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Turbidity Removal from Kaolin Synthetic Wastewater via Coagulation Process Using Sludge from Water Treatment Plant

Noor Ainee Zainol[1,2*], Putri Al Balqis Khalilullah[2], Azlinda Abdul Ghani[1,2], Najihah Abdul Rashid[2,3], Salwa Mohd Zaini Makhtar[2,3]

[1]Faculty of Chemical Engineering and Technology,
Universiti Malaysia Perlis, 02100 Padang Besar, Perlis, MALAYSIA

[2]Centre of Excellence Water Research and Sustainability Growth, Faculty of Civil Engineering and Technology,
Universiti Malaysia Perlis, 02600 Arau, Perlis, MALAYSIA

[3]Faculty of Civil Engineering and Technology,
Universiti Malaysia Perlis, 02600, Arau, Perlis, MALAYSIA

*Corresponding Author

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Abstract: This study aims to treat kaolin synthetic wastewater by using sludge from water treatment plant as a coagulant via the coagulation process. Water treatment sludge (WTS) was characterized by using Fourier transform infrared (FTIR), Scanning electron microscope (SEM), and X-ray diffraction (XRD). WTS had an amorphous structure and contained high aluminium metal constituents at 25.3%, which can be recovered as a coagulant. Acidification was conducted to recover the aluminium in WTS using sulphuric acid and produced water treatment sludge coagulant (WTSC). The optimum condition of WTSC prepared from WTS was evaluated through sludge concentration and acid dosage parameters. It has been observed that 0.5% sludge concentration acidified with 0.08 ml/ml sulphuric acid and normality constant at 0.5N was the optimum condition as it can remove turbidity at 99.5%. The performance of WTSC in the coagulation process using a standard jar test was studied based on the effect of pH and dosage of coagulant. The highest percentage turbidity removal (98.66%) was obtained at pH 12, kaolin turbidity was 117NTU, and WTSC dosage at 40mg/L. The percentage turbidity removal for alum and WTSC was almost similar. Thus, WTSC can potentially replace alum as a coagulant in water treatment plant (WTP).

Keywords: Coagulation, water treatment sludge, kaolin synthetic wastewater, turbidity, aluminium leaching

1. Introduction

Waste generation has increased, from municipal waste to wastewater, as the country has become more industrialized and its population has grown. As a consequence of development, there has been a significant amount of pollution. WTS is a waste produced by the industrial or municipal waste treatment plant at the last water treatment step. Sludge is a by-product of the wastewater process that is widely used as fertilizer. However, the usage of sludge for fertilizer has disadvantages which can cause contamination or pollution to the environment as well as harm living things as it contains heavy metals, microbes, impurities and chemicals. Discharges of WTS into rivers, streams, lakes, drains, and other water bodies, as well as dewatered WTS deposits, are not environmental disposal options [1].

*Corresponding author: aineezainol@unimap.edu.my

The coagulation process is a must to get rid of the colloidal particles and suspended solids which in turn resulting production of WTS. It is a method of chemical water treatment that includes electrostatic charges for the removal of suspended particles in water. The dosage of coagulant is added into the wastewater destabilizes the colloidal particles that cause turbidity in raw water. The destabilized colloidal particles cluster into larger aggregates that are effectively sediment and further removed during the filtration process. By improving filter efficiency and clarification, the right coagulant for the system will improve overall system performance, particularly solid removal efficiencies. Due to its easy operations, simple design, and low energy consumption, coagulation process is one of the most common processes to treat drinking water [2].

Commonly, the coagulants used to treat wastewater are metal, synthetic coagulants and biopolymer coagulants. Coagulants of aluminium or iron salts are commonly used [3]. Aluminium sulphate or known as alum is a widely used commercial coagulant. The wide use of alum due to low cost and easy to obtain compared to other coagulants. Alum is hydrolyzed into water to form the aluminium hydroxide. The charging neutralization and the sweep mechanism [4] are examples of mechanisms in the coagulation process.

This research aims to recover and reuse of WTS as coagulant from the water treatment plant for turbidity removal of kaolin synthetic wastewater and compare performance of WTSC with commercial alum via coagulation process. Therefore, the usage of sludge in a water treatment plant as a coagulant can be viable alternative as it can save the management cost and environmental friendly.

2. Material and Methods

The experiment was started with the preparation of water treatment sludge to produce WTSC and preparation of kaolin synthetic wastewater. Moisture content, SEM, pH, FTIR, and XRD were used for analysing physical and chemical characteristics of WTS. The preparation of WTSC was performed by acid digestion treatment by sulphuric acid and One-Factor-At-A-Time (OFAT) analysis used to find the optimum WTSC. The jar test was performed to find the best pH and coagulant dosage for removing turbidity from kaolin synthetic wastewater. The percentage of turbidity removal was calculated by using the formula in order to assess the efficiency of turbidity removal.

2.1 Preparation of WTS

The sludge was heated at 105℃ in the oven for 24 hours to dry the sludge. This is to reduce the moisture content of the sludge to a constant weight. The dry sludge was sieved through a 2mm wire mesh pan for removal of any stones and debris that is in the sieve. Then, the raw sludge was grinded and sifted with a 126µm wire mesh sieve pan a second time.

2.2 Characterization of WTS

WTS was characterized by measuring pH and moisture content. Then, the sludge's physiochemical properties were determined by using FTIR, SEM, and XRD.

2.2.1 pH of Solution

5 g of powdered WTS was dissolved in 500 mL of distilled water and stirred for 10 minutes. Then, the pH was measured by using Mettler Toledo pH meter. The measurement was repeated 3 times in order to get the accurate data.

2.2.2 Moisture Content

Firstly, the initial weight of petri dish was measured using analytical balance and then the initial weight of petri dish and 10g of raw WTS was measured again. Next, it was dried in an oven at 105℃ for 2 hours to remove water content in the samples. The weight was measured for every 2 hours until a constant weight obtained. The moisture content of WTS is calculated by using Eq. (1).

$$\text{Moisture content (\%)} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (1)$$

where W_2 stands for weight of petri dish and sample before dried while W_3 stands for weight of petri dish and sample after dried. W_1 stands for weight of petri dish.

2.2.3 Total Suspended Solid

The aluminium foil was weighed using analytical balance. Next, the weight of raw water treatment sludge was weighed at 10g. The 10g raw water treatment sludge was heated in the oven for 2 hours. The initial weight and after dried were weighed and noted. The total suspended solid was calculate using Eq. (2).

$$\text{Total solid} = \frac{(A-B)\times 1000}{\text{mL (sample volume)}} \tag{2}$$

where *A* is weight of sample and aluminium foil (mg) while *B* is weight of aluminium foil (mg).

2.2.4 Functional Group Analysis

FTIR analysis was done to determine the functional group of WTS. WTS was mixed with potassium bromide (KBr) to produce pellet form. The KBr-sample mixture was compressed under vacuum for several minutes to form clear pellets. Next, FTIR sample holder was used to hold the pellet exposed to the infrared light needed to conduct the analysis. FTIR analysis was performed in a KBr chamber at frequencies 4000 to 400 per cm with a spectral resolution of 4 cm^{-1} [1].

2.2.5 Surface Morphology Analysis

SEM was used to analyse the surface morphology of raw WTS. When a sample surface was scanned with an electron beam that is focused on it, SEM produced images of the surface [5]. In order to increase the conductivity, a sticky carbon disc was used to mount the dewatered sludge sample on the metal stub. Before SEM observation, the sample was coated with a conducting layer such as gold to prevent charge accumulation in the specimen surfaces. SEM uses an applied voltage of 15 kV to accelerate the electron beam, which scans the sample. High - resolution images are created by detecting and utilising electrons backscattered from the object and those knocked off its near surface region [6].

2.2.6 Component and Structure Analysis

XRD is a non-destructive technique that provides detailed information about the crystallographic structure, chemical composition, and physical properties of materials [7]. 5 g of powdered water treatment sludge (WTS) was placed into the XRD sample holder plate. Then, it was placed in the XRD chamber prior analysis from 2θ of 10˚ to 80˚. The analysis was performed at 30 kV and 10 mA with a scanning rate of 3˚ 2θ/min. The analysis of XRD spectrum was conducted using High Score Plus software.

2.3 Preparation of WTSC

The preparation of WTSC was used using OFAT method. It was to determine the ideal condition for WTSC production from WTS. In this research, the value of one variable was altered while maintaining the values of the others constant throughout the experiment [8]. A jar test apparatus was used to dissolve the 1 L of distilled water to produce sludge in liquid mixture. For sludge acidification, it was treated with 0.5 N H_2SO_4 ranging from 0.02 ml/ml to 0.12 ml/ml of sludge with an increment of 0.02 ml/ml. the sludge concentrations used were 0.5 % to 3.0 %. Finally, the mixture was stirred for 30 minutes at 30 rpm to remove the aluminium from the sludge dissolved in the mixture. After the reaction, the liquid sludge mixture was filtered through 16 µm filter paper and filter funnel. The filtration procedure was repeated two times to ensure that the WTSC was free of any impurities [9].

2.4 Preparation of Kaolin Synthetic Wastewater

5 g of kaolin powder was weighed and mixed with 500 mL distilled water. First, the mixture was stirred for 1 hour at 200 rpm using a magnetic stirrer. Next, the mixture was let down for 24 hours [10], and the turbidity value was checked. Next, the mixture was diluted with distilled water by 800 mL of kaolin added with distilled water until 1 L of mixture got. If the turbidity is still high, the method was repeated until it got the turbidity value needed. The turbidity must be around 117 NTU, a standard turbidity value from the industry [11]. Lastly, the pH of kaolin suspension was noted and adjusted with 0.1 M sodium hydroxide (NaOH) and 0.1 M hydrochloric acid (HCl).

2.5 Jar Test

This procedure was performed with a regular jar test apparatus, equipped with six paddle- rotors with rectangular blades that will remain throughout the experiments, and six 1000 mL beakers containing kaolin synthetic wastewater. The automatic controller was set up to the required time and speed for the coagulation process. Firstly, six beakers were filled with 500 mL of kaolin suspension, and the pH for five beakers was adjusted as needed. The pH adjustment for kaolin suspension was required 0.1 M NaOH and 0.1 M HCL [10]. The pH needed in this experiment were 2, 4, 6, 7, 9, and 12. The dosage of WTSC and alum added were 20 mg/ L, 40 mg/L, 60 mg/L, 80 mg/L, 100 mg/L, and 120 mg/L. Two minutes of mixing at high speed (100 rpm) were followed by 30 minutes of low speed (40 rpm) for the coagulation-flocculation process. Prior to turbidity testing, the mixtures were allowed to settle for 30 minutes to fix the flocs [12]. The experiment was repeated with an aluminium sulphate solution was used in the exact dosage and pH as WTSC. One beaker of kaolin synthetic wastewater was unchanged in pH and coagulant dosage for the experiment's control.

2.6 Turbidity Removal

The procedure was according to APHA Method 2130B and Nephelometric Turbidity Units (NTU) were used for measuring the turbidity. The calculation of the turbidity removal as follows in Eq. (3).

$$\text{Turbidity removal efficiency} = \frac{A-B}{A} \times 100\% \qquad (3)$$

3. Results and Discussion

3.1 Characterization of WTS

Water treatment sludge is an alkali as the pH for WTS is 9.30. It is reciprocating with aluminium or metal oxides which are positively charged. This indicates that WTS contained aluminium or metal oxides and suitable to act as a coagulant. Besides, the moisture content of WTS is 13.9% which had more solid content and suitable for acidification process. The total solids for WTS were 891.402mg/L. It shows WTS had more solid. Table 1 shows the physiochemical properties of WTS.

Table 1- Physiochemical properties of WTS

Parameters	Values
pH	9.30
Moisture content (%)	13.9
Total suspended solid (TSS) (mg/L)	891.402

3.1.1 Functional Group Analysis

FTIR was used to analyse the functional group in WTS before coagulation process was proceed. Fig. 1 shows the graph transmittance versus wavenumber that depicts the FTIR data. The FTIR spectrum of WTS shows the strong absorption There are three wavenumber regions in the IR spectrum: the far-IR spectrum (less than 400), the mid-IR spectrum (400-4000), and the near-IR spectrum (less than 400) (4000-13000). However, samples can also be analysed using far- and near-IR spectrums, which provide valuable information [13]. Therefore, the mid-IR FTIR spectrum was the focus of this study. From the graph, the high absorption can be found at 3679.5 which shows the medium or sharp free O-H stretching bond. Besides, the medium C-H stretching was found at 2982 and 2832.5 which indicate the alkane. Next, strong S=O stretching was found at absorption value, 1413. This bond indicates the sulphate. The strong C-N stretching which corresponds to aromatic amine also can be found from the graph at 1288.5. The carbonate ion was found at 874, and C-S stretch at 661. It is concluded that WTS was suitable as a coagulant to treat kaolin synthetic wastewater as kaolin is ionic charge. It is because there is sharp O-H bond which indicates that in water treatment sludge contained ions to form a variety of soluble species such as $Al(OH)^{2+}$ or $Al(OH)^{3+}$. It is effective as coagulants as they adsorb very strongly onto the surface of most negative colloids [14].

3.1.2 SEM

The surface morphology of water treatment sludge was investigated using SEM. WTS was examined under 1mm and 100µm magnifications. When the structure of WTS was investigated in this study, it was discovered to be non-uniform and irregular in shape, with the absence of a well-crystalline phase. Thus, WTS has an amorphous structural configuration as a result. In the study by [15], it was found that alum sludge exhibited no distinct shape or form and that the crystalline phase was absent within alum sludge, even though pure possesses a regular crystalline structure. WTS can form complexes with positive charges that have enhanced adsorption properties to adsorb negatively better-charged colloids. Thus, it enhances the coagulant properties of the drug [16], [17]. Fig. 2 to Fig. 4 show the SEM image under 1mm and 100μm at different position.

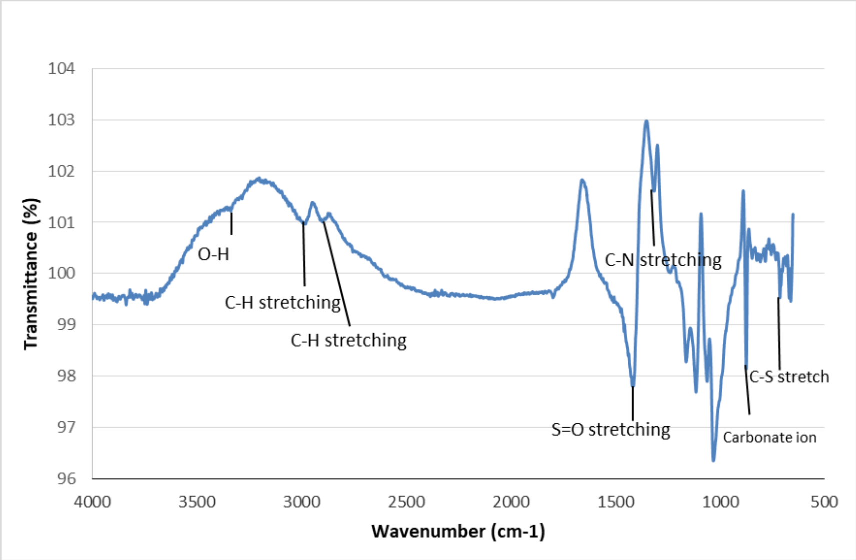

Fig. 1 - Graph of transmittance versus wavenumber

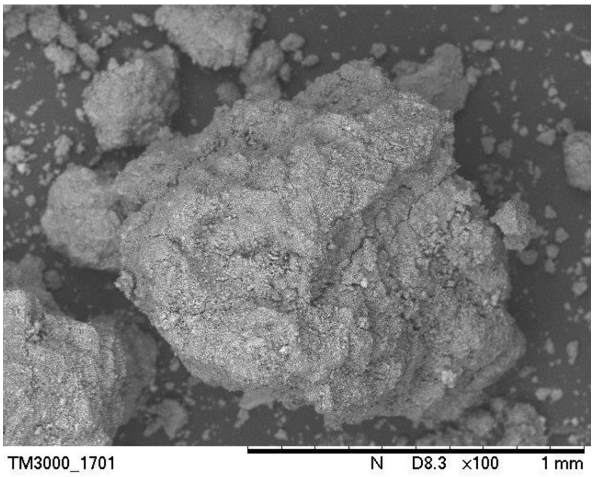

Fig. 2 - SEM image of WTS under 1mm magnification

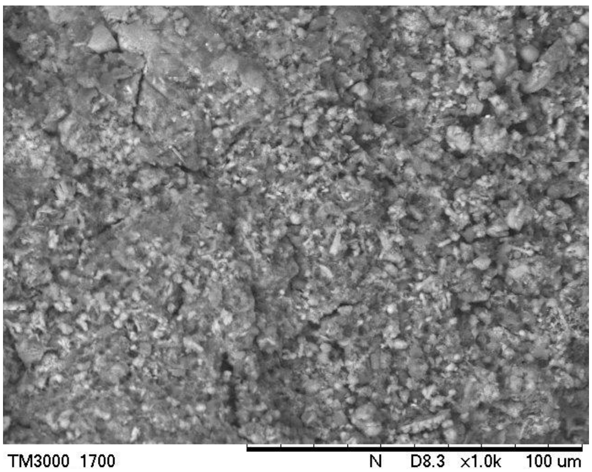

Fig. 3 - SEM image of WTS under 100 μm

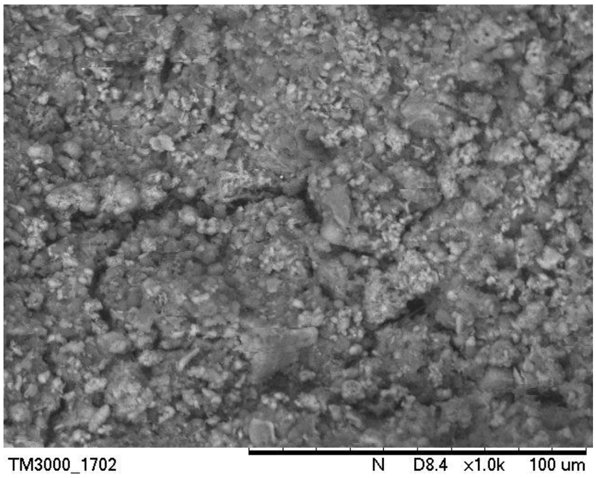

Fig. 4 - SEM image of WTS under 100 µm

3.1.3 XRD

XRD detected the inorganic compounds in the WTS that important in order to act as coagulant. The XRD pattern of water treatment sludge is shown in Fig. 5. The reflections at 37.76˚, 25.63˚, and 43.70˚ 2θ were characteristics of aluminium oxide (Al_2O_3). Next, the reflections at 20.90˚, 26.69˚, 36.30˚, 39.82˚, 42.97˚, and 50.89˚ 2θ were characteristics for silicon oxide (SiO_2). Besides, 27.33˚ and 31.57˚ 2θ were reflections for sodium chloride (NaCl). While the reflections for calcium carbonate ($CaCO_3$) were 29.39˚, 31.69˚, 57.52˚, and 39.93˚ 2θ. Lastly, the reflections for chromium oxide (Cr_2O_3) were 36.42˚, 39.57˚, and 50.61˚ 2θ. Table 2 shows the percentage composition of metal compounds in WTS. The major metal compositions in water treatment sludge (WTS) were aluminium oxide at 16.2%, silicon oxide at 32.3%, sodium chloride at 7.1%, calcium carbonate at 30.3%, and chromium oxide at 14.1%. The elements of metal oxide especially aluminium were the crucial to act as a coagulant (Wei et al., 2018). It is because the metal oxide can form hydroxide ions that good as a coagulant. Thus, WTS was the suitable and efficient to be used as a coagulant in water treatment plant.

Table 2 - Percentage composition of metal compounds in WTS

Metal compounds	Percentage (%)
Aluminium oxide (Al_2O_3)	16.2
Silicon oxide (SiO_2)	32.3
Sodium chloride (NaCl)	7.1
Calcium carbonate ($CaCO_3$)	30.3
Chromium oxide (Cr_2O_3)	14.1

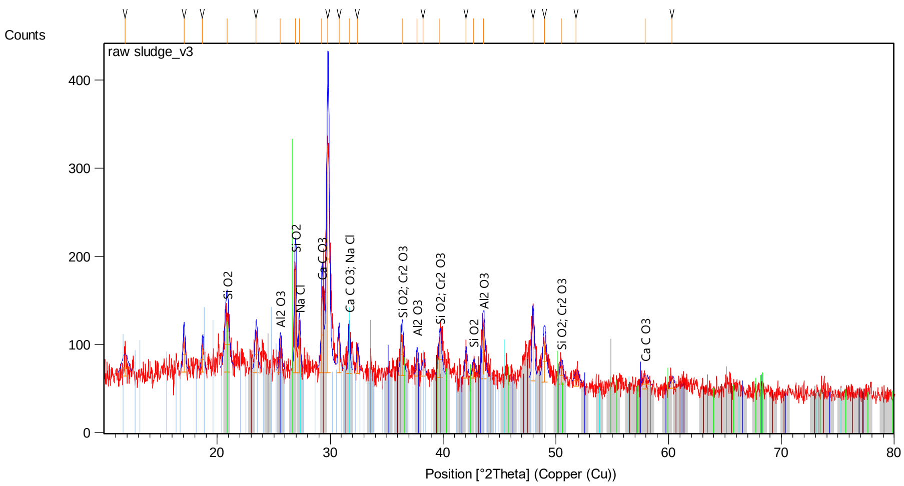

Fig. 5 - XRD pattern for water treatment sludge

3.2 Preparation of WTS

Acidification was used to remove the water treatment sludge's high aluminium. A reaction between aluminium hydroxide in WTS and sulphuric acid generated aluminium sulphate, which can be used as an effective coagulant in wastewater treatment plant (WTP) [18]. The effect of acid dosage and sludge concentration on turbidity removal of kaolin solution was studied to determine the best WTSC conditions.

3.2.1 Effect of Acid Dosage

The water treatment sludge coagulant was prepared with different sulphuric acid dosage to investigate the efficiency as a coagulant in treating kaolin synthetic wastewater. The method addition sulphuric acid was called acid digestion or acidification. The normality of sulphuric acid was constant at 0.5 N. Fig. 6 shows the graph of percentage turbidity removal with WTSC dosage. The highest percentage turbidity removal was 99.5% at 120 mg/L WTSC dosage for 0.08 ml/ml sulphuric acid dosage. Thus, the effective WTSC was 120mg/L dosage with 0.08 ml/ml sulphuric acid dosage for acidification. It is because the water treatment sludge become acidic in the addition of acid. In fact, the process increased the proton activity and aluminium leaching efficiency. From this, the sulphate ion increased in concentration which may decrease the dissolution rate of aluminium due to precipitation of aluminium sulphate [19-20]. However, the water treatment sludge did not recover highest aluminium because sulphuric acid which is the strong acid can act as an absorbent which leach out the other minerals such as phosphorus [21].

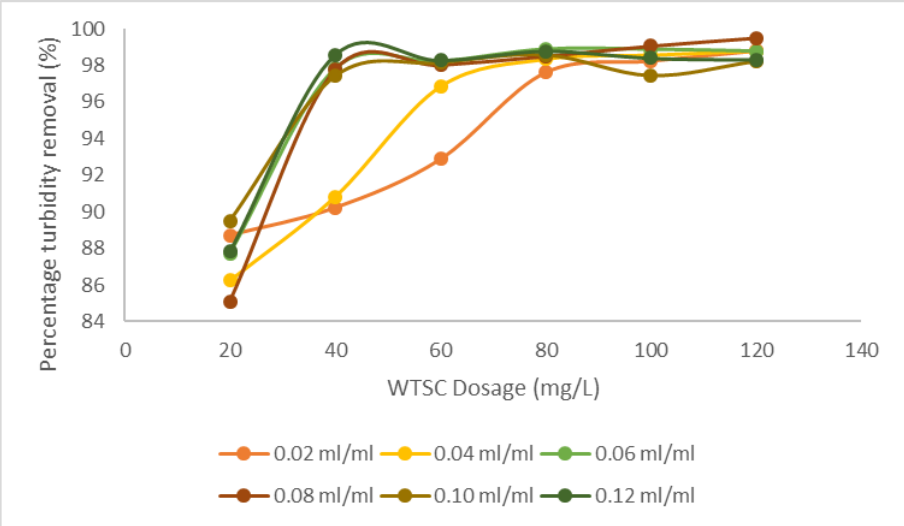

Fig. 6 - Turbidity removal with WTSC dosage for acid dosage

3.2.2 Effect of Sludge Dosage

It was decided what concentration of water treatment sludge to use when making the coagulant for WTS. An illustration of the relationship between percentages of turbidity removal and WTSC dose in sludge concentration factor is depicted in Fig. 7. The graph demonstrated that the percentage turbidity reduction rose by the dosage of 40 mg/L WTSC until it remained steady or dropped. The maximum percentage turbidity removal was achieved at 120 mg/L WTSC and a sludge concentration of 0.5 g/L when the WTSC was used as a coagulant. Consequently, the sludge concentration required for acidification is 0.5 g/L with 0.08 ml/ml of 0.5 N sulphuric acid to achieve the desired pH. It was discovered that 0.5 g/L provided the maximum efficiency because the lower the sludge concentration, the higher the rates of aluminium recovery because the lower the sludge concentration allows for greater contact between the acid and sludge particles [22]. The rate of acid consumption and the amount of aluminium leaching out of the sludge are proportional to the sludge concentration [21]. As a result, the greater the WTSC dosage, the greater the percentage of turbidity removed.

3.3 Performance of WTSC and Aluminium Sulphate (Alum) Coagulants in Coagulation Process

In this study, the performance of WTSC with alum as coagulant was evaluated in reducing turbidity of kaolin synthetic wastewater via coagulation process. A jar test was used in this research as it is a simple equipment for coagulation process. Several parameters were used in jar test which are pH and dosage of coagulant.

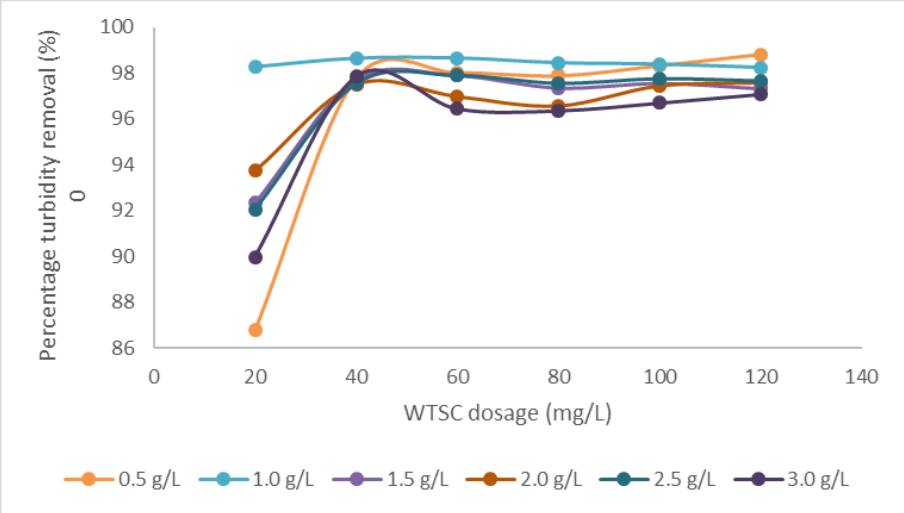

Fig. 7 - Turbidity removal with WTSC dosage for sludge concentration

3.3.1 Effect of pH and Dosage of WTSC

The pH values were modified from 2 to 12 in the coagulation process, which serves as a parameter in treating kaolin synthetic wastewater using WTSC. WTSC's chemical reaction with kaolin and aluminium is dependent on the pH of the water used in the procedure. Fig. 8 depicts the relationship between the percentage turbidity removal and the WTSC dosage for various pH and coagulant dosages. The trend for percentage turbidity removal was increased accordingly at first. Then it was decreased and constant at some point. With a 40 mg/L WTSC dosage, the highest turbidity was removed at a rate of 98.66 % when the pH was set to 12. Because alkaline water is preferable for the coagulation process because it tends to include more positively charged ions that can interact with the negatively charged colloids, water with high alkalinity is used. OH^- ions was present in significant concentrations, which resulted in the development of insoluble metal hydroxides, which in turn predominated the sweep-floc processes, capable of achieving high coagulation efficiencies in alkaline conditions [23]. WTS has the potential to be used as a coagulant because it removed a high percentage of turbidity in a wide range of pH solutions. With its ability to destabilise colloidal particles of kaolin suspension, it created enormous hard flocs that saved water treatment plant running costs in a short period of time, as a result.

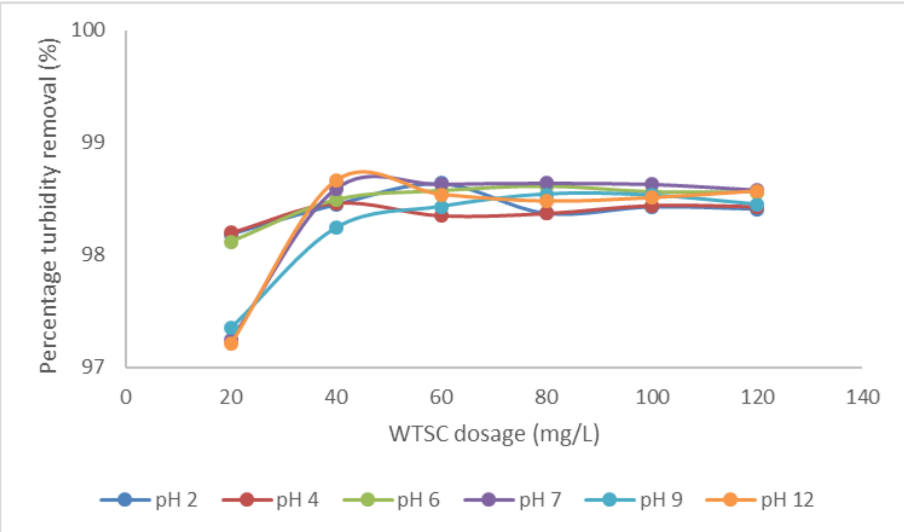

Fig. 8 - Turbidity removal with WTSC dosage for pH and dosage of coagulant

3.3.2 Effect of Dosage and pH for Aluminium Sulphate (Alum)

Aluminium sulphate (alum) was used in Jar test for coagulation process in this research to compare the performance with WTSC. Fig. 9 shows the graph of percentage removal turbidity with alum dosage. There were two factors in this process which are pH and coagulant dosage. The highest performance of alum was at pH 6 and 80 mg/L alum dosage at 99.74%. From the graph, performance of alum was great at pH 7 and pH 6. However, the performance was decreased when alum dosage increase. The pH 12 at first gave slow reaction in coagulation-flocculation process but it was increased when the alum dosage increased. When the aluminium concentration increased from 40 to 50 mg/L, the efficiency of turbidity removal decreased. For example, the removal of turbidity at pH 6 decreased from 97.1 to 95.7% when the aluminium concentration increased from 40 to 50 mg/L (initial turbidity of 100 NTU). This reduction may help to reverse and destabilise colloidal particles due to excess dosages, which they also hypothesised, according to [24].

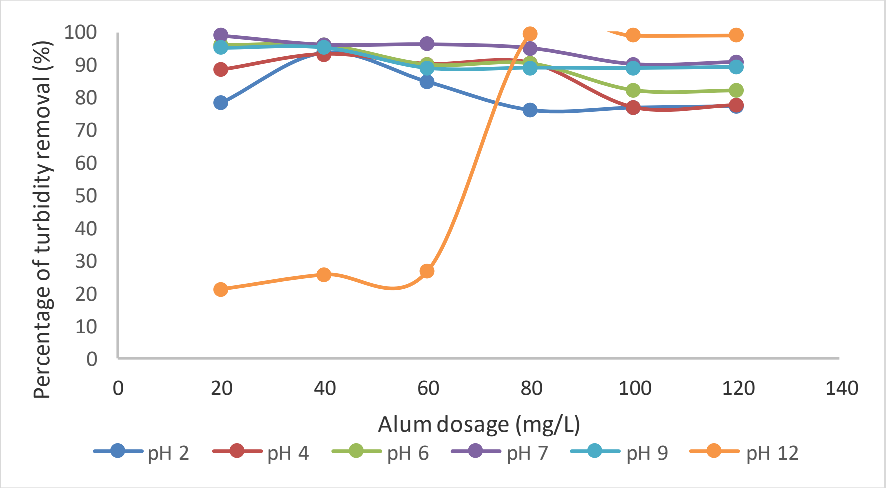

Fig. 9 - Turbidity removal with alum for pH and coagulant

4. Conclusion

This study was successfully examined and evaluated coagulation process for the turbidity removal using WTS from water treatment plant. The characteristics of WTS with high aluminium constituents, amorphous surface and presence of O-H bond made them possible to be recovered and reused as coagulant in wastewater treatment. The performance of WTSC and alum was nearly similar as both removed around 99 % turbidity. In a nutshell, WTSC from water treatment plant can be utilised as a coagulant to remove colloidal particles in water.

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