

Analysis of the Efficiency of Sludge Dewatering Using *Moringa oleifera* as Natural Phytocoagulant

Qabas Marwan Abdulazeez¹, Mohammed Saedi Jami^{*1}, Md. Zahangir Alam¹, and Masashi Iwata²

Abstract— In order to evaluate the efficiency of sludge dewatering, specific parameters such as settling velocity (V_s) and sludge volume index (SVI) have to be measured. However, problems arise when these parameters are used for the evaluation of efficiency using *M. oleifera* as natural phytocoagulant or sludge conditioner. Using statistical optimization, it was found that despite good results of V_s and SVI, the concentration of residual suspended solids in supernatant liquid or turbidity was very high. Thus, turbidity of supernatant liquid was selected as a criteria to evaluate the efficiency of dewatering process. In this research, two optimization steps were run under two factors for each optimization; i.e., mixing time and concentration of *M. oleifera* seeds extract with NaCl (1 M). The range of these factors was (100 – 1000 mg/L) for *M. oleifera* seeds extract concentration, and (5 – 30 min) for mixing time. In the first optimization, V_s and turbidity were used as responses. While in the second optimization SVI and turbidity were used as response parameters. Both optimization steps were run under (pH = 7), mixing speed (120 rpm) for the first minute, and (40 rpm) during the rest period of experiment. By using Design-Expert software v9, for the first optimization of settling velocity, the optimum V_s was found to be (1 cm/min) and the turbidity of the supernatant was (350.7 NTU). Whereas in the optimization of the sludge volume index (SVI), the optimum value was (24.7 mL/g), corresponding turbidity value of suspended solids in supernatant liquid was (341.5 NTU). Since the turbidity was very high, second optimization by redesigning the factors was conducted resulting in optimum values of dosage of (462.8 mg/L), mixing time of (13.4 min), turbidity of (67.2 NTU) (further (80.2 %) reduction compared to the (350.7 NTU) of the first optimization) and settling velocity of (0.93 cm/min) were obtained. For the optimization of SVI, dosage of (447.5 mg/L) and mixing time of (8.3 min) gave (33.5 mL/g) of SVI with (67.2 NTU). These optimized dewatering parameters can be used to improve the efficiency of sludge dewatering.

Keywords—*Moringa oleifera*, sludge dewatering, settling velocity, sludge volume index, phytocoagulant, environmentally friendly.

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I. INTRODUCTION

SEWAGE sludge is produced from different wastewater treatment processes, such as physical, chemical and biological treatment methods. Generally, the main component of sludge is water, solids represent only (0.25 to 12%) from sewage sludge weight [1]. Wastewater treatment produces high quantities of sludge every day. For wastewater treatment plant with flow of (0.5 m³/s), the mass of daily dry solids produced from sludge is (2,035.58 kg/d) [2]. The production and handling processes of sludge make it very expensive with a cost reaching up to half of the total wastewater treatment cost [3]. A process called sludge dewatering is responsible for water separation from solids. It can be done through physical, chemical, biological and also natural methods. The physical (mechanical) methods such as belt press are very expensive due to equipment cost and energy consumption [4]. The chemical methods which include chemical conditioners such as polyaluminium chloride (PAC) and aluminum sulphate (alum) have a negative impacts on human health and environment [5]. For biological methods, the use of some microbial cultures can help sludge dewatering process, such as *Acidithiobacillus ferrooxidans* culture [6].

Recently, many scientists are interested with the natural dewatering methods to protect human life and preserve the environment. The most common natural conditioners are plant-based coagulants. Some parts in specific plants have the ability to coagulate the solids and forming flocs, such as seeds powder of *Moringa oleifera*. This seeds have cationic polyelectrolyte which flocculate particles with negative charge by providing a strong adsorption, led to flocs formation then sedimentation [7]. *Abelmoschus esculentus* (okra) also has been used in sludge dewatering as a natural coagulant. Under the optimum conditions, more than (98%) of suspended solids were removed and (68%) of water was recovered during sludge dewatering [8]. Another natural conditioner is *Opuntia ficus Indica* (prickly pear cactus), the juice of this cactus was used to dewater sewage sludge using (0.4 g/kg) as optimum dosage, with response SRF = (0.13 × 1012 m/kg) [9].

