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Research Article

Extraction of Water Treatment Coagulant from Locally Abundant Kaolin Clays

Fidelis Chigondo,[1] Benias Chomunorwa Nyamunda,[2] and Vuyo Bhebhe[1]

[1]*Department of Chemical Technology, Midlands State University, Private Bag Box 9055, Gweru, Zimbabwe*
[2]*Department of Chemical and Processing Engineering, Manicaland College of Applied Sciences, Private Bag Box 9055, Gweru, Zimbabwe*

Correspondence should be addressed to Benias Chomunorwa Nyamunda; nyamundab@gmail.com

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Rapid industrialisation is contributing to water pollution. There is a need to identify cheaper and efficient methods of removing contaminants as the demand for clean water rises. A study is carried out to investigate the extraction of alum from locally abundant kaolin clays using sulphuric acid. Alum is a coagulant that is used for raw water treatment. The kaolin clay and alum were characterized by Fourier transformation infrared spectroscopy (FTIR). The effects of particle size, calcination temperature, calcination time, acid-kaolin clay ratio, acid concentration, leaching temperature, and leaching time on extraction efficiency were investigated. The optimum leaching conditions for the calcined kaolin clay were found to be particle size 100 μm, acid-kaolin clay weight ratio 6 : 1, acid concentration 4 M, leaching temperature 100°C, and leaching time 90 min. Under optimised conditions, 66.95% (w/w) aluminum sulphate was extracted. The results showed that sulphuric acid could be used on a large scale to extract alum from kaolin clay. The extracted alum showed similar structural and physical characteristics compared with commercial alum. A dosage of 40 mg/L of the extracted alum showed effective coagulant properties with a great potential of treating raw water.

1. Introduction

Alum (aluminum sulphate) is a coagulant that is used in water treatment plants for supply of safe domestic and industrial water [1–4]. Alum is traditionally extracted from bauxite, an aluminosilicate mineral by the Bayer process [5]. During this process, the ground bauxite mineral is pressure leached with an alkali such as sodium hydroxide to obtain an aluminate solution. Pure aluminum hydroxide is then precipitated from the aluminate solution by seeding.

Another aluminosilicate mineral is kaolin which is found among different types of clays [6, 7]. Kaolin has a high Al content compared to other types of clay such as smectite, illite, and chlorite. Kaolin is a naturally hydrated aluminum silicate mineral that has a variety of colours ranging from white to red due to presence of variable amounts of iron oxide [8].

Aluminum has been extracted from kaolin clays using different mineral acids [9]. The use of hydrochloric acid for leaching alumina compared to other acids offers several advantages such as the ease of filtration of slurries, ease of iron removal, and the insolubility of titanium dioxide present in many clays [10]. The main setback of using hydrochloric acid is the severe corrosion of equipment. However, the development of corrosion resistant rubbers and plastics has solved this problem to a large extent. Both hydrochloric and sulphuric acids extract approximately the same amounts of alumina from same quantities of clay [11, 12]. The effects of reaction parameters on alumina extraction from kaolin clay have been reported in the literature [13–16].

Other aluminosilicate forms such as coal fly ash can be utilised for the extraction of alumina. Alkali treatment on desilicated coal fly ash was reported to extract 89-90% alumina [17, 18]. Wu et al. [19] reported 82.4% $Al_2(SO_4)_3$ extraction efficiency using sulphuric acid. Both alkali and acid dissolution processes on fly ash suffer high energy consumptions drawbacks that are not economical at industrial scales [17].

This study focuses on determining the feasibility of leaching aluminum from locally abundant Zimbabwean kaolin clays using sulphuric acid. It aims at optimizing the conditions for acid extraction of low iron content alumina. The extracted alum would be tested for industrial wastewater treatment. Currently the country imports aluminum sulphate which is costly. There is need to identify local sources of the coagulant to reduce or eliminate import costs.

