



EFFECTIVE SLUDGE DEWATERING USING *Moringa oleifera* SEED EXTRACT COMBINED WITH ALUMINIUM SULFATE

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ABSTRACT

This research aims to find an optimum mixture ratio that consists of natural and chemical coagulants, and using it for sludge dewatering. *Moringa oleifera* seed extract by NaCl solution (1 M) was used as natural coagulant due to its characteristics such as availability, low cost, biodegradable, and it is environmentally friendly. This natural coagulant was combined with aluminium sulphate $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ (alum) as chemical coagulant. Sludge samples were prepared from kaolin suspension (5% w/v) with distilled water. The purpose of the mixed coagulant is to produce a mixture with low chemical content and high dewatering efficiency, and to involve the natural coagulant in sludge dewatering process. The use of mixed coagulant can achieve less hazardous impacts to human health and environment. Optimization of process conditions (the first optimization) was done using three factors: *M. oleifera* dosage, pH, and mixing time. The second optimization was done to determine the optimum mixture ratio between alum and *M. oleifera* using Quadratic Mixture Model optimization. This optimization (mixture ratio optimization) was run under the same optimum values of dosage, pH, and mixing time obtained from the first optimization to optimize the coagulation efficiency of *M. oleifera* seed extract. The specific resistance to filtration (SRF) was used as response for both optimization steps, and the results were analysed using Analysis of Variance (ANOVA) approach by Design-Expert v9 software. Results showed that the optimum SRF value obtained from the first optimization was $1.1\text{E}+11$ m/kg at dosage of 235.58 mg/L, pH of 6.5, and mixing time of 21.2 min at $R^2 = 95.8\%$. For the second optimization, the optimum SRF value was $0.8\text{E}+11$ m/kg for the ratio of 50:50 for alum and *M. oleifera* seed extract. At this ratio, dewatering efficiency was the same as the efficiency of alum alone ($0.8\text{E}+11$ m/kg). Using mixed coagulant at ratio of 50:50 for alum and *M. oleifera* seed extract, sludge dewatering efficiency can be high with 50% elimination of alum use.

Keywords: *Moringa oleifera*, alum, sludge dewatering, SRF, natural coagulant, environmentally friendly.

1. INTRODUCTION

One of the most costly processes in wastewater treatment plants is sludge treatment, which represents approximately 50% of wastewater treatment cost [1]. Sludge dewatering process is the most effective and economic process. This process responsible for separating solids from liquid by combining solids to each other and forming larger particles called flocs. These flocs can be settled down using gravity or supplied force. Flocs formation can be done using chemical coagulants such as aluminium sulphate (alum) and polyaluminium chloride (PAC). These chemicals have negative impacts on the environment and hazardous effects on human body [2]. Mechanical dewatering such as conventional belt press can run without any chemicals, but these techniques have some limitations especially in developing countries due to the high cost of equipment and energy consumption [3]. The best solution is to use natural coagulants such as *Moringa oleifera* seeds.

M. oleifera (also known as horseradish tree) is a non-toxic plant; it grows naturally in tropical areas in Asia, India, Africa and Latin America [4]. The seeds of this plant have the ability to flocculate any suspended particles with negative charge in water and wastewater bodies. This seeds have cationic polyelectrolyte, which provide strong adsorption with negative particles. Thus,

the particle surface will be neutralized [5]. *M. oleifera* has the potential to be used side by side with chemical coagulants because of its availability, low cost, biodegradable, and environmentally friendly [6].

Currently, the use of combinations consist of natural and chemical coagulants is not widely used in water and wastewater treatment. A recent study [7] showed that more than 90% of concrete wastewater turbidity was removed using a combination of *M. oleifera* with alum at ratio of 20:80 (w/w), respectively. Other study showed the significant effect of water turbidity removal by using a mixed coagulant consist of *M. oleifera* with PAC as coagulant. The optimum dosage for 95.7% of turbidity removal was 30 mg/L with 300 mg/L for PAC and *M. oleifera* [8]. These combinations give chance to minimize the cost and usage of these chemical coagulants, which led to less hazardous effects on humans and environment. In this study, the use of mixed coagulant consist of *M. oleifera* and alum was investigated in sludge dewatering. Design of Experiment (DOE) was used to optimize the process conditions, such as the ratio of the mixed coagulant, dosage, pH and mixing time.



