INVESTIGATION OF BIOFLOCCULANT AS DEWATERING AID IN SLUDGE TREATMENT

MOHAMMED SAEDI JAMI*, MAIZIRWAN MEL, AYSHA RALLIYA MOHD ARIFF, AND QABAS MARWAN ABDULAZEEZ

Department of Biotechnology Engineering, Faculty of Engineering,
International Islamic University Malaysia,
Jalan Gombak, 53100 Kuala Lumpur, Malaysia.

**Corresponding author: saedi@iium.edu.my*

(Received: 15th Sept 2016; Accepted: 5th May 2017; Published on-line: 1st June 2018)

https://doi.org/10.31436/iiumej.v19i1.735

ABSTRACT: Sludge treatment is one of the most important and expensive steps in water and wastewater treatment plants. Chemical conditioners such as polyaluminum chloride, aluminum sulfate, Fenton's reagent, gypsum, and polyacrylamide can produce byproducts that cause health and environmental problems. *Moringa oleifera* (MO) seeds can be used as a natural alternative to chemical conditioners. The bioactive materials have to be extracted from MO seeds for better performance. In this study, the treatment methods of MO seeds were the bioactive extraction by NaCl (1 M) and oil extraction by hexane solvent, as well as the untreated (crude) seeds powder. Synthetic sludge samples were prepared using kaolin suspension (5% w/v). The most effective coagulant-form was determined based on the values of settling velocity (V_s) and sludge volume index (SVI). Results showed that extraction by NaCl gave the best results of 0.41 cm/min of settling velocity and 63.39 ml/g of SVI. A SVI value greater than 150 ml/g indicates poor settling qualities whereas the control sludge of the current study was 100 ml/g. The most effective coagulant-form was optimized with respect to three process conditions: MO seeds dosage, mixing speed, and contact time. The experiments were designed using 2 Level Factorial-Design by Design-Expert software. The optimum process conditions were seeds dosage of 3246 mg/l, mixing speed of 102 rpm, and mixing time of 29 min. MO seeds can be considered as a natural coagulant that can be used as main sludge conditioner.

ABSTRAK: Rawatan kotoran mendapan adalah salah satu rawatan penting dan termahal dalam merawat air dan sisa kumbahan loji. Perapi kimia seperti poli-aluminium klorida, aluminium sulfida, reagen Fenton, gipsum, dan poli-akrilamida menghasilkan sisa, di mana memberi kesan kepada kesihatan dan alam sekitar. Benih *Moringa oleifera* (MO) boleh digunakan sebagai bahan ganti semula jadi kepada perapi kimia. Bahan bio-aktif perlu diekstrak daripada benih MO bagi memberi kesan terbaik. Dalam kajian ini, kaedah rawatan menggunakan benih MO adalah dari ekstrak bio-aktif NaCl (1 M) dan ekstrak minyak dari bahan larut hexane, serta serbuk benih tidak dirawat (mentah). Sampel sintetik kotoran mendapan disediakan dengan menggunakan ampaian kaolin (5% w/v). Bentuk kogulan yang paling efektif didapati berdasarkan nilai halaju malar (V_s) dan indeks ketumpatan kotoran mendapan (SVI). Keputusan menunjukkan ekstrak NaCl memberi keputusan terbaik pada halaju malar 0.41 cm/min dan bacaan pada SVI 63.39 ml/g. Nilai SVI lebih besar daripada 150 ml/g menunjukkan kualiti kadaran malar kurang baik berbanding 100 ml/g kajian kawalan semasa kotoran mendapan. Bentuk kogulan yang paling efektif telah dioptimumkan pada tiga keadaan proses: dos benih MO, halaju campuran dan tempoh campuran. Eksperimen dibentuk menggunakan 2 Level Factorial-Design daripada perisian Design-Expert. Keadaan optimum proses adalah pada 3246 mg/l dos benih, 102 rpm halaju campuran, dan tempoh campuran selama 29 min. Benih MO boleh di kategori sebagai kogulan semula jadi dan boleh digunakan sebagai perapi utama bagi kotoran mendapan.

