3900, while iron was measured via atomic absorption spectroscopy (AAS) (Perkin Elmer AA400). Turbidity was assessed with a turbidimeter (HACH 2100AN), and pH was measured using a pH meter (Metrohm 827 pH Lab, USA).

RESULTS AND DISCUSSION

COAGULANT SCREENING

Figure 2 illustrates the removal efficiency of CPME parameters at various dosages using different approaches. COD was reduced by 8%, 10%, and 22% with alum dosages at of 500 mg/L, 750 mg/L, and 1000 mg/L, respectively, while other coagulants showed no significant COD reduction. Furthermore, alum consistently removed 100% iron at all doses, whereas chitosan removed 70%, 90%, and 100% of the iron at 500 mg/L, 750 mg/L, and 1000 mg/L, respectively. Chitosan's efficiency is due to its capacity to adsorb metal ions through both chemical and physical mechanisms (Pradhan et al. 2005).

Phosphate and NH_3-N removal can occur through charge neutralization and sweep flocculation mechanisms. Figure 2(b) shows that the combination of alum and lime achieves the highest phosphate removal. This is due to the addition of lime, which forms an insoluble compound, calcium hydroxyapatite, aiding in the removal of phosphate. As the removal of organic components is minimal, there is no significant change in the color of the coffee effluent.

Turbidity has a linear relationship with suspended solids (Hannouche et al. 2011). Consequently, the trend in turbidity removal closely follows that of suspended solids. As shown in Figure 2(d), alum, ferric chloride, PAC, and ferrous sulphate effectively removed turbidity. This removal is primarily attributed to charge neutralisation and sweep flocculation mechanisms.
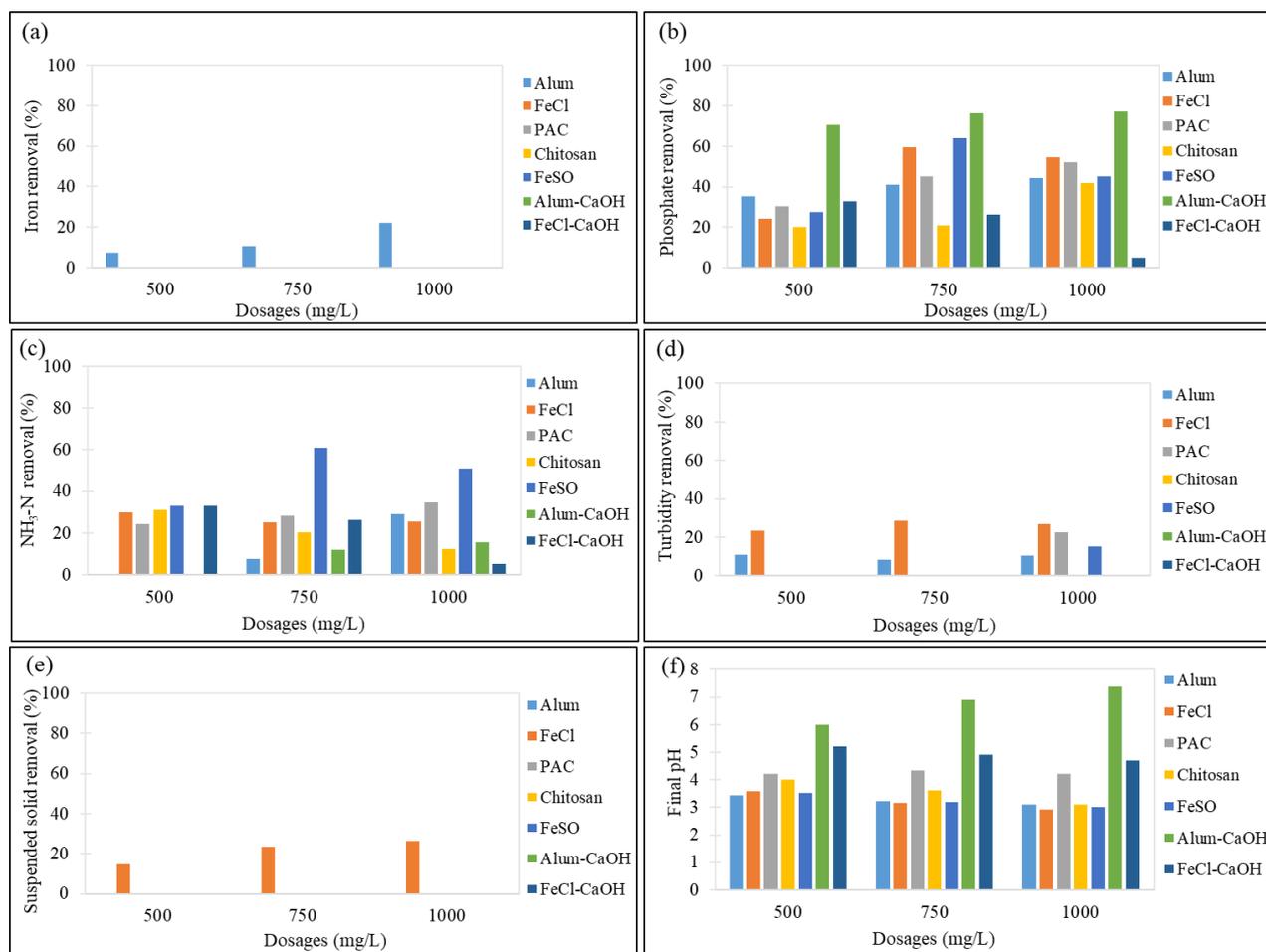
FIGURE 2. Removal efficiencies of (a) iron, (b) phosphate, (c) NH$_3$-N, (d) turbidity, and (e) suspended solids at various dosages using different coagulant