Sludge dewatering evaluation can be measured with many parameters, such as: settling velocity (V_s), sludge volume

index (SVI), specific resistance to filtration (SRF), capillary suction time (CST), bound water content, and dry solids content. When natural coagulants are evaluated with V_s and SVI, some mistakes can occur. Because of natural coagulants are not strong as chemical coagulants, the residual suspended solids in supernatant liquid will be different in concentration, which led to error readings in V_s and SVI values.

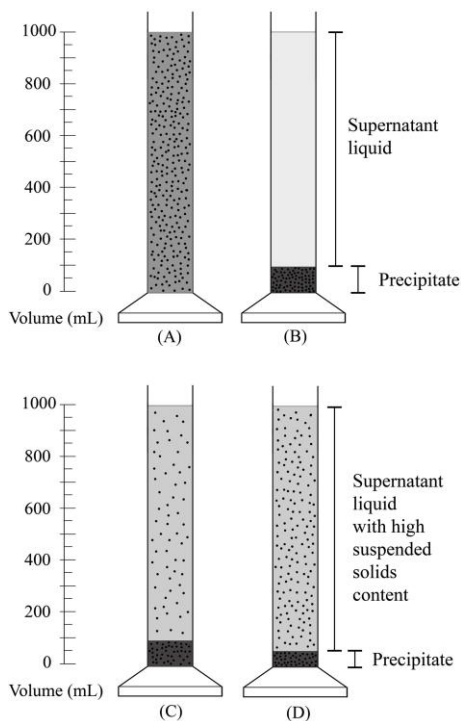


Fig. 1

- (A): Sludge sample without any coagulant
 (B): An efficient sludge dewatering process with very low supernatant turbidity
 (C): Sludge dewatering process with moderate-high supernatant turbidity
 (D): Inefficient sludge dewatering process with very high supernatant turbidity

In Fig. 1, (A) and (B) represent raw sludge sample (standard) and very efficient sludge dewatering process, respectively. (C) represents sludge dewatering with moderate-high supernatant turbidity at the upper part of the cylinder. And (D) illustrates sludge dewatering process with very high supernatant turbidity. In (C) and (D), settling velocity V_s is very high and sludge volume index SVI is very low, means that both parameters represent an effective dewatering process. In fact, both of these parameters have to be classified as inefficient dewatering process, due to the high suspended solids concentration in the supernatant. This situation will cause problem in dewatering evaluation through the use of V_s and SVI as efficiency parameters. This research will explain the relation between these parameters with dewatering efficiency through using *Moringa oleifera* as natural coagulant, and illustrate the possible solution for this problem.

Because of the low cost, availability, and eco-friendly of *M. oleifera* plant, it is possible to be used alone or side by side with chemical coagulants [1]. *M. oleifera* is innocuous plant, grows naturally or manually in tropical areas such as Asia and Africa. It was investigated for sludge dewatering as natural conditioner. A study by [10] showed the dewaterability of sewage sludge using *M. oleifera* seeds with $SRF = (1.22 \times 10^{11} \text{ m/kg})$ and $CST = (4.5 \text{ s})$ under optimum conditions.

II. MATERIALS AND METHODS

A. Materials

1) *M. oleifera* seeds, sludge sample and chemicals

M. oleifera seeds were collected from Serdang, Selangor – Malaysia, dried and kept inside the pods for 4 months. Kaolin suspension (R&M Chemicals - UK) was used as sludge sample. Hexane solvent (n-Hexane – 99%, SYSTEM) was used for *M. oleifera* oil extraction, and sodium chloride NaCl (Bendosen) was used to extract active component from *M. oleifera* seeds. Sludge pH values were adjusted using hydrochloric acid (HCl) and sodium hydroxide (NaOH). To get the exact pH, three values of molarity were used: 3 M, 1 M and 0.3 M.

