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Parametric study of coagulant recovery from water treatment sludge towards water circular economy

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ABSTRACT

This study aims to recover the used coagulants from two water treatment plants via acidification technique. The water treatment sludge (WTS) was acidified with sulfuric acid (H2SO-4) at variable normalities (0.5, 1, 1.5, 2.0 and 2.5 N). The surface morphology and functionalities of both recovered coagulants were analysed using scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). The performance of recovered coagulants was tested for turbidity removal in surface water treatment at different coagulant dosages and pH. It was found that the optimum normality of H2SO4 for recovered alum was 1.5 N, where 66% turbidity removal was recorded. The recovered PAC treated with 1.0 N H2SO4 indicated high turbidity removal percentage, which was 50.5%. The turbidity removal increased with increasing coagulant dosage. More than 80% turbidity removal was achieved with 40 mg/L dosage of recovered alum and recovered PAC. Maximum removal (85%) was observed with 50 mg/L dosage of recovered alum. For commercial coagulant, the turbidity removal was higher, with a difference of up to 6% in favor of recovered alum. The potential reuse of coagulants can be explored in order to reduce the operating costs and promotes the reduction of WTS disposal.

Key words: alum, coagulant, flocculation, poly aluminum chloride, sludge, turbidity, water treatment

HIGHLIGHTS

- Coagulation in water treatment process generates a large amount of sludge.
- Water treatment sludge (WTS) are commonly discharged into rivers.
- Coagulant recovery from WTS can reduce environmental impact.
- Coagulant recovery is a great approach to support water circular economy.

1. INTRODUCTION

The domestic water treatment process through the circular economy and resilience values promotes opportunities to tackle water challenges through a transformative approach to provide more sustainable, comprehensive, and structured water and sanitation services. Circular economy attempts are responses to the current unsustainable linear model of 'take, make, consume, and waste' to reduce pressure on natural resources and eliminate waste (Gude 2021; Bouziotas *et al.* 2023). Global demand for drinking water has increased with the increase in the world's population, economic development, rapid urbanization, and changes in consumption patterns.

In conventional water treatment plants, the coagulation–flocculation process effectively removes impurities in water, such as chemical oxygen demand, turbidity, color, suspended solids (SS), heavy metals, oil, and organic matter, where chemicals are introduced and small particles begin to clump and become large flocs; thus, safe and clean drinking water is produced for the public (El-Gaayda *et al.* 2021; Abujazar *et al.* 2022). The coagulation process can be considered one of the most typical physicochemical processes in water treatment due to its ease of operation, relatively simple design, and low-energy

consumption (Ahmad *et al.* 2016). This is attributed to the combined cost of coagulant and pH adjustment chemicals as well as the disposal of the resulting sludge or water treatment sludge (WTS) (Keeley *et al.* 2016).

Coagulation generates large amounts of sludge. In fact, almost 10,000 tonnes of sludge are generated per day (globally). WTS undergoes the dewatering process before being discarded into landfill or is directly discharged into water bodies. WTS has a high concentration of organic matter, inorganic salts, and various oxides (Shahin *et al.* 2019). Sludge has pathogenic microorganisms and heavy metals, such as zinc, mercury, chromium, and lead, which are difficult to degrade and potentially contaminate soil and water resources (Xu *et al.* 2019). It can significantly affect water quality and threaten aquatic ecosystems. In addition, the production of WTS is likely to increase as clean drinking water becomes a standard resource. Therefore, coagulant recovery is crucial, so that the metals contained in WTS can be reused and recycled for the subsequent treatment. There are a few methods, including acidification using hydrochloric acid (HCl) and sulfuric acid (H_2SO_4), basification by caustic soda, ion exchange, and electrodialysis processes, that can be employed to recover aluminum and other coagulants from WTS (Ahmad *et al.* 2021). The acidification process is the most investigated process as it is a cost-effective method to recover coagulants. Yang *et al.* (2014) studied the potential of recovered coagulant (treated using H_2SO_4) to remove phosphorus in which the phosphorus removal rates reached nearly 100%. Dahasahastra *et al.* (2022) reported turbidity removal using coagulant recovered from polyaluminum chloride water treatment plants (PAC-WTP) and acidified using HCl.