KEYWORDS: *sludge treatment; Moringa oleifera; dewatering; environmental friendly*

1. INTRODUCTION

The major aim of wastewater treatment is to eliminate or minimize the dangerous effects on public health and the environment. Various types of contaminants should be removed from wastewater, such as suspended solids, biodegradable matter (organic compounds), inorganic solids, nutrients, metals and pathogenic microorganisms. In order to remove these contaminants, wastewater treatment requires physical, chemical, and biological processes. The physical and chemical processes are usually used for potable water treatment while the biological process is mainly for wastewater treatment due to the high organic concentration [1].

Sludge can be generated from settling tanks during primary, secondary and advanced treatment processes. These processes separate the suspended solids and colloidal materials from the liquid body and produce sludge at high solids content of 0.25 - 12% by weight [2]. With the high range of solid concentration, sludge management is considered to be one of the most difficult and expensive processes in wastewater treatment [3]. Sludge should pass through many treatment stages before disposal, which may include thickening, anaerobic and aerobic digestion, and dewatering. Thickening and dewatering processes reduce the content of water and increase solid concentration. The dewatering process occurs during use of mechanical systems such as belt filter presses, centrifuges or gravity drainage, and evaporation in sand drying beds. Usually, mechanical dewatering equipment is expensive and consumes high amounts of energy [1]. Dewatering by chemical conditioners is very efficient and it is used worldwide, but has negative impacts on human health and the environment associated with the formation of byproducts [4].

Moringa oleifera (MO) seed is a natural polymer and a highly biodegradable coagulant that is affordable and environmentally friendly. The seeds have potential to be used as sludge conditioner for thickening and dewatering. These seeds were investigated for the removal of turbidity, heavy metal ions, color, and as disinfectant [5-8]. MO seeds with a cationic property are expected to be efficient in adsorbing the sludge particles and neutralizing the charge on their surface resulting in effective coagulation and elimination of the hazardous impacts on human health and the environment.

2. MATERIALS AND METHODS

2.1 Materials and Equipment

Dry MO seeds were imported from Genius Herbs Company, India. Kaolin suspension was used as the synthetic sludge, which was bought from R&M Chemicals Sdn Bhd. NaCl from HmbG was used for bioactive material extraction. A kitchen blender was used for seed blending. Soxhlet apparatus was used for oil extraction from MO seeds. Jar test apparatus was used for process condition optimization. Vacuum filtration with a Buchner-funnel and Whatman-filter-paper #3 were used to determine TSS. A 500 ml graduated cylinder and stopwatch were used for the sludge volume index (SVI). Other laboratory equipment included a magnetic stirrer, a drying oven, and a weighing balance.

2.2 Methods

2.2.1 Preparation of Sludge Sample

Sludge samples were prepared from kaolin suspension by adding 5 g of kaolin to 1 liter of distilled water in order to generate a sludge with 5% total solids (w/v). The sample was mixed at a speed of 200 rpm for about 10 min using jar test apparatus [9,10].

2.2.2 Preparation of Different Forms of MO Seeds

The shelled MO seeds were ground by kitchen blender and screened using a 212 µm sieve. Three types of MO seeds were prepared as natural conditioners: Crude MO seeds, defatted MO seeds, and MO extracted by NaCl (1 M). All coagulant forms were used as an aqueous solution. The crude MO seed powder was used for coagulation without any treatment after mixing with distilled water. The second type was oil-extracted by hexane solvent using 10 g of MO seed powder with 200 ml of hexane. Soxhlet extraction apparatus was used for oil extraction [6,9,11].

The first two types (crude and defatted MO seed powder) were mixed with distilled water by adding 10 g of powder into 1 liter distilled water, then, the mixture was rapidly mixed for 10 min to extract the bioactive materials. Finally, the mixture was filtered using filter paper and the insoluble powder particles were removed.

For salt extraction, 58.44 g of NaCl was added to 1 liter of distilled water and mixed using jar test apparatus. The NaCl solution was mixed with 10 g of crude MO seeds for 10 min. Finally, the mixture was filtered using filter paper and the insoluble powder particles were removed [12].

2.2.3 Experimental Procedures

Jar test apparatus was used for sludge conditioning using six beakers of 1 liter capacity. The selection of the most effective coagulant form was determined through the sludge volume index (SVI) and settling velocity (V_s). Experiments were run at MO dosages of 2000, 4000, and 6000 mg/l. The most effective coagulant form was optimized under different process conditions.