2. Materials and Methods

2.1. Sample Collection. Kaolin samples were collected from Indiva Siding quarry which is located 38 km east of Gweru, Zimbabwe. The random point sampling method was applied for sample collection using a metal scoop and a metal container. Four samples were collected into 5 kg metal containers. A composite sample was then prepared by uniformly mixing the four samples using a shovel. Cone and quartering method was used to obtain smaller fractions of the composite sample. The samples were thoroughly mixed and collected into labelled plastic bags [17].

2.2. Sample Preparation and Activation. The clay samples were ground using a ball mill to particle sizes in the range 50–200 μm. The ground clay samples (20 g) of different particle sizes (50–200 μm) were put in vitreous crucible and subjected to high temperatures in a muffle furnace (carbolite) for thermal activation [17]. The calcination temperature was varied between 500 and 900°C for one hour.

2.3. Kaolin Characterisation. The elemental composition of powdered kaolin was determined using XRF (Panalytical Zetium) according to a previously reported procedure [20, 21]. Kaolin samples were pressed into pellets using boric acid before XRF analyses. The kaolin samples (1 g) were subjected to a temperature of 900°C in a muffle furnace to determine the loss on ignition (LOI).

KBr pressed pellets solid kaolin samples were analysed using FTIR spectrophotometer (Nicolet 6700).

2.4. Acid Leaching. All leaching experiments were performed in triplicate. The calcinated kaolin samples (5 g) of different sizes (50–200 μm) were leached using wide ranging concentrations of H_2SO_4 (1–5 M) over varying durations (10–180 min), at different temperatures (25–100°C) and various acid/clay ratios (4 : 1 to 7 : 1) w/w under reflux and constant stirring (120 rpm). A separating funnel was used to slowly add 100 mL of ethanol into the acid leach liquor under continuous stirring to precipitate aluminum sulphate. After leaching, the mixture was filtered using Whatman number 1 filter paper. The residue was washed thrice with 30 mL aliquots of deionised water. The filtrate and washings were separately analysed for aluminum ion content using Flame Atomic Absorption Spectrometer (Shimadzu AA-6800).

2.5. Coagulation Tests. Four jar test beakers were filled with 1 L industrial wastewater. Different dosages (10–40 ppm) of the extracted and commercial aluminum sulphate were separately added to the wastewater [1]. The jars were continuously stirred for 10 minutes before allowing water to settle for 15 minutes. The turbidity, conductivity, total dissolved solids, and pH of the settled water were determined. Coagulations tests were repeated.

Table 1: Chemical composition of kaolin.

Compound	Composition (wt.%)
Al_2O_3	20.34
CaO	2.29
Fe_2O_3	1.35
K_2O	2.02
MgO	1.99
SiO_2	58.02
TiO_2	2.05
Mn_2O_3	0.01
Na_2O	0.13
K_2O	0.17
SO_3	0.23
P_2O_5	0.45
LOI	10.95

3. Results and Discussion

3.1. Characterisation of Kaolin by XRF. The chemical composition of kaolin is shown in Table 1. Aluminum oxide and silicon dioxide were the major components of kaolin. The percentage composition of alumina (20.34%) was lower than values reported in previous studies. Kaolin samples containing 29.4% aluminum oxide were reported to achieve an extraction of 32% $Al_2(SO_4)_3$ [19]. Numluk and Chaisena [20] used kaolin samples containing 22.7% Al_2O_3. High silica content (58.02%) formed greater part of the insoluble residue after acid treatment. Numluk and Chaisena [20] reported samples containing 66.30% silica content. Oxides of iron, calcium, magnesium, manganese, sodium, potassium, and titanium had compositions below 2.5%.

Loss on ignition (LOI) value of 10.95% was recorded. The value is attributed to bound hydroxyl ions in calcined kaolin [16]. Aderemi et al. [22] reported a loss on ignition of 14.15% from a kaolin sample calcined at 750°C for 2 h. These values compare closely to the theoretically expected value of 13.96% (w/w) attributed to kaolin hydroxyl composition [7].

