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Potential of Using *Hibiscus Sabdariffa* in Treating Greywater

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Abstract. Greywater recycling can help to curb water shortages especially in developed countries as they consumed high amount of water for their activities. Greywater can be treated using coagulation process due to its simplicity and effectiveness with usage of coagulant. Studies show that the usage of conventional coagulant has bad effect on human health, produces large volume of sludge and affect pH of treated water. Therefore, a replacement on conventional coagulant with natural coagulant is preferable as it is safer for human health, biodegradable and toxic-free. In this study, *Hibiscus Sabdariffa* seeds as natural coagulant is used due to high protein content compared to other parts of the plant which important for coagulation process. The objectives of this study are to investigate the performance of the seeds in treating greywater by varying pH, coagulant dosage and mixing effects and compare the performance with conventional coagulant. Jar test experiment was carried out. It was found that the seeds work the best at pH 2, 100 mg/l of coagulant dosage, 30 rpm for slow mix and 140 rpm for rapid mix which resulted in 93.83% and 57.98% removal efficiencies of turbidity and COD respectively. Alum coagulant was used to compare the coagulation performances. Alum coagulant works the best at pH 6, 200 mg/l of coagulant dosage, 40 rpm for slow mix and 150 rpm for rapid mix which resulted in 97.48% and 65.26% removal efficiencies of turbidity and COD respectively Therefore, it shows that *Hibiscus Sabdariffa* seeds is comparable with alum coagulant and hence, it has potential in treating greywater.

INTRODUCTION

The global demand for freshwater is increasing every year and it is set to exceed the supply by 2050 [1]. For developed countries, this caused is driven by activities from development in terms of urbanization, industrialization, tourism and communications [2]. Study done by [3] reported that the extensive drainage basin development for large scale projects in Malaysia causing the level of river beds to rise which could result in flooding and thus reduced the amount of freshwater supplies. In regards with this, treated greywater can be reused for toilet flushing and garden irrigation to curb water shortages [4]. In Malaysia, greywater constitutes 43% of total water use which is equivalent to 97 liters of greywater generated per person in a day [5] and it indicates that the freshwater demand can drop significantly if greywater being recycled widely.

Greywater is defined as untreated household wastewater that not mix with sewage and usually generated from shower, toilet, sink, washing machine, dishwasher and kitchen sink [6]. Generally, the main difference of these wastewaters is greywater has lower organic pollutant concentration and pathogens due to the absence of feces, urine and toilet paper [7]. These characteristics of greywater made it relatively easy to be treated compare to sewage [8].

Decentralized treatment system is more practical when it comes to greywater treatment for domestic reuse [9, 10]. It can be operated in small scale which helps to reduce financial burden on people living in low-income urban areas [9, 10]. It is more suitable for sustainable development apart from ease the duty operation on centralized treatment system [11]. The most common treatment that can be applied on decentralized treatment system is coagulation process

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due to its simplicity, effectiveness [12] and ability to be implemented widely in either urban or rural area [13]. Furthermore, the price of coagulant is cheap and widely available.

The coagulation process is aimed to reduce the amount of pollutants in wastewater such as turbidity, color, organic matters as well as metal ion contents [14]. The coagulant is added into the wastewater to neutralize the charges of the colloidal particles and the flocs formed will be settled at the bottom [14]. Alum is one of the most common coagulants used in water treatment industry, however, the studies showed that the usage of alum can cause bad effect for human health as well as it produces large volume of sludge and affects pH of treated water [13]. Therefore, natural coagulant is preferable to treat the wastewater as it is safer for human health, biodegradable and toxic-free [15].

