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Performance of Recovered Coagulant from Water Treatment Sludge by Acidification Process

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Abstract Aluminium sulphate $(Al_2SO_4)^3$ or commonly known as alum has been used as a coagulation-flocculation agent by water treatment industries around the world, including Malaysia. Regardless of its effectiveness in purifying the raw water, it produces a large volume of alum sludge. In Malaysia, alum sludge is categorized as scheduled waste where it requires to be transported only to approved landfills and the costs could be high. Hence, reusing and recycling the alum sludge may be beneficial for water treatment industries in reducing the disposal cost. In this study, acidification method using nitric acid at a molarity of 0.5M – 2.0M is employed to recover a maximum percentage of aluminium from alum sludge. The findings showed that the difference in molarity of nitric acid is proportional to the recovery rate of aluminium up to 99% of the recovery. The performance of recovered coagulant in removing turbidity from raw water was evaluated at variations of dosage (0.5 – 12 mg/l) and pH (2 – 13). At an optimum dosage of 2 mg/l, the recovered coagulant efficiently removes the turbidity of raw water at 93.32% and achieved 99.47% at pH 7. Consequently, the use of recovered coagulant can promote a sustainable environmental approach to converting waterworks residuals into usable resources, particularly in water/wastewater treatment industries.

Keywords Recovered Coagulant, Recovered Aluminium, Alum Sludge, Acidification, Turbidity Removal

1. Introduction

Drinking water treatment undergoes coagulation and flocculation process to enhance the ability of a water treatment process to eliminate all colloidal and suspended solids from raw water. In the coagulation-flocculation process, a chemical coagulant such as aluminium sulphate $(Al_2SO_4)^3$ or alum is added in untreated water and hydrolysed to form a range of hydrolysis species, which will facilitate colloidal and suspended impurities to accumulate into larger flocs via charge neutralization and sweep-flocculation mechanisms [1]. For many years, alum has been used as a coagulation and flocculating agent by water treatment industries around the world including Malaysia for raw water purifications. This is because alum is the most basic aluminium-based coagulant and inexpensive product available in the water treatment industry. Despite its ability to purify raw water, it generates a significant amount of alum sludge. Consequently, alum sludge has been recognized as the most massive by-product generated and locally available by the water industries globally [2].

Due to the high volume of sludge production and its environmental drawbacks, the most persistent problem associated with alum sludge is finding the most cost-effective and efficient disposal method. As a

common practice in most water treatment plants in Malaysia, water treatment sludge was left to dry in the sludge lagoon with inadequate cover soil before final disposal to the sanitary landfill [3]. Almost two million tonnes of water treatment sludge were produced annually in Malaysia [4] and it will exponentially increase in line with the demand for treated water in the future. Alum sludge is categorized as scheduled waste in Malaysia where it requires to be disposed off in a sanitary landfill. It is estimated that if all alum sludge were handled in drums and sent to Kualiti Alam, the annual cost of disposal of the residue for the entire country could exceed RM10 billion [5]. On top of that, a new water treatment plant would require additional expenses to provide a larger footprint for treatment and storage of the residue. Therefore, the current research aimed at profitable recycles to bridge the gap between the enormous amounts of alum sludge and reduce environmental pollution. The most common disposal methods still rely on land utilization and reuse for agricultural purposes, but there is no attempt to recycle it as a coagulant [6]. Consequently, sustainable approaches are in need, including aluminium recovery from WTS to resolve its environmental impacts and change these residuals into usable resources.

In a comprehensive review by Babatunde and Zhao [7] and Dassanayake et al. [2], suggested three main beneficial applications of alum residual including; (1) environmental usage such as pollutant adsorbent, coagulant aid in wastewater treatment process and as wetland substrates (2) industrial utilization as construction materials, cement and brick manufacturing and (3) land application involving agricultural sector and soil improvements. Dassanayake et al. [2] also highlighted that the aluminium recovery method can be considered as an alternative approach in managing aluminium in WTS for safe utilization in line with sustainability development.

In recent years, acidification, alkalization, ion exchange, and membrane separation are among the common methods used in the recovery of aluminium from water treatment sludge [8]. However, membrane separation and ion exchange are too expensive among these methods. By using these techniques to extract a large amount of alum turns out to be very costly and impractical. Alternatively, the recovery of coagulant using the acidification and alkalization process is inexpensive and very efficient [8].