This study focused on the recovery of alum and PAC coagulants in WTS from two water treatment plants via acidification using H_2SO_4 at different normalities. The performance of recovered alum and recovered PAC was compared with commercial alum in terms of their ability to remove turbidity in surface water treatment under different coagulant dosages and pH values.

2. METHODS

2.1. Sampling

WTS was obtained from two water treatment plants (WTPs) in Malaysia. PAC sludge was generated when PAC was used as a coagulant, while alum sludge was generated when alum salt was employed as a coagulant. Each sludge was collected in the WTP sludge lagoon and stored in plastic containers. Raw water was collected at the inlet of the storage tank. The alum and PAC sludges were oven-dried at 105 °C for 3 h to remove the moisture content. The mass/weight of these two sludges before and after heating were measured and recorded. Then, the sludge cake was crushed to produce sludge powder. This work was conducted at the Water and Wastewater Engineering Laboratory, Universiti Malaysia Terengganu.

2.2. Coagulant recovery from WTS via the acidification method

The alum and PAC sludges were modified using the acidification method, where 97% H_2SO_4 was used. H_2SO_4 with normality values of 0.5, 1.0, 1.5, 2.0, and 2.5 N was prepared by mixing them with 50 mL of deionized water. The alum and PAC sludges were treated with H_2SO_4 and were oven-dried at 105 °C for 3 h to remove the moisture content. The dried sludge cake obtained was ground into powder form and denoted as recovered alum and recovered PAC. The performance of both recovered alum and recovered PAC were evaluated using the jar test experiment.

2.3. Sludge characterization

The powdered sludge samples were characterized using several methods. The chemical composition and surface morphology of the sludge were analyzed using scanning electron microscopy (SEM), JEOL (JSM 6360LA). The sludge samples were also subjected to Fourier-transform infrared (FTIR) spectroscopy in the range of 4,000–600 cm^{-1} to determine the surface functionalities of the sludge. The pH value and solid content of the sludge were measured according to the Standard Methods for Examination Water and Wastewater (APHA 2017).

2.4. Performance evaluation of recovered coagulants in surface water treatment

Turbidity removal was tested using the jar test experiment, where 500 ml of raw water sample was poured into six beakers. Water quality parameters, including turbidity, pH, temperature, and total dissolved solids, were recorded before the experiment was conducted. The prepared coagulants, namely alum, PAC, recovered alum, and recovered PAC, were added into a beaker containing 500 mL of water. The mixture was then stirred at 120 rpm for 2 min, followed by slow stirring at

80 rpm for 20 min. The mixture was kept stagnant for 45 min to allow the flocs to settle down. The sediments formed were removed using the filtration system equipped with a vacuum pump to collect the water.

The performance of both recovered PAC and recovered alum prepared with 0.5, 1.0, 1.5, 2.0, and 2.5 N H_2SO_4 was tested using the method described above. The test was repeated by adding different dosages of recovered PAC and recovered alum (10–60 mg/L). Experimental work was also conducted at different pH values (3, 5, 7, 9, and 11). Water quality was measured and recorded. The best acid concentration was chosen due to the higher percentage of turbidity removal.

3. RESULTS AND DISCUSSION

3.1. Sludge characteristics

The morphology of sludge and recovered sludge was determined using SEM. Figure 1(a) and 1(b) shows the SEM images of both recovered alum and recovered PAC after treatment with H_2SO_4 at 1,500× magnification. SEM analysis displays some of the features of surface morphology, topography, and material structure (Akhtar *et al.* 2018). The SEM images indicated that the recovered coagulant had a crystalline structure and was known to have an amorphous composition as discovered in the previous study (Awab *et al.* 2012). Both images of the coagulants before and after recovery exhibited similar crystal shapes. The alum and PAC (Figure 1(c) and 1(d)) were irregularly shaped. The recovered coagulant surface was covered by acid due to sludge acidification. The sludge surface has highly porous structures which makes the sludge surface sites more adsorptive in nature for most anions (Zand & Hoveidi 2015).

