Performance Study of Dragon Fruit Foliage as a Plant-based Coagulant for Treatment of Palm Oil Mill Effluent from Three-phase Decanters

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This study focused on the performance of dragon fruit foliage as a natural, plant-based coagulant to replace chemical coagulants for the treatment of three-phase decanter palm oil mill effluent (POME). Palm oil mill effluent is a high strength wastewater with high chemical oxygen demand (COD), suspended solids (SS), and turbidity as compared to other sources of POME in mills. The first objective of this study was to determine the optimum operating conditions of the coagulation-flocculation process using the plant-based coagulant in terms of pH and dosage. The second objective was to measure the percentage removal of COD, SS, and turbidity using the coagulant to treat the POME. The pH values and dosages used in the coagulation-flocculation process were in the range of two to ten and 300 mg/L to 800 mg/L, respectively. This study revealed that the optimum percentage removals of COD, SS, and turbidity were 48.7%, 98.8%, and 99.2%, respectively, at the best operating conditions of pH 2 and a coagulant dosage of 300 mg/L. In conclusion, the dragon fruit foliage, plant-based coagulant was found to have potential and could be used in the pretreatment stages to substitute chemical coagulants for the treatment of POME.

Keywords: Natural coagulant; Plant-based coagulant; Dragon fruit foliage; Coagulation-flocculation; POME treatment; Three-phase decanter

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INTRODUCTION

Malaysia is currently the world’s second largest palm oil producer and exporter, after Indonesia. One of the wastes generated from this industry is palm oil mill effluent (POME). The sources of POME include sludge condensers, sludge pits, sludge hydrocyclones, *etc*. Untreated POME is a major source of water pollution when it is discharged in rivers or lakes (Oswal *et al*. 2002). Palm oil mill effluent is a highly polluted wastewater that is particularly harmful to the environment if discharged directly into rivers because of its high chemical oxygen demand (COD), biochemical oxygen demand (BOD), phenol concentration, and color (Mohammed and Chong 2014). Water pollution has been a crucial issue; based on recorded cases, more than six million people die because of diarrhea related to this type of pollution (Asrafuzzaman *et al*. 2011).

A three-phase decanter is a piece of equipment used in the palm oil industry. This decanter is used to separate crude palm oil from POME and cake. Additionally, the decanter is used to recover the remaining oil from the underflow (sludge fraction) during the oil clarification process (Sahad *et al*. 2014, 2015; Yunos *et al*. 2015) and reduce the solid loading in the palm oil mill effluent before being sent for further biological treatment in an open ponding system (Adam *et al*. 2014).

Today, the palm oil industry uses chemical-based coagulants, such as aluminum sulphate ($Al_2(SO_4)_3$) and polyaluminium chloride (PAC), to treat POME. However, these inorganic coagulants can only function within a narrow range of wastewater pH. Sometimes the coagulant may also lead to pH depression in the wastewater stream due to the formation of their acidic salts. Nonetheless, the disadvantages associated with the use of these coagulants include high procurement costs, ineffectiveness in low temperatures, production of large chemical sludge in the form of scheduled waste, changes in the pH of treated water, as well as detrimental effects on human health and the environment (Sanghi *et al*. 2006; Yin 2010; Vijayaraghavan *et al*. 2011). Furthermore, prolonged exposure to water with high residual aluminum content could result in the possibility of Alzheimer's disease (Singh and Kumar 2011). Therefore, it is desirable that these chemical coagulants are replaced with cost-effective, natural coagulants to counteract the aforementioned disadvantages. Chitosan, a natural coagulant, has excellent properties for coagulants and flocculants in water and wastewater treatment. It has long polymer chains, a high cationic charge density, and high precipitates formation. A high concentration of chitosan leads to the complete neutralization of anionic charges; however, excessive chitosan dosage has caused dye suspension. High molecular weight chitosan slightly decreases the process efficiency of dye removal (Oladoja 2015). Seed extracts from European chestnuts and common oak acorns are another possible substitute. This coagulant exhibits the highest coagulation activities in both low and medium investigated water turbidities approximately 80% and 70%, respectively, at the lowest coagulant dose (0.5 mg/L). Although the value was below the European standards' maximum admissible concentration, a minimal increase in the organic matter content of the water treated with the extracts was noted (Oladoja 2015). The use of dragon fruit foliage as a natural coagulant for treating wastewater concentrated with latex has been found to be effective (Idris *et al*. 2012). In this study, dragon fruit foliage was used as a plant-based coagulant to replace chemical coagulants due to the existence of $Al_2(SO_4)_3$ discovered in the dragon fruit foliage as confirmed by an X-ray diffractometer (XRD) analysis (Som *et al*. 2007). However, for high strength wastewaters (*i.e.*, POME), the use of this coagulant has not been reported in literature.