2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Sludge sample, chemicals and *M. oleifera* seeds

Kaolin suspension (R&M Chemicals, UK) was used as synthetic sludge samples. Aluminium sulphate (HmbG) was used as chemical coagulant. *Moringa oleifera* seeds used in this research were imported from India and kept dry inside their pods for about 3 months. Hexane solvent (n-Hexane – 99%, SYSTEM) was used for oil extraction, and sodium chloride NaCl (Bendosen) was used for salt extraction. pH values were calibrated using sodium hydroxide (NaOH) and hydrochloric acid (HCl) with three values of molarity: 3 M, 1 M and 0.3 M to get the exact pH values.

2.1.2. Equipment

Vacuum filtration apparatus (GAST, MODEL DDA-V111-ED, USA) with 90 mm funnel diameter, filter papers (Whatman, Qualitative 1, 90 mm), soxhlet extraction apparatus for oil extraction, cylinder beakers (HmbG, 1 L), laboratory weighing balance (METTLER TOLEDO, B204-S), viscometer (BROOKFIELD), laboratory oven, stopwatch, pH meter (SARTORIUS), magnetic stirrer, jar test apparatus (Stuart flocculator sw6, UK) and sieve with 212 μ m pore size (Retsch).

2.2 Methods

2.2.1 Preparation of sludge sample

Kaolin suspension was used as synthetic sludge sample by adding 5 g of kaolin powder to 1 L distilled water (5% w/v). The mixture was mixed at 200 rpm for about 10 minutes using jar test device [9].

2.2.2 Preparation of *M. oleifera* seed extract

Good seeds of *Moringa oleifera* were selected, pods were removed, and seeds were grinded and sieved with 212 μ m pore size. Then, 10 g of *M. oleifera* powder was defatted by 170 mL of hexane solvent for about 90 minutes using soxhlet extraction apparatus [10]. After drying the defatted seeds powder using oven, 5 g of the

dried powder were mixed with 1 L of NaCl (1 Molar) using magnetic stirrer for about 60 minutes [11]. Finally, the solution was filtered by filtration paper using vacuum filtration apparatus.

2.2.3 Preparation of alum solution

1 g of alum powder was dissolved in 100 mL distilled water. The mixture was mixed using magnetic stirrer for about 10 min. The dosage will be 10 mg of alum for each mL stock solution [8].

2.3 Design of experiment and statistical analysis

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

OFAT was used for the determination of the possible optimum dosage for the three process conditions: dosage of *M. oleifera* seed extract, pH and mixing time. Each factor was investigated at different range of values. The suitable range of these factors was determined depend on SRF values. Mixing speed was fixed at two values: rapid mixing at 125 rpm for the first minute, followed by slow mixing at 40 rpm to the end of the experiment [10].

2.3.2 Design of Experiment

2.3.2.1 First optimization (process conditions opt.)

Depending on OFAT results, the range of the process conditions was determined, as shown in Table-1. The experiments were designed using Response Surface Methods by Design-Expert software v9 (Stat-Ease Inc). Table-2 shows the optimization of process conditions at 20 experiments in total.

Table-1. Range of levels for parameters used in jar test.

Parameters	Range of levels		
	-1	0	1
Conc. of seed extract (mg/L)	100	300	500
pH	4	7	10
Mixing time (min)	5	17.5	30

**Table-2.** Optimization of process conditions (first opt.).

Std	Run	Factor 1 A: dosage (mg/L)	Factor 2 B: pH	Factor 3 C: mixing time (min)	Response 1 SRF (m/kg)
14	1	300	7.00	30.00	
16	2	300	7.00	17.50	
4	3	500	10.00	5.00	
3	4	100	10.00	5.00	
8	5	500	10.00	30.00	
20	6	300	7.00	17.50	
2	7	500	4.00	5.00	
17	8	300	7.00	17.50	
15	9	300	7.00	17.50	
11	10	300	4.00	17.50	
19	11	300	7.00	17.50	
5	12	100	4.00	30.00	
1	13	100	4.00	5.00	
18	14	300	7.00	17.50	
9	15	100	7.00	17.50	
6	16	500	4.00	30.00	
10	17	500	7.00	17.50	
7	18	100	10.00	30.00	
13	19	300	7.00	5.00	
12	20	300	10.00	17.50	