The effectiveness of aluminium recovery can vary based on the characteristics of raw water. Generally, the physical and chemical characteristics of raw WTS are mainly affected by the dosage of coagulant added during the coagulation-flocculation stage and the source of raw water. Additionally, impurities discharged into the river from anthropogenic and natural sources such as catchment bedrock minerals will influence the source of raw water at water treatment intake [9]. Table 1A and 1B show the physical and chemical properties of raw WTS on in dry-weight basis taken from a local water treatment plant in Johor, Malaysia reported by Awab et al. [10]. The dominant metallic constituents that exist in the raw WTS is aluminium (Al) followed by iron (Fe) at the half amount of the Al. Other elements were also present based on the trace quantities. Similar findings from Ooi et al. [3] as Al and Fe are among the major elements present in calcined WTS. In Malaysia, the use of alum sludge as recovered coagulant has not been implemented as the alum sludge is classified as a scheduled waste, but the findings from this research can be employed as a guide for its application and a high recovery rate of aluminium from WTS is expected with suitable recovery methods and parameters.

Table 1A. Physicochemical properties of raw WTS [10]

Parameter	Unit	Concentration
pH	-	4.28 ± 0.012
Moisture	%	28.67
Bulk density	kg/m^3	831
Particle density	kg/m^3	2660
Porosity	%	68 - 69
Surface area	m^2/kg	38.92 x 10^3

Table 1B. Chemical properties of raw WTS [10]

Parameter	Concentration (g kg^{-1})
Al	12.8033 ± 0.2052
Fe	7.3517 ± 0.0155
Mn	0.0395 ± 0.0013
Pb	0.0371 ± 0.0002
Zn	0.0339 ± 0.0007
Cu	0.0171 ± 0.0005
Cr	0.0155 ± 0.0006
Cd	0.0024 ± 0.0002

Acidification plays a significant role in neutralizing alum flocs by leaching the aluminium concentration to the supernatant solution [8][11][12]. The basic concept of coagulant recovery using the acidification process is represented in equation (1), proposed by Vishal et al. [13] where insoluble aluminium hydroxide reacts with acid to form a diluted alum solution.

$$2Al(OH)_3.3H_2O + 3H_2SO_4 + 2H2O \rightarrow Al_2(SO_4)_3.14H_2O \quad (1)$$

However, other substances which soluble under a high concentration of acid will recover as well such as manganese, zinc and lead [13][14]. In the acidification process, various factors such as pH range, acid concentration, reaction time, and solid to liquid ratio would affect the quantity and recovery rate of aluminium. Recent research has proved that using the acidification method, the aluminium recovery efficiency ranged between 72 to 99.5% of alum has recovered at different

molarity of acid from 1M to 8M [8][15][3], 65 – 90% of recovery rate at pH between 1.0 to 2.5 [14][16][17][18], 68% of recovery rate at 28% of solid to liquid ratio [3], and range of acidification reaction time between 15 – 150 minutes, with 30 minutes as the lowest possible optimum contact time [19][20].

Recovery of alum coagulant is pH dependent and pH around 2.5 showed better recovery rate results [8][16][21]. The recovered alum can be reused for water treatment. To sum up, acidification is a highly efficient and low-cost process for extracting coagulant [8]. From the literature, it has been observed that sulphuric acid (H_2SO_4) is the common acid chemical reagent used by researchers for the recovery of aluminium in the acidification method. However, a few previous researchers used nitric acid in pre-treatment alum sludge. Sanga et al. [22] used a combination of concentrated nitric acid and perchloric acid at a ratio of 2:1 as sludge pre-treatment before using the alkalization method as the main recovery process. Ooi et al. [3] mixed alum sludge with hydrochloric acid and nitric acid at a ratio of 1:4 in the recovery process. However, limited research on utilizing nitric acid as the sole leaching solvent in the acidification of WTS. Hence, in this research, an acid digestion method using nitric acid is adopted for the recovery of coagulant from WTS.

On the other hand, the potential of reusing recovered coagulant in several industries including water/wastewater, construction, and agricultural industries has been reviewed by Dassanayake et al. [2]. Xu et al. [8] used recovered coagulant from alum sludge in both water and wastewater treatment. The efficiency of recovered coagulant in turbidity removal is above 96% and it shows similar trends with the fresh alum coagulant used in the study. Sanga et al. [22] evaluated the effects of recovered coagulant towards turbidity, total suspended solids (TSS), conductivity, and total dissolved solids (TDS) of raw water from Yanze River, China. The recovered coagulant has shown an impressive reduction of turbidity level at a dosage of 50 ml. Ahmad et al. [16] analysed the performance of recovered coagulant in turbidity removal of untreated water from Yamuna River from lowest pH of 2 to highest pH of 13. The turbidity removal rate ranges from 20 – 99% where the highest turbidity removal occurred at pH 13. The development of Al ion is greater at lower pH while the development of Al(OH) is greater at higher pH [16]. Furthermore, the performance of recovered coagulant in turbidity removal efficiency is highly comparable with pure coagulant with only a small removal difference [19][23]. These findings proved that recovered coagulant from alum sludge shows an excellent capability in improving the quality of raw water up to the drinking water standard.