3.2. FTIR Analysis of Kaolin before and after Acid Treatment. The FTIR spectra of kaolin before and after acid extraction are shown in Figures 1 and 2. The FTIR spectrum of kaolin before acid treatment showed a band at 3460 cm^{-1} which is assigned to inner hydroxyl stretch. This band, attributed to water physisorbed on kaolin surface, disappeared after acid treatment. The peak was a result of bonding between protons and oxygen atoms coordinated to aluminum ions in the octahedral structural layer [6]. The band observed at 465 cm^{-1} was attributed to Si–O–Si bond deformation.

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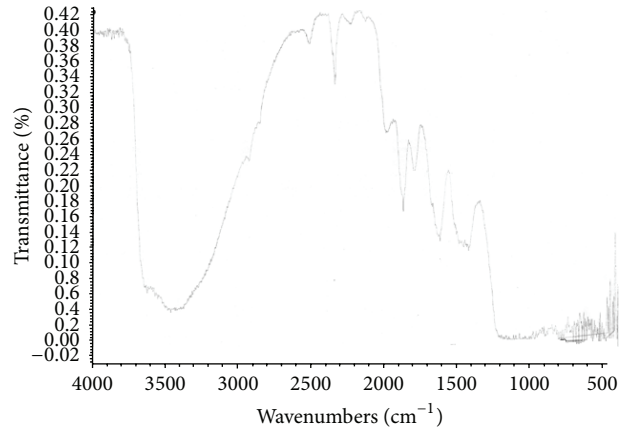


FIGURE 1: FTIR spectrum of kaolin clay before acid leaching.

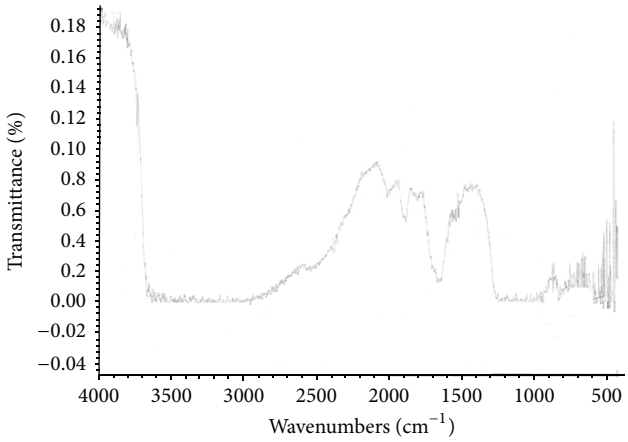


FIGURE 2: FTIR spectrum of kaolin clay after acid leaching.

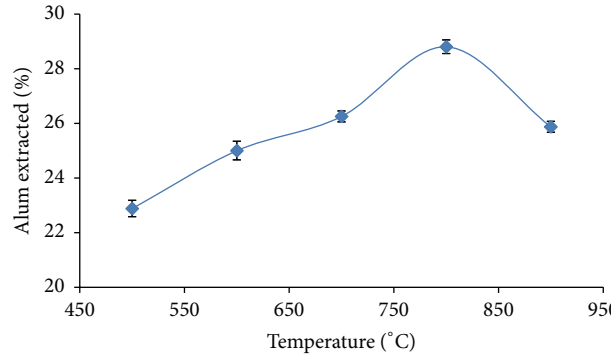


FIGURE 3: Effect of calcination temperature on the extraction of aluminum sulphate (time: 60 min; particle size: 100 μm; error bar = ±SD; and $n = 3$).