Dragon fruits are also known as pitayas or strawberry pears and are originally native to Mexico as well as Central and South America. The popularity of dragon fruit has prompted countries to grow this crop for export beyond the local markets of Hong Kong, Singapore, Taiwan, and Malaysia. Consequently, large amounts of dragon fruit foliage are produced by dragon fruit farms. It is an agricultural by-product of priming the dragon fruit trees and is mostly treated as waste (Abidin *et al.* 2014). Today, research has even been done on the application of agro-industrial and industrial wastes to produce microalgal biodiesel for future renewable energy in Malaysia (Jayakumar *et al*. 2017). Dragon fruit foliage is suitable for use as a coagulant because of its low price, abundance, innocuousness, versatility, and biodegradability (Idris *et al.* 2012). Coagulation is a very common process in wastewater treatment and well known for its capability of destabilizing and aggregating colloids (Ahmad *et al*. 2006). By using coagulation coupled with different biological methods to treat wastewater from the vegetable oil refining industry, overall removal efficiencies of 92% to 96% COD, 83% to 98% TSS, and 93% to 95% oil and grease were achieved (Azbar and Yonar 2004).

Coagulant selection is very important for the efficiencies of treatment processes (Tan *et al*. 2006). The jar test mimics full scale operations well. Thus, good system operators can use jar tests to help determine which treatment chemicals will work best with their system's raw water. Because it is a natural resource available in abundance, the cost of using dragon fruit foliage is low compared to chemical coagulants. Furthermore, dragon fruit foliage is environmentally friendly and not harmful to human health as it does not leave any toxic residue (Idris *et al*. 2012).

The first objective of this study is to determine the optimum operating conditions of the coagulation-flocculation process using the dragon fruit foliage plant-based coagulant in terms of pH and dosage. The second objective is to measure the percentage removals of COD, SS, and turbidity of three-phase decanter POME treated with the plant-based coagulant.

EXPERIMENTAL

Materials

Preparation of the POME sample

The sample of POME was collected from the discharge outlet of a three-phase decanter at the palm oil mill at Felda Jengka 27, Pahang, Malaysia. The sample was stored in a refrigerator prior to its use in the experiment.

Preparation of dragon fruit foliage

The dragon fruit foliage was collected from the Bertam Dragon Fruit Farm at Gua Musang, Kelantan, Malaysia. Thorn from foliage was removed and tap water was then used to wash the foliage. Foliage was cut, milled, and sieved to a particle size of 0.45 mm to 1.25 mm, and then oven-dried at 50 °C (3 days) or 80 °C (1 day). Finely powdered foliage was stored in a tight container and kept in the refrigerator at 4 °C.

Jar test

Six beakers of 600 mL were obtained, and a 250 mL sample of POME from the three-phase decanter was placed in each beaker. To obtain the adjusted pH values, sulfuric acid (3 M) or potassium hydroxide (5 M) was used. The solutions were adjusted to pHs of 2, 4, 6, 8, and 10. The finely powdered dragon fruit foliage was added into every beaker with different dosages starting at 300, 400, 500, 600, 700, and 800 mg/L. The mixing process was executed in three stages, starting with a high-speed mixing at 100 rpm for 4 min, followed by slow speed mixing at 40 rpm for 25 min, and ending with 30 min for sedimentation purposes. After sedimentation, the sample was collected at the top layer and recorded for observation. All analyses were done in triplicates, and only their averages were reported in the results and discussion section.