2) Equipment

Commercial grinder, sieve with (212 μm) pore size (Retsch), vacuum filtration apparatus (DDA-V111-ED, USA) with filter papers (Whatman, Qualitative 1) to filter *M. oleifera* solution after salt extraction, soxhlet extraction apparatus for *M. oleifera* seeds oil extraction, cylinder beakers (HmbG, 1 L), laboratory weighing balance (TOLEDO, B204-S), pH meter (SARTORIUS), turbidimeter (Tn-100, Eutech), stopwatch, magnetic stirrer, long pipette, and jar test apparatus (Flocculator sw6, UK) were used.

B. Methods

1) Preparation of sludge sample

Sludge sample was prepared by adding (5 g) of kaolin powder to (1 L) of distilled water (5% w/v), followed by rapid mixing at (200 rpm) for (10 min) using jar test apparatus [11].

2) Preparation of *M. oleifera* seeds extract

After selecting good seeds of *M. oleifera*, pods were removed, seeds were grinded to fine powder and sieved using (212 μm) pore size. Then, (10 g) of this powder was defatted using (170 mL) of hexane solvent. Oil extraction operation took about (90 min) using soxhlet extraction apparatus [12]. After oil extraction, (5 g) of defatted seeds powder were mixed with (1 L) of NaCl solution (1 M) using magnetic stirrer, mixing duration continued for one hour to ensure the total extraction of active components from *M. oleifera* seeds [10]. Finally, the solution was filtered using vacuum filtration apparatus with filtration paper to remove all *M. oleifera* seeds particles and produce clear solution.

C. Design of experiment and statistical analysis

1) One-Factor-At-a-Time (OFAT)

To view the relation between V_s , SVI, and dewatering efficiency using 3D contour plot, two factors for each optimization were set. The first chosen factor was the dosage of seeds extract, the other factor was selected between three factors using OFAT. The three factors are: pH, mixing speed, and mixing time, and the selection was set depend on the most effective factor on SVI values.

2) Design of experiment (DOE)

Depending on OFAT values, mixing time was set for optimization design with the dosage of *M. oleifera* seeds extract. The responses were: V_s , SVI, and supernatant turbidity. This turbidity was set as response to determine sludge dewatering efficiency level. For design of experiment, two optimization were run with concentration (dosage) of *M. oleifera* seeds extract and mixing time as factors. Table I Shows the range of levels for parameters used in jar test for both optimization experiments. The first optimization included V_s and turbidity as responses, as shown in Table II. The second optimization was included SVI and turbidity as responses, as shown in Table III. For other factors, pH was fixed at 7.00 and mixing speed was fixed at (125 rpm) for the first minute, followed by (40 rpm) during the rest period of experiment. The two experiments were designed using Design-Expert software v9 (Stat-Ease Inc.).

TABLE I
RANGE OF LEVELS FOR PARAMETERS USED IN JAR TEST

Parameters	Range of levels		
	-1	0	1
Dosage of M.O seeds extract (mg/L)	100	550	1000
Mixing time (min)	5	17.5	30
pH	7.00		
Mixing speed (rpm)	125 (1 st min) - 40		

TABLE II
THE FIRST OPTIMIZATION DESIGN.

Std	Run	Factor 1 A: M.O conc. (mg/L)	Factor 2 B: Mixing time (min)	Response 1 Turbidity (NTU)	Response 2 Settling velocity (cm/min)
6	1	1000	17.5		
10	2	550	17.5		
8	3	550	30		
1	4	100	5		
7	5	550	5		
12	6	550	17.5		
11	7	550	17.5		
13	8	550	17.5		
9	9	550	17.5		
3	10	100	30		
2	11	1000	5		
5	12	100	17.5		
4	13	1000	30		

TABLE III
THE SECOND OPTIMIZATION DESIGN

St d	Run	Factor 1 A: M.O conc. (mg/L)	Factor 2 B: Mixing time (min)	Response 1 Turbidity (NTU)	Response 2 SVI (mL/g)
6	1	1000	17.5		
10	2	550	17.5		
8	3	550	30		
1	4	100	5		
7	5	550	5		
12	6	550	17.5		
11	7	550	17.5		
13	8	550	17.5		
9	9	550	17.5		
3	10	100	30		
2	11	1000	5		
5	12	100	17.5		
4	13	1000	30		

3) Analytical methods

a) Settling velocity (V_s) measurement

A sticker ruler was fixed on the upper surface of the cylinder with (34 cm) in height (same height for the cylinders with 1 L capacity). Using stopwatch, the height of the upper sludge dewatering surface was recorded every two minutes. This process continued for (30 min). After that, settling velocity (cm/min) was recorded by plotting the sludge surface height in cm (axis y) versus the time in minutes (axis x). Settling velocity is equal to the slope value of axis y and axis x, as shown in Fig. 2 [13]. Data and slope were calculated using Microsoft office excel (2013).