Therefore, this paper provides a study of the available literature on the potential for active recovery of alum sludge towards a sustainable approach in reducing the issues of sludge disposal. Hence, the aims of this study are (1) to investigate the effect of the acidification process at various molarity of nitric acid in recovering aluminium from the WTS and (2) to determine the performance of recovered coagulant in turbidity removal of surface water at various dosage and pH.

2. Materials and Methods

This study was conducted in three stages. In the first stage, sampling and preparation of sludge were done. In the second stage, the aluminium recovery process from WTS was conducted using the acidification method. In the third stage, a jar test was conducted to determine the performance of recovered coagulant in removing turbidity of raw water. Table 2 below summarise the stages of the experimental set-up for this study.

Table 2. Experimental set-up

Stage I: Preparation of Materials		
Collection of raw WTS from sludge lagoon and preparation of sludge sample		
Stage II: Aluminium recovery using acidification method		
Independent variable	Control variable	Dependent variable
• Molarity of HNO_3 for acidification: 0.5M, 1.0M, 1.35M, 1.5M and 2.0M	• Constant weight of WTS: 100 g • Mixing rate: 350 rpm • Mixing duration: 120 mins. • Mixing temperature: 100^{o}C	Concentration of aluminium in the supernatant layer (recovered coagulant)
Stage III: Performance of recovered coagulant		
Independent variable	Control variable	Dependent variable
• Coagulant dosage: 0.5, 1.0, 1.5, 2, 4, 6, 8, 10, 12 mg/l • pH: 2, 4, 6, 7, 8, 10, 13	• Rapid mixing: 80 rpm for 1 min • Slow mixing: 30 rpm for 15 mins. • Settlement: 20 mins.	Settled turbidity of surface water (% removal)

2.1. Stage I: Preparation of Materials

The water treatment sludge (WTS) was taken from the local water treatment plant in Sungai Petani, Kedah, Malaysia. The wet sludge was taken from the sludge lagoon and stored in the plastic container for transportation to the laboratory. The alum sludge was dried using an oven (Model: Binder ED720) at 105^{o}C for 24 hours [15][24] to remove the moisture content until a constant weight was achieved. After the dewatering process, the sludge samples are crushed using a mortar and pestle to disintegrate the sludge boulders into smaller sizes and grind until fine sludge is obtained. The sludge was then sieved using a 16μm sieve pan to obtain powdered sludge. The powdered sludge sample was weighed at a constant weight of 100g and mixed with 500 ml of distilled water using a magnetic stirrer. The distilled water works as a solvent to dissolve the dried powdered sludge [18][15] prior to the acidification process.

2.2. Stage II: Acidification of WTS

In this stage, nitric acid (HNO_3) was used as a leaching solvent with variations of molarity; 0.5M, 1.0M, 1.35M, 1.5M, and 2.0M. To begin the digestion process, the magnetic stirrer was pre-heated to 100^{o}C for 15 minutes [9]. The diluted sludge samples were mixed with HNO_3 and stirred using a magnetic stirrer at the constant speed of 350 rpm for 2 hours until they became homogenous [18] [25][26]. This makes the reaction time and speed as the constant variable. The steps were repeated for all samples with different molarity. Finally, the digested alum sludge was left to settle for 45 minutes [26] until the supernatant layer is separated from the solid. The supernatant was extracted using a graduated glass pipette and filtered using a 0.45 μm membrane filter [27] to ensure that the sample is free from particles that may interfere with the reading of aluminium concentration.

The concentration of aluminium for each molarity was determined using Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). The amount of aluminium recovered from the ICP-OES represents the efficiency of aluminium recovery. The recovery rate was calculated based on the initial concentration of elements present in the dissolved WTS. The filtrates solution extracted from the supernatant layer that achieved the highest recovery rate of aluminium was used as a recovered coagulant in Stage III and the performance was evaluated in terms of turbidity reduction in untreated surface water.

2.3. Stage III: Performance of Recovered Coagulant

For this stage, the performance of the recovered coagulant was tested using a jar test experiment. The efficiency of recovered coagulant in removing turbidity was evaluated at various dosage ranges of 0.5 – 12 mg/l and various pH of 2 - 13. Surface water from Sungai Muda, Kedah, Malaysia was collected using the grab sampling method and the initial physicochemical properties were as recorded as in Table 3. In this stage, the first experiment was conducted under variation of recovered coagulant dosage. pH range of 6 to 8 is recommended for the application of recovered coagulant in raw water treatment [15], however constant pH of 6.5 is employed in this stage. Nine jars containing 500 mL of raw water sample were placed in the jar test apparatus and the recovered coagulant was added with the dosage of 0.5, 1, 1.5, 2, 4, 6, 8, 10, and 12 mg/l. H_2SO_4 and NaOH were used to set the constant pH.