2.3 Design of Experiment and Statistical Analysis

2.3.1 Process Conditions Optimization

The design of experiment (DOE) was used to optimize the three process conditions: conditioner dosage, mixing speed and contact time, and the settling velocity (cm/min) was used as the response. The primary ranges were determined using one-factor-at-a-time (OFAT) preliminary optimization. The experimental results were analyzed using Design-Expert software v10 (Stat-Ease Inc.). The variables and their range of levels for the experimental design are listed in Table 1. Experimental design for process conditions optimization is listed in Table 2.

Table 1: Ranges of levels for experiment parameters

Parameters	Unit	Range of levels		
		-1	0	+1
Concentration of MO seeds	mg/l	500	3250	6000
Mixing speed	rpm	50	100	150
Contact time	min	5	17.5	30

2.3.2 Settling Velocity (Vs) Measurement

Settling velocity (V_s) was used to determine the most effective coagulant form together with SVI. V_s was also used as a response for process condition optimization. V_s was measured using graduated beakers and a stopwatch and one reading was recorded each 1 min for 30 min. Then, the settling rate was determined by plotting the V_s values versus the time, and the slope of the curve represents the final settling velocity [10].

2.3.3 Sludge Volume Index (SVI) Measurement

The sludge volume index (SVI) is the volume of one gram of sludge after 30 min of settling. The SVI was determined by placing the samples into cylinder beakers and measuring the settled sludge after 30 min [10]. The values of SVI was determined using Eqn. (1):

$$\mathrm{SVI} = \frac{\text{settled sludge volume (ml/l) x 1000 (mg/g)}}{\text{TSS (mg/l)}} \tag{1}$$

2.3.4 ANOVA Analysis

The results were analyzed using analysis of variance (ANOVA) approach and *P-value*. The significance of each coefficient was analyzed by comparing the obtained *P-value* with 0.05 (95% confidence level). The model is significant if *P-value* was less than 0.05 [13]. The optimum V_s value was obtained using a regression model equation, and 2D and 3D plot analysis.

3. RESULTS AND DISCUSSION

3.1 The Most Effective Coagulant Form

Settling rates were estimated from the slope of the straight-line portion of the plot interface height versus settling time. From the results obtained, V_s for MO salt extract was the highest settling velocity of 0.41 cm/min at 4000 mg/l dosage, as shown in Fig. 1. Settling velocities for salt extraction were 0.37 and 0.366 cm/min for 2000 and 6000 mg/l, respectively. The defatted MO seeds showed lower settling velocities compared to the salt extract. The settling velocities were 0.35, 0.36, 0.34 cm/min for dosages of 2000, 4000, and 6000 mg/l, respectively. The lowest settling velocities were recorded using crude MO seeds. Settling velocities of 0.33, 0.34, and 0.32 cm/min were recorded for dosages of 2000, 4000, and 6000 mg/l, respectively. The control sludge sample recorded a settling velocity of 0.32 cm/min.

Previous studies also reported the high efficiency of bioactive extraction using NaCl. A study by Okuda et al. showed that the bioactive extraction using salts improved the coagulation efficiency due to the increase in the dissociation of MO proteins, which then led to the increase of the protein solubility in the salt medium. The study also showed that there was no difference in the coagulation efficiency through the extraction of bioactive compounds using NaCl, KNO_3, KCl, and $NaNO_3$ [14].

The sludge volume index (SVI) is an indicator for characterization of settled sludge. The lower SVI value indicates better settling efficiency. The lowest SVI value of 63.39 ml/g was obtained using MO salt extraction at 2000 mg/l, as shown in Fig. 2. Unexpectedly, the highest SVI value of 143.34 ml/g was also for MO salt extraction at a 4000 mg/l dosage. The defatted MO seeds recorded the lowest SVI value at a dosage of 4000 mg/l. An SVI range of 50-150 ml/g indicates good sludge-sedimentation properties. The SVI values higher than 150 ml/g indicate that the sludge sedimentation is poor and that there is the possibility of bulking problems [15]. However, the obtained SVI values for all MO forms were in the normal range.