pH plays a significant role in the coagulation and flocculation process. When alum, ferric chloride, or ferrous sulfate are added to CPME, they undergo hydrolysis, releasing hydrogen ions into the water and causing a reduction in pH. According to Świderska-Bróz and Rak (2009), PAC can effectively destabilize negatively charged colloidal particles. As a result, PAC causes the pH to decrease more gradually than alum. Chitosan, which is synthesized in an acidic medium, lowers the pH when introduced. In contrast, lime is used to raise the alkalinity of the effluent. As seen in Figure 2(f), increased lime dosages result in higher pH values.

The removal trends for phosphate, NH$_3$-N, and suspended particles were identical. Alum and ferric chloride outperformed other compounds in terms of turbidity removal. Furthermore, alum demonstrated superior effectiveness in reducing COD and iron levels during the coagulation-flocculation treatment of CPME.

EFFECT OF PH ON PERFORMANCE OF ALUM

The effectiveness of alum as a coagulant in the coagulation-flocculation process was evaluated under the pH effects. This study used 0.25M NaOH to modify the pH of CPME. The study's results were explained by removing the residual parameters.

Figure 3 shows the performance of COD removal using alum at 500 mg/L, 750 mg/L, and 1000 mg/L at different pH values. The trend shows that at 500 mg/L and 750 mg/L, the highest COD removal occurred at pH 7, whereas at 1000 mg/L the optimal removal was at pH 8. The reason for this trend lies in the coagulation mechanism of alum, which is dosage- and pH-dependent (Liu et al. 2024). Under reduced dosages, charge neutralization dominates, where alum species effectively destabilize organic matter at pH 7. With the alum dosage increasing