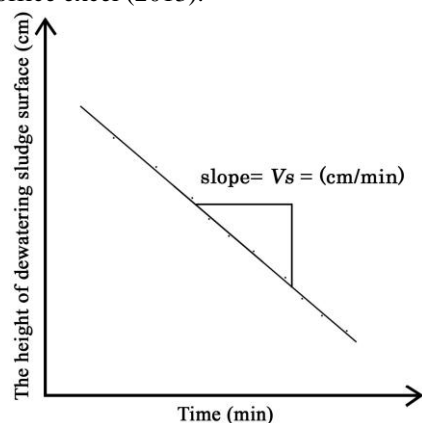


Fig. 2 Scheme for settling velocity calculation

b) Sludge volume index (SVI) measurement

According to the Standard Methods [13], sludge volume index (SVI) is “the volume in milliliters occupied by (1 g) of a suspension after (30 min) settling”.

$$\text{SVI (mL/g)} = \frac{\text{Settled sludge volume (mL/L)} \times 1000}{\text{suspended solids (mg/L)}} \quad (1)$$

c) Turbidity measurement

Supernatant turbidity was measured using turbidimeter (Tn-100, Eutech - Singapore). The supernatant samples were withdrew using a long pipette at depth (10 cm) from the

surface of the supernatant liquid. Each sample was measured three times, then the average of these values was calculated to reduce error rate.

d) Data analysis

From analysis of variance (ANOVA) test and probability value (P -value), the significance of coefficients was analyzed. If a model has P -value less than 0.05, that model is considered significant. For optimizations, the optimum values were obtained by analyzing the 3D response plot for V_s , SVI, and turbidity.

III. RESULTS AND DISCUSSION

A. OFAT Results

OFAT was applied on three factors to select the factor with high effectiveness on SVI values. Factors are: mixing speed, pH, and mixing time. Between these factors, mixing time was selected, it showed highest differential efficiency on SVI values, as shown in Fig. 2. Mixing time range was selected from (5 – 30 min). Although SVI values were low below (5 min) and above (30 min), it had very high supernatant turbidities, as shown in Fig. 3. Thus, the range was set only between (5 – 30 min). For mixing speed, it almost has no effect on SVI values, as shown in Fig. 4. pH has shown some change in SVI values, but comparing with mixing speed, the last factor has higher effectiveness.

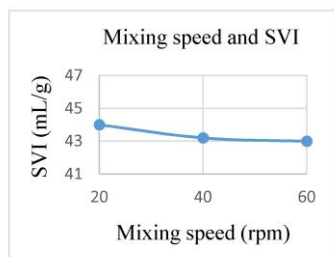


Fig. 3 OFAT, mixing speed with SVI

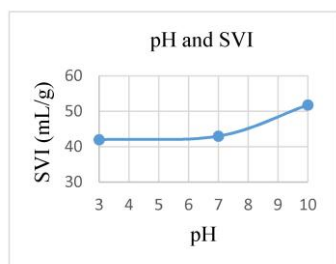


Fig. 4 OFAT, pH with SVI

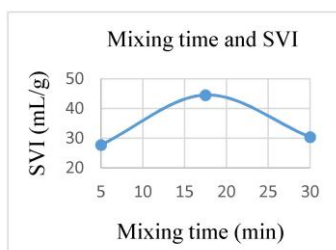


Fig. 5 OFAT, mixing time with SVI

B. Settling velocity (V_s) optimization

According to ANOVA analysis, factors: A, B, AB, A^2 , and B^2 are significant because P -value is less than 0.05 ($\text{Prob} > F$). The quadratic model is also significant because R^2 value is 0.9849. The predicted R^2 which is (0.8968) is in reasonable agreement with adjusted R^2 which is (0.9742). The lack of fit (F -value) of (0.1042) implies the lack of fit is not significant. Final equation in terms of coded factors is:

$$\text{Settling velocity } (V_s) = 0.93 - 0.17 * A + 0.05 * B - 0.018 * AB - 0.19 * A^2 + 0.01 * B^2 \quad (2)$$