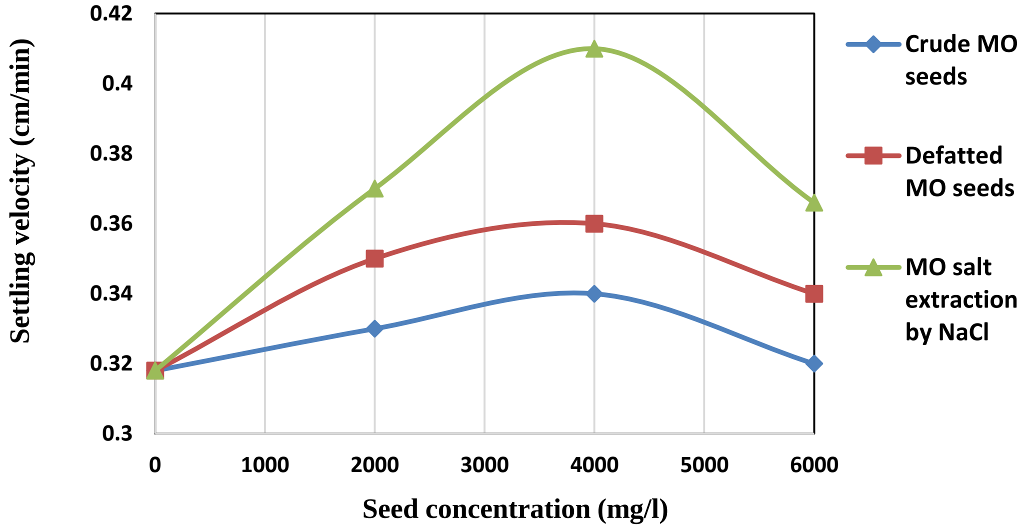

Fig. 1: Different forms of MO seeds and their settling velocities at different dosages.

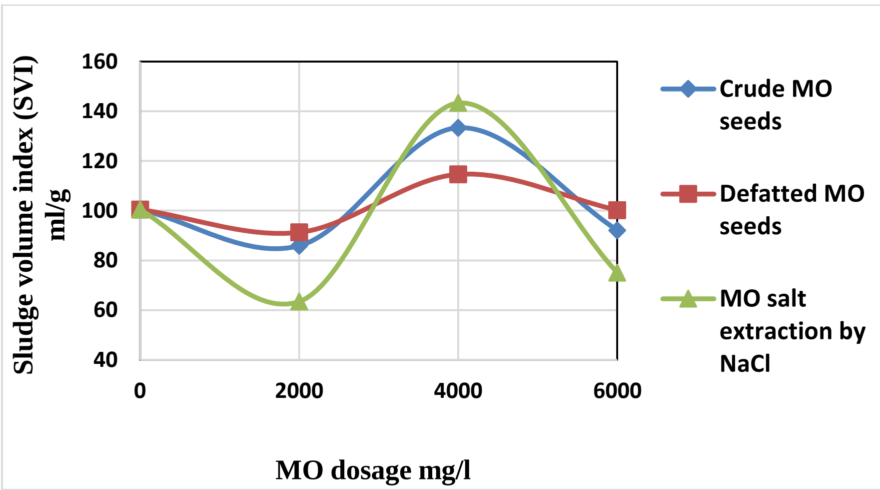

Fig. 2: Different MO coagulant-forms and their SVI values at different dosages.

3.2 Optimization and Statistical Analysis

The results of process condition optimization are listed in Table 2. Regression analysis was used to predict the optimal settling velocity using a third order factorial model. The predicted settling velocity was determined through regression model equation developed by Design-Expert software v10. From the experimental results, the highest settling velocity was 0.32 cm/min for experiment number 7 under process conditions of 3250 mg/l dosage, 17.5 minutes of contact time at 100 rpm of mixing speed. The 2D and 3D contour plots of the response surface are the graphical representation of the regression equation used to determine the optimum values of the variables within the ranges considered. The two plots were presented in Fig. 3 and Fig. 4. Each contour curve represents an infinitive number of combinations of the two test variables.