The protein is one of the important features for natural coagulant to carry out coagulation process which help to reduce the turbidity. The protein content inside *Hibiscus Sabdariffa* seeds is the highest which is 31.02% compare to the other parts of the plant [16] and thus, making it one of the natural coagulants. Most of the studies showed that *Hibiscus Sabdariffa* seeds can remove the turbidity more than 80% and the highest turbidity removal efficiency is 96% for turbid wastewater (300 NTU) [17]. *Hibiscus Sabdariffa* seeds also showed potential in removing dye in textile wastewater up to 96.67% [18] and able to reduce the COD level by 29% in pharmaceutical wastewater [19]. However, the performance of the *Hibiscus Sabdariffa* seeds in treating greywater is yet to be unknown. To bridge this knowledge gap, this paper investigates the potential of *Hibiscus Sabdariffa* in treating greywater. The effect of pH, coagulant dosage and mixing effects on coagulation process are also studied in this research and single factor method is used to determine the best combination. The performance of the natural coagulant is compared with conventional coagulant, aluminum sulphate (alum).

METHODOLOGY

Preparation of Natural Coagulant from *Hibiscus Sabdariffa* Seeds

Hibiscus Sabdariffa was obtained from Pusat Memproses Makanan FAMA, Rengit, Johor. The seeds of *Hibiscus Sabdariffa* was removed from its capsule and dried in the oven for 3 hours at 60 °C to remove the moisture content [19]. The sample of the dried seeds was taken to measure its moisture content using Moisture Analyzer XM50. Constant reading of residual weight of the sample measured by moisture analyzer indicates that the seeds are fully dried or else, the seeds will be going through drying process again for an hour until it fully dried. 1 g of the dried seeds and 100 ml of extractive solvent were blended using food blender for 2 minutes [19] to make 1% (w/v) suspension. The suitable extractive solvent is 0.5 M of sodium chloride solution as the binding sites of sodium chloride solvent is enough to extract all the proteins fractions [20]. The seeds residue was filtered out by pouring the milky mixture through Smith Qualitative Medium Speed 102 filter paper. The coagulant was prepared and used on the same day to ensure optimum performance as the microbial decomposition of organic compounds might present in the coagulant [21].

Preparation of Conventional Coagulant from Alum

Aluminium sulphate or alum was used in this project to compare the performance between the natural coagulant and conventional coagulant. The alum coagulant was prepared by dissolving 1 g of alum powder with 100 ml of distilled water to make 1% (w/v) suspension. The suspension was mixed using magnetic stirrer for 10 mins at room temperature [22].

Collection of Greywater

All the greywater used in this study was collected from the bathroom, Pangsapuri Mutiara Perdana, Selangor. 12 liters of water samples were collected where the same products were used to minimize the variation of greywater characteristics. The greywater was recollected if the stored greywater exceed 48 hours before running the jar test due to the deterioration of greywater quality [23, 24]. The turbidity of the greywater was measured using turbidimeter TN-100 EUTECH Instruments and the COD was measured using photometer Prove 300 Spectrometer before running the jar test experiment. The characteristics of the greywater used in this study are tabulated in Table 1.

TABLE 1. Characteristics of greywater

Parameters	Unit	Min	Max
pH	n.a	6.5	6.8
Turbidity	NTU	77.8	86.9
COD	mg/L	376	414

Jar Test Experiment

Jar tests were conducted using a standard apparatus compromising six 500 ml beakers to evaluate the performance of natural coagulant and conventional coagulant. There is a total of 4 parts in the experiment, namely to determine the optimum pH, optimum coagulant dosage, optimum slow mixing and optimum rapid mixing for *Hibiscus Sabdariffa* coagulant and alum coagulant. The range of parameters carried out in this study for both coagulants is summarized in Table 2. The experiment was done part by part where each test will involve rapid mixing, slow mixing and sedimentation process. The pH of the greywater was adjusted using 1 M of hydrochloric acid and 1 M of sodium hydroxide before running the jar test and measured using pH meter. 500 ml of greywater was added with coagulant and rapidly mixed for 4 minutes to disperse the coagulant effectively. The mixing speed was then reduced for further 20 minutes to allow the growth of the flocs. The mixture was then allowed to settle for 1 hour for sedimentation [22]. The treated water was filtered using filter paper. The optimum condition is determined based on the highest turbidity and COD removal efficiencies.