TABLE IV
ANOVA FOR RESPONSE SURFACE QUADRATIC MODEL – SETTLING VELOCITY (V_s)

Analysis of variance table [Partial sum of squares - Type III]					
Source	Sum of Squares	d f	Mean Square	F Value	p-value Prob > F
Model	0.30	5	0.060	91.46	< 0.0001
A-concentration	0.17	1	0.17	256.51	< 0.0001
B-mixing time	0.015	1	0.015	22.84	0.0020
AB	1.260E-003	1	1.260E-003	1.93	0.2071
A^2	0.10	1	0.10	156.41	< 0.0001
B^2	2.926E-004	1	2.926E-004	0.45	0.5245
Residual	4.566E-003	7	6.523E-004		
Lack of Fit	3.440E-003	3	1.147E-003	4.07	0.1042
Pure Error	1.126E-003	4	2.815E-004		
Cor Total	0.30	12			

not significant

TABLE V
EXPERIMENTAL DESIGN FOR PROCESS OPTIMIZATION OF V_s

Std	Run	Factor 1 A: M.O conc. (mg/L)	Factor 2 B: Mixing time (min)	Response 1 Turbidity (NTU)	Response 2 Settling velocity (cm/min)
6	1	1000	17.5	83.1	0.54
10	2	550	17.5	77.6	0.961
8	3	550	30	220	0.946
1	4	100	5	125	0.835
7	5	550	5	97	0.892
12	6	550	17.5	68	0.92
11	7	550	17.5	68.9	0.928
13	8	550	17.5	67.2	0.93
9	9	550	17.5	69.6	0.921
3	10	100	30	495.1	0.993
2	11	1000	5	195	0.546
5	12	100	17.5	226	0.893
4	13	1000	30	131	0.633

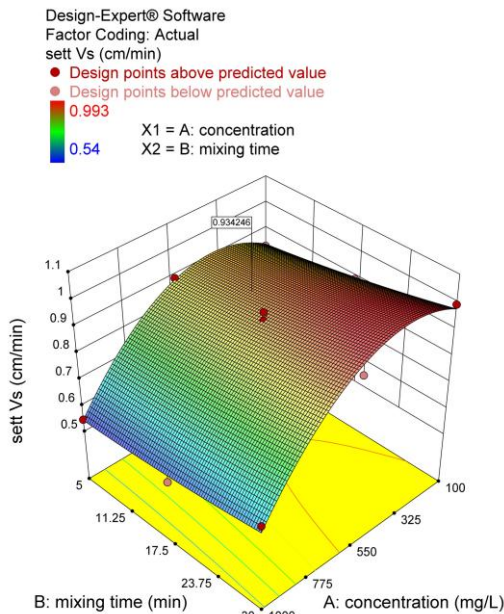


Fig. 6 3D contour plot of the interaction of concentration and mixing time with Vs as response

C. Sludge volume index (SVI) optimization

ANOVA analysis shows that factors of A, B, AB, A², and B² are significant because *P*-value of the model is less than 0.05. The quadratic model is also significant because R² value is 0.9992. The predicted R² which is 0.9947 is in reasonable agreement with adjusted R² which is 0.9987. The lack of fit (*F*-value) of 0.1462 implies the lack of fit is not significant. Final equation in terms of coded factors is:

$$\text{Sludge volume index (SVI)} = 45.49 + 37.03 * A - 0.65 * B + 4.4 * AB + 36.15 * A^2 - 12.4 * B^2 \quad (3)$$

TABLE VI
ANOVA FOR RESPONSE SURFACE QUADRATIC MODEL – SLUDGE VOLUME INDEX (SVI)