Table 2: Experimental design for process conditions optimization

std	Run	Factor 1 A: concentration of seed (mg/l)	Factor 2 B: contact time (min)	Factor 3 C: mixing speed (rpm)	Response Settling velocity (cm/min)	Predicted Settling velocity (cm/min)
14	1	3250	17.5	150	0.31	0.31
12	2	3250	30	100	0.3	0.29
6	3	6000	5	150	0.19	0.20
15	4	3250	17.5	100	0.3	0.31
1	5	500	5	50	0.19	0.21
10	6	6000	17.5	100	0.2	0.22
20	7	3250	17.5	100	0.32	0.30
13	8	3250	17.5	50	0.28	0.31
17	9	3250	17.5	100	0.31	0.31
4	10	6000	30	50	0.28	0.28
9	11	500	17.5	100	0.23	0.22
18	12	3250	17.5	100	0.3	0.31
19	13	3250	17.5	100	0.3	0.31
5	14	500	5	150	0.25	0.25
2	15	6000	5	50	0.22	0.22
3	16	500	30	50	0.23	0.22
7	17	500	30	150	0.24	0.25
11	18	3250	5	100	0.3	0.28
8	19	6000	30	150	0.27	0.28
16	20	3250	17.5	100	0.3	0.31

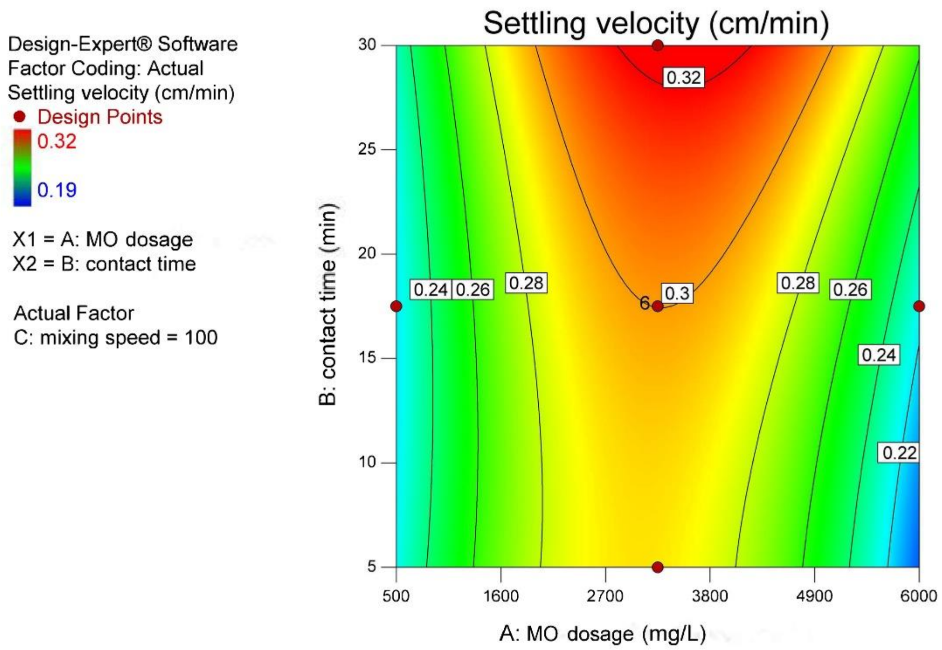

Fig. 3: The 2D contour plot of the interaction of dosage with contact time at mixing speed of 100 rpm.