Next, the second experiment was conducted to determine the effect of pH variation using the optimum dosage acquired from experiment 1. Seven jars containing 500 mL of raw water sample were placed in the jar test apparatus, the recovered coagulant at optimum dosage was added and the desired pH of 2, 4, 6, 7, 8, 10, 13 was set for each beaker.

Table 3. Physicochemical properties of surface water

Parameter	Unit	Value
Temperature	^{o}C	30
pH	-	7.5
Turbidity	mg/l	83.05
TDS	mg/l	171.5
Conductivity	uS/cm	59.7
Salinity	‰	0.1

The jar test experiment is intended to simulate the conventional coagulation-flocculation process in a water treatment plant. At both conditions of varying dosage and pH, all water samples were set to have rapid mixing of 1 min at 80 rpm followed by slow mixing for 15 min at 30 rpm to flocculate the colloidal suspension during the mixing process [16]. Then, the flocs were allowed to settle down for 20 min before the settled turbid water was extracted for determination of turbidity removal.

3. Results and Discussion

3.1. Recovery of Aluminium

The recovery rates of aluminium were observed at different molarity of nitric acid. Figure 1 shows the recovery trend of elements in WTS under different molarity of HNO_3. During the acidification process of WTS, other cations elements such as Fe, Ca, Mn and K were dissolved out. Thus, the reduction of sludge volume is expected as the rate of coagulant recovery is also correlated to sludge reduction [8]. The initial concentration of aluminium, iron, calcium, manganese and potassium are 9.882 mg/l, 3.727 mg/l, 18.570 mg/l,

1.312 mg/l and 5.351 mg/l respectively. The order of heavy metals released from the acidification process follows the same pattern of solubility of such metal hydroxides, Al > Fe > Ca > Mn > K. The concentration of all recovered metals increases with the increasing of acid concentration. All elements obtained highest final concentration at 2.0M of nitric acid (Al = 4117 mg/l, Fe = 1903 mg/l, Ca = 231.26 mg/l, Mn = 35.69 mg/l, K = 29.72 mg/l) while the lowest final concentration after acidification process is at 0.5M (Al = 2068 mg/l, Fe = 423.76 mg/l, Ca = 145.26 mg/l, Mn = 26.81 mg/l, K = 21.70 mg/l). Table 4 shows the final concentration, pH and recovery rate of Al from 0.5M to 2.0M where the recovery rate corresponds significantly to the increment of acid concentration. The recovery rate was calculated based on the initial concentration of Al in the dissolved WTS before the acidification process.

Al shows a significant recovery rate from the acidified WTS, from 99.52% to 99.76% as the acid concentration increased from 0.5M to 2.0M as shown in Figure 2. This is followed by the recovery of Fe, with the rate ranging from 91% to 99.8%. From the findings, Al and Fe are the major metallic elements present in the acidified sludge. This is expected as these are also the key element present in the dry WTS [3][9]. Higher molarity/normality of acid is expected to produce better efficiency of aluminium recovery [28]. Fouad et al. [24] used hydrochloric acid as a leaching agent in the acidification process of WTS at different normality (0.1N – 2.0N) and the maximum recovery rate of aluminium was achieved at 2.0N. Comparable trend described by Ooi et al. [3], as the recovery rate of aluminium from WTS also increases as the molarity of sulphuric acid increases from 2M to 4M. From the results, HNO_3 yield a similar trend of recovery with H_2SO_4 and HCl as leaching agent especially on Al and Fe, where the increment of acid concentration is proportioned with the recovery of cations. A high molarity of acid will have a high concentration of H+ ions and this indicates that the pH value of the acid solution is low. As the acid concentration increases, more H+ ions will react with the sludge samples hence greater recovery of aluminium will be produced [25]. The recovery rate of alum is effectively completed at the pH range of 1.0 – 1.6 whilst the recovery may not be easy to achieve at the pH range of 2.0 – 2.5 but it is more cost-effective [13]. Vishal et al. [13] highlighted that at the pH range of 1.0 – 1.5, the recovery of aluminium does not rebound which indicates the complete reaction of aluminium sulphates. However, from the economic point of view, 2M is the maximum molarity of acid solvent that will be more cost-effective in aluminium recovery [24][28]. Hence, 2M of nitric acid was chosen as the best condition of acid concentration and was used in the second stage to evaluate the performance in turbidity removal of the raw water samples.