Analysis of variance table [Partial sum of squares - Type III]						
Source	Sum of Squares	d f	Mean Square	F Value	p-value Prob > F	signifi cant
Model	11923.81	5	2384.76	1779.63	< 0.0001	signifi cant
A- concentration	8228.81	1	8228.81	6140.75	< 0.0001	
B- mixing time	2.53	1	2.53	1.89	0.2114	
AB	77.44	1	77.44	57.79	0.0001	
A ²	3608.98	1	3608.98	2693.20	< 0.0001	
B ²	424.79	1	424.79	317.00	< 0.0001	
Residual	9.38	7	1.34			
Lack of Fit	6.61	3	2.20	3.19	0.1462	not signifi cant
Pure Error	2.77	4	0.69			
Cor	11933.1	1				
Total	9	2				

TABLE VII EXPERIMENTAL DESIGN FOR PROCESS OPTIMIZATION OF SVI					
St d	Run	Factor 1 A: M.O conc. (mg/L)	Factor 2 B: Mixing time (min)	Response 1 Turbidity (NTU)	Response 2 SVI (mL/g)
6	1	1000	17.5	83.1	118.6
10	2	550	17.5	77.6	43.9
8	3	550	30	220	34
1	4	100	5	125	37
7	5	550	5	97	33.5
12	6	550	17.5	68	45.4
11	7	550	17.5	68.9	45
13	8	550	17.5	67.2	46
9	9	550	17.5	69.6	45.8
3	10	100	30	495.1	26
2	11	1000	5	195	103
5	12	100	17.5	226	46
4	13	1000	30	131	109.6

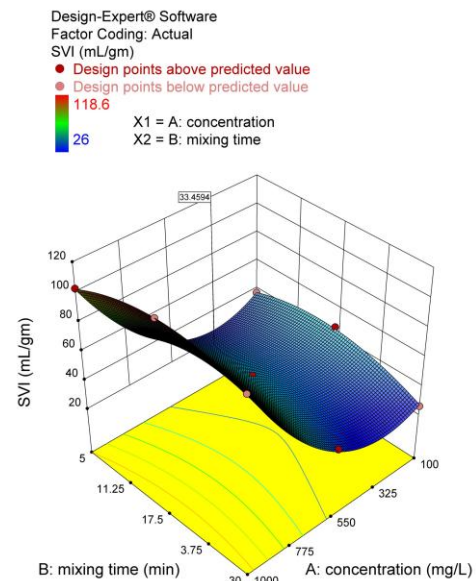


Fig. 7 3D contour plot of the interaction of concentration and mixing time with SVI as response

D. Relation between SVI, Vs and turbidity

Settling velocity (*V_s*) and sludge volume index (SVI) values are linked together. Under specific model conditions, when *V_s* value is optimal (*V_s* value is high), SVI value will also be optimal (SVI value is low), and vice versa. From Table V and Table VII, the best values of *V_s* and SVI were (0.993 cm/min) and (26 mL/g). These values were obtained from the same experiment (no.10) under the same dosage and mixing time. Contrary, the first experiment (no.1) has the lowest efficiency values of *V_s* and SVI, which are (0.54 cm/min) for *V_s* and (118.6 mL/g) for SVI. The reason of these similarities can be understood from sludge volume index equation. From Eq. 1, settled sludge volume (mL/L) is depending on the velocity of sedimenting particles which is *V_s*. For example, when settling velocity value is high (means that *V_s* is effective), the sedimentation process will be fast, which lead to small volume of settled sludge (mL/L). The small amount of settled sludge will give small SVI value (means that SVI is effective).

For supernatant turbidity, results showed that turbidity range values was from (67.2 – 495.1 NTU). These values were measured after (30 min) form dewatering process starting. Contour plot in Fig. 8 represents supernatant turbidity values for both V_s and SVI optimizations. ANOVA for turbidity showed high values of R^2 with 0.9976 and adjusting R^2 with 0.9802. The model was significant and lack of fit of 0.0537 was not significant with only 5.37% chance that F-value this large could occur due to this noise.