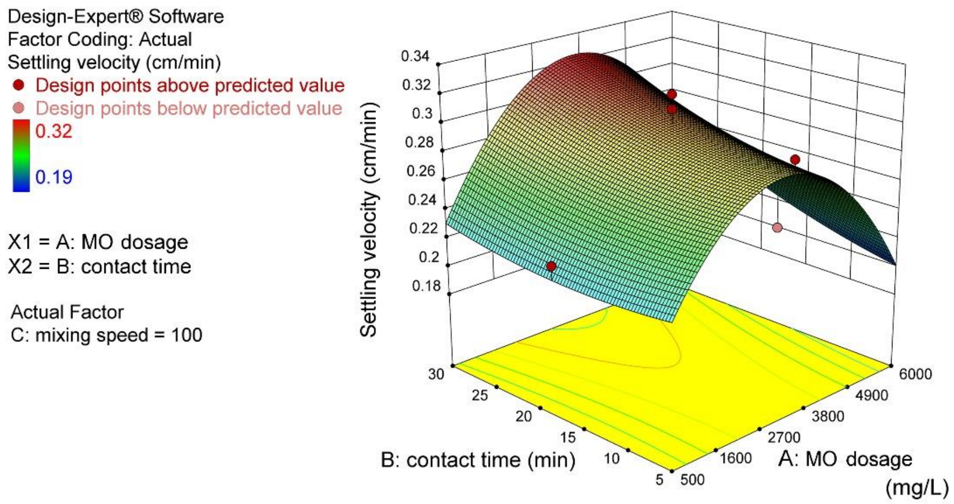

Fig. 4: The 3D contour plot of the interaction of dosage with contact time at mixing speed of 100 rpm.

Figure 5 shows the regression value of actual and predicted settling velocity. Using ANOVA analysis, the Model F-value of 11.65 implies the model is significant. There is only a 0.03% chance that an F-value this large could occur due to noise. Values of "Prob > F" lower than 0.05 indicate that the model terms are significant. Due to noise, the "Lack of Fit F-value" of 7.88 implies the Lack of Fit is significant. There is only a 2.04% chance that a "Lack of Fit F-value" this large could occur. The "Predicted R-Squared" of 0.1695 is not as close to the "Adj R-Squared" of 0.8345. The Final Equation, in terms of the actual factors, is:

$$\text{Settling velocity} = 0.15646 + 7.05342\times10^{-5} A - 9.69091\times10^{-4} B + 3.68182\times10^{-4} C + 4\times10^{-7} AB - 1\times10^{-7} AC - 6\times10^{-6} BC - 1.0278\times10^{-8} A^2 + 4.65455\times10^{-5} B^2 + 9.09091\times10^{-7} C^2 \quad (2)$$

Where *A* is the MO dosage, *B* is the contact time, and *C* is the mixing speed.

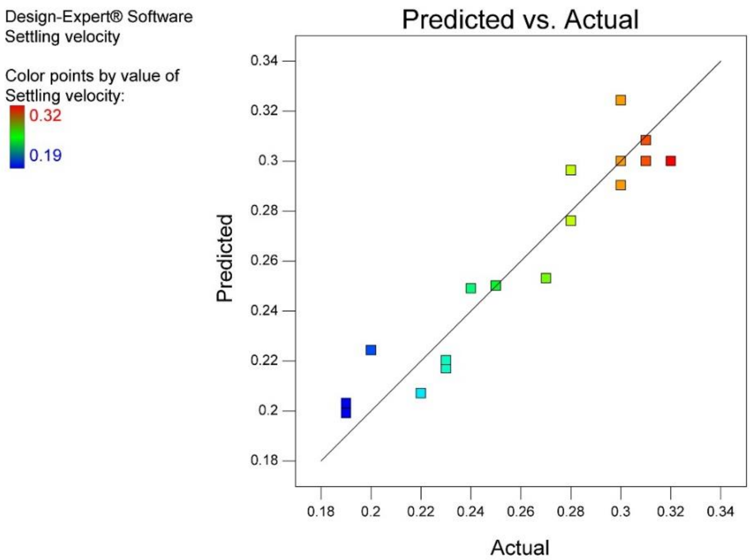

Fig. 5: Regression value of actual and predicted settling velocity.

Table 3: Regression value and standard deviation

Std. Dev.	0.018	R-Squared	0.9129
Mean	0.27	Adj R-Squared	0.8345
C.V. %	6.63	Pred R-Squared	0.1695
PRESS	0.030	Adeq Precision	10.045

From the optimization step, multiple solutions were obtained with a desirability of 1. The first five solutions were listed in Table 4.

