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Application of Response Surface Methodology (RSM) in Production of Potassium Aluminium Sulphate from Egyptian Kaolin

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Abstract

Alum is a commonly a hydrated double sulfate salt of aluminum with the general formula $XAl(SO_4)_2 \cdot 12H_2O$, wherein X is a monovalent cation that includes Ammonium or Potassium. It is used in water purification, medicines, cosmetics, food preparation, and flame-retardant paper and textiles. The goal of the present work is to produce potassium aluminum sulfate (PAS) from local kaolin. Alum is produced through leaching of metakaolin with H_2SO_4 to obtain liquid aluminum sulphate (AS), which crystallized with potassium sulphate to produce crystalline PAS. Response surface methodology (RSM) (Box-Behnken) technique is used to optimize the leaching conditions to maximize the yield. The studied parameters are temperature (70 –210°C), acid concentration (2– 6 Molar), and Time (60 –120 min). A predicted maximum yield of 97.3062 % was obtained on using 5.3 M acid concentration, 180°C temperature of reaction and of 92 min for time. The suggested model (quadratic) was found to be highly significant, with R^2 (0.9764) and P value < 0.005. The determined maximum yield is tested experimentally through five runs at optimum conditions. The average yield of the five runs is 94.592 % with a standard deviation 0.89, the percentage error did not exceed 2.49 %. Which confirms the validity of the model.

Keywords: *Leaching, RSM, optimization, Crystallization*

1. Introduction

Water is a natural resource that is necessary for the survival of humanity and the socio-economic development of nations [1]. There is a growing need to use all available forms of water, including their recovery from all types of waste streams. [2].

Raw water can have varying characteristics regarding suspended solid,colloids, organic matter c and sometimes of high mineralization (chlorides, sulfates). To eliminate totally or partially these elements, the conception of the treatment facility may appeal to several processes [3, 4]. Coagulation-flocculation is the most common treatment to eliminate the suspended solid and colloids present in water[3, 5]. One of the traditional techniques used in removing colloids and particles is coagulation; Alum coagulation is a common process used in water treatment to remove suspended solids.

Potassium Alum is a crystallinewhite to colourless substance that constitute of the mineral aluniteKAl$(SO_4)_2 \cdot 2Al(OH)_3$ and naturally found as (Kalunite) KA1$(SO_4)_2 \cdot 12H_2O$ [6].

Other common alums are ferric ammonium alum $NH_4Fe(SO_2)_2 \cdot 12H_2O$, and sodium chrome alum $NaCr(SO_4)_2 \cdot 12H_2O$.

Potassium aluminum sulphate (PAS) or Potassium alum is commonly used in water purification, leather tanning, dying, fireproof textiles, baking powder, and it has cosmetic uses as a deodorant.

It is one of the earliest and still efficient coagulants).it has been a standard raw material for water treatment in industry because of its ability to form multi charged polynuclear complexes with enhanced adsorption characteristics[7].

Alum can be prepared from different raw materials; such as bauxite, aluminium dross, and kaolin clay. Bauxite oreundergo an expensive and energy intensive procedure to be converted into alum for industrial use [8–11].

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In Egypt, due to rarity of bauxite ores and the availability of kaolin raw materials, research was directed towards producing of alumina and alum from kaolin [12–16].

Leaching is the process of extracting substances from a solid by dissolving them in a liquid. One of the most frequently applied techniques in hydrometallurgy for the extraction of metals in solutions is the leaching process. Leaching processes are economic, show less energy consumption, and enable the treatment ores of law grade [2, 17].

(RSM) technique isa software that can be usedtobuild model empirically. These techniques are intended to choose experimental conditions so as to minimize the number of required runs; the final target is to optimize the responsevariable; all the independent variables must affect the response. In the selected runs, input variables are changed in such a way so as to enable determining the effect of these changes on the output response[18, 19].

Alum is the most commonly used chemical coagulant in water treatment plants worldwide. [20, 21].The demand for alum increases due to its frequent use in several different fields, including paper making, water treatment, dyes, and other industrial processes[22, 23].