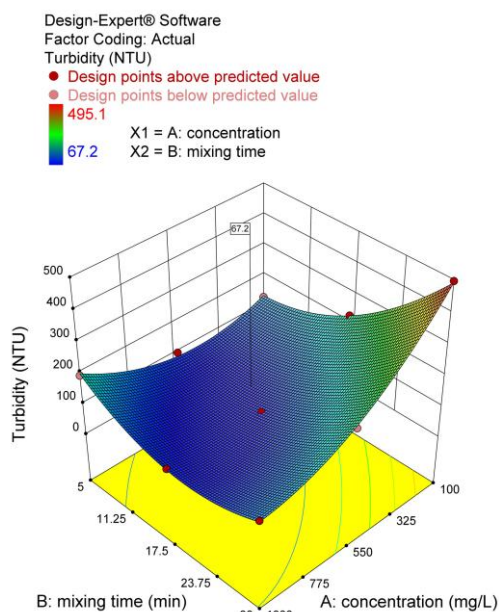


Fig. 8 3D contour plot of the interaction of concentration and mixing time with turbidity as response

The optimum V_s and SVI values have high supernatant turbidity. From fig. 6, the optimum V_s is (1 cm/min) and the turbidity of the supernatant is found to be (350.7 NTU). Whereas SVI optimum value is (24.7 mL/g) (as shown in Fig. 7), corresponding turbidity value of suspended solids in supernatant liquid was (341.5 NTU). At optimum values of V_s and SVI, sludge dewatering cannot be considered as effective due to the high supernatant turbidities. This high concentration of suspended solids in supernatant require another treatment, by which the cost for sludge treatment will increase.

To get optimum V_s and SVI values with low supernatant turbidity, a turbidity range of (67.2 – 70 NTU) was set during optimization. With this range, only values of V_s and SVI with supernatant turbidity between (67.2 – 70 NTU) will be obtained. During optimization, the goal of V_s was set to maximum and SVI was set to minimum, and both V_s and SVI was set under turbidity response with range of (67.2 – 70 NTU). Fig. 9 shows holographic contour which represents the interaction of settling velocity with limited range of turbidity. The optimization solutions of V_s under turbidity range was listed in Table VIII. The optimum values of dosage of (462.8 mg/L) and mixing time of (13.4 min) gave turbidity of (67.2 NTU) and settling velocity of (0.93 cm/min). Through using these values of dosage and mixing time, the efficiency of

settling velocity is high with minimum value of supernatant turbidity. At these values, the moderate turbidity of supernatant can be treated with lower cost comparing with the cost of the high supernatant turbidity.

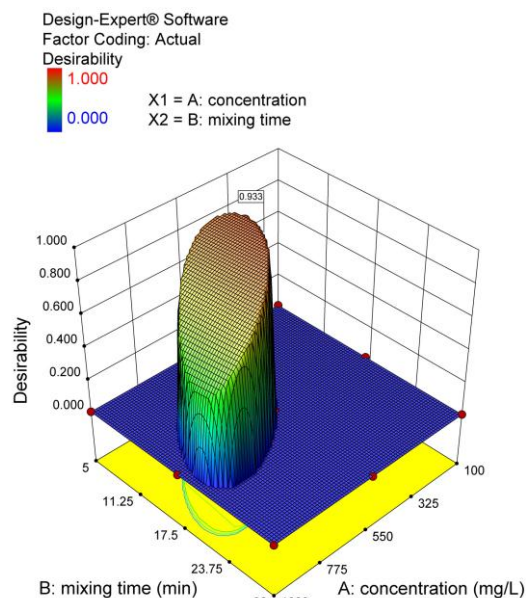


Fig. 9 Settling velocity with limited range of supernatant turbidity

For SVI optimization under the same range of supernatant turbidity, the dosage of (447.5 mg/L) and mixing time of (8.3 min) gave SVI of (33.4 mL/g) and moderate turbidity of (67.2 NTU). The holographic contour which represents the interaction of SVI with limited range of turbidity was shown in Fig. 10. The optimization solutions of SVI under turbidity range was listed in Table IX.