2.3.2.2 Second optimization (mixture ratio opt.)

The mixture ratio optimization was run under pH of 6.5 because it is suitable for both *M. oleifera* and alum [7]. The total dosage of the mixture was 235.58 mg/L and the mixing time was determined to be 21.2 min. The minimum and maximum ratios for each component in the mixture were 10% (23.56 mg/L) and 90% (212.04 mg/L), respectively. Optimization was done using Mixture–Simplex Lattice type by Design-Expert software v9 (Stat-Ease Inc). Table-3 shows the range of levels of each parameter, while Table-4 shows the experimental design for the mixed coagulant.

Table-3. Range of levels for mixture parameters.

Parameters	Range of levels		
	-1	0	1
Dosage of <i>M. oleifera</i> (mg/L)	23.558	117.79	212.022
Dosage of alum (mg/L)	23.558	117.79	212.022
pH	6.564		
Mixing time (min)	21.238		
Mixing speed (rpm)	125 for 1 st min - 40		

**Table-4.** Experimental design for the mixed coagulant.

Std.	Run	Component 1 A: <i>M. oleifera</i> dosage (mg/L)	Component 2 B: alum dosage (mg/L)	Response 1 SRF (m/kg)
4	1	164.906	70.674	
7	2	23.558	212.022	
3	3	117.79	117.79	
1	4	212.022	23.558	
5	5	70.674	164.906	
6	6	212.022	23.558	
8	7	117.79	117.79	
2	8	23.558	212.022	

2.3.3 Analytical methods

2.3.3.1 SRF measurement

SRF measurements for sludge samples were done after two hours of settling time for complete sedimentation [12]. Pressure of 300 mmHg was applied using vacuum filtration with 90 mm funnel diameter. From Darcy's law for liquid flow through porous media, an equation to calculate SRF can be obtained:

$$\alpha = \frac{a * 2\Delta P * A^2}{\mu * P_c} \quad (1)$$

Where:

α : specific resistance to filtration (SRF) (m/kg).

a : slope (s/m⁶).

ΔP : pressure (N/m²).

A : area of filter medium (m²).

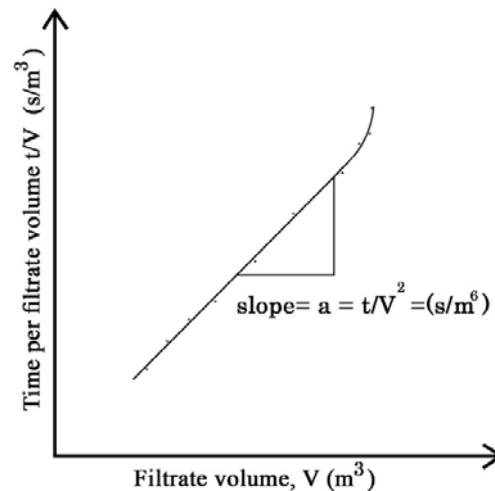
μ : viscosity of the filtrate (N.s/m²).

P_c : the mass of dry cake per filtrate volume (kg/m³).

The viscosity for each sample was measured by viscometer. Slope was determined by plotting filtration time per filtrate volume (t/V) versus filtrate volume (V). The plot should give a straight line [9], as shown in Figure-1.

2.3.3.2 Data analysis

The significance of coefficients was analysed using ANOVA approach and P -value (Probability > F). If P -value for a model was less than 0.05, that model can be considered as significant. The optimum values for the first optimization were obtained using regression model equation and analysing the 3D response plot. The optimum values for the second optimization were obtained using regression model equation and analysing the 2D response plot for the mixed coagulation.