to 1000 mg/L, sweep flocculation is favored, where excess coagulant forms larger flocs that enhance pollutant removal, particularly at pH 8. However, under such optimum conditions, excessive alum can lead to restabilization of particles or disrupt coagulation and hence reduce COD removal efficiency. Despite these removal efficiencies, the COD levels remained above the discharge limit, highlighting the need for additional treatment measures. This explains the observed variation of peak performance with rising alum dosage.
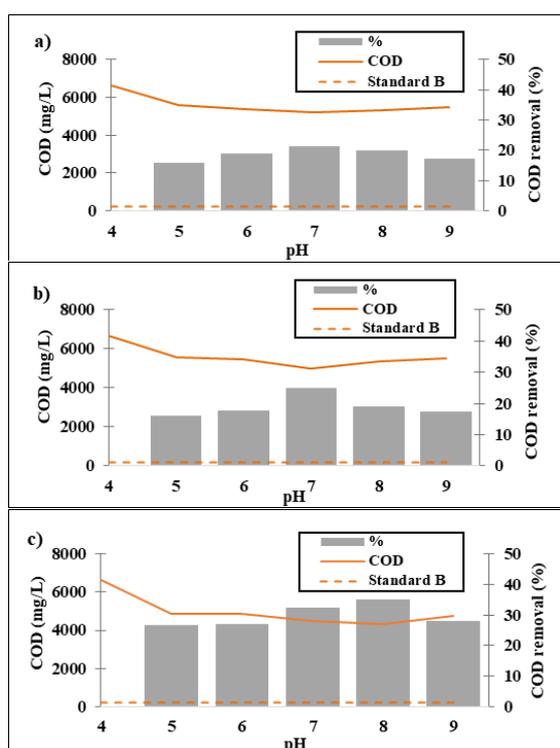
FIGURE 3. COD removal efficiency using alum at different dosages: (a) 500 mg/L, (b) 750 mg/L, and (c) 1000 mg/L.

Figure 4 illustrates iron removal using alum as a coagulant at dosages of 500 mg/L, 750 mg/L, and 1000 mg/L. The figure shows that the final iron concentration for all dosages was significantly below Standard B, demonstrating alum's strong capability in iron removal. The observed trend can be attributed to the formation of insoluble iron hydroxides, which readily precipitate from solution in the presence of alum (Krupińska 2021). At lower dosages of 500 mg/L and 750 mg/L, alum species facilitate adsorption and co-precipitation of iron, leading to effective removal. As the alum dose increases to 1000 mg/L, the excess aluminum ions enhance precipitation, resulting in greater iron removal. In contrast to COD removal, which varies depending on pH and its interaction with organic matter, iron removal remains consistently high across different dosages due to the strong affinity between iron

and hydroxide precipitates. Consequently, iron removal efficiency remains stable across all alum dosages while staying within discharge standards.
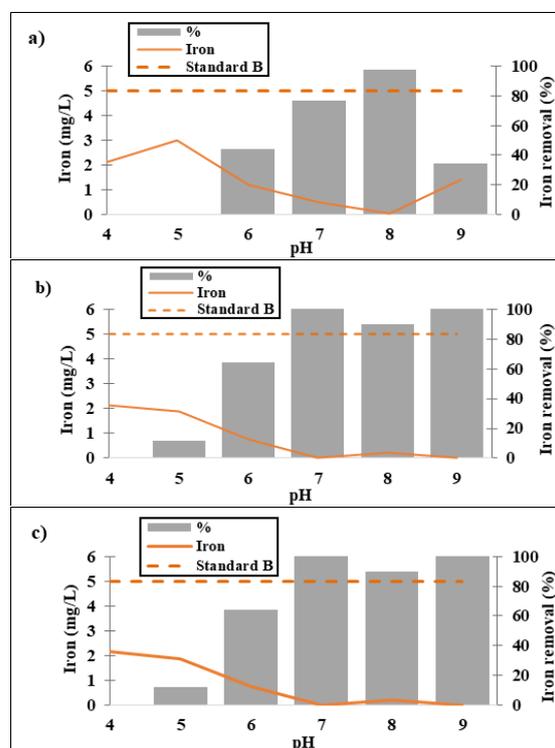

FIGURE 4. Iron removal efficiency using alum at different dosages: (a) 500 mg/L, (b) 750 mg/L, and (c) 1000 mg/L.

Figure 5 illustrates NH_3-N removal using alum at concentrations of 500 mg/L, 750 mg/L, and 1000 mg/L. The highest removal of NH_3-N for all alum dosages was more than 80% at pH 7. The NH_3-N concentration at pH 7 with alum doses of 500 mg/L and 1000 mg/L significantly lower than Standard B, indicating effective NH_3-N removal. The trend is dominated by the adsorption and entrapment of ammonium (NH_4^+) in aluminum hydroxide flocs, rather than direct chemical precipitation (Muscarella et al. 2021). At pH 7, the dominant species present in solution is NH_4^+, and its removal efficiency is higher at higher concentrations of alum since there is a greater amount of available aluminum hydroxide flocs, which are utilized as adsorption surfaces. The marginal increase in removal efficiency from 750 mg/L to 1000 mg/L suggests that the excess alum promotes the floc formation, thereby increasing NH_4^+ entrapment in the settling flocs. However, unlike COD and iron removal, NH_3-N removal is more pH-dependent since an increase in pH above 7 would facilitate the conversion of NH_4^+ to gaseous NH_3. This would reduce the efficiency of removal by coagulation-flocculation. For this reason, NH_3-N removal is most effective at a pH of 7, with efficiency improving as alum