TABLE 2. Summary of jar test experiment for *Hibiscus Sabdariffa* coagulant and alum coagulant

Parameters	pH	Coagulant dosage (mg/l)	Slow mixing velocity (rpm)	Rapid mixing velocity (rpm)
Part 1	2-11	300	30	150
Part 2	*	100-700	30	150
Part 3	*	*	20-50	150
Part 4	*	*	*	100-160

*optimum condition

Analytical Method

Turbidity

The measurement of turbidity is to show the amount of colloidal matter present in water and it is the widely used method to easily screen the result. The turbidity was measured using turbidimeter TN-100 EUTECH Instruments [19]. The turbidity removal efficiency was calculated using formula below:

$$\text{Turbidity removal efficiency} = \frac{T_c - T}{T_c} \times 100\% \tag{1}$$

where T_c is the value of initial turbidity and T is the value of final turbidity.

Chemical oxygen demand (COD)

COD measures the amount of oxygen that is required to oxidize organic materials. High COD values mean the amount of oxidizable organic material is large and can lead to low dissolved oxygen which in further can pose threat to aquatic life. Thus, lower COD is preferable. The procedure to measure COD removal efficiency is first started with 3 ml of the sample was added into reaction cells of range 25-1500 mg/l. The cells were heated using thermoreactor at 150 °C for 2 hours. The cells were removed from thermoreactor and allowed to cool to room temperature. Then, the COD was measured using photometer Prove 300 Spectrometer [19]. The COD removal efficiency was calculated using formula below:

$$\text{COD removal efficiency} = \frac{COD_c - COD}{COD_c} \times 100\% \tag{2}$$

where COD_c is the initial COD value of the sample and COD is the final COD value of the sample.

RESULTS AND DISCUSSION

Coagulation Activity using *Hibiscus Sabdariffa* Seeds Coagulant

Effect of pH on coagulation activity

pH is one of the factors that has significant effect on coagulation activity as it affects the stability of protein of the seeds [22]. Study done by [21] proves that the coagulation process using *Hibiscus Sabdariffa* seeds is highly pH-dependent. This explained the result obtained as shown in Fig. 1 and Fig. 2 where the highest turbidity removal and COD removal efficiencies is 90.69% and 54.38% respectively at pH 2. Lower turbidity and COD removal efficiencies are achieved in alkaline conditions, pH 8 -11 compare to acidic condition where the values are less than the lowest values achieved in acidic condition which are 68% and 33% for turbidity and COD removal efficiencies respectively.

The charges of the wastewater particles are typically consisting of more negative charges while amino acids of the protein which consist of glutamic acid and aspartic acid, are the cationic amino group [22]. In acidic condition, the density of positive chargers (H+) around coagulant hydrolysates is high and it enhances the cationic amino acids group to act as coagulant. The cationic coagulant protein will neutralized the dissociated organic compounds inside the greywater and encourages the particles to stick together [25]. Hence, it resulted in higher turbidity removal efficiency in acidic condition, especially at pH 2. This finding is supported by the previous study done by [19, 26] in which the seeds work efficiently under acidic condition.

In neutral and alkaline conditions, the turbidity and COD removal efficiencies are lower than in acidic condition. In neutral condition, the particles in greywater are merely absorbed on the surface of coagulant when they collide with each other. However, the absorption can occur due to weak chemical bonding but not attraction of charges as in coagulant in acidic condition [25] and thus, low turbidity removal efficiency. The turbidity removal efficiency is slightly increased from pH 8 to 11 due to the increase in density of (OH-) charges which promotes the dissociation of small amount of arginine present in the coagulant. The greywater particles will coagulate with this protein and hence, reduce the turbidity of treated greywater [22].

The graph of COD in Fig. 2 shows similar trend to graph of turbidity in Fig. 1. Generally, it can be concluded that pH 2 is the most suitable for *Hibiscus Sabdariffa* seeds to act as coagulant effectively based on the highest turbidity and COD removal efficiencies and hence, it was kept constant for the following parts of the experiment.

