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Treatment of waste emulsion using coagulation method

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Abstract. Waste emulsion is one of the major toxic wastes that are generated from the metal processing industry. Emulsions are commonly used in the metal processing production as emulsified coolant and in power plant as lubricating oil. This paper presents a research to suggest the best coagulant in treating waste emulsion from manufacturing plant via coagulation method. Chitosan and bio-solvent will represent the natural coagulants, alum and polyaluminium chloride (PAC) will represent the industrial coagulant. Jar Test apparatus was used to treat the waste emulsion. It will be conducted in a batch test with a row of six beakers. One jar will be used as a control and the other five will be of different dosages of coagulant at 150 rpm of mixing rate, 30 minutes of mixing time and at room temperature. From the results, PAC showed to be the best coagulant with 82.29%, 95.45%, 99.95%, 96.4%, and 53% removal of COD, BOD O&G, TSS, and Turbidity, respectively, at a pH of 3.7 and minimum dosage of 2wt%. In conclusion, the PAC managed to break the oil and water bonding from the waste emulsion. Thus, it was suitable to treat the industrial waste emulsion.

1. Introduction

Water is one of the most important substances on earth. All plants and creatures must have water to survive. In the event that there is no water, there would be no life on earth. It covers around 71% of the Earth's surface, and is vital for all for all known forms of living. However, just 2.5% of the Earth's water is fresh water. Economic growth and industrialisation in Malaysia has led to better living quality but at a large cost of excessive waste and pollutants. With the current population rate at 1.4%, rapid progress in urbanisation and industrialisation has become a challenge in scheduled waste management [1]. According to the Department of Environment (DOE), a total of 2,918,478.34 metric tonnes of scheduled wastes were generated in Malaysia in 2015, all of which are dross/slag/clinker/ash, heavy metal sludge, gypsum, and oil and hydrocarbon were again the main categories. Waste emulsion is one of the major toxic wastes that are generated from the metal processing industry. Emulsions are commonly used in metal processing productions as emulsified coolant and also in power plant as lubricating oil. They are also commonly referred as cutting fluid, cutting oil, coolant, or lubricant. Usually, after they have lost their efficiency, these emulsion waste will be discharged or sent for treatment [2]. It is categorised as oil and hydrocarbon type of scheduled waste. Even so, oil and hydrocarbon type of scheduled waste is only 2.69% from the total 100%. This type of scheduled waste must not be looked at lightly, where this type of scheduled waste can rise to the top of the list if improper disposal is done.

Majority of the industrial wastes in Malaysia is sent to Kualiti Alam Sdn. Bhd. for treatment and disposal periodically. From a conducted survey, a manufacturing plant will produce around 20 tonnes of wastewater consistently in a month. This implies that 240 tonnes of wastewater is produced each year. This scheduled wastewater is send to Kualiti Alam Sdn. Bhd. at a rate of RM3,000.00 for every tonne; henceforth, each plant will spend more than RM720,000.00 every year. In Malaysia, there are more than 100 manufacturing plants operating and still developing. In this way, the plant will have to spend RM72 million yearly to treat this scheduled waste [2]. Since a huge amount of money is being spent by the industry to treat the wastewater, it is feasible to have a treatment facility that runs on an affordable budget, yet has high treatment performance. This study is going to evaluate the coagulation performance of chitosan, alum, bio-solvent and polyaluminium chloride (PAC) in handling this matter. Breakthroughs on the water treatment technologies have been developed over the years. Chemical treatment is one of the most useful and utilised techniques in treating waste emulsion [3]. Chemical treatment usually involves coagulation and flocculation process as a primary treatment, followed by a secondary stage treatment. As for the secondary stage treatment, it involves filtration, foam flotation, ion exchange, aerobic and anaerobic, electrolysis, solvent extraction, and adsorption treatment as a selection for a secondary stage treatment [4]. For this study, coagulation and filtration method will be employed by using common industrial coagulant and natural coagulant as primary treatment, followed by filtration using filter paper as the secondary stage treatment. Chitosan and bio-solvent will represent the natural coagulant, alum and polyaluminium chloride (PAC) will represent the common industrial coagulant. Coagulation method is selected because of its ability to deal with organic matter. In this case, the dissolved oil inside the waste emulsion can be broken using the coagulation method, based on the previous study [5]. Once the bond between oil and water is broken, it is easy to recover the water or oil for further usage. There are two types of coagulants selected because, according to a handful of previous studies, the common industrial coagulant used is chemical-based coagulant. Chemical coagulant may enhance the toxicity of the treated waste emulsion due to the reaction between the colloidal particles inside the waste emulsion with the chemical coagulant. This is different from the natural coagulant that will not enhance the toxicity of the treated waste emulsion because the coagulant is made from bio-material [6].