Methods

Determination of suspended solids

A sample of 1 mL was diluted with 9 mL of distilled water and added into the vial. The blank sample of 10 mL distilled water was prepared and added into the vial. The blank vial was placed in a spectrophotometer (Model DR 2000; HACH, Loveland, CO, USA) and the reset button was then pushed. The sample vial was placed in the spectrophotometer and the reading was recorded.

Determination of chemical oxygen demand

A sample of 1 mL was diluted with 199 mL of distilled water. The cap of the high range COD digestion reagent vial was removed, and 2 mL of sample was pipetted into the vial. A blank sample was prepared by substituting 2 mL of deionized water for the sample. The vial cap was replaced tightly. The outside of the COD vial was raised with deionized water, and the vial was wiped clean with a paper towel. The vial was held by the cap over a sink. The contents were inverted and mixed gently several times. The vial was placed in the preheated COD reactor. The vial was then heated for 2 h. The COD reactor was then turned off. It was required to wait for approximately 20 min for the vials to cool down by placing the vials into a rack. Once the vials cooled to room temperature, the spectrophotometer was switched on, and the program for COD tests was selected. The sample was placed into a spectrophotometer (Model DR 2000; HACH, Loveland, USA) and its reading was taken in mg/L.

Determination of turbidity

The turbidity meter (Model 2100P; HACH, Loveland, USA) was switched on for at least 30 s prior to the experiment. The blank sample was prepared with distilled water that was poured into the vial. A sample of 0.5 mL was diluted with 18 mL of distilled water into the vial. The vial sample was closed and shaken for 30 s. The outside of the vial was then cleaned and placed into the turbidity meter prior to pressing the reset button. The sample was placed into the turbidity meter and its reading was taken in mg/L.

RESULTS AND DISCUSSION

POME characteristics

When POME is compared with domestic water, POME has a COD and BOD 100 times higher than that of domestic water (Sethupathi 2004). Although POME has high contaminants with COD and BOD, POME is not classified as toxic. Based on a previous study, POME is a brown slurry containing 95% to 96% water, 0.6% to 0.7% residual oils, and 4% to 5% solids (mainly organic) (Lim *et. al*. 2014). The temperature of the POME at discharge is in the range of 80 °C to 90 °C and is naturally acidic with a pH of 4 to 5 (Sethupathi 2004). Table 1 shows the characteristics of POME from the three-phase decanter at the palm oil mill in Felda Jengka 27, Pahang, Malaysia.

Table 1. Raw Characteristics of Three-phase Decanter POME from a Palm Oil Mill

Parameter	Average Value
pH	4.47
Color	Brownish
COD	97,400 mg/L
Suspended Solids	32,050 mg/L
Turbidity	54,420 mg/L

Dragon fruit foliage characteristics

The dragon fruit foliage was chosen as a coagulating agent because of its characteristics being similar to those of a coagulant, which includes having positive charges to interact with the negative charges present in wastewater, as reported by Som *et al*. (2007)

and Idris *et al*. (2012). After the foliage was dried using an oven for three days with a temperature of 50 °C, its size and weight decreased. This was done so that the dragon fruit foliage would have a very low moisture content, so that it was expected to last longer.

Experimental results from jar tests

Sulfuric acid and potassium hydroxide were added to adjust the pH of the POME sample. When the POME was adjusted to basic pHs, the colour of POME changed from brownish to darker. When the alkalinity of the POME solution increased, the dark colour of POME also increased. This may have been due to the reaction of potassium hydroxide and a POME contaminant that affects the colour change (Ahmad *et al.* 2006). In other experiments for the treatment of POME, the color changed when the pH concentration was increased from an acidic to an alkali value (Bhatia *et al.* 2007).