dosage increases. However, excessive dosages result in diminishing returns. Excess NH_3-N contributes to eutrophication, leading to algal blooms, oxygen depletion, and imbalances in aquatic ecosystems. Therefore, optimizing NH_3-N removal is crucial to meeting discharge limits in CPME treatment plants, where nitrogen-rich effluent poses significant environmental risks.
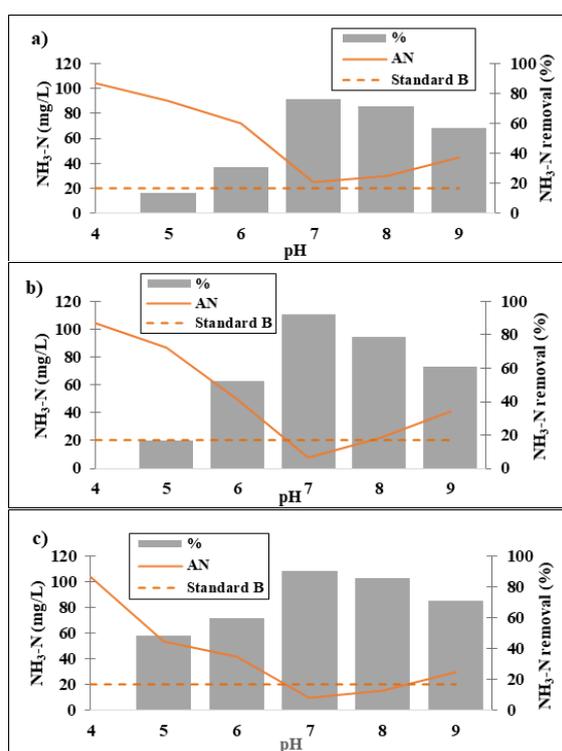

FIGURE 5. NH_3-N removal efficiency using alum at different dosages: (a) 500 mg/L, (b) 750 mg/L, and (c) 1000 mg/L.

In this study, color removal using alum was tested at dosage levels of 500 mg/L, 750 mg/L, and 1000 mg/L. It was found that even when there was an increment in alum dosage, color reduction was below 5% and was an ineffective treatment. The reason is that the color-generating compounds are likely dissolved organic molecules that tend not to coagulate using alum. Unlike metal ions or suspended solids, these dissolved constituents weakly interact with aluminum hydroxide flocs and hence cannot be efficiently removed through typical coagulation-flocculation. In addition, stabilizing functional groups of such compounds, as in the case of humic or fulvic acids, could inhibit destabilization effectiveness and consequently lower removal even further (Zhu et al. 2023).

Figure 6 illustrates turbidity removal using alum at doses of 500 mg/L, 750 mg/L, and 1000 mg/L. At pH 7, all the doses exhibited the highest percentage of turbidity removal, which shows that the coagulation-flocculation process is most effective at near-neutral conditions. This trend is primarily controlled by the physical agglomeration of suspended particles, rather than chemical interactions like in COD, iron, or NH_3-N removal. Aluminum hydroxide flocs are most effectively created at pH 7, which has the capability of trapping and bridging suspended solids and leading to successful sedimentation (Asharuddin et al. 2023). Under low or high pH levels, solubility in alum is higher, which decreases the supply of solid hydroxide flocs needed for efficient turbidity removal (Verma et al. 2022). This leads to poor floc structure and a reduction in removal effectiveness. Moreover, increasing the alum dosage enhances turbidity removal to a certain extent. However, excessive dosage can lead to the restabilization of colloids due to the formation of overly dispersed aluminum species. This explains the situation where high turbidity removal achieved at pH 7 regardless of dosing but applying more alum does not necessarily improve performance. High turbidity in CPME reduces sunlight penetration, disrupting aquatic ecosystems. The study's findings confirm that alum effectively clarifies water, making it more suitable for discharge or reuse in agriculture and industry, highlighting its potential as a low-cost treatment option.