Therefore, the objective of this research is preparation of potassium aluminium sulfate$KAl(SO_4)_2$through leaching locally kaolin utilizing response surface methodology (Box-Behnken method). crystallization of aluminium sulphate produce PAS.Characterization of the produced samples. Application of potassium aluminium sulphate in water treatment as coagulant.

2. Experimental:

2.1. Materials

The material used in this research includes:

1) Egyptian kaolin (Kalabsha, Aswan area). This was obtained from the main quarry in Kalabsha.
2) Sulphuric acid (98%) purchased from El-sharq El-Awsat office in El Kasr El Aini Street for Chemicals (Cairo).
3) Potassium sulphate purchased from El-sharq El-Awsat office in El Kasr El Aini Street for Chemicals (Cairo).

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2.2. Methods

50 g of Meta kaolin calcined at 700°C for two hours was used for each run[12].Then leaching of process was carried out according to statistical design at the following conditions fixed for all runs:

1. Solid to liquid ratio (1:5)
2. The reaction mixture consists of 50 gm of calcined kaolin and 250 ml of dilute H_2SO_4 (At different concentrations according to excremental designis put in 1000 ml flask connected to condenser.
3. The reaction mixture is heated using oil bath
4. Stirring speed is fixed at a speed of 250 rpm. [24, 25].
5. The reaction product is filtrated to separate solid from liquid product using vacuum pump.

$$Al_2O_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2O \quad (1)$$

The conversion is calculated by measuring the concentration of aluminiumsulphate in filtrate solution.Calculation of the mass of aluminium extracted then division on the initial weight of aluminum in the kaolin sample that calculated using XRF according to the following equation;

$$\%\ \text{Conversion} = \frac{mi - mf}{mi}\ x\ 100 \quad (2)$$

m_i= initial mass of Al

m_f= mass of Al in filtrate solution

Crystallization:

It is one of the important steps to produce high purity crystals (PAS) from a concentrated solution which contains equivalent amounts of aluminium sulfate and potassiumsulfate. The chemical reaction is given below.

$$K_2SO_4(aq) + Al_2(SO_4)_3(aq) \rightarrow 2KAl\ (SO_4)_2.12H_2O\ (s) \quad (3)$$

The PAS crystals are separated from the solution, washed with a small amount of ethanol, then dried[26–28].

2.3. Apparatuses:

- Alumina and kaolin composition was determined by using X-ray fluorescence (XRF), Pananalytical XRF (Model Advanced Axios, Netherland).
- X-ray diffractometry techniques (XRD) in order to determine the essential peaks of the crystal of alum formed.
- The temperature of calcination was determined by Thermogravimetric measurements (TGA, 20–1000 °C) were recorded on a DTG-50 Shimadzu thermogravimetric analyzer at a heating rate of 10 °C min^{-1} and nitrogen.
- Fourier transform infrared spectroscopy (FTIR) was used within the range (400–4000 cm^{-1}) using Nicolet Avatar FTIR 370 CSI using KBr pellets.
- Particle Size Distribution (PSD)

The approach for evaluating the distribution was based on the standard test method for particle-size analysis of soils (ASTM D 422). The Tyler series. The sieves' opening is shown in Table 1 [29].

Table 1: Standard screen openings [29].

Mesh size	4	10	14	20	28
Opening mm	4.7	1.651	1.168	0.833	0.589
Mesh size	42	60	100	115	200
Opening mm	0.351	0.246	0.147	0.124	0.074

2.4. Statistical Model for the Leaching Process

The variables investigated in acid leaching were the temperature of reaction (T°C), the concentration (c molar) and reaction time (t h) shown in table 2 . The Box - Behnken statistical design is used as a tool for simulation and optimization of the dissolution process[12, 18].Statistical Design-Expert 11 was used to relate the percent recovery (Y) to the three variables by a second order equation in the form:

$$Y= \beta_0+ \beta_1X_1+ \beta_2X_2+ \beta_3X_3+ \beta_{12}X_1X_2+ \beta_{23}X_2X_3+$$
$$\beta_{31}X_3X_1+ \beta_{11}X_{12}+ \beta_{22}X_{22}+ \beta_{33}X_{32} \quad (4)$$

The chosen values of the three parameters are shown in Table 2. These were selected following a set of preliminary trials.