A comparison of the suspended solids removals from all of the pH values used is shown in Fig. 1. The five pH values used showed more than 60% suspended solids removal at all dosages used. The best pH value to remove suspended solids was at pH 2, while the lowest percentage removal of suspended solid was at pH 10. As the pH decreased, the percentage removal of suspended solid thus increased. This is because a higher pH value resulted in a lesser tendency for the coagulation species to form positive charges, which makes the coagulant less attracted to anionic compounds (Bhatia *et al.* 2007). At different dosages, only small changes in the percentage removal of suspended solids were recorded for all observed pH values.

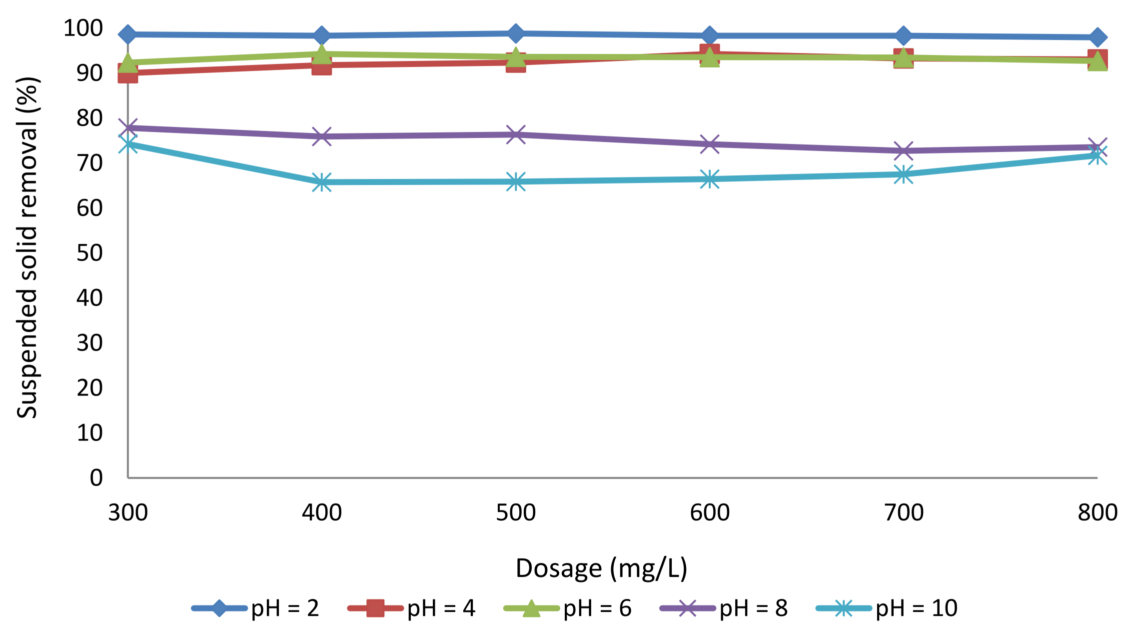

Fig. 1. Percentage removal of suspended solids for all pH values (2, 4, 6, 8, and 10) *versus* different dosages of coagulants

A comparison of the percent decreases of COD at varying levels of coagulant dosage for the pH values used is shown in Fig. 2. The range of percentage COD removal was fairly low, between 25% to 50%. The three-phase decanter unit is an oil separation process that is responsible for producing the high COD levels in POME discharge. Moreover, it was the highest contributor to COD in POME as compared to other POME sources in the mill. The highest percentage of COD removal was at pH 4, while the lowest percentage COD removal was at a pH of 6, as can be seen in Fig. 2, and the COD removals were 40.8% (at a dosage of 300 mg/L) and 48.7% (at a dosage of 500 mg/L), respectively. The dosage of powdered dragon fruit foliage did not result in further effects on the percentage COD removal, unlike suspended solids. A dosage of 500 mg/L was considered to be not efficient for pHs 4, 6, and 8; but for pH 2 and 10, they were still higher as shown in Fig. 2. Because pH values of 4, 6, and 8 are approaching neutral, at these pH conditions, the dragon fruit foliage coagulant was not suitable to be used to treat the three-phase decanter POME for COD removal purposes.