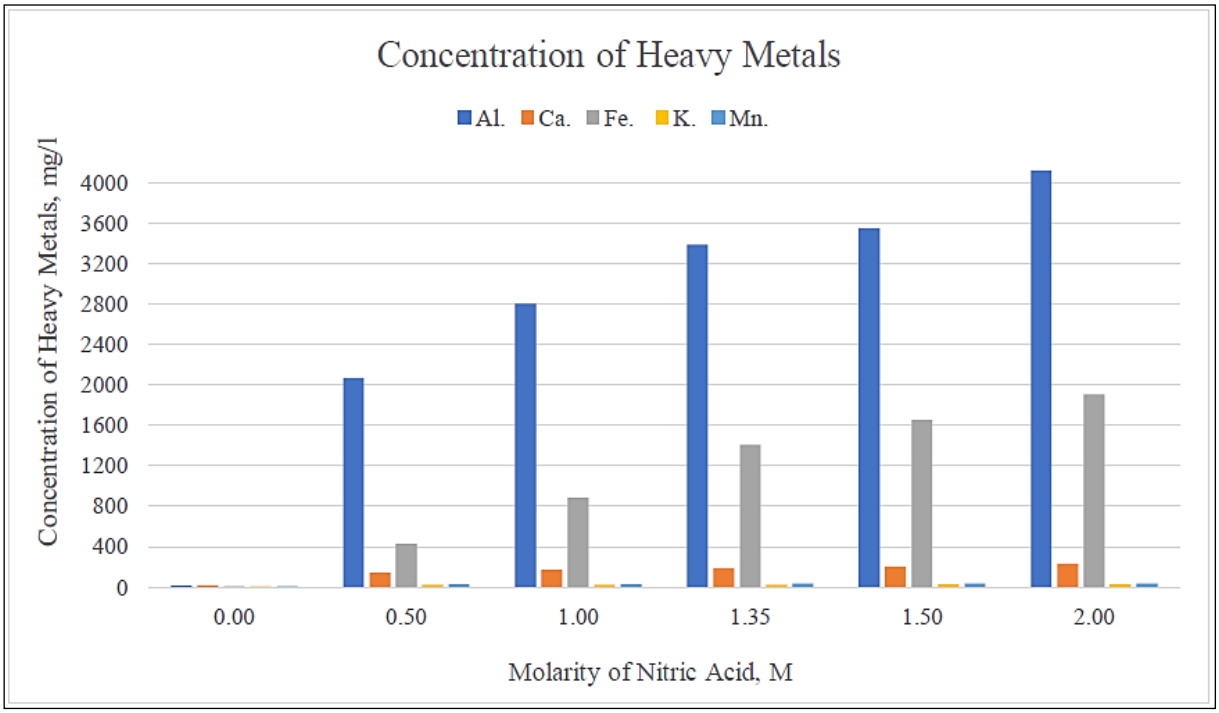

Figure 1. Recovery of aluminium and other chemical elements at different molarity of HNO_3