Table 2: Selected reaction parameters

Variable	Min.	Center	Max.
T	70	140	210
c	2	4	6
t	60	90	120

2.5. Application of Produced Alum in Wastewater Treatment

Water treatment was done using the produced alum against the commercial alum that used in under the same conditions, which are alum dose 250 ppm, pH 6.5 and time 20 min to compare between them and to know the quality and efficiency of the produced alum[30].

3. Results and Discussion

Applying the experimental work techniques, this part shows the characteristics of raw material and alum products. It also presents and discusses the leaching reaction results, optimum conditions of leaching process obtained with the aid of statistical design expert, the interaction between the reaction parameters in 2D and 3D models as well as yield and rate equations, also crystallization results.

3.1 Characterization of kaolin

Different characterization techniques were applied on the kaolin clay under study. The results are reported in the following section.

3.1.1 Chemical Analysis (XRF)

AsampleofEgyptiankaolin(Kalabsha,Aswanarea)wasusedthechemicalcompositionofwhich is shown in Table 3 as obtained usingXRF.

Table 3: XRF chemical composition of Kalabsha kaolin

Component	Content, wt.%	Component	Content, wt.%
Al_2O_3	32.906	TiO_2	5.918
SiO_2	48.931	Fe_2O_3	1.193
Na_2O	0.094	SO_3	0.291
K_2O	0.014	Cr_2O_3	0.138
CaO	0.505	ZrO_2	0.465
MgO	0.09	LOI (Loss on Ignition)	9.2
TOTAL		99.745	

The two main components of the sample as show in Table 3 are silica (48.931%) and aluminum oxide (32.906%) which is required to be extracted as liquid aluminum sulphate using sulphuric acid leaving silica as solid residue which can be separated by filtration.

3.1.2 Mineralogical Analysis (XRD)

The mineralogical composition of raw kaolin was assessed using XRD Figure 1. This figure has main constituent, which is kaolinite, a result in agreement with XRF findings. Also, free silica shows up as quartz.

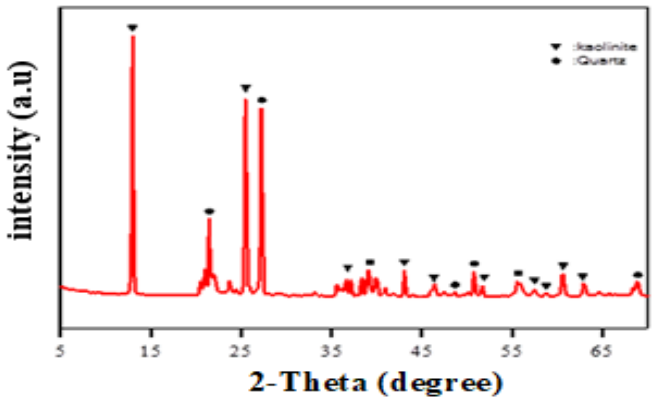

Figure 1: XRD pattern of Kalabsha kaolin[12].

3.1.3 Thermogravimetric Analysis (TGA)

A kaolin sample was subjected to thermogravimetric analysis to determine the minimum temperature of de-hydroxylation. The heating rate was 10 ^{o}C.min^{-1}. From Figure 2, it appears that the major loss in weight takes place starting from about 500 ^{o}C up to about 770 ^{o}C, whereby the loss in weight is about 10%[12, 31, 32].