As shown in Figures 2(a) and 2(b), there were some similarities in terms of surface functionalities of both recovered PAC and recovered alum. There was a narrow band at 3,600 cm^{-1}, indicating the presence of oxygen-related bonds in the FTIR spectra of the recovered PAC (Hu *et al.* 2021). The peak observed at 3,000 cm^{-1} indicated the presence of C–C bond. In addition, a weak absorption band found from 2,200 to 2,400 cm^{-1} indicates the presence of a triple bond between carbon atoms (C triple bond C).

In the double bond region (1,500–2,000 cm^{-1}), a medium bond peak was detected around 1,635.64 cm^{-1}. This indicated the presence of a carbonyl double bond. There is a specific and unique region called the fingerprint region (600–1,500 cm^{-1}). Moreover, a weak bond between 912.33 and 1,002.98 cm^{-1} is represented by the multiple absorption band. The number of peaks for recovered alum in Figure 2(b) showed a single bond region (2,500–4,000 cm^{-1}), where a medium

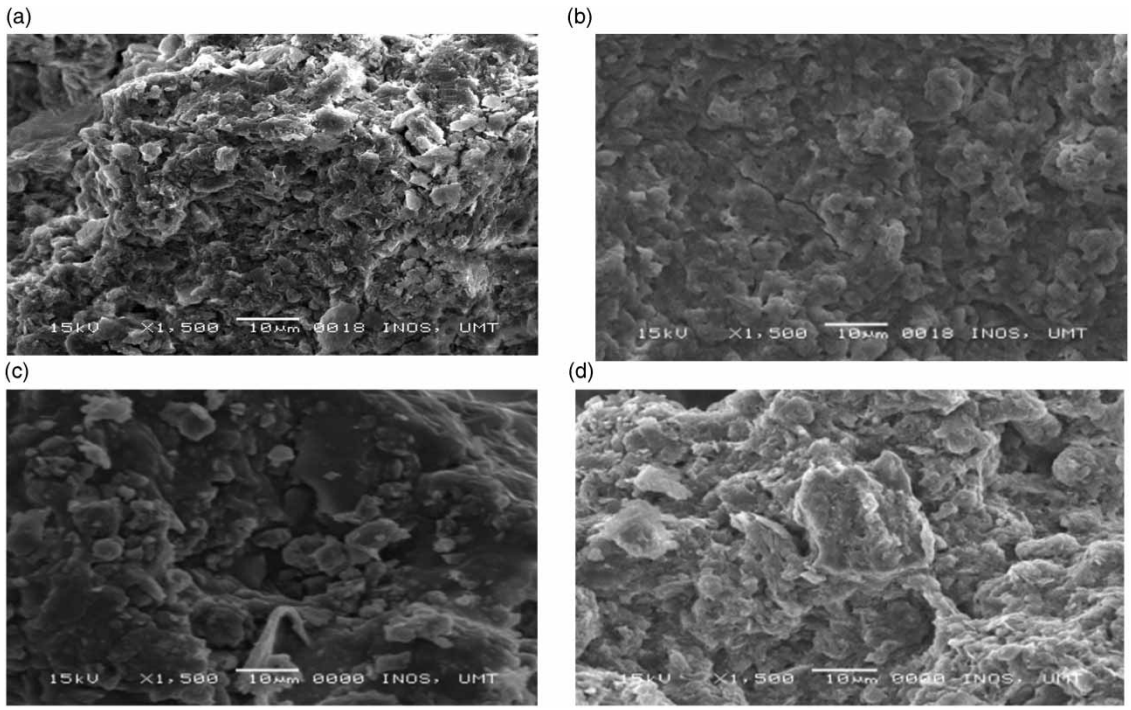

Figure 1 | SEM images at 1,500× magnification of (a) recovered alum, (b) recovered PAC, (c) alum sludge, and (d) PAC sludge.