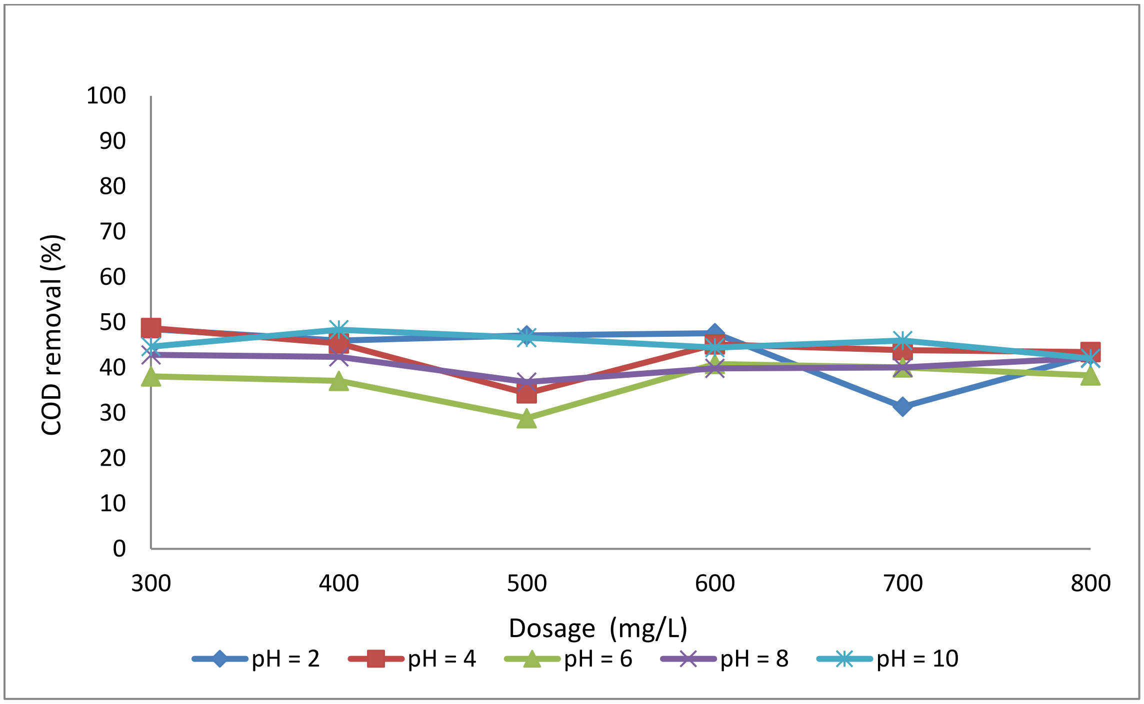

Fig. 2. Percentage removal for all pH values (2, 4, 6, 8, and 10) *versus* different coagulant dosages for COD removal

The percentage removals of turbidity with different pH values and coagulant dosages are shown in Fig. 3. The highest percentage removal of turbidity (more than 98%) was at pH 2 and the lowest (less than 90%) was at pH 10 and 8. As the pH increased, the percentage removal of turbidity decreased. Naturally, turbidity and suspended solids are always caused by the same contaminants in the solution. Accordingly, when the level of suspended solids increases, turbidity also increases. The percentage removal of turbidity, for acidic solutions of POME, showed a minimal dependency on dosage. Only small changes in percentage turbidity removal were achieved by varying the dosage at high pH values.

Effect of pH value

The effects of pH were investigated relative to the removal of suspended solids, COD, and turbidity. Coagulation by dragon fruit foliage particles occurred because these positively charged particles were attracted to the negative charges of the sludge or contaminants in POME and they combine. The combination of these particles produced flocs that settled down to the bottom of the beaker. The percentage removals for suspended solids and turbidity were relatively high at every pH used as these contaminants are mainly present in the form of sludge particles. For example, turbidity from suspended and colloidal matter in POME are made up of clay, silt, finely divided organic and inorganic matter, plankton and other microscopic organisms (Ibrahim 2009).