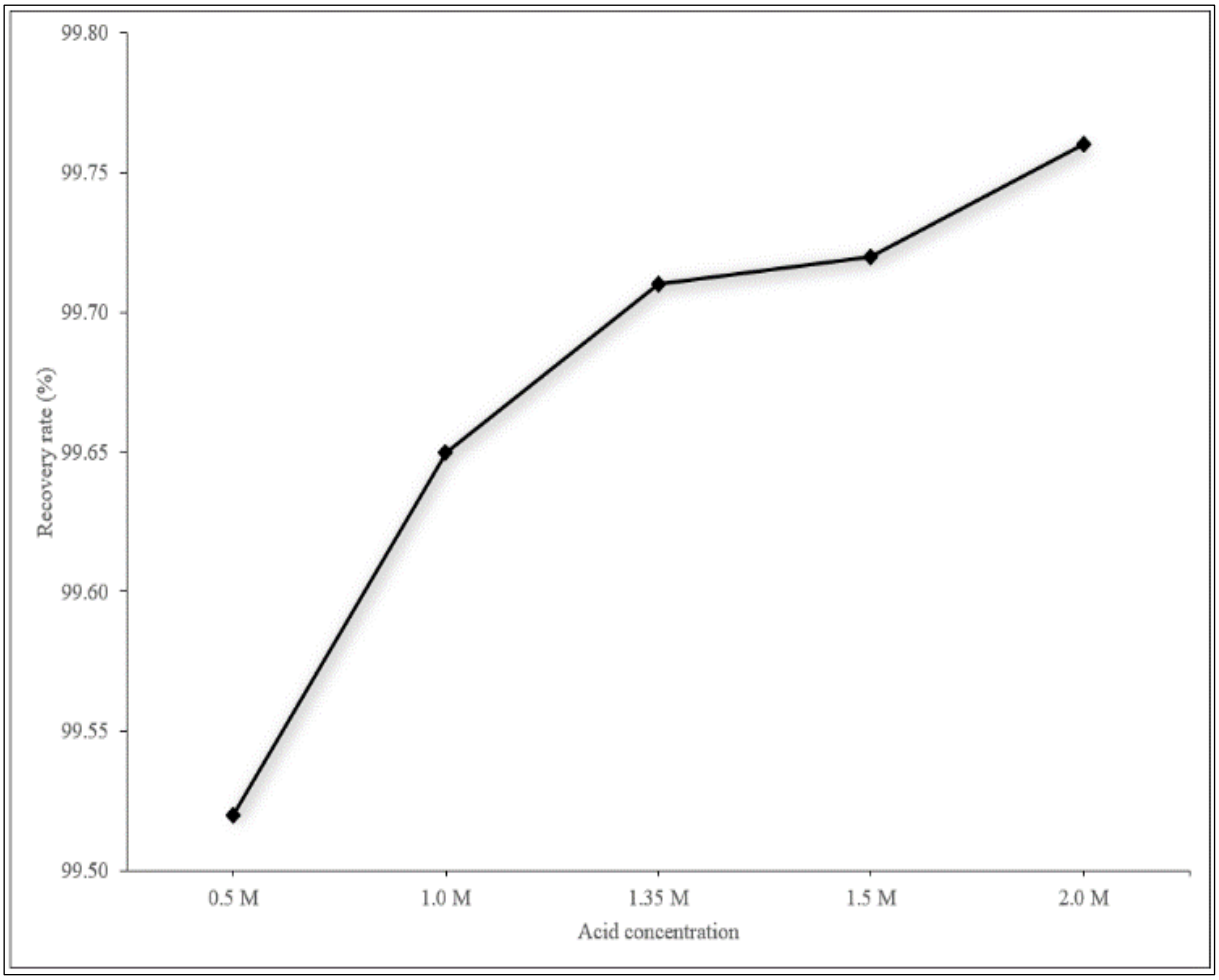

Figure 2. Recovery rate of aluminium at different molarity of HNO_3

Table 4. Recovery efficiency of Al

Molarity of HNO_3 (M)	Final conc. of Al (mg/l)	Recovery rate % (Based on initial conc. of Al)	Final pH
0.50	2068	99.52	3.21
1.00	2801	99.65	1.61
1.35	3384	99.70	1.24
1.50	3549	99.72	1.19
2.00	4117	99.76	1.07

From the acidification process, most of the elements dissolved out are metals. Therefore, the coagulant recovery rate should have close relation with the reduction of sludge volume [8] which is very useful in reducing the operating costs [18]. However, the release of other metals such as Mn shall limit the usage of recovered coagulant [18].

3.2. Performance of Recovered Coagulant

The performance of recovered coagulant was analysed in turbidity reduction of surface water at different dosages and pH. The physicochemical properties of surface water are summarised in Table 3.

3.2.1. Performance of Recovered Coagulant at the Variation of Dosage

The effectiveness of recovered coagulant for turbidity removal is shown in Figure 3. The turbidity removal ranges from 10% to 93% at the variation of dosage. The highest turbidity removal of raw water is 93.32%, achieved at the dosage of 2.0 mg/l whilst the lowest is 10.09% at the dosage of 0.5 mg/l. From the graph, the turbidity removal increases when the recovered coagulant dosage increases starting from the dosage of 0.5 mg/l until 2.0 mg/l. However, turbidity removal is found to decrease at the dosage of 4.0 mg/l (84.43%) and 10 mg/l (86.91%), but it further increases as the dosage of recovered coagulant increases. At the dosage of 8 mg/l and 12 mg/l, the turbidity removal achieved 90.93% and 91.15% respectively. The results have coincided with the trend by Ahmad et al. [16] achieved 94.2% turbidity removal at pH 6 and 7 with the dosage of 8ml/l.

Xu et al. [8] recommended that the best dosage range of coagulant should be between 3 – 6 mg/l. In this study, the

optimum dosage was achieved at 2 mg/l and the recovered coagulant was able to reduce the turbidity level up to 5.5 NTU. These results almost achieved the recommended turbidity level in the drinking water standard in Malaysia (5 NTU). It is also expected that the recovered coagulant should exhibit low performance in turbidity reduction compared to the pure alum due to the presence of other metals and dissolved solids [19][8]. As the recovery rate of aluminium increased, the performance of recovered coagulant is comparable with the pure alum as the recovery of aluminium increased [19]. Nevertheless, the comparison with pure alum is not examined in this study. Therefore, it can be concluded that the efficiency of aluminium recovery rate will affect the performance of recovered coagulant in turbidity reduction.