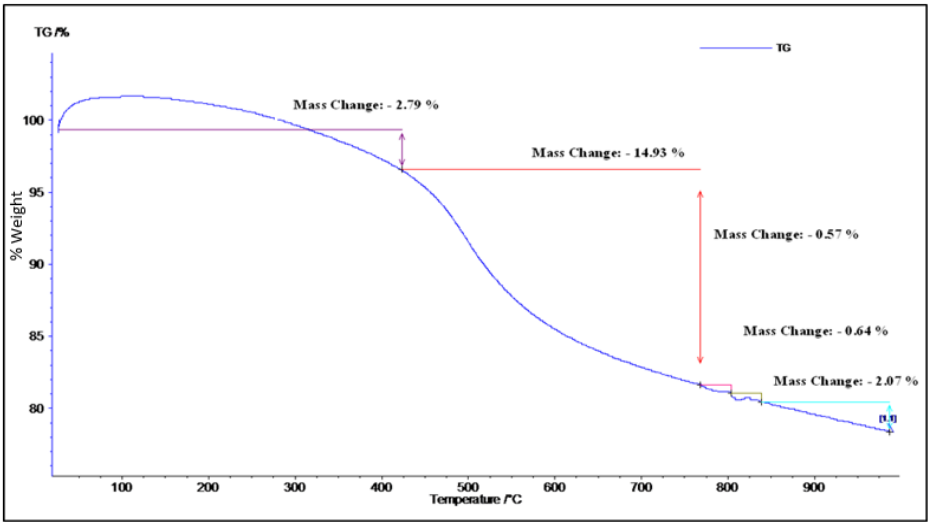

Figure 2: TG – Curve of Kaolin sample[12].

3.1.4 Particle Size Distribution(PSD)

Wet size analysis of the sample is shown in Figure 3 where it appears that about 45% of themass passes 0.075 mm screen (200 mesh). The median particle size D_{50}) as obtained from the cumulative plot = 0.053mm.

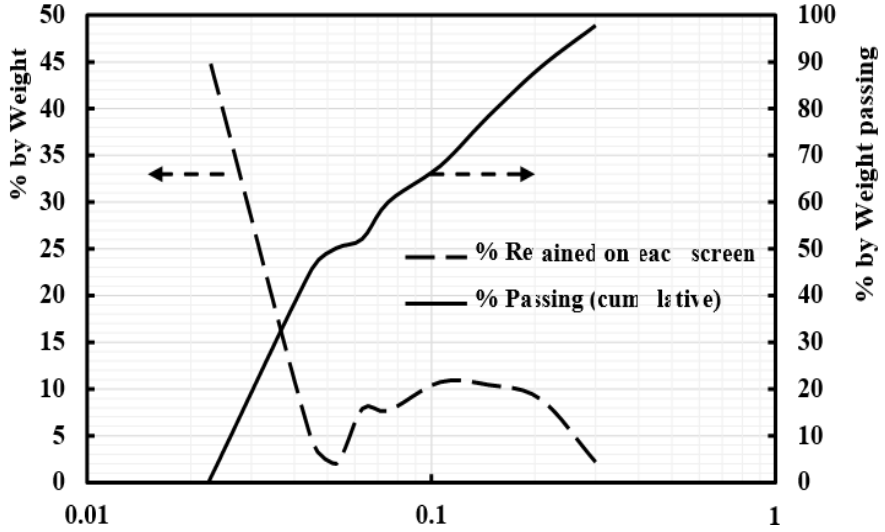

Figure 3: Screen analysis of kaolin sample[12].

3.2 Leaching Reaction Results (Statistical Model Result)

The results of the leaching reaction are illustrated in actual value in Table 4. Where A is temperature (℃), B is acid concentration (M), and C is time (min), and the response is yield (Y).

Table 4: Actual experimental results with different parameters

Model	Temp. (℃)	Acid concentration (M)	Time (min)	Response Yield%
1	140	4	90	93.418
2	140	6	120	87.667
3	140	2	120	64.914
4	140	4	90	89.69
5	70	4	120	57.998
6	70	4	60	49.068
7	140	4	90	92.086
8	140	4	90	87.314
9	210	4	60	81.17
10	140	4	90	90.42
11	70	6	90	41.267
12	70	2	90	32.25
13	210	4	120	87.208
14	210	2	90	65.14
15	140	6	60	73.03
16	210	6	90	92.16
17	140	2	60	44.966

It can be noticed from the Table 4 that the highest yield (93.418 %) is obtained at the center of design and the lowest yield at run 12 where minimum values of temperature and concentration of acid, which refers to the highly effect of both parameters on leaching process.