Alum or ($Al_2(SO_4)_2$) is the commonly used chemical coagulant in the industry. Since ancient times, this coagulant is popular to be used to treat suspended solid (TSS), turbidity and volatile suspended solid (VSS) in the wastewater. Alum was found to be the most effective coagulant for this application as compared to ferric chloride and lime. Alum was also the most effective coagulant in treatment of white and red wine wastewater [7]. Chitosan or poly-(1-4)-2-amino-2-deoxy-b-d-glucose is a biopolymer that can be chemically expressed as non-toxic, heterogeneous, linear, cationic, and biodegradable polysaccharide with high molecular weight produced by alkaline deacetylation of chitin [8], where chitin or poly(β-(1-4)-N-acetyl-D-glucosamine) is the major component of crustaceans shell such as crab, lobster, shrimp, and crawfish. Chitin and chitosan are both insoluble in water as well as in most organic solvents due to it hydrogen bonds between its molecules. However, it is soluble in acidic solutions due to the protonation of its amine groups. Due to that property, chitosan potentially has high affinity to adsorb pollution such as heavy metals and dye [9]. Bio-solvent is water-based dispersant that are formulated with an active ingredient called surfactants. Surfactant is design to have hydrophilic (water liking) & oleophilic (oil liking). Moreover, surfactants can reduce the surface tension of hydrocarbon molecules in oil, and the oil will spread out and be collected in droplets. Surfactant molecules possess hydrophilic head groups that associate with water molecules, while the oleophilic tails associate with oil molecules. This oil dispersant or degreaser product contains many different surfactants ingredient blended with it to solve all oil characteristic matters (EURO-CIRCUIT, 2017). PAC is a coagulant that derives from the action of its basic active constituent, namely, polyaluminium chloride. As a pre-hydrolysed coagulant, PAC is becoming increasingly popular in the coagulation-flocculation process. PAC has many benefits over the conventional hydrolysing aluminium or iron salts, such as better performance at low temperature, less aluminium residuals, less sludge volume, less effect on the pH value of raw water, and more rapid flocculation. Furthermore, there are large differences between the alum and PAC in morphology and precipitation in water [10]. This paper is a study done to suggest the best coagulant in treating waste emulsion from manufacturing plants via the coagulation method.

2. Materials and methods

2.1. Experimental materials

Approximately 50 kg of waste emulsion were collected from the CNC Machine at FTEK Lab. The collected samples were placed in a plastic container, sealed tight and labelled, before it was transported back to the laboratory. Samples were then refrigerated at about 4 ℃ to prevent bio-activities from occurring. Analysis was done within 6 to 24 hours of collection from the plant [11]. Chitosan was purchased from Hunza Nutriceuticals Sdn. Bhd and obtained from the FKKSA lab in powder form with viscosities and DDA of about 30–3000 mPa.S (at 25 ℃) and 85–99%. 15 L of polyaluminum chloride in liquid form were purchased from i-Chem Solution Sdn. Bhd. 1 L of bio-solvent in liquid form was purchased from Isnas Resources Sdn. Bhd. 3 kg of alum in powder form was purchased from Train Aid Global Sdn. Bhd.

2.2. Experimental procedure

2.2.1. Preparation of chitosan. Chitosan powder was used as a starting material in this work. The procedure for the activation of chitosan was prepared by diluting it with acetic acid to produce a gel-like substance. Dilution was conducted by using a self-estimated 7:2:91 ratio of chitosan to acetic acid to distilled water to produce a certain chitosan concentration. 35 ml of acetic acid was added into 455 ml of distilled water. 10 g of chitosan powder was later added slowly into the stirring solution to improve or promote well mixing and was stirred rapidly for 15 minutes. The prepared 500 ml of chitosan coagulant was stored in a beaker for further use [2].

2.2.2. Preparation of alum. Alum powder was used as a second material in this work. The procedure for the activation of alum was prepared by diluting it with diluted water to produce alum solution. Dilution was conducted by using concentrations of alum obtained from journals. The desired concentration of alum solution is 5000 mg/L [11]. 5 g of alum powder was added slowly into 1000 ml of distilled water to improve well mixing. The solution was stirred rapidly for 15 minutes. The prepared 1000 ml of alum coagulant was stored in a beaker for further use.