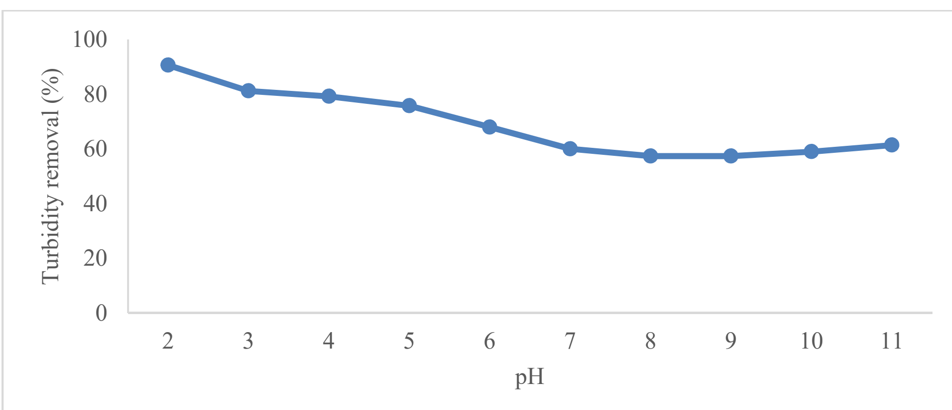

FIGURE 1. The effect of pH on turbidity of greywater after treatment

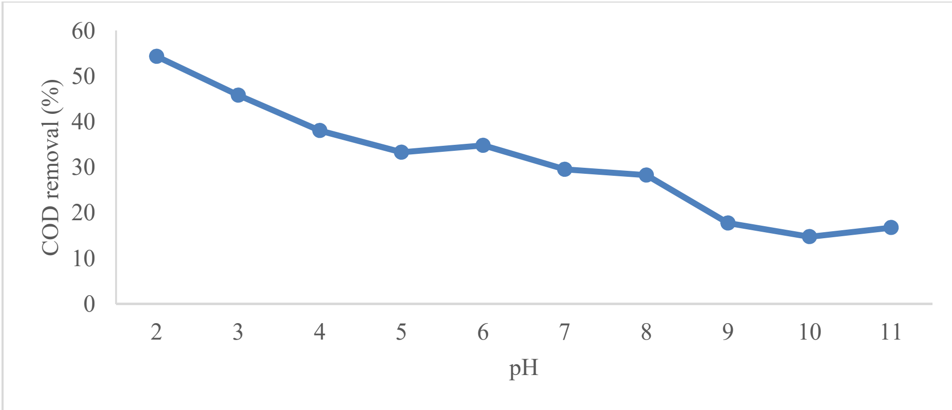

FIGURE 2. The effect of pH on COD of greywater after treatment

Effect of coagulant dosage on coagulation activity

The flocs formation depends on coagulant dosage where the charges of coagulant will neutralize the greywater particles and encourages the particles to stick together. Low amount of dosage will neutralize low greywater particles as it has low energy and thus, form weak and small flocs [27]. Overdosing the greywater might result in charge reversal which caused poor performance of coagulation activity [28]. In this experiment, the optimum amount of coagulant dosage is 100 mg/l where the highest turbidity and COD removal efficiencies are 93.06% and 57.89% respectively as shown in Fig. 3 and Fig. 4.

It can be observed that the flocs formation increases as coagulant dosage is heightened during the experiment. This is due to the high amount of coagulative components that are available as coagulant dosage increases that can neutralize the greywater particles charges more effectively [13]. The turbidity removal efficiency should be increasing as well however, in this experiment, it turns out that the result is the opposite as show in Fig. 3. This is might due to the high amount of flocs that was left unsettled inside the beaker which can be observed after 1 hour sedimentation time. The unsettled flocs might pass through the filter paper which affected the turbidity reading. Hence, it can be concluded that longer sedimentation time is needed for the flocs to be settled at the bottom of the beaker as the volume of flocs is higher as coagulant dosage increases.

From Fig. 4, the COD removal efficiency drops significantly at 700 mg/l of coagulant dosage. This condition is caused by charge reversal where the greywater particles are undergoing re-dispersion and re-stabilization due to overdosing of coagulant protein. The same condition occurred in removing dye in textile wastewater when the *Hibiscus Sabdariffa* coagulant dosage reaches up to 709 mg/l [21].