The suggested model (quadratic) was found to be highly significant, its determination coefficient R^2 being (0.9764) and P value < 0.005 (confidential level) Table 5 shows the analysis of variance (ANOVA) for the quadratic model.

Table 5: ANOVA for Quadratic model

Source	Mean Square	F-value	P-value	
Model	736.62	32.16	< 0.0001	Significant
A	2631.57	114.89	< 0.0001	Significant
B	942.95	41.17	0.0004	Significant
C	306.94	13.4	0.0081	Significant
AB	81.03	3.54	0.1020	Not-Significant
AC	2.09	0.0913	0.7713	Not-Significant
BC	7.05	0.3079	0.5963	Not-Significant
A^2	1055.42	46.08	0.0003	Significant
B^2	1223.87	53.43	0.0002	Significant
C^2	146.19	6.38	0.0394	Significant

In case of P-values are less than 0.05 indicate model terms are significant, in this case, A, B, C, A^2, B^2 and C^2 are significant terms. However, in case of P-values are greater than 0.05 indicate the model terms are not significant. So, AB, AC and BC are not significant terms.

The correlation between yield (Y) and the three studied parameters was obtained in coded and actual equations which include the significant model terms only as shown in equation 6 and equation 7 respectively. Where, A is the temperature of reaction (°C), B is the acid concentration (M), and C is the reaction time (min).

$$Y = 90.59 + 18.14\,A + 10.86\,B + 6.19\,C - 15.83\,A^2 - 17.05\,B^2 - 5.89\,C^2 \quad (5)$$

The coded parameters can be converted to actual parameters by using the following equations

$$A = \frac{T-140}{70} \quad (6)$$

$$B = \frac{C-4}{2} \quad (7)$$

$$C = \frac{t-90}{30} \quad (8)$$

$$Y = -164.84162 + (1.06619 \times \mathbf{T}) + (37.01735 \times \mathbf{C}) + (1.52165 \times \mathbf{t}) - (0.003231 \times \mathbf{T^2}) - (4.26226 \times \mathbf{C^2}) - (0.006547 \times \mathbf{t^2}) \quad (9)$$

The rate of reaction(R) can be obtained by partial differentiation of the yield with respect to time

$$\frac{dR}{dt} = 1.52165 - 0.013094 \times \mathbf{t} \quad (10)$$

3.2.1 Interaction Between Selected Parameters

The interaction between the studied parameters were studied using contour figures. The contours display the effect of changes of two parameters on the yield at different values of the third parameter (minimum, average and maximum values). The dissection of this effect is illustrated in the following sections.

3.2.2 Contour Statistical Model Graphs

It is a 2D graph that is generated by projecting 3D graph model, it shows not only the maximum, minimum points but also the effect of parameters on a certain response. Contour is a 2D model that has two changing parameters and a constant oneat its minimum, average, and maximum value. This can be used to get maximum yield that can be obtained at these values. The colors on the graph is codded as thehigher the yield, the closer to red-colored regions is obtained, and the lower the yield the closer to blue regions is obtained. Each line shows the yield obtained at any condition.

a) Effect of the Temperature and Acid Concentration on Aluminium sulphate (AS) Yield

Figure 4 (a, b, c) indicates the effect of temperature and acid concentration on the aluminium sulphate yield at minimum time (a, 60 min), average time(b, 90 min)), and maximum time(c, 120 min). As can be shown from the figure 4, increasing temperature and concentration increases the AS yield. At minimum time (figure 4a) the yield increased from 40 % at minimum concentration and temperature to 80 % at about4.5 M of acid and 140°C. At average value of time (figure 4b), the yield increased from 40% at minimum values of acid concentration and temperature to 90 % at about4.2 M of acid and 140°C which displays the effect of increasing time on the percentage yield at the same conditions of acid concentration and temperature. At maximum time 120 min (figure4c), the change of yield is as the same as at average value of time which means there is no need for increasing time above 90 min[33].