**Figure-1.** Plot of time/volume vs volume.

3. RESULTS AND DISCUSSIONS

3.1 Process conditions and dewatering efficiency

Three process conditions (factors) were used in the first optimization: *M. oleifera* dosage, pH, and mixing time, and each factor had different effect on response value. The effect of different dosages of *M. oleifera* seed extract on dewatering process was investigated using dosage range of 100-500 mg/L, which was obtained by OFAT preliminary optimization. The optimum dosage at lowest SRF value was 288 mg/L with SRF = 1.045E+11 m/kg, pH = 7.43, and mixing time = 24.78 min. The curve of SRF values increased in both sides, as shown in Figure-2.

The effect of pH on SRF values was not significant. The pH range was used from 4-10. The optimum pH was 6.25 with SRF = 1.023E+11 m/kg and the curve of SRF values increased slightly in both sides of pH range, as shown in Figure-3. Because HCl can release cations at pH < 3, the pH values below 4 were not used in



the optimization. These cations with the positive charge can be combined with negative charge particles and form flocs [13]. The range of mixing time was selected from 5-30 min. The optimum mixing time was 24.88 min at SRF of $1.14\text{E}+11$ m/kg, as shown in Figure-4.

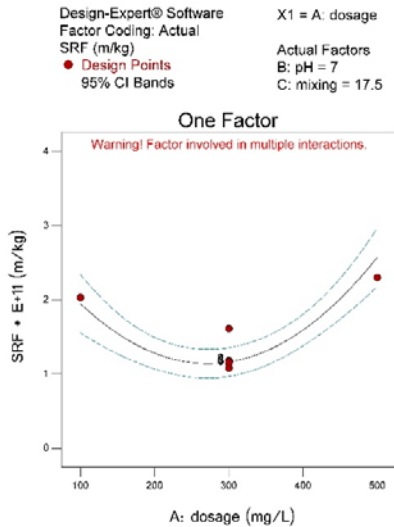


Figure-2. SRF vs dosage.

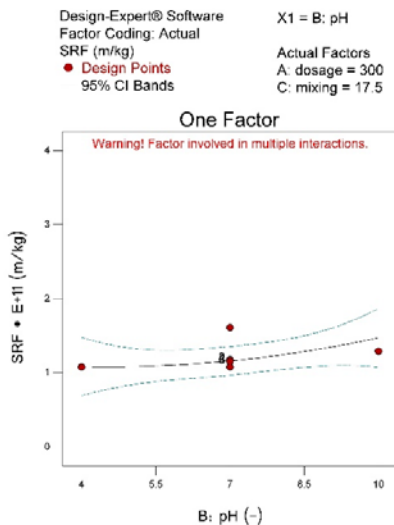


Figure-3. SRF vs pH.

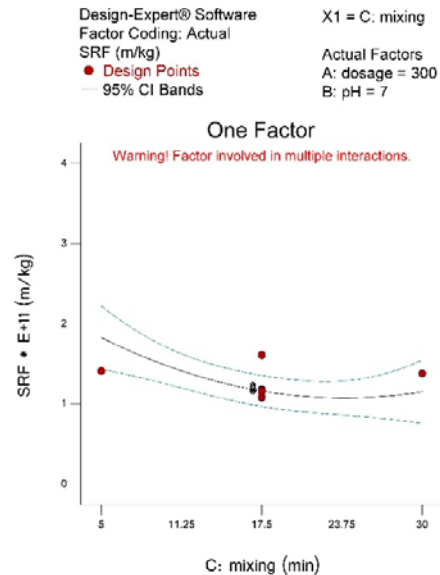


Figure-4. SRF vs mixing time.

3.2 First optimization (process conditions opt.)

The results of process conditions optimization were listed in Table-5. The lowest SRF value was $1.1\text{E}+11$ m/kg for experiments number 9 and 10, while the highest SRF value was $3.9\text{E}+11$ m/kg for experiment number 3.