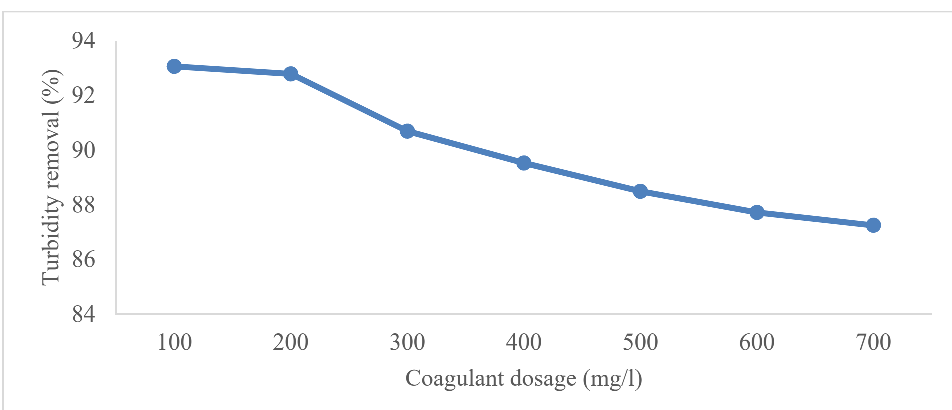

FIGURE 3. The effect of coagulant dosage on turbidity of greywater after treatment

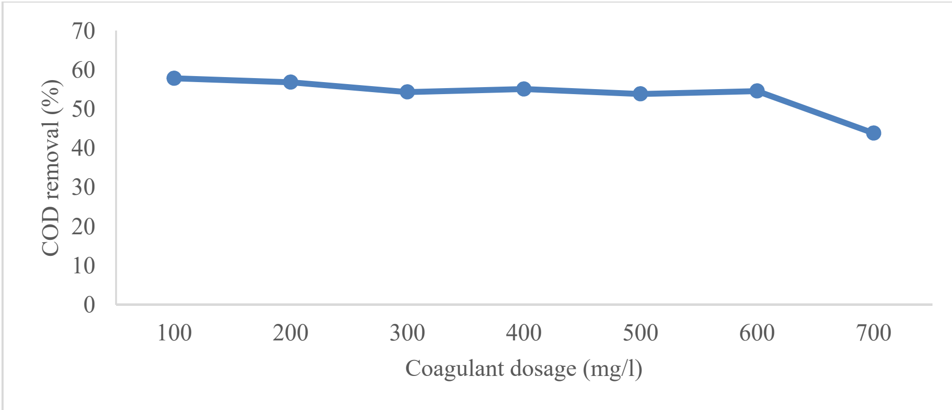

FIGURE 4. The effect of coagulant dosage on COD of greywater after treatment

In previous study by [26] which tested the *Hibiscus Sabdariffa* coagulant in acidic condition resulted in 93.13% turbidity removal efficiency for industrial wastewater with 40 mg/l coagulant dosage and study done by [19] resulted in 35.8% and 29% of turbidity and COD removal efficiencies respectively in pharmaceutical wastewater with 190 mg/l coagulant dosage. Thus, the optimum coagulant dosage in this experiment is acceptable as it is lower as compared to the coagulant dosage in more severe condition of pharmaceutical wastewater. However, the experiment should be tested at dosage lower than 100 mg/l as it has the possibility in more efficient coagulation activity concerning study done by [26].

Effect of mixing effects on coagulation activity

Mixing effects can be divided into two categories which are slow mixing and rapid mixing. The purpose of slow mixing is to allow the maximum growth of the flocs and the purpose of rapid mixing is to ensure the highest collision between the greywater particles and the coagulant [19]. The suitable mixing effects is important to coagulation process as turbidity and TSS removal efficiencies depend on mixing effects [29].