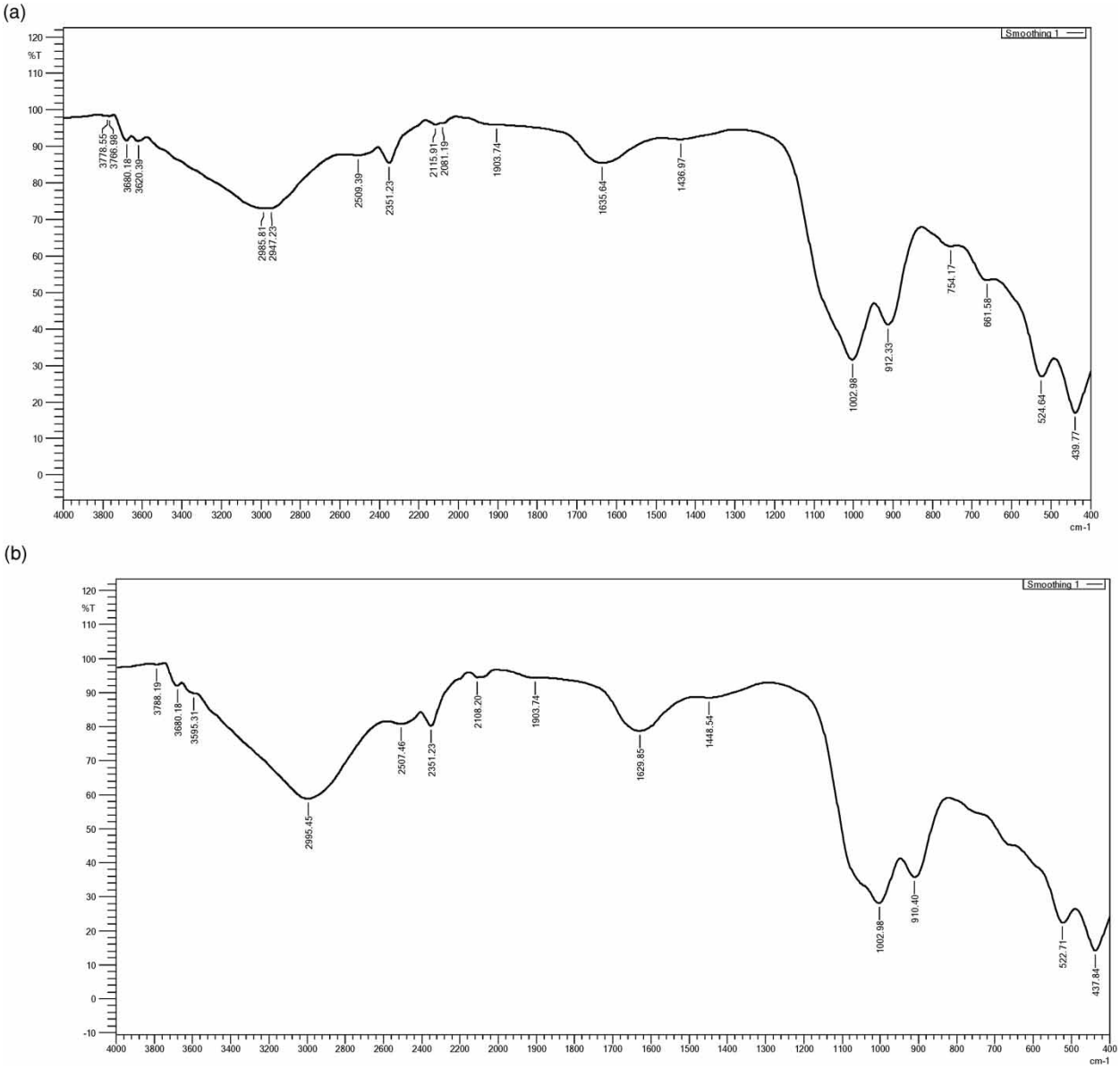

Figure 2 | FTIR spectra of (a) recovered PAC and (b) recovered alum.

narrow bond was found, which defined that there were hydrogen bonds in the material. A medium absorbance peak was detected around 1,629.85 cm^{-1} in the presence of a double bond region. This signified that several carbonyl double bonds existed. The absorption bands from 910.40 to 1,002.98 cm^{-1} indicated multi-band absorption. The IR spectra for both recovered coagulants were almost similar.