The response was analysed using analysis of variance (ANOVA) approach, as shown in Table-6. According to the results, the factors A, B, C, AB, AC, BC, A^2 , C^2 were significant because P -values were less than 0.05 ($\text{Prob} > F$), where A is dosage, B is pH, and C is mixing time. The high value of R^2 at 0.958 showed that the quadratic model was also significant. The Predicted R^2 which is 0.7601 is in reasonable agreement with adjusted R^2 which is 0.9202. The lack of fit (F-value) of 2.46 implies the lack of fit is not significant, and there is only a 17.32% chance that a "Lack of fit F-value" this large could occur due to noise. The final equation in terms of actual factors is:

$$\begin{aligned} \text{SRF} = & 1.77023 - 8.60359\text{E-}003 \text{ dosage} + 0.17875 \\ & \text{pH} - 0.019135 \text{ mixing} - 6.0725\text{E-}004 \text{ dosage pH} - \\ & 1.1764\text{E-}004 \text{ dosage mixing} - 6.598\text{E-}003 \text{ pH} \\ & \text{mixing} + 2.74602\text{E-}005 \text{ dosage}^2 + 0.013101 \text{ pH}^2 \\ & + 2.10182\text{E-}003 \text{ mixing}^2 \end{aligned} \quad (2)$$

**Table-5.** Optimization of process conditions (first opt.) with results.

Std	Run	Factor 1 A: dosage (mg/L)	Factor 2 B: pH	Factor 3 C: mixing time (min)	Response 1 SRF (m/kg)
14	1	300	7.00	30.00	1.4E+11
16	2	300	7.00	17.50	1.2E+11
4	3	500	10.00	5.00	3.9E+11
3	4	100	10.00	5.00	3.7E+11
8	5	500	10.00	30.00	2E+11
20	6	300	7.00	17.50	1.2E+11
2	7	500	4.00	5.00	3.7E+11
17	8	300	7.00	17.50	1.2E+11
15	9	300	7.00	17.50	1.1E+11
11	10	300	4.00	17.50	1.1E+11
19	11	300	7.00	17.50	1.8E+11
5	12	100	4.00	30.00	1.95E+11
1	13	100	4.00	5.00	1.6E+11
18	14	300	7.00	17.50	1.2E+11
9	15	100	7.00	17.50	2E+11
6	16	500	4.00	30.00	2.7E+11
10	17	500	7.00	17.50	2.3E+11
7	18	100	10.00	30.00	2.5E+11
13	19	300	7.00	5.00	1.4E+11
12	20	300	10.00	17.50	1.3E+11

Table-6. ANOVA for response surface quadratic model.

Analysis of variance Table [Partial sum of squares - Type III]						
Source	Sum of squares	df	Mean square	F Value	p-value Prob > F	
Model	14.55	9	1.62	25.36	< 0.0001	<i>significant</i>
A-dosage	0.98	1	0.98	15.32	0.0029	
B-pH	0.37	1	0.37	5.88	0.0358	
C-mixing	1.14	1	1.14	17.93	0.0017	
AB	1.06	1	1.06	16.65	0.0022	
AC	0.69	1	0.69	10.85	0.0081	
BC	0.49	1	0.49	7.68	0.0197	
A ²	3.32	1	3.32	52.02	< 0.0001	
B ²	0.038	1	0.038	0.60	0.4567	
C ²	0.30	1	0.30	4.65	0.0564	
Residual	0.64	10	0.064			
Lack of Fit	0.45	5	0.091	2.46	0.1732	<i>not significant</i>
Pure Error	0.18	5	0.037			
Cor Total	15.19	19				



The first optimization results showed 10 solutions with desirability = 1, as shown in Table-7. To check the efficiency of optimization, five solutions were tested. The

results of these solutions were similar to the predicted SRF values.

Table-7. First optimization results.