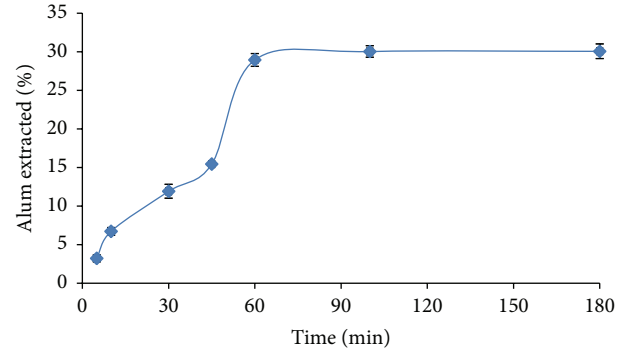


FIGURE 4: Effect of calcination time on the extraction of aluminum sulphate (particle size: 100 μm; temperature: 800°C; error bar = ±SD; and $n = 3$).

The bands at 3735, 3663, and 3629 cm^{-1} observed in acid treated kaolin could be due to OH stretching of inner surface hydroxyl groups in the Al–OH in the octahedral layers of kaolin [23]. The peak intensity of 913 cm^{-1} band decreased after acid leaching showing weakening of the kaolin structure due to neutralisation of the hydroxyl groups and leaching of Al^{3+} ions. A new band appeared at 911 cm^{-1} after acid treatment which could be attributed to OH deformation of inner hydroxyl groups in the bonding of Al–Al–OH octahedral sheet [24]. The 489 and 463 cm^{-1} bands were attributed to Si–O–Si deformations. The band intensity at 489 cm^{-1} increased after acid leaching.

3.3. Effect of Calcination Temperature. Calcination temperature is a very important parameter when investigating the extraction of aluminum from kaolin clays. Calcination thermally activates the kaolin to a more reactive form [16]. The effect of calcination temperature on the extraction of aluminum sulphate is shown in Figure 3. The quantity of alumina extracted increased with increasing calcination temperature from 22.9% at 500°C to a maximum of 28.8% at 800°C. Above 800°C the quantity of alumina extracted decreased. Thermal treatment leads to loss of water molecules within the kaolin clay structure. Above 800°C, total dehydration of kaolin clay occurs resulting in disorderly phase transformation and disruption of kaolin structure into metakaolin amorphous solid that is less prone to acid attack [16, 23]. Similar work done by Ajemba and Onukwuli [25] showed that calcined clays at 750°C exhibited more alumina dissolution rates compared to those at lower temperatures. Active extraction was observed to begin at 500–600°C. Numluk and Chaisena [20] reported that the percentage of alumina extracted increased with increasing calcination temperature in the range 550–850°C and decreased sharply above 850°C. Numluk and Chaisena [20] managed to achieve high alumina dissolution of 52.2% at a calcination temperature of 750°C. Studies carried out by Aderemi et al. [22] on kaolin reported maximum alumina extraction at 800°C.

3.4. Effect of Calcination Time. Figure 4 shows the effect of calcination time on the yield of aluminum sulphate. The percentage of alum extracted increased up to 60 minutes. Beyond 60 minutes the amount of alum extracted leveled off at 30%. Low calcination times could result in insufficient thermal treatment of the kaolin sample. Increasing time ensures that kaolin samples are adequately exposed to calcination. Similar

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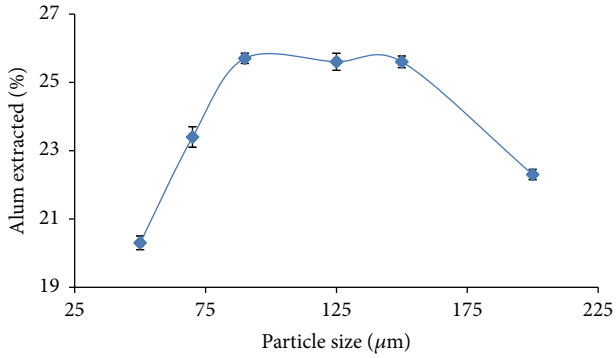


Figure 5: Effect of particle size on extraction of aluminum sulphate (time: 1.5 h; leaching temperature: 100°C; acid concentration: 4 M; error bar = ±SD; and $n = 3$).