Figure 7 shows the final pH values following alum treatment at dosages of 500 mg/L, 750 mg/L, and 1000 mg/L. The results indicate a slight decrease in pH upon adding alum, with the decrease becoming more pronounced as the alum dosage increases. This trend is due to alum being acidic and hydrolyzing in water to release hydrogen ions (H^+) that lower the pH (Zhang et al. 2021). At lower dosages, the amount of H^+ released is minimal and only slightly lowers the pH. As the dosage of alum increases, however, more sulfate (SO_4^{2-}) ions are broken down, releasing more H^+ and lowering the pH more. Unlike the removal processes shown in Figures 3–6, which rely on coagulation-flocculation, precipitation, or adsorption, the trend in Figure 7 is driven solely by a chemical reaction between water and alum. The extent of the pH drop also depends on the alkalinity of the CPME sample. Highly alkaline CPME will neutralize some of the acidity, resulting in a smaller reduction in pH, whereas low-alkalinity CPME will experience a more significant drop. This is why the pH drops more as the alum dosage increases, but the extent of the drop depends on the composition of the water. pH fluctuations can impact downstream treatments, as biological processes rely on stable pH for microbial activity, while adsorption systems may require pH adjustments for optimal performance. This can increase operational costs, making alum dosage optimization crucial for cost-effective CPME treatment.
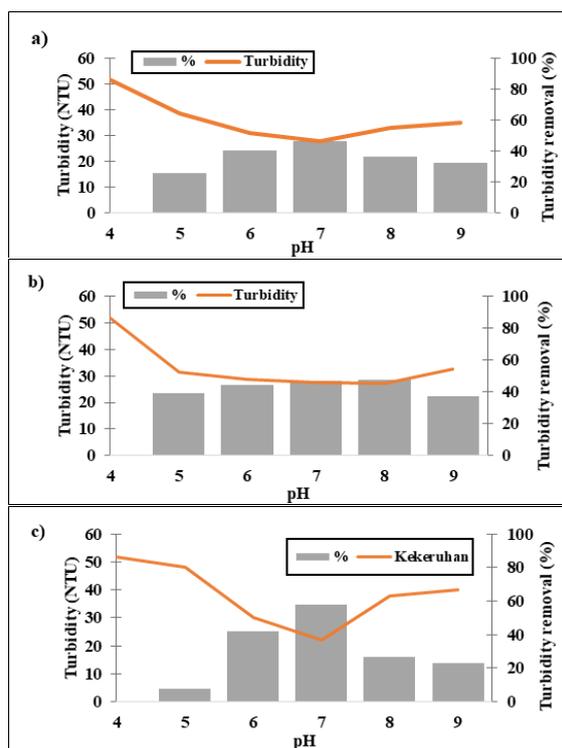
FIGURE 6. Turbidity removal efficiency using alum at different dosages: (a) 500 mg/L, (b) 750 mg/L, and (c) 1000 mg/L.
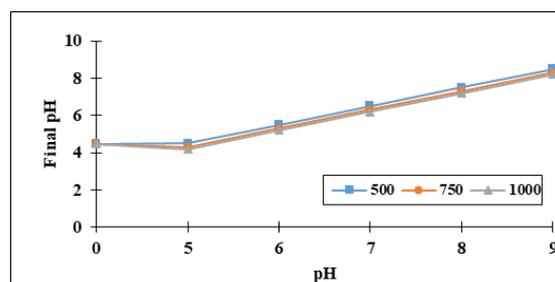

FIGURE 7. Final pH after treatment using different alum dosages: (a) 500 mg/L, (b) 750 mg/L, and (c) 1000 mg/L.