Constraints						
Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A:conc	is in range	100	500	1	1	3
B:pH	is in range	4	10	1	1	3
C:mix time	is in range	5	30	1	1	3
SRF	minimize	1.078	3.86	1	1	3
Solutions						
Number	Conc.	pH	mixing time	SRF	Desirability	
1	235.58	6.5	21.2	1.1E+11	1.000	Selected
2	255.55	7.05	22.1	1.1E+11	1.000	
3	217.04	5.74	16.4	1.1E+11	1.000	
4	233.33	5.8	15.83	1.1E+11	1.000	
5	286.11	7.59	25.55	1.1E+11	1.000	
6	225.68	5.01	22.64	1E+11	1.000	
7	300	4	17.5	1.1E+11	1.000	
8	286.67	7.6	27.5	1.1E+11	1.000	
9	249.2	5.41	13.85	1.1E+11	1.000	
10	313.33	5	19.17	1.1E+11	1.000	

Figure-5 shows the 2D contour plot, which represented the interaction between *M. oleifera* dosage and pH at mixing time = 17.5 min. Figure-6 shows the 3D contour plot for the same interaction. Both figures were obtained using Design-Expert v9 software.

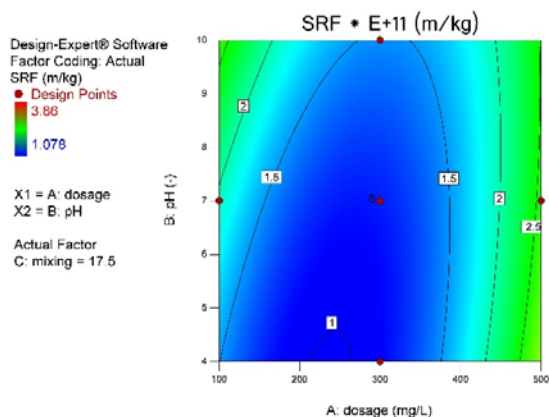


Figure-5. 2D contour plot of the interaction of dosage with pH.

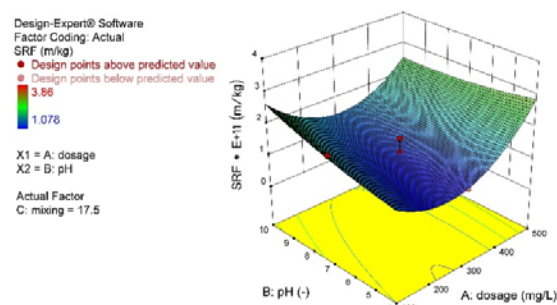


Figure-6. 3D contour plot of the interaction of dosage and pH with SRF as response.

As reported in [11], the dewatering ability of *M. oleifera* seeds was studied on real sludge samples. The samples were collected from sewerage treatment plant, Kuala Lumpur, Malaysia. Optimum process conditions were mixing speed of 100 rpm, mixing time of 1 min, and *M. oleifera* dosage of 4695 mg/L. The response (SRF) was 1.22E+11 m/kg which is near from SRF value obtained from the current study (SRF = 1.1E+11 m/kg) as shown in Table-7. The study used *M. oleifera* seed extract by



distilled water and applied on real sludge sample, while the current study used *M. oleifera* seed extract by salt extraction (NaCl) and applied on synthetic sludge sample (5% kaolin suspension). The reason of using high dosage of *M. oleifera* seed extract by water (4695 mg/L) is the extraction and sludge sample. The real sludge sample consist of different types of organic and inorganic constituent. The mixing speed of 100 rpm is different from the mixing speed used in the current study, which is 40 rpm. The different properties of real sludge sample require different process conditions.

3.3 Second optimization (mixture ratio opt.)

Experimental design for the mixed coagulant ratio with SRF values were listed in Table-8. SRF value for alum alone was found to be 1.1E+11 m/kg, which is more effective by 27.3% than *M. oleifera* seed extract alone at SRF of 0.8E+11 m/kg. The mixed coagulant at

ratio of 50:50 for alum and *M. oleifera* was effective as alum alone. At ratio of 90:10 for alum and *M. oleifera*, the mixture also had the same efficiency for alum alone.

The responses were analysed using ANOVA approach, as shown in Table-9. The model is significant because *P*-values were less than 0.05 (Prob > *F*). The factors A, B, and AB were also significant.