For slow mixing velocity, the highest turbidity and COD removal efficiencies are 93.06% and 57.89% respectively at 30 rpm as shown in Fig. 5 and Fig. 6. These findings are supported by previous study done by [30] that shows the highest turbidity removal occurred at 30 rpm for low initial turbidity (50 NTU) and 40 rpm for medium initial turbidity (150 NTU) when using natural coagulant. The study also noted that the slow mixing velocity depends on the initial turbidity of the wastewater and since the initial turbidity of greywater falls between low and medium initial turbidity, thus the result obtained in this experiment is acceptable. From Fig. 5 and Fig. 6, the coagulation activity decreases as slow mixing speed increases from 30 rpm to 50 rpm. This is due to the disturbance of floc formation where the particles undergo re-dispersion which caused less efficient coagulation activity. Study done by [31] noted that re-dispersion of particles of already formed flocs occurred when slow mixing is above 60 rpm.

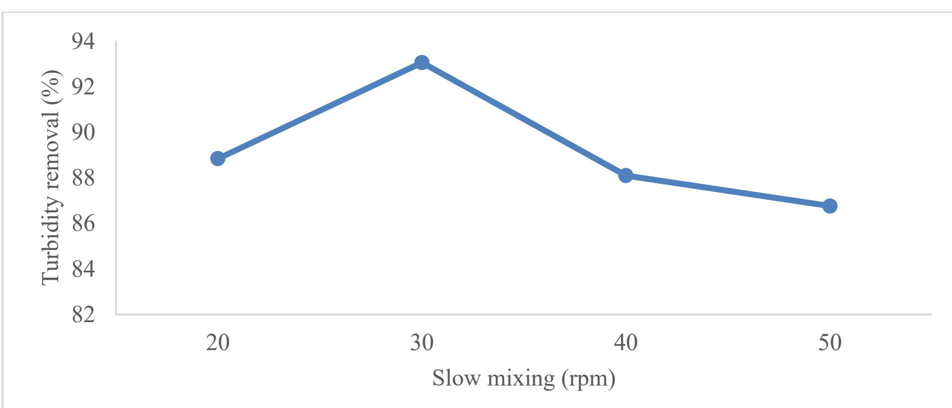

FIGURE 5. The effect of slow mixing on turbidity of greywater after treatment

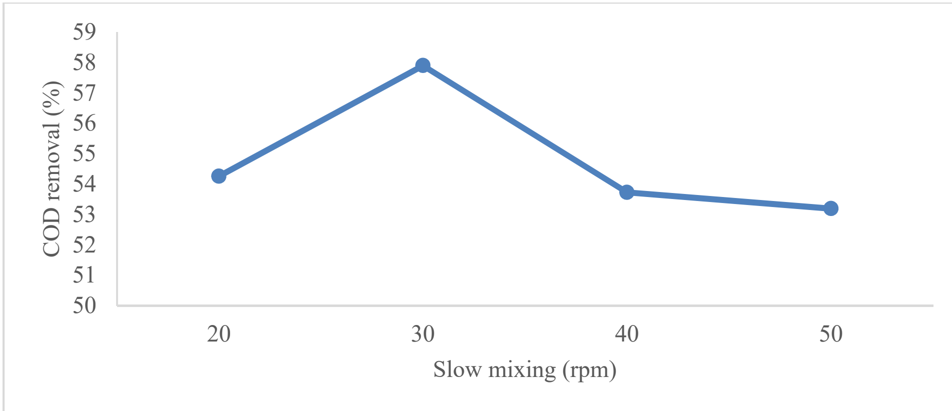

FIGURE 6. The effect of slow mixing on COD of greywater after treatment

For rapid mixing velocity, the highest turbidity and COD removal efficiencies are 93.83% and 57.98% respectively at 140 rpm as shown in Fig. 7 and Fig. 8. This result is in an agreement with [30] in which the highest turbidity removal efficiency occurred at 120 rpm and 140 rpm for low initial turbidity (50 NTU) and medium initial turbidity (150 NTU) respectively when using natural coagulant. Study done by [32] noted that the rapid mixing depends on initial turbidity of wastewater where less speed needed for low turbidity and high speed needed for high turbidity. Rapid mixing causes the total particles count decreases as the formation of flocs increases. The size of flocs in the wastewater increases with increment in rapid mixing velocity up to optimization and further increment will cause the re-dispersion of already formed flocs [30, 32] and thus, reduce the efficiency of coagulation activity at 150 rpm and 160 rpm as shown in Fig. 7 & Fig. 8. Therefore, it can be concluded that the 140 rpm of optimum rapid mixing velocity is acceptable according to the initial turbidity of greywater which falls in between low and medium initial turbidity.