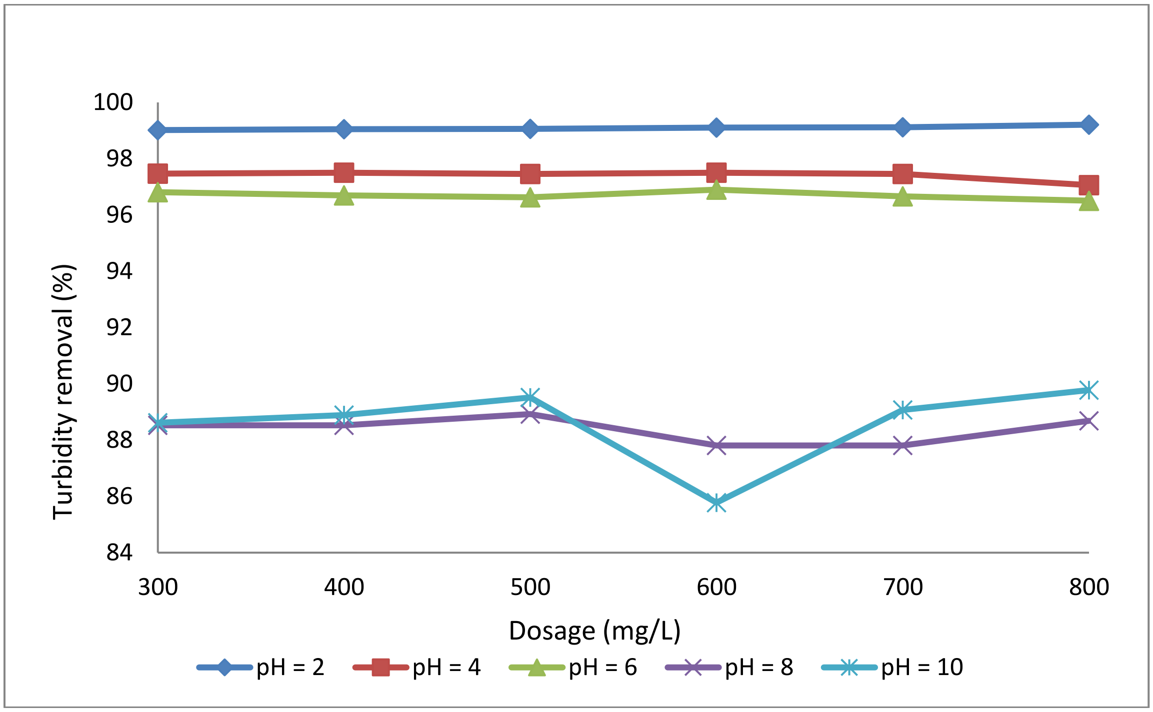

Fig. 3. Percentage removal for all pH values (2, 4, 6, 8, and 10) *versus* different coagulant dosages for turbidity removal

The turbidity and suspended solids of a water sample are caused by some of the same contaminants. Thus, a higher turbidity results in higher suspended solids; the contaminant of suspended solids in water, such as small salts, will cause the water to turn cloudy (Myre and Shaw 2006). When the sludge particles or suspended solids in POME were removed, the turbidity also decreased. The overall results showed that a pH value of two gave the best removal as compared to pH values of 4, 6, 8, and 10. Therefore, this result showed that the acidic conditions from a pH of two were the best operating parameter for treatment of POME. Based on a previous result, the plant seeds are the most effective natural coagulant used in water with lower pH because suspended pollutants tend to coagulate better at lower pH values (Yongabi 2010).

The sample with a pH of 2 did not change colour when it was adjusted with sulfuric acid. However, for pHs 8 and 10, the colour of the POME changed from brown to a dark colour when the pH was made more alkaline by using potassium hydroxide. When it was dark in colour, the coagulation process using the dragon fruit foliage was not efficient; the suspended solids and turbidity experiments showed that it was very difficult for the flocs to settle down.