Although the R^2 of 0.9626 is high, it is not close enough to the adjusted R^2 , which is 0.9477. It is very difficult to get close values of SRF, especially when the differences between these values are small. The final equation in terms of actual components is:

$$\text{SRF} = (4.53109\text{E-}003 * \text{Alum}) + (3.32013\text{E-}003 * \text{MO}) - (6.67751\text{E-}006 * \text{Alum} * \text{MO}) \quad (3)$$

Where MO is the dosage of *M. oleifera* seed extract.

Table-8. Experimental design for the mixed coagulant with results.

Std.	Run	Component 1 A: M.O (mg/L)	Component 2 B: Alum (mg/L)	Response 1 SRF (m/kg)
4	1	164.906	70.674	0.89E+11
7	2	23.558	212.022	0.8E+11
3	3	117.79	117.79	0.8E+11
1	4	212.022	23.558	1E+11
5	5	70.674	164.906	0.8E+11
6	6	212.022	23.558	0.1E+11
8	7	117.79	117.79	0.8E+11
2	8	23.558	212.022	0.8E+11

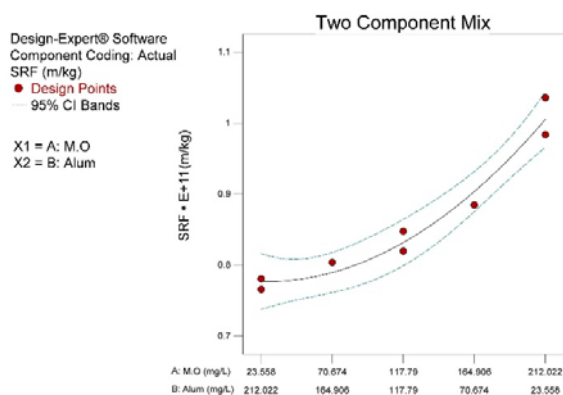


Figure-7. Optimization for different ratios of the two components mixture.

A study of [14] used mixed coagulant of alum and *M. oleifera* at ratio of 50:50. This mixture was tested on real sludge sample at high TSS of 3.19 g/L. *M. oleifera* seeds powder was extracted using 1 M of NaCl. Comparing the efficiency of the three types of coagulants: alum, *M. oleifera*, and the mixture; the combined coagulant had almost similar dewatering efficiency comparing with alum alone. Although sludge samples and total suspended solids content were different from the current study, it showed that the efficiency of the mixture was better comparing with *M. oleifera* alone.

**Table-9.** ANOVA for Quadratic Mixture model.

Mixture component coding is L_Pseudo						
Analysis of variance Table [Partial sum of squares - Type III]						
Source	Sum of squares	df	Mean square	F Value	p-value Prob > F	
Model	0.064	2	0.032	64.39	0.0003	significant
<i>Linear Mixture</i>	<i>0.059</i>	<i>1</i>	<i>0.059</i>	<i>117.54</i>	<i>0.0001</i>	
<i>AB</i>	<i>5.603E-003</i>	<i>1</i>	<i>5.603E-003</i>	<i>11.24</i>	<i>0.0203</i>	
Residual	2.493E-003	5	4.985E-004			
<i>Lack of Fit</i>	<i>6.362E-004</i>	<i>2</i>	<i>3.181E-004</i>	<i>0.51</i>	<i>0.6428</i>	<i>not significant</i>
<i>Pure Error</i>	<i>1.857E-003</i>	<i>3</i>	<i>6.188E-004</i>			
Cor Total	0.067	7				

4. CONCLUSIONS

The results showed that the optimum process conditions obtained from the first optimization were 235.58 mg/L for dosage, 6.5 for pH, and 21.2 min for mixing time at SRF of 1.1E+11 m/kg. R^2 was 95.8% and the model was significant. The optimum ratio of the mixed coagulant was 50:50 (w/w) for *M. oleifera* and alum at SRF of 0.8E+11 m/kg. R^2 was 96.26% and the model was significant. The recorded efficiency of alum alone was the same as SRF value of the mixed coagulant at ratio 50:50. By using combined coagulant consisting of 50% alum and 50% *M. oleifera* seed extract, the efficiency will be high with eliminating the use of alum up to half. Involving natural coagulants in sludge treatment will maintain the dewatering efficiency and decrease the risk on human health and environment.

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