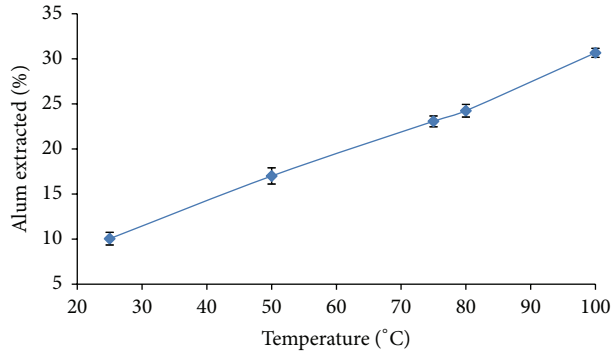


Figure 6: Effect of leaching temperature on the extraction of aluminum sulphate (particle size: 100 μm; leaching time: 1.5 h; acid concentration: 4 M; error bar = ±SD; and $n = 3$).

results were reported by Al-Zahrani and Abdul-Majid [26] giving maximum of 62.94% alumina yields.

3.5. Effect of Particle Size. The particle size of kaolin has an influence on the extraction efficiency of aluminum sulphate. Figure 5 shows the effect of kaolin particle size on the quantity of alum extracted. The quantity of aluminum sulphate extracted increased with increasing particle size before leveling off at 90 μm. The maximum extraction efficiency (25.7%) was observed between 90 and 150 μm. Above 150 μm particle size, a decrease in the quantity of aluminum sulphate extracted was observed. The decline could be attributed to decreased clay surface area exposed for acid attack. Smaller particle sizes exhibit a larger surface area. The work done by Al-Zahrani and Abdul-Majid [26] concluded that a particle size of 149 μm resulted in maximum aluminum sulphate extraction (32%). Particle sizes of 100 μm were recommended in this work for further leaching experiments.

3.6. Effect of Leaching Temperature. Conventional aluminum sulphate extraction method using heated water depends on the bath temperature. Figure 6 shows the effect of water bath temperature on aluminum sulphate extraction. Increasing leaching temperature results in increased yield

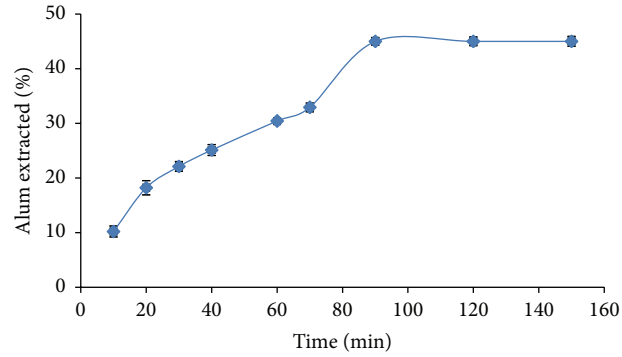


Figure 7: Effect of leaching time on the extraction of aluminum sulphate (acid concentration: 4 M; particle size: 100 μm; leaching temperature: 100°C; error bar = ±SD; and $n = 3$).

of aluminum sulphate. An increase in leaching temperature increases the kinetic energy of the kaolin-acid solution resulting in increased collision frequency. The highest degree of aluminum sulphate extraction was observed at boiling temperature (100°C). A study done in comparing conventional and microwave heating methods showed that the latter produced higher extraction yields due to attainment of elevated leaching temperature [27]. Other studies concluded that kaolin leaching requires high temperatures [25]. Another study investigating the effect of leaching temperature also showed that the highest yield of aluminum sulphate was obtained at boiling temperature [26].

3.7. Effect of Leaching Time. The effect of leaching time on the extraction process is shown in Figure 7. The yield of alum increased with increasing leaching time. The percentage yield of alum leveled off at 45% after 90 minutes. Prolonged leaching time allowed ample time of interaction between the acid and kaolin particles. Al-Zahrani and Abdul-Majid [26] reported that the percentage of aluminum sulphate extracted attained a constant value after 1 h at all leaching temperatures. Numluk and Chaisena [20] attained maximum Al extraction after 120 minutes.