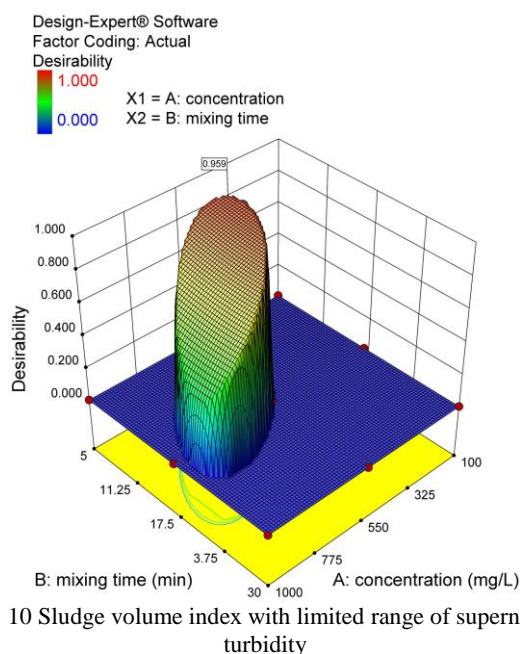


Fig. 10 Sludge volume index with limited range of supernatant turbidity

TABLE VIII
OPTIMIZATION SOLUTIONS OF V_s WITH LIMITED RANGE OF SUPERNATANT TURBIDITY

no	Dosage (mg/L)	Mixing time (min)	Turbidity (NTU)	Settling velocity (cm/min)	Desirability
1	462.834	13.435	67.200	0.934	0.933
2	460.457	13.310	67.200	0.934	0.933
3	465.756	13.584	67.200	0.934	0.933
4	458.665	13.214	67.200	0.934	0.933
5	469.134	13.750	67.200	0.934	0.933
6	471.277	13.852	67.199	0.934	0.933
7	452.104	12.842	67.200	0.934	0.933
8	476.185	14.080	67.199	0.934	0.932
9	436.304	11.728	67.199	0.932	0.931
10	432.467	11.360	67.199	0.932	0.930

TABLE IX
OPTIMIZATION SOLUTIONS OF SVI WITH LIMITED RANGE OF SUPERNATANT TURBIDITY

n	Dosage (mg/L)	Mixing time (min)	Turbidity (NTU)	SVI (mL/g)	Desirability
1	447.528	8.326	67.200	33.459	0.959
2	444.650	8.415	67.200	33.467	0.959
3	452.056	8.206	67.200	33.477	0.959
4	441.288	8.535	67.200	33.499	0.959
5	438.980	8.631	67.199	33.538	0.958
6	461.124	8.020	67.200	33.600	0.958
7	434.604	8.853	67.200	33.662	0.958
8	428.655	9.337	67.200	34.039	0.956

IV. CONCLUSION

Natural phytocoagulants such as *M. oleifera*, being safe and environmental friendly, can be used for sludge dewatering even though their efficiency is not as strong as chemical coagulants. After sludge dewatering for a certain period, the supernatant turbidity has to be monitored to observe the progress of dewatering. This is necessary because as the turbidity level increases, it will result in higher cost for subsequent treatment of the supernatant. In case of *M. oleifera*, the optimum settling velocity (V_s) was found to be (1 cm/min) and the turbidity of the supernatant was measured to be (350.7 NTU). By re-designing the factors of the model (dosage of phytocoagulant and mixing time), the supernatant turbidity decreased up to 80.8% (from 350.7 NTU to 67.2 NTU) while the settling velocity (V_s) decreased from (1 cm/min) to (0.93 cm/min). Re-designing the model was done through avoiding the results with high values of supernatant turbidity, even if the values of SVI and V_s for these results were very good. With this procedure of re-designing the model, the efficiency of sludge dewatering can be considered as high with low supernatant turbidity output.

For sludge volume index (SVI), the re-designing of the model resulted in decreasing the supernatant turbidity up to 80.3% (from 341.5 NTU to 67.2 NTU) with SVI value varying from (24.7 mL/g) to (33.5 mL/g). Optimizing the dosage and mixing time of a given phytocoagulant and monitoring supernatant the turbidity level of the supernatant is a good strategy to reduce the cost of further treatment supernatant

before disposal. Thus, for sludge dewatering process, suitable natural phytocoagulant can be selected with high efficiency and eco-friendly character.

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