3.2.2. Performance of Recovered Coagulant at the Variation of pH

The efficiency of recovered coagulant in turbidity removal of surface water was measured at various pH ranging from 2 to 13 and at the optimum dosage of 2 mg/l achieved earlier. The desired pH was set using H_2SO_4 and NaOH. Figure 4 shows the turbidity removal of surface water at a different range of pH. Minimum turbidity removal was attained at pH 10 with 69.28% efficiency while the maximum turbidity removal was achieved at pH 7 with 99.47%. In acidic conditions, turbidity removal is high at pH 4 (94.38%) but reduced to 82.06% at pH 6 before it reached maximum removal at pH 7. The charge neutralization mechanism is expected to occur at pH levels lower than 7 [29] causing the resultant charge to almost neutralize at pH 4 and hence producing high percentage turbidity removal. The removal efficiency decreased when approaching alkaline conditions where the lowest occurred at pH 10 (69.28%). However, higher turbidity removal is observed at pH 12 and 13 with percentage removal of 90% and 94% respectively. The sweep-floc mechanism is expected to occur at higher pH due to the larger concentration of hydroxyl ion (OH^-) in solutions which may have caused the formation of insoluble metal hydroxides [16]. Hence, as the sweep flocs are generated, higher removal of pollutants will occur as the colloids enmeshed in these flocs. Acidified WTS has not only stimulated the recovery of aluminium, but also the iron salts. Therefore, the presence of Al^{3+} and Fe^{3+} in the recovered coagulant has promoted better coagulation efficiency in turbidity removal [16].

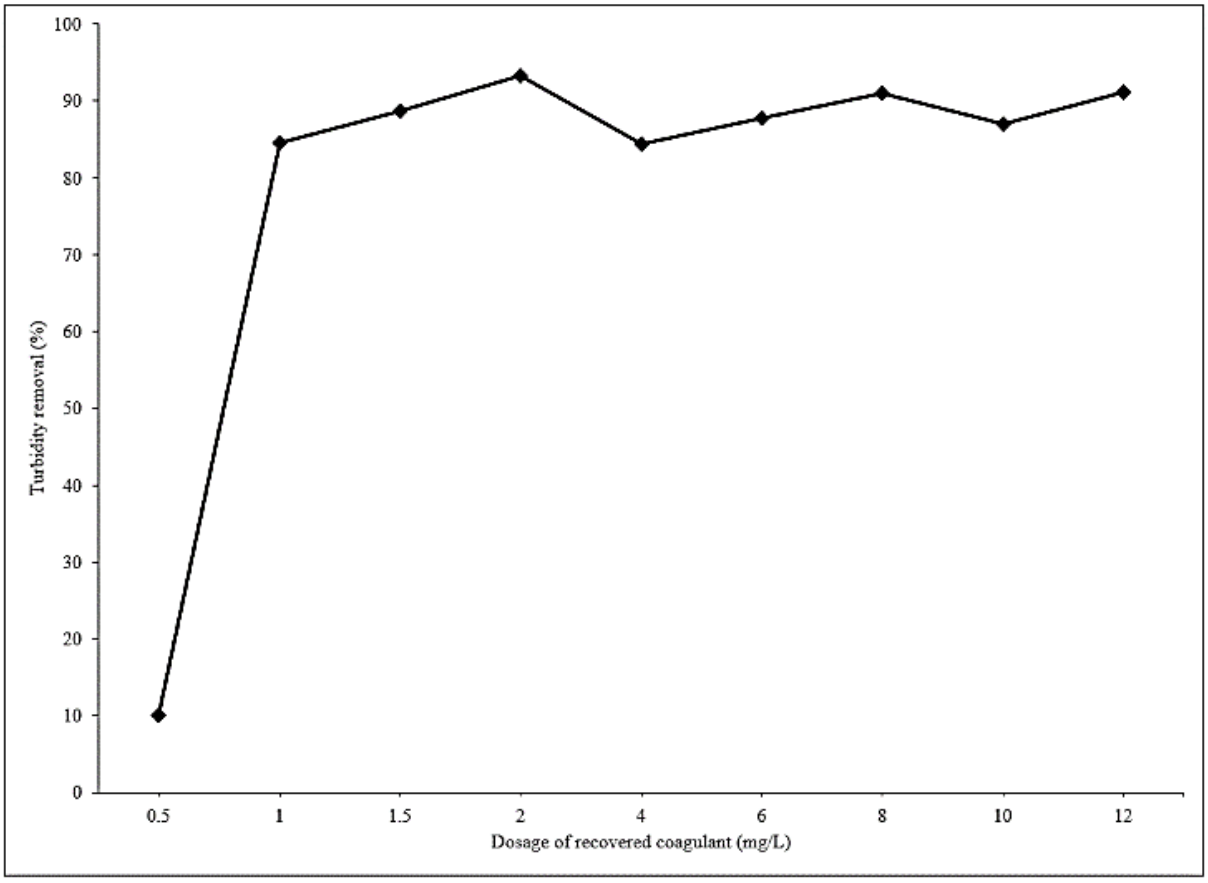

Figure 3. Turbidity removal efficiency (%) at a different dosage of recovered coagulant