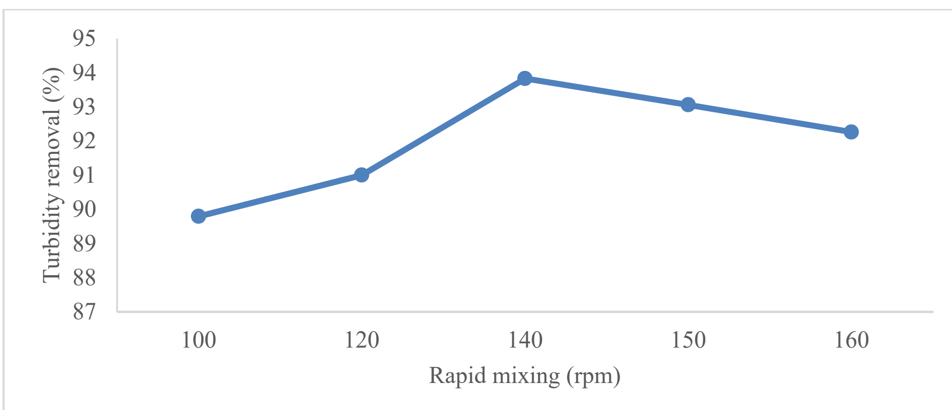

FIGURE 7. The effect of rapid mixing on turbidity of greywater after treatment

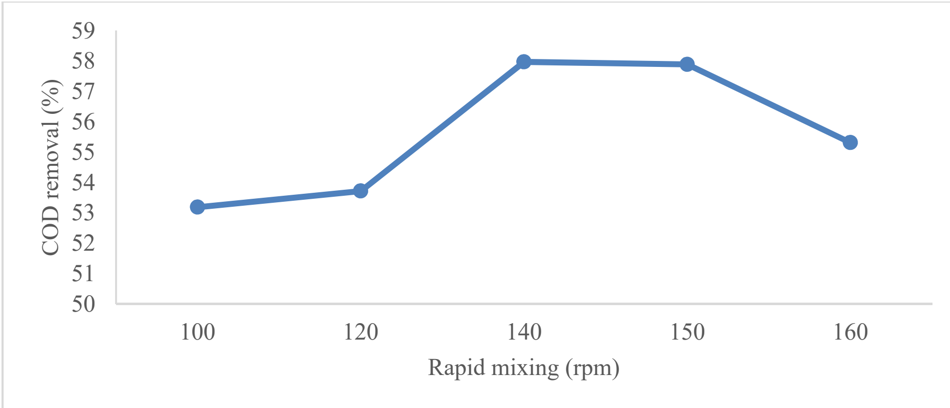

FIGURE 8. The effect of rapid mixing on COD of greywater after treatment

Comparison of *Hibiscus Sabdariffa* Seeds and Alum Coagulant

Alum coagulant is used to compare the performance between the natural coagulant and conventional coagulant. In this experiment, the highest turbidity and COD removal efficiencies are achieved at pH 6 with optimum alum dosage of 200 mg/l. The result is almost similar to [33] where the alum coagulant works the best at pH 5.5 with 204 mg/l when tested in greywater which resulted 92.2% of turbidity removal efficiency. For slow mixing and rapid mixing, the optimum speeds are found to be 40 rpm and 150 rpm respectively. The findings are almost similar to [30] where the optimum slow mixing is 30 rpm and rapid mixing is 140 rpm. The study from [34] noted that the residual turbidity is the result of combination between rapid mixing and coagulant dosage. Hence explained slight difference between mixing effects values between the studies which is might due to the amount of coagulant dosages used in [30] that is less than 100 mg/l of alum coagulant.