Alkaline conditions were not suitable for dragon fruit foliage to extract the suspended solids, turbidity, and COD from the three-phase decanter POME. Furthermore, strongly acidic conditions aggravated POME to break oil droplets and destabilize the suspended solids that were initially in suspension (Ahmad *et al*. 2006). In this study, pH 2 was recorded as the best operating parameter, which gave higher percentage removals of 98.8% of suspended solids, 48.7% of COD, and 99.2% of turbidity. It is applicable in practice to maintain acid conditions during the coagulation-flocculation process using dragon fruit foliage (DFF) as both POME and DFF are already somewhat acidic. Even though DFF is considered pH sensitive, similar to inorganic coagulants, it still offers environmentally friendly characteristics due to its plant origin. Further research needs to be carried out to overcome this pH sensitivity issue in the case of DFF. Suspended solids and turbidity were at the highest removal percentages (almost 100%) in treating three-phase decanter POME. While only half of COD was removed from the three-phase decanter POME using coagulation, the rest would be removed through biological treatment such as activated sludge processes.

Effect of dosage

The dosage used was very important to coagulation efficiency when coagulant was used to remove suspended solids, decrease COD, and lower turbidity. The different dosages were used for every pH so that the performance of the coagulant at the optimum dosage and pH could be evaluated. The dosage performance depends on the type of coagulant used (*i.e.* cationic, anionic or nonionic) and strong positive charges of the coagulant as well as the materials to be treated. Based on the results, different dosages did not have a large effect on the percentage removal.

Therefore, an increase in dosage did not give more positive charges to extract suspended solids, COD, and turbidity in POME. In choosing the best dosage for dragon fruit foliage coagulant, other factors should also be considered such as cost of the coagulant. Based on the cost of coagulant within the Malaysian context and quantity of dosage, 300 mg/L was the best choice for dosage because the cost and quantity were relatively low to treat POME.

Comparison study with other natural coagulants

The comparison between the dragon fruit foliage coagulant used in this study and other natural coagulants used to treat POME, based on their performance in terms of suspended solids and COD percentage removals, can be seen in Table 2. Three types of coagulants were used: *Moringa oleifera*, chitosan, and wheat germ.

Table 2. Comparison with Previous Studies of Other Types of Natural Coagulants

Type of Natural Coagulant	Operating Condition, pH	Coagulant Dosage, mg/L	Removal Efficiency (%)			Reference
			Suspended solids	COD	Turbidity	
Moringa oleifera	5	6000	95.0	52.2	NA	Bhatia *et al*. 2007
Chitosan	4	500	95.0	NA	NA	Ahmad *et al.* 2006
Wheat germ	2	12000	95.6	61.7	99.1	Daud *et al*. 2014
Dragon fruit foliage	2	300	98.82	48.73	99.21	This study

NA = Not available

Recommendation for future works

It is recommended to carry out further research to minimize the sensitivity of the DFF coagulant on the pH variation. This is important, as the primary aim of this study is to provide a substitute for inorganic coagulants, which are highly pH sensitive as one of its defects. Besides pH, the effect of different temperature conditions on DFF performance would have to be studied further as chemical reaction is very much temperature dependent. As DFF is considered agro-waste and abundantly available, it is also recommended to combine those with any commercial coagulants or flocculants so as to minimize operating costs as well as propagating green material for sustainability purposes.

CONCLUSIONS

1. Dragon fruit foliage has potential and could be commercialized as a coagulant to substitute for chemical coagulants in the pretreatment of three-phase decanter POME.
2. The suspended solids were removed with the highest percent removal of 98.8% at a pH of 2 and dosage of 500 mg/L. To remove COD, 48.7% COD removal was achieved at a pH of 4 and a 300 mg/L coagulant dosage. Turbidity decreased 99.2% from the three-phase decanter POME at pH 2 and a coagulant dosage of 800 mg/L.
3. Small effects on the percentage removal were shown when different dosages were used. This might have been due to small differences in the coagulant dosage that were not suitable to test the performance of the coagulant. All dosages used had shown the same result and only a small difference was reported among them. Therefore, 300 mg/L of dosage was judged to be the best choice based on the quantity used and cost of the coagulant dosage. In summary, the dragon fruit foliage could be used as a pretreatment prior to employing primary or secondary treatments in treating POME.

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