3.2. Performance of recovered coagulant in water treatment

Both PAC and alum sludges underwent acidification treatment using 0.5–2.5 N H_2SO_4. As the normality of the acid increased, the percentage of turbidity removal increased to a certain degree and then decreased due to the Al-flocs dissolution process, affecting the coagulation process (Zand & Hoveidi 2015). As shown in Figure 3, the highest turbidity removal percentage was recorded at 1.5 N H_2SO_4 (50.51%) for recovered alum and 1.0 N H_2SO_4 (66.07%) for recovered PAC. Meanwhile, the lowest turbidity removal percentage was observed at 2.0 N H_2SO_4 for recovered alum and 1.5 N H_2SO_4 for recovered PAC, which was suitable for sludge acidification. Turbidity removal increases monotonically for different

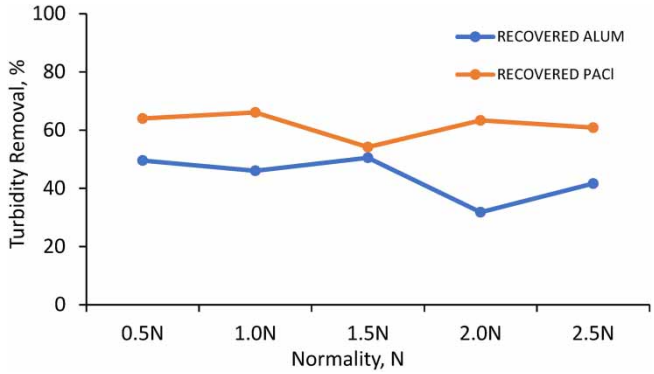

Figure 3 | Turbidity removal percentage of recovered coagulants with different normality of H_2SO_4 (dosage: 40 mg/L).

rates of acid as the coagulant dose also increases (Ahmad *et al.* 2016). The optimum condition of Al-flocs, which is comparatively predominant and adsorbs more colloidal particles, may be due to the presence of more adsorptive sites on aluminum hydroxide flocs (Zand & Hoveidi 2015). Therefore, sludge acidification using 1.5 and 1.0 N H_2SO_4 for each sludge was found to be less efficient in removing suspended colloids from river water.

Figure 4 shows the comparison of turbidity removal for commercial coagulants (alum and PAC) and recovered alum and recovered PAC from WTS. The percentage of turbidity removal increased with increasing coagulant dosage until the maximum removal was achieved and stagnation occurred. It showed significant turbidity removal with several coagulant dosages. More than 80% turbidity removal was achieved with a 40 mg/L dosage of recovered alum and recovered PAC. Low turbidity removal indicates the low concentration of stable particles and it is difficult to coagulate (Wei *et al.* 2015).

The result also showed that the highest turbidity removal of 86% was obtained by recovered alum with 50 mg/L dosage. This study compared the performance of both commercial and recovered alum, where recovered alum exhibited higher turbidity removal of up to 6% compared to commercial alum. The turbidity removal decreased slightly with the addition of more recovered alum from 50 to 60 mg/L due to the destabilization of colloidal particles. Moreover, the turbidity removal percentage of recovered PAC and commercial PAC with 40 mg/L dosage was almost similar at 80%. The percentage of turbidity removal recorded was almost similar for both commercial and recovered PAC, i.e., 97% at 30 mg/L dosage. This proved that that recovered coagulant has the potential to be used in the water treatment process, thus reducing the dependency on commercial coagulant and reducing WTP-operating costs.

From these results, the turbidity removal efficiency was relatively high for both recovered coagulants compared to commercial coagulants. As the coagulant dosage increases, positive charges become more dominant leading to electrostatic repulsion