Table 4: Optimization solutions

No.	dosage of MO seeds (mg/l)	contact time (min)	mixing speed (rpm)	Settling velocity (cm/min)	Desirability	
1	3245.993	28.794	101.906	0.321	1.000	Selected
2	3641.710	27.892	52.399	0.321	1.000	
3	4148.779	29.743	149.907	0.321	1.000	
4	3299.708	26.667	146.186	0.321	1.000	
5	3474.074	29.444	125.185	0.325	1.000	

4. CONCLUSION

Different coagulant-forms derived from MO seeds were investigated as sludge conditioners. The MO coagulant extracted by NaCl showed the highest settling velocity of 0.41cm/min and lowest SVI of 63.39 ml/g, compared to the crude and defatted MO seeds. Due to the soluble proteins of the MO seed, salt extraction can improve the coagulation efficiency through the salt-in mechanism. The coagulant derived from salt extraction was used in the optimization stage and the optimum process conditions were 3246 mg/l, 29 min, 102 rpm for dosage, contact time, and mixing speed, respectively, at an optimum V_s of 0.321 cm/min. All the coagulant forms showed a significant improvement compared to the control sample. *Moringa oleifera* can be used as a natural conditioner, and it has the opportunity to be used as a single coagulant or combined with chemical conditioners.

ACKNOWLEDGEMENT

This work was partially supported by International Islamic University Malaysia, (PRGS 15-011-0021) from Ministry of Higher Education of Malaysia.

REFERENCES

[1] Henderson JP, Heinecke JW. (2003) Waste water treatment and reuse in the mediterranean region. The Handbook of Environmental Chemistry, D. Barcelo and M. Petrovic, Eds Springer, 3:201-214.

[2] Verlicchi P, Zambello E. (2015) Pharmaceuticals and personal care products in untreated and treated sewage sludge: occurrence and environmental risk in the case of application on soil - A critical review. Sci. Total Environ., 538:750-767.

[3] Abdul Hamid SH, Lananan F, Khatoon H, Jusoh A, Endut A. (2016) A study of coagulating protein of *Moringa oleifera* in microalgae bio-flocculation. Int. Biodeterior. Biodegrad, 113:310-317.

[4] LeBlanc RJ, Matthews P, Richard RP. (2008) Global atlas of excreta, wastewater sludge, and biosolids management: moving forward the sustainable and welcome uses of a global

resource.

[5] Bichi MH. (2013) A review of the applications of *Moringa oleifera* seeds extract in water treatment. Civ. Environ. Res., 3(8):1-10.

[6] Abdulazeez QM, Jami MS, Alam MZ. (2016) Effective sludge dewatering using *Moringa oleifera* seed extract combined with aluminium sulfate. J. Eng. Appl. Sci., 11(1):372-381.

[7] Gold M, Dayer P, Faye MCAS, Clair G, Seck A, Niang S, Morgenroth E, Strande L. (2016) Locally produced natural conditioners for dewatering of faecal sludge. Environ. Technol., 37(21):2802-2814.

[8] Kansal SK, Kumari A. (2014) Potential of *M. oleifera* for the treatment of water and wastewater. Chem. Rev., 114(9):4993-5010.

[9] Hussain J, Jami MS, Muyibi SA. (2012) Enhancement of dewatering properties of kaolin suspension by using cationic polyacrylamide (PAM-C) flocculant and surfactants. Aust. J. Basic Appl. Sci., 6(1):70-73.

[10] Abdulazeez QM, Jami MS, Alam MZ, Iwata M. (2015) Analysis of the efficiency of sludge dewatering using *Moringa oleifera* as natural phytocoagulant. Int. J. Res. Chem. Metall. Civ. Eng., 2(2):111-117.

[11] Ali EN, Muyibi SA, Salleh HM, Alam MZ, Salleh MRM. (2010) Production of natural coagulant from *Moringa oleifera* seed for application in treatment of low turbidity water. J. Water Resour. Prot., 2(3):259-266.

[12] Muyibi SA, Alfugara AMS. (2003) Treatment of surface water with *Moringa oleifera* seed extract and alum- A comparative study using a pilot scale water treatment plant. Intern. J. Environ. Stud., 60(6):617-626.

[13] Yusoff MM, Gordon MH, Ezeh O, Niranjan K. (2016) Aqueous enzymatic extraction of *Moringa oleifera* oil. Food Chem., 211:400-408.

[14] Okuda T, Baes AU, Nishijima W, Okada M. (1999) Improvement of extraction method of coagulation active components from *Moringa oleifera* seed. Water Resour., 33(15):3373-3378.

[15] Turovskiĭ IS, Mathai PK. (2006) Wastewater sludge processing. New Jersey: John Wiley & Sons, Inc.