3.8. Effect of Acid to Clay Weight Ratio. The effect of acid to kaolin clay ratio on aluminum sulphate extraction is illustrated in Figure 8. The acid-solid ratio plays a crucial role in the extraction of aluminum sulphate from kaolin. This parameter indicates the amount of acid needed to be in contact with solid for optimum extraction. There was an increase in percentage of aluminum sulphate extracted as the acid-solid ratio increased. The maximum yield of alum (33.1%) was achieved at an acid-solid ratio of 6 : 1. Above 6 : 1 acid-clay ratio the alum yield remained constant probably due to a saturation of available clay sites with hydrogen ions. Increasing acid-clay ratio increased the amount of H^+ ions that interact with kaolin particles until saturation was attained. Previous studies done by Al-Zahrani and Abdul-Majid [26] showed that increasing the ratio from 4 : 1 to 10 : 1 increased quantity of aluminum sulphate extracted. Similar studies revealed that the percentage of extraction of

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Table 2: Optimised sulphuric acid extraction conditions for alum from kaolin clay.

Particle size (μm)	Acid conc. (M)	Acid : kaolin ratio (w/w)	Leaching		Calcination		Extraction (wt.%)
			Temperature (°C)	Time (min)	Temperature (°C)	Time (min)	
100	4	6 : 1	100	90	800	60	66.95

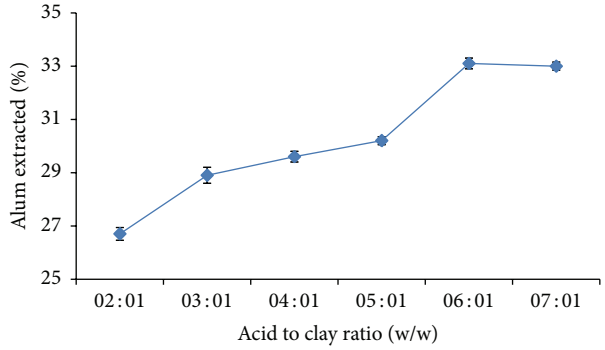


Figure 8: Effect of acid volume on the extraction of aluminum sulphate (leaching time: 1.5 h; particle size: 100 μm; acid concentration: 4 M; leaching temperature: 100°C; error bar = ±SD; and $n = 3$).

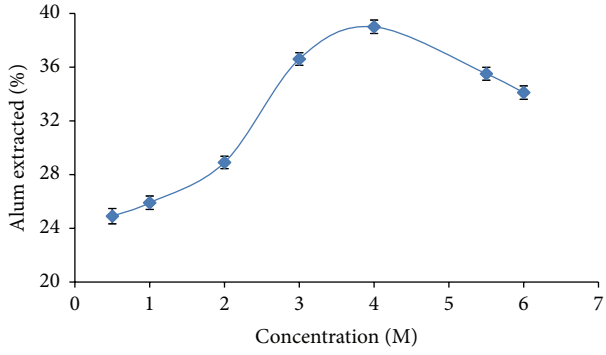


Figure 9: Effect of acid concentration on the extraction of aluminum sulphate (leaching time: 1.5 h; leaching temperature: 100°C; particle size: 100 μm; error bar = ±SD; and $n = 3$).

alumina increased by increasing the acid-clay ratio reaching a maximum value of 30.16% and 65.8% for uncalcined and calcined samples, respectively, at the ratio of 10 : 1 [28].