COMPARISON WITH PREVIOUS STUDIES

At pH 7, alum shown good turbidity removal (98%), surpassing the 66.9% obtained by Piper sarmentosum (Shah et al. 2023). However, COD removal (30%) is far lower than the 98.2% COD reduction achieved by Getahun et al. (2024a) using natural coagulants at the same pH (Table 2). This clear disparity implies that alum is significantly less efficient in removing dissolved organic pollutants than natural coagulants, most likely because of variations in coagulating processes and pollutant interactions.

TABLE 2. Performance of certain coagulants/flocculants in coffee wastewater treatment using coagulation–flocculation steps

Coagulant/flocculant materials	Dosage	Wastewater characteristics	Experimental conditions	Removal efficiency (%)	Reference
Alum	1 g/L	pH: 4.54, Color: 1848 ADMI, NH_3-N: 104 mg/L, Phosphate: 10.2 mg/L, COD: 6657 mg/L, Turbidity: 51 NTU, Iron: 2.14 mg/L,	pH: 7-8, Rapid mixing for 1 min, slow mixing for 30 min, settling time of 30 min	Turbidity: 98%, COD: 30%, NH_3-N: 85%, Iron: 100%	This study (2025)
Moringa oleifera	0.04 g/L	pH: 4, Turbidity: 600 NTU	pH: 4, Rapid mixing at 150 rpm for 2 min, slow mixing at 40 rpm for 20 min, sedimentation period of 24 h	Turbidity: 62%	Said et al. (2024)
Three natural coagulants: Acanthus sennii C., Moringa stenopetala B., Aloe vera L.	1.25 g/L	pH: 4.75, Color: 3 Abs, Turbidity: 145 NTU, COD: 7603 mg/L, Nitrate: 20.21 mg/L, Phosphate: 9.10 mg/L	pH: 7, Mixing at 80 rpm for 30 min	COD: 98.24%	Getahun et al. (2024a)
Three natural coagulants: Acanthus sennii C., Moringa stenopetala B., Aloe vera L.	0.75 g/L	pH: 4.75, Color: 3 Abs, Turbidity: 145 NTU, COD: 7603 mg/L, Nitrate: 20.21 mg/L, Phosphate: 9.10 mg/L	pH: 8.76, Mixing at 80.73 rpm for 19.23 min	Color: 99.99%, Turbidity: 98.7%	Getahun et al. (2024b)

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Piper sarmentosum	2 g/L	pH: 3.41, TSS: 746.67 mg/L, Turbidity: 247.12 NTU, Color: 19,278 ADMI, COD: 5861 mg/L, BOD: 233 mg/L, NH$_3$-N: 224 mg/L	pH: 7, Rapid mixing at 300 rpm for 5 min, slow mixing at 180 rpm for 30 min, settling time of 30 min	TSS: 51.0%, Turbidity: 66.9%	Shah et al. (2023)

Although this study obtained 100% iron and 85% NH$_3$-N removal, which are not stated in other studies, its poor COD removal efficiency raises questions on effluent quality. After treatment, the initial COD concentration (6,657 mg/L) remained high, which confirmed that alum mostly targets suspended particulates rather than dissolved organic matter. Conversely, Getahun et al. (2024b) used natural coagulants at a slightly higher pH (8.76) to exhibit nearly complete color removal (99.9%), suggesting improved pollutant aggregation and removal. Since Abdessemed et al. (2000) combined coagulation-flocculation with adsorption to reduce COD by 82.7%, further treatment is obviously required. The persistent COD levels in this study imply that depending solely on alum is insufficient to meet discharge regulations. Without additional treatment, particularly adsorption, the effluent may continue to pose environmental hazards due to high organic pollutant levels.

CONCLUSIONS

This study examined the effects of pH on coagulation-flocculation processes. At a dosage of 1000 mg/L, alum effectively removed 98% of turbidity, 30% of COD, 85% of NH$_3$-N, and 100% of iron at pH 7-8. The effluent analysis indicated that increasing alum dosage improved treatment results, with notable reductions in NH$_3$-N, iron and turbidity at 500 mg/L, 750 mg/L, and 1000 mg/L alum. However, none of the treatment outcomes met the limits of Environmental Quality (Industrial Effluent) Regulation 2009 Standard B, indicating the need for further treatment, with previous studies suggesting that adsorption could be an effective solution.

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DECLARATION OF COMPETING INTEREST

None.

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