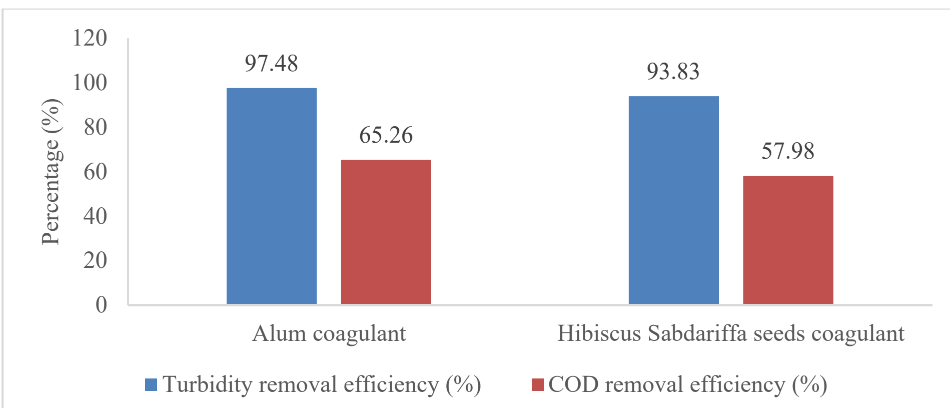

FIGURE 9. Comparison of alum coagulant and *Hibiscus Sabdariffa* seeds coagulant

From Fig. 9, it can be seen that the turbidity and COD removal efficiencies for alum coagulant are 97.48% and 65.26% respectively. These values are higher than coagulation activity for *Hibiscus Sabdariffa* seeds. However, the findings show that the result obtained from using *Hibiscus Sabdariffa* seeds is comparable to alum coagulant. The results can be improved by using ion-exchange column chromatography for protein purification which done by [35].

Thus, the use of *Hibiscus Sabdariffa* seeds in greywater treatment as a natural coagulant may be an option with many advantages over chemical coagulant, particularly biodegradability, low toxicity and safer for human health. Hence, the decentralized greywater treatment with this natural coagulant seems to be possible to be applied in the future in Malaysia. Additional equipment might be needed if it were to be commercialized considering the seeds works the best at acidic condition.

CONCLUSION

High consumption of water in developed country for large projects has become an issue in which these activities could reduce freshwater supplies. Hence, recycling greywater for toilet flushing and garden irrigation can help to curb water shortages. In Malaysia, the greywater recycling can be initiated from ablution activities and bathroom [8] which can be treated using coagulation process. Studies show that chemical coagulant such as alum that used in coagulation process, has bad effect on human health, produces high amount of sludge and change pH of treated water. Therefore, natural coagulant such as *Hibiscus Sabdariffa* is more preferable as it is safer for human health, biodegradable and toxic-free. In natural coagulant, protein is the one of the crucial features to carry out coagulation process. For *Hibiscus Sabdariffa*, the seeds possess the highest protein content compare to other parts of the plant and hence, the usage of seeds as natural coagulant in this study. From the experiment, the seed works the best at pH 2, 100 mg/l of coagulant dosage, 30 rpm for slow mix and 140 rpm for rapid mix which resulted in 93.83% and 57.98% removal efficiencies of turbidity and COD respectively. *Hibiscus Sabdariffa* coagulation performance is slightly lower than alum coagulant performance where the turbidity removal efficiency is 97.48% and COD removal efficiency is 65.26% at pH 6, 200 mg/l of coagulant dosage, 40 rpm for slow mix and 150 rpm for rapid mix. Therefore, it can be concluded that *Hibiscus Sabdariffa* has potential in treating greywater as the performance is comparable to conventional coagulant.

For future work, further analysis can be conducted to study the volume of flocs formed and sedimentation time by using *Hibiscus Sabdariffa* seeds coagulant. Purification of protein extracted from seeds can be considered to achieve better performance. The synthetic greywater produced from [36] is not suitable for the experiment as it resulted in inconsistent coagulation activity due to improper dilution as in real life.

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