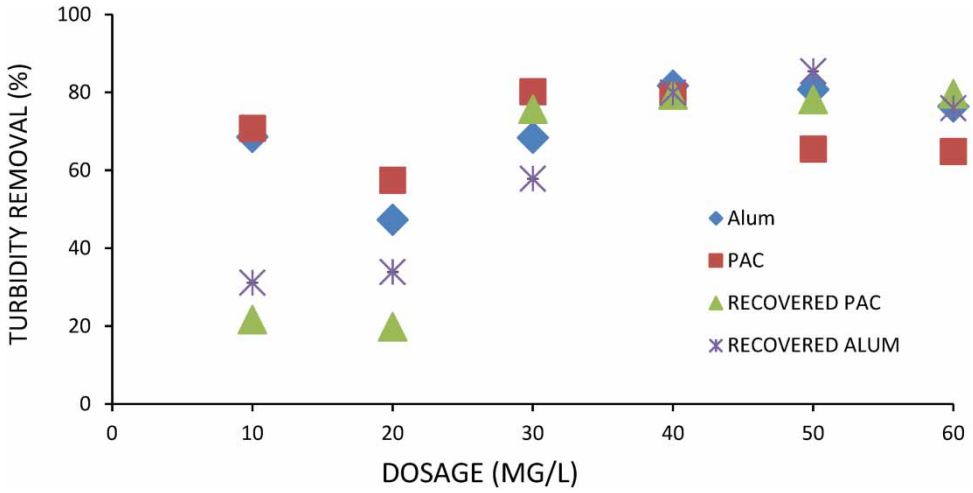

Figure 4 | Effect of different dosages on recovered alum (1.5 N), recovered PAC (1.0 N), commercial alum, and commercial PAC.

Table 1 | ANOVA: the effect of dosage on turbidity removal

ANOVA

Source of variation	SS	df	MS	*F*	*p*-Value	*F*-crit
Between groups	633.7434	3	211.2478	0.500825	0.685973	3.098391
Within groups	8,435.991	20	421.7995			
Total	9,069.734	23				

between particles (Wang *et al.* 2021; Khazaie *et al.* 2022). Table 1 shows the analysis of variance (ANOVA) performed at a significant level (α) of 0.05 with a *p*-value of 0.685973, which was higher than 0.05. It indicated that the rejection of H_o was weak, and there was no statistically significant difference in the mean of the four types of coagulants due to increased dosage.

After the jar test experiment, the pH value of the water samples decreased between 8.4 and 7.49 for recovered alum. and the pH value increased between 6.33 and 7.97 as the dosage increased from 20 to 60 mg/L for recovered PAC (Figure 5). With increasing final pH, the optimum dosage of PAC increased at pH 8 and decreased at pH 6. This suggested that the production of PAC precipitates affects the charge neutralization of coagulation (Mcyotto *et al.* 2021; Zhou *et al.* 2021). The best pH for PAC coagulation is associated with the pH value at which the formation of PAC precipitates is maximized (Ebrahimi *et al.* 2014).

The best performance of alum was seen at pH 7, and its performance decreased to some extent at pH values of 5 and 8. The highest performance of PAC was observed at pH 5 and 6 (Wei *et al.* 2015). Based on Figure 5, it demonstrated that when the dosage increased, the final pH of PAC and recovered PAC increased, but the final pH of alum and recovered alum increased to some extent and then decreased. The pH disturbed the balance of reactions between organic functional groups and hydrogen ions and Al hydrolysis products. At low pH, hydrogen ions compete more vigorously with metal hydrolysis products (Demissie *et al.* 2021; Guo *et al.* 2023). The results showed that turbidity removal was dependent on pH and coagulant dosage. The highest turbidity removal was achieved at pH 5–6 for PAC and pH 6–7 for alum (Wei *et al.* 2015). From Table 2, the significance level (α) of 0.05 and the *p*-value of 0.054202, which was greater than 0.05, implied that there was no statistically significant difference. This result also proved that the rejection of H_o was weak. There was no statistically significant difference in the mean of the final pH with increasing dosage with the four types of coagulants.

Figure 6 displays the curve of turbidity removal percentage at different pH values of water samples. It was revealed that the optimum pH values for the highest turbidity removal for PAC and alum coagulants were between 5 and 7, respectively. For both recovered coagulants, turbidity removal increased in an acidic medium (pH 3–7) and decreased as the pH increased. The maximum turbidity removal rate of 84.32% was achieved at 40 mg/L dosage and pH 7 for recovered PAC, and 82.95% was achieved at 50 mg/L dosage and pH 5 for recovered alum. In a previous study, the recovered coagulant was able to remove 96% of turbidity via chemical treatment with H_2SO_4 (1 M) at a dosage of 12 g/L (Xu *et al.* 2009). Another study conducted by Dahasahastra *et al.* (2022) reported a turbidity removal rate of 74%, which was comparable to the findings of this study. These previous studies underlined the potential of recovered coagulants, such as PAC, in water treatment.