2.2.3. Jar Test procedures. Jar Test apparatus was used treat the waste emulsion. It will be conducted in a batch test with a row of six beakers, each having a stirring device. One jar will be used as a control and the other five will be with different dosages of coagulant (2 wt%, 4 wt%, 6 wt%, 8 wt%, 10 wt%, and 12 wt%) at 150 rpm of mixing rate and 30 minutes of mixing time and at room temperature. Experiment was repeated by varying the coagulant types and dosages. Analysis for the desired parameter was done with treated water with all the coagulants.

2.2.4. Laboratory analysis. The analysis of the treated sample was carried out in the Central Lab of Universiti Malaysia Pahang. Parameters of pH, TSS, O&G, COD, BOD, and turbidity were analysed. These methods are based on the Examination Manual of Water and Wastewater [12]. Table 1 summarises the analysis method.

Table 1. Summarised experimental analysis method.

Parameter	Test Method	Equipment
pH	ASTM D1293-18 (ASTM, 2018)	Seven Easy pH-METTLER TOLEDO
TSS	ASTM D5907-18 (ASTM, 2018)	Laboratory apparatus
O&G	ASTM D3921 (ASTM, 2018)	Laboratory apparatus
Turbidity	ASTM D7315 - 17 (ASTM, 2018)	HACH 2100P
COD	ASTM D1252 - 06(2012)e1 (ASTM, 2018)	HACH Model DR/2400 and HACH Reactor
BOD	ASTM D 1252 - 60 (ASTM, 2018)	Yellow Springs Model 5010 and BOD Incubator

3. Results and discussion

3.1. Characterisation of coagulants

Characterising the coagulants is important to be conducted before further investigation can be performed in order to understand the nature and behaviour of the coagulants. It is also important, as it would explain the reason behind its response toward specific treatment that is adopted in this study. Hence, the study was conducted to examine the two critical parameters of the coagulant, as shown in Table 2. The table shows the conductivity and pH of the coagulants measured in the laboratory.

Table 2. Characteristic of coagulant

No	Coagulant	pH	Conductivity	Unit
1	Low molecular weight chitosan with DDA 99% (LMWC)	3.7	4.34	mS/cm
2	Medium molecular weight chitosan with DDA 99% (MMWC)	3.45	4.12	mS/cm
3	High molecular weight chitosan with DDA 99% (HMWC)	3.49	4.32	mS/cm
4	Chitosan powder with DDA 85%	3.3	2.72	mS/cm
5	Chitosan powder with DDA 75%	3.24	2.63	mS/cm
6	Poly aluminium chloride (PAC)	2.03	4.78	mS/cm
7	Bio Solvent	12.22	3.84	mS/cm
8	Alum	3.65	0.0245	mS/cm

Reasons to study both of the parameters in Table 2 are: first, is to see whether the coagulants have enough positively-charged particles or not to neutralise the negatively-charged or colloidal suspension in the waste emulsion for coagulation to occur. Second, is to select the best type of chitosan to be used in this study. From the result, it showed that low molecular weight chitosan with DDA 99% (LMWC) has a higher conductivity value compared to other types of chitosan, therefore, it will be chosen as one of the coagulants that is going to be used in this study. Lastly, the pH value will indicate whether the coagulant can reduce or increase the waste emulsion pH value.

3.2. Characterisation of coagulants

Characterising the waste emulsion is important to be conducted before further investigation can be performed in order to understand the nature and behaviour of the coagulants. It is also important, as it would explain the reason behind its response toward specific treatment that is adopted in this study. The waste emulsion was compared with the Standard A and Standard B water quality parameter limits set by the Department of Environmental Malaysia (DOE). Discrepancy and comparison of the focused parameters are discussed in this chapter. In addition, from the comparison, it will provide a standard guideline to be compared with the treated wastewater.

Table 3. Characteristics of waste emulsion

No	Parameter	Unit	Water Quality Standards		Raw CNC Wastewater
			A	B	
1	Temperature	℃	40	40	23.3
2	pH Value	-	6.0-9.0	5.5-9.0	5.1
3	Suspended Solids	mg/l	50	100	More than 750
4	COD	mg/l	50	100	14,000
5	BOD5 at 20 ℃	mg/l	20	40	352
6	Oil and Grease	mg/l	1.0	10	969.0

Table 4. Physical characteristics of waste emulsion

No	Parameter	Unit	Result
1	Turbidity	NTU	100

Table 3 and Table 4 listed the values for the tested parameters of waste emulsion sample before the treatment processes. Standard A is a more stringent list compared to Standard B. This is because Standard A is applied as a regulation at the water catchment areas, which includes areas upstream of surface or above sub-surface of waterways, mainly directed to the community and for human use.