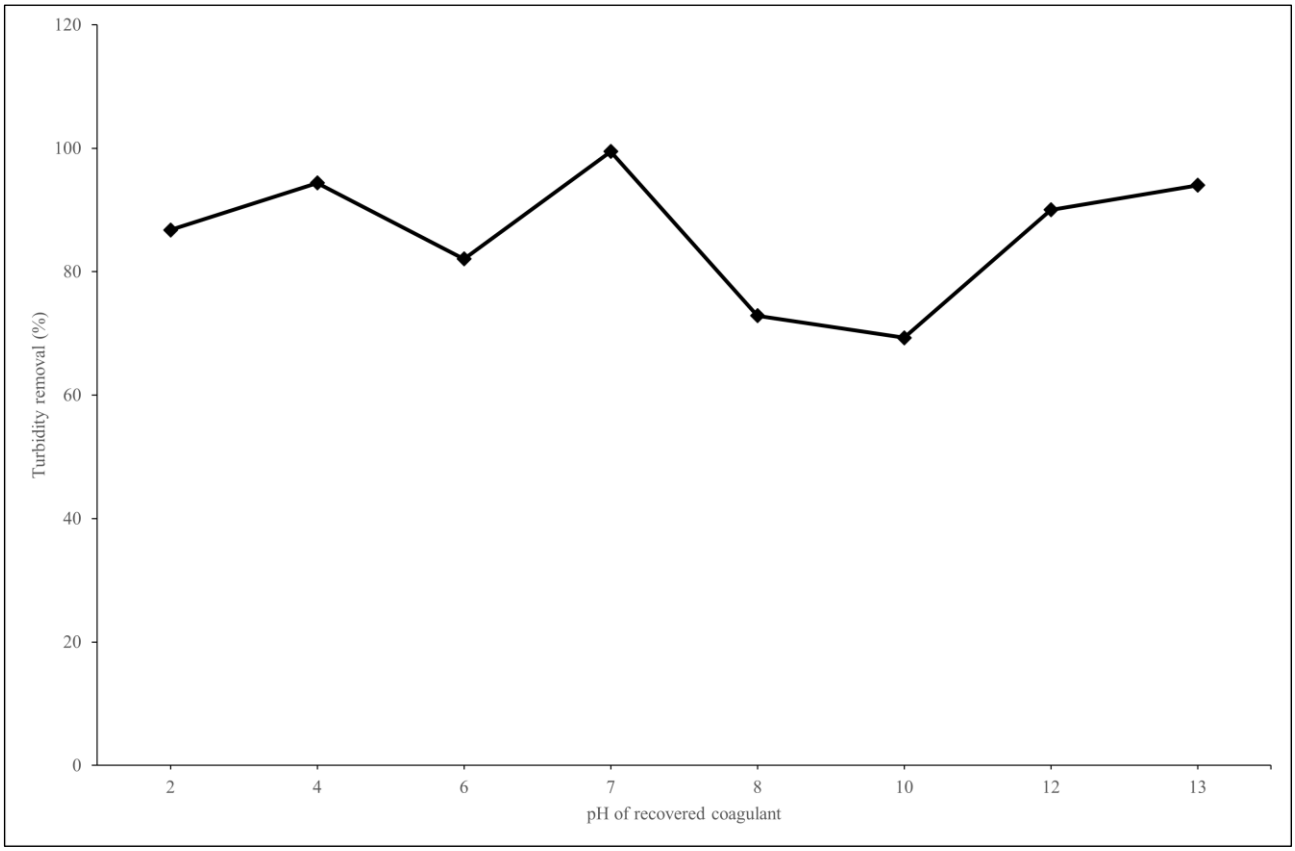

Figure 4. Turbidity removal efficiency (%) at the variation of pH

4. Conclusions

In a nutshell, based on the findings, the acidification method using nitric acid (HNO_3) as leaching solvent is capable in the recovery of aluminium from WTS. At variation of HNO_3 from 0.5M to 2.0M, it shows that the recovery rate of aluminium is corresponding to the increase of molarity. The order of heavy metals released from the acidification process follows the same pattern of solubility of such metal hydroxides, Al > Fe > Ca > Mn > K. High rate of coagulant recovery will lead to the reduction of sludge volume, hence this may reduce the need for sludge lagoon for a new treatment plant. As for the performance of recovered coagulant in treating raw water, it achieved 93.32% of turbidity removal at an optimum dosage of 2 mg/l and 99.47% at neutral conditions (pH 7). The recovered coagulant able to remove turbidity in raw water effectively and the quality of treated water are still within the prescribed limit of drinking water. Therefore, it can be concluded that the high recovery rate of aluminium and iron salts in recovered coagulant will affect the performance of recovered coagulant in turbidity reduction. However, as other metals such as manganese could be released during the acidification process, this shall limit the usage of recovery coagulant in water treatment industries but shall be feasible for pollutant removal in wastewater treatment. Besides, further research on the removal of other impurities shall be needed to maximise the potential of recovered coagulant.

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