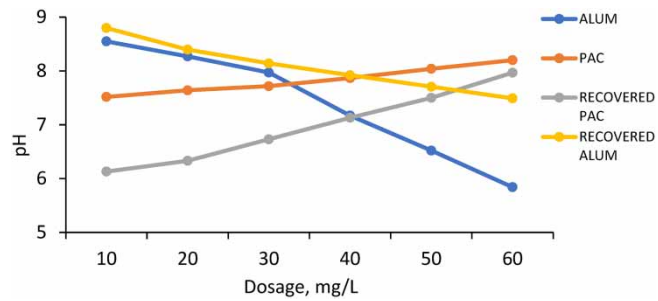

Figure 5 | Final pH against coagulant dosage.

Table 2 | ANOVA: the effect of final pH on dosage

ANOVA

Source of variation	SS	df	MS	*F*	*p*-value	*F*-crit
Between groups	4.336757375	3	1.445585792	3.01274032	0.054202	3.098391
Within groups	9.596484507	20	0.479824225			
Total	13.93324188	23				

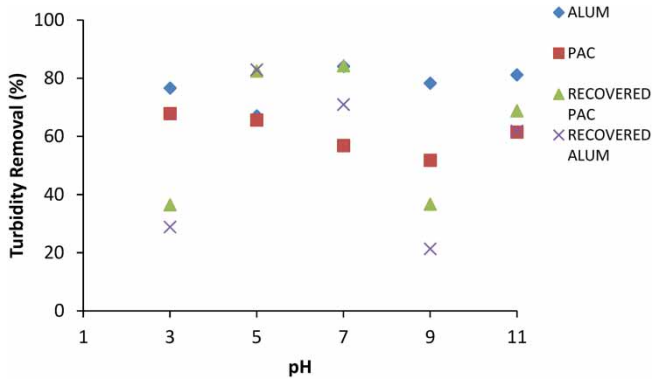

Figure 6 | Turbidity removal percentage against the initial pH of raw water.

Table 3 | ANOVA: the effect of turbidity removal on pH

ANOVA

Source of variation	SS	df	MS	*F*	*p*-value	*F*-crit
Between groups	938.4838572	3	312.827952	2.351433	0.11080052	3.2388715
Within groups	2,128.594884	16	133.03718			
Total	3,067.078741	19				

The effect of pH on turbidity removal using PAC may be related to the formation of high polymeric positive hydrolyzates and $Al(OH)_3$ in the solution with a pH between 6 and 8 (Gong *et al.* 2022). Therefore, the turbidity removal percentage was lower at pH 3, 9, and 11 for both recovered coagulants.

Table 3 presents the ANOVA results at a significant level (α) of 0.05. The *p*-value of 0.11080052 indicated that there was no statistically significant difference. Since the *p*-value was greater than 0.05, the evidence to reject was weak. Thus, there was no statistically significant difference in the mean of the turbidity removal percentage on pH.

Coagulant recovery from WTS is crucial and contributes to the circular economy in the water sector. The WTS collected from the WTP can be recycled and reused in the subsequent cycle of water treatment. The acid digestion method proposed in this study was able to promote high recovery efficiency with good quality recovered coagulant and low treatment cost. This study also promotes the reduction of WTS disposal in the landfills and dependency on commercial coagulants.

4. CONCLUSIONS

The alum and PAC present in WTS sludge could be recovered with the addition of H_2SO_4 with various normalities. The optimum H_2SO_4 normality for recovered alum and recovered PAC were 1.5 and 1.0 N with 66 and 50.5% turbidity removal, respectively. More than 80% turbidity removal was achieved with a 40 mg/L dosage of recovered alum and recovered PAC. The quality of water treated with recovered alum and recovered PAC were almost similar to the commercial alum and PAC (80%). Increasing the dosage of recovered alum to 50 mg/L exhibited an increase in turbidity removal to 85%.

This study compared the performance of commercial and recovered coagulants and found that the recovered alum showed higher turbidity removal of up to 6% compared to the commercial alum. The potential of recovered PAC and recovered alum as coagulants can be further explored to obtain water quality comparable to that treated with commercial coagulants and also beneficial for the reduction of WTP-operating costs. In addition, this study can be the baseline for the future development of more sustainable waste management, particularly in Malaysia. Nevertheless, the performance of recovered alum and recovered PAC must also be tested for the removal of total suspended solids, volatile suspended solids, biochemical oxygen demand and chemical oxygen demand before commercialization and scale-up for the pilot study.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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