3.9. Effect of Acid Concentration. Figure 9 shows the effect of acid concentration on the extraction of aluminum sulphate. The degree of extraction increased with increase in acid concentration. The percentage yield of alum increased from 24.9% at 0.5 M to 31.1% at 4 M H_2SO_4. A decrease in aluminum sulphate extracted was observed above 4 M H_2SO_4. An increase in acid strength increases the diffusion of H^+ ions into the octahedral layer of kaolin resulting in increased leaching of aluminum ions. At very high acid concentration the structure of sample collapses and the Al^{3+} ions block the diffusion of H^+ [26]. Numluk and Chaisena [20] obtained a high degree of aluminum sulphate extraction of 39.0%.

Table 3: Some physical properties of commercial and in-house extracted alum.

Property	Extracted alum	Commercial alum
pH	2.5	3.0
Melting point (°C)	84.0	86.5
Appearance	White	Off-white

3.10. Optimum Conditions for Aluminum Sulphate Extraction. Table 2 shows the alum yields obtained under optimum extraction conditions. Leaching kaolin clay under these conditions yielded 66.95% alum. The yield compares favourably with previously reported extractions. Al-Zahrani and Abdul-Majid [26] reported 63% alum extraction efficiency from kaolin using hydrochloric acid.

3.11. Comparative and Physical Properties of Extracted and Commercial Alum. The melting points, pH, and colour of the commercial and extracted alumina are compared closely as illustrated in Table 3.

3.12. Coagulation Tests. The coagulation tests using locally extracted (I) and commercial (C) alum on industrial wastewater are shown in Table 4. The wastewater had a turbidity of 16.7 Nephelometric Turbidity Units (NTU) depicting high amounts of organic matter, mud, silt, and other inorganic precipitates that gave it a cloudy appearance. Commercial alum was more effective in removing turbidity than the extracted coagulant. A coagulant dose of 40 mg L^{-1} of the local alum resulted in reduction of turbidity to 6.4 NTU whereas only 25 mg L^{-1} of the commercial alum was required to obtain WHO recommended turbidity of 0–5 NTU [3]. The conductivity of the water after coagulation increased due to additional ions from the coagulants. There were no significant differences in conductivity changes using both alums. The pH of the clarified water decreased after alum treatment using both alums which is in agreement with previous studies due to partial hydrolysis of aluminum sulphate [1, 28]. The two coagulants exhibited similar tendencies in removing natural organic matter as reflected by similar changes in total organic content (TOC). The in-house alum therefore exhibits good coagulation tendencies and compares favourably with the commercial alum with the exception of its effectiveness in removing turbidity.

4. Conclusion

This study demonstrated that aluminum sulphate can be efficiently extracted from local kaolin. The extracted alum has shown great potential in removing pollutants from industrial

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Table 4: Coagulation tests using commercial and local alum.

Alum dose (mg L^{-1})	0	10		20		25		30		40	
Alum type	I/C	I	C	I	C	I	C	I	C	I	C
Turbidity (NTU)	16.7	15.8	10.1	9.9	6.5	8.8	4.3	7.5	3.5	6.4	3.1
pH	7.7	7.5	7.4	7.3	7.2	6.9	7.0	6.8	6.9	6.7	6.7
Temperature (°C)	18.5	18.4	18.6	19.6	19.5	19.4	19.6	19.6	19.5	19.6	19.4
Conductivity (μSm^{-1})	101	114	112	115	116	116	120	121	122	124	125
TOC (mg L^{-1})	5.2	4.1	4.3	3.3	3.4	2.5	2.7	2.3	2.5	2.3	2.4

wastewaters. Approximately 67% of alum was extracted from kaolin clay calcined at 800°C for 1 h. The optimum leaching conditions were leaching 100 μm clay particle sizes with 4 M sulphuric acid at 100°C for 1.5 h using acid to clay ratio of 6 : 1. The coagulant produced demonstrated huge potential for treating industrial wastewater. The coagulant however fell short in lowering turbidity to recommended levels making the treated water unsuitable for human consumption. Extracting alum from local kaolin at industrial scale has great potential in reducing coagulant import costs. Further studies aimed at converting the extracted alum to more effective polyaluminum chlorides coagulant are recommended.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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