3.3. Effect of dosage on reduction of Chemical Oxygen Demand (COD)

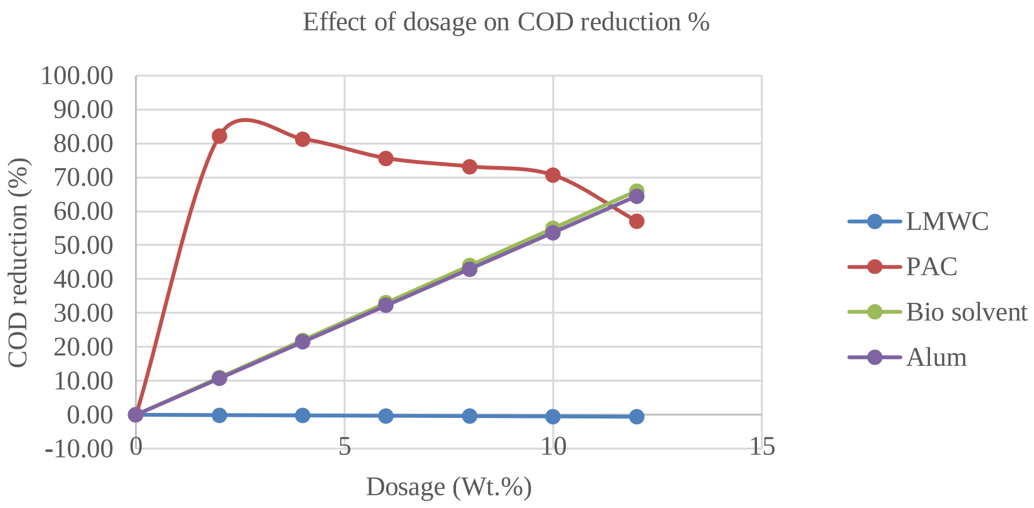

Figure 1. Effect of dosage on COD reduction (%)

From Figure 1, it can be seen that at the highest coagulant dosage (12 wt%), LMWC showed the lowest reduction of COD compared to other coagulant. Meanwhile, at the same dosage, bio-solvent showed the highest reduction of COD. PAC has 57.07% of COD reduction, while bio-solvent reduced 66.00%, LMWC reduced 0%, and alum reduced 64.50% at the maximum dosage. On the other hand, at the lowest coagulant dosage (2 wt%), PAC showed the highest reduction of COD compared to other coagulants. Meanwhile, at the same dosage, LMWC also showed the lowest reduction of COD. PAC has 82.29% of COD reduction, while bio-solvent reduced 11.00%, LMWC reduced 0% and alum reduced 10.57% at the minimum dosage. Based on the results, PAC proved to be the best coagulant in reducing COD from waste emulsion with a minimum dosage of injection.

Waste emulsion with high PAC dosage contains highly positive charged particle in exceed, that will make them repel to each other thus lowering the chance for them to attract to the negatively charged particle inside the waste emulsion. For Bio Solvent and Alum, as the dosage increase, the more the positively charged particle can settle down the negative charged, At this point, the chemical compound inside the waste emulsion used less oxygen to break the organic matter due to the organic matter is attracted to the positive charge particle. As the pH value of the waste emulsion becoming more acidic, the higher the effectiveness of coagulation, the higher the probability of organic matter to be attracted to the positive charge particle. Thus reducing the COD of the waste emulsion. As for LMWC the COD increase as the dosage increase.

3.4. Effect of dosage on reduction of Biological Oxygen Demand (BOD)

From Figure 2, it can be seen that at the highest coagulant dosage (12 wt%), PAC showed the highest reduction of BOD compared to other coagulants. Meanwhile, at the same dosage, LMWC showed the lowest reduction of BOD. PAC has 94.03% of BOD reduction, while bio-solvent reduced 75.10%, LMWC reduced 68.09% and alum reduced 71.47% at the maximum dosage. On the other hand, at the lowest coagulant dosage (2 wt%), PAC also showed the highest reduction of BOD compared to other coagulants. Meanwhile, at the same dosage, LMWC also showed the lowest reduction of BOD. PAC has 95.59% of BOD reduction, while bio-solvent reduced 19.76%, LMWC reduced 18.55% and alum reduced 19.16% at the minimum dosage. Based on the results, PAC proved to be the best coagulant in reducing BOD from waste emulsion with a minimum dosage of injection.

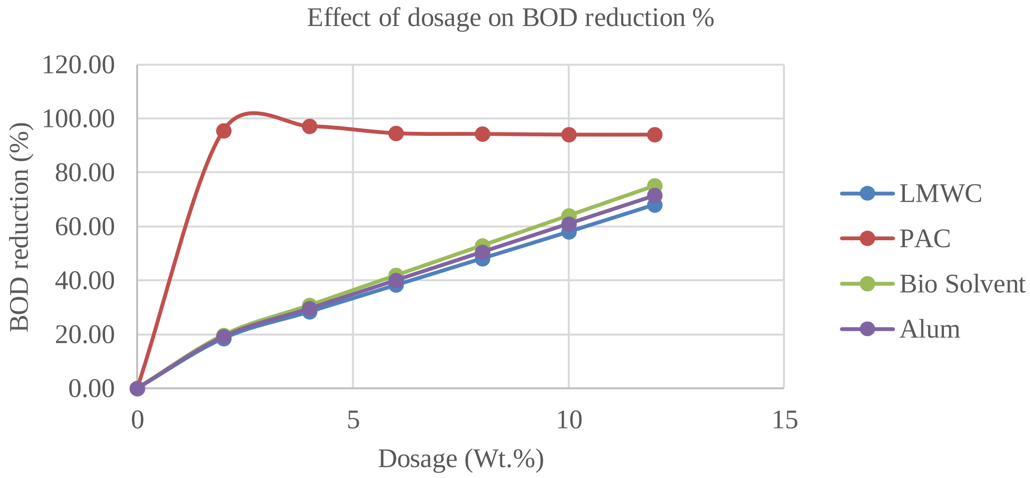

Figure 2. Effect of dosage on BOD reduction (%)

The results show that PAC have highest removal at minimum dosage due to this type of coagulant and have advantages to coagulate with a minimum number of dosage. At this point, the biological compound inside the waste emulsion used less oxygen to stabilize the organic matter. The organic matter is adsorbed by the positive charge particle as the pH value of the waste emulsion becoming more acidic. The higher the effectiveness of coagulation, the higher the probability of organic matter to be attracted to the positive charge particle thus reducing the BOD of the wastewater.

3.5. Effect of dosage on reduction of oil residue (O&G)

From Figure 3, it can be seen that at the highest coagulant dosage (12 wt%), LMWC showed the lowest reduction of O&G compared to other coagulants. Meanwhile, at the same dosage, alum showed the highest reduction of O&G. PAC has 93.29% of O&G reduction, while bio-olvent reduced 97.52%, LMWC reduced 92.98%, and alum reduced 99.48 % at the maximum dosage. On the other hand, at lowest coagulant dosage (2 wt%), PAC showed the highest reduction of O&G compared to other coagulants. Meanwhile, at the same dosage, LMWC also showed the lowest reduction of O&G. PAC has 99.90% of O&G reduction, while bio-solvent reduced 16.20%, LMWC reduced 15.48%, and alum reduced 16.62% at the minimum dosage. Based on the results, PAC proved to be the best coagulant in reducing O&G from waste emulsion with a minimum dosage of injection.

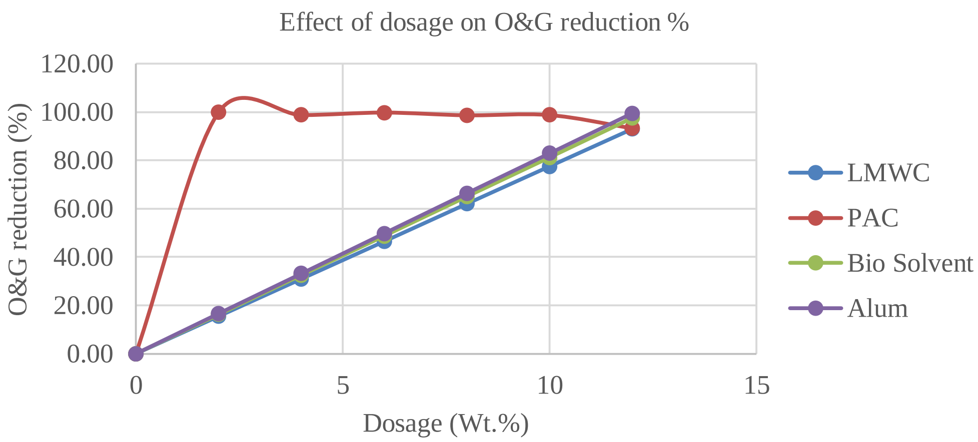

Figure 3. Effect of dosage on O&G reduction (%)

The results indicated that PAC able to break oil bond with water at a lower dosage. At this point, the dissolved solids in the oil residue in the waste emulsion are absorbed by the positively charged particle in the coagulants. Therefore the waste emulsion loss it emulsion characteristic thus reducing the O&G of the wastewater.

3.6. Effect of dosage on the reduction of TSS

From Figure 4, it can be seen that at the highest coagulant dosage (12 wt%), PAC showed the lowest reduction of TSS compared to other coagulants. Meanwhile, at the same dosage, bio-solvent showed the highest reduction of TSS. PAC has 3.07% of TSS reduction, while bio-solvent reduced 77.20%, LMWC reduced 53.20%, and alum reduced 2.40% at the maximum dosage. On the other hand, at the lowest coagulant dosage (2 wt. %), PAC showed the highest reduction of TSS compared to other coagulant. Meanwhile at the same dosage, alum showed the lowest reduction of TSS. PAC has 96.40% of TSS reduction, while bio-solvent reduced 12.80%, LMWC reduced 8.80%, and alum reduced 0.40% at the minimum dosage. Based on the results, PAC proved to be the best coagulant in reducing TSS from waste emulsion with a minimum dosage of injection.

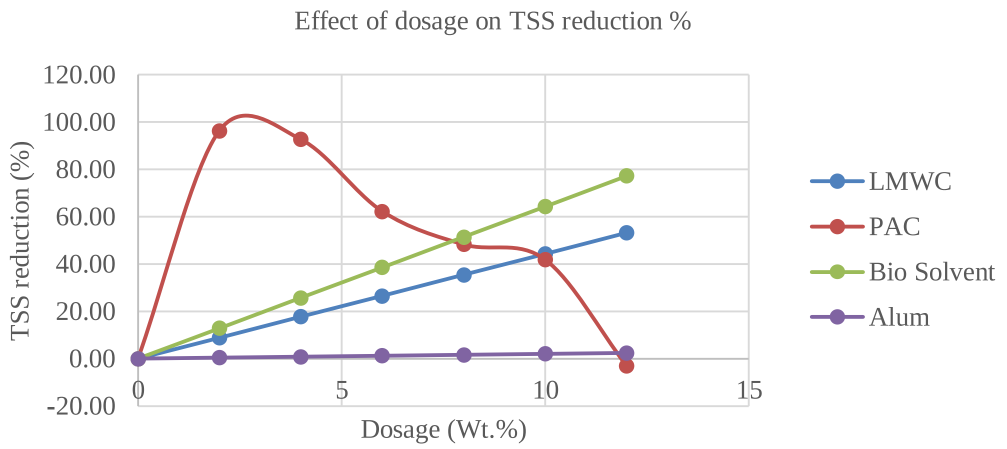

Figure 4. Effect of dosage on TSS reduction (%)

The results show that PAC showed to have an increasing and decreasing trend. Because when the waste emulsion is rich with PAC dosage, then the positively charged particle is in exceed, that will make them repel to each other thus lowering the chance for them to attract to the negatively charged particle inside the waste emulsion. At this point, the colloidal particle in the sample is absorbed by the positively charged thus reducing the TSS of the wastewater.

4. Conclusion

The present exploratory works showed that the PAC coagulant can be used for waste emulsion treatment. The optimum dosage needed for PAC, for coagulation of 500 mL of waste sample is 2wt%. The optimum operating condition for coagulation is at 150 rpm of mixing rate and 30 minutes of mixing time and at room temperature. The PAC coagulant showed satisfying results with 82.29%, 95.45%, 99.95%, 96.4%, and 53% removal of COD, BOD O&G, TSS and Turbidity, respectively, at a pH of 3.7. Based on the results, it showed that the coagulation method using PAC had managed to break the oil and water bonding from waste emulsion. Thus, it was found suitable to treat industrial waste emulsion. Therefore, it is recommended to use PAC to ensure a feasible treatment for treating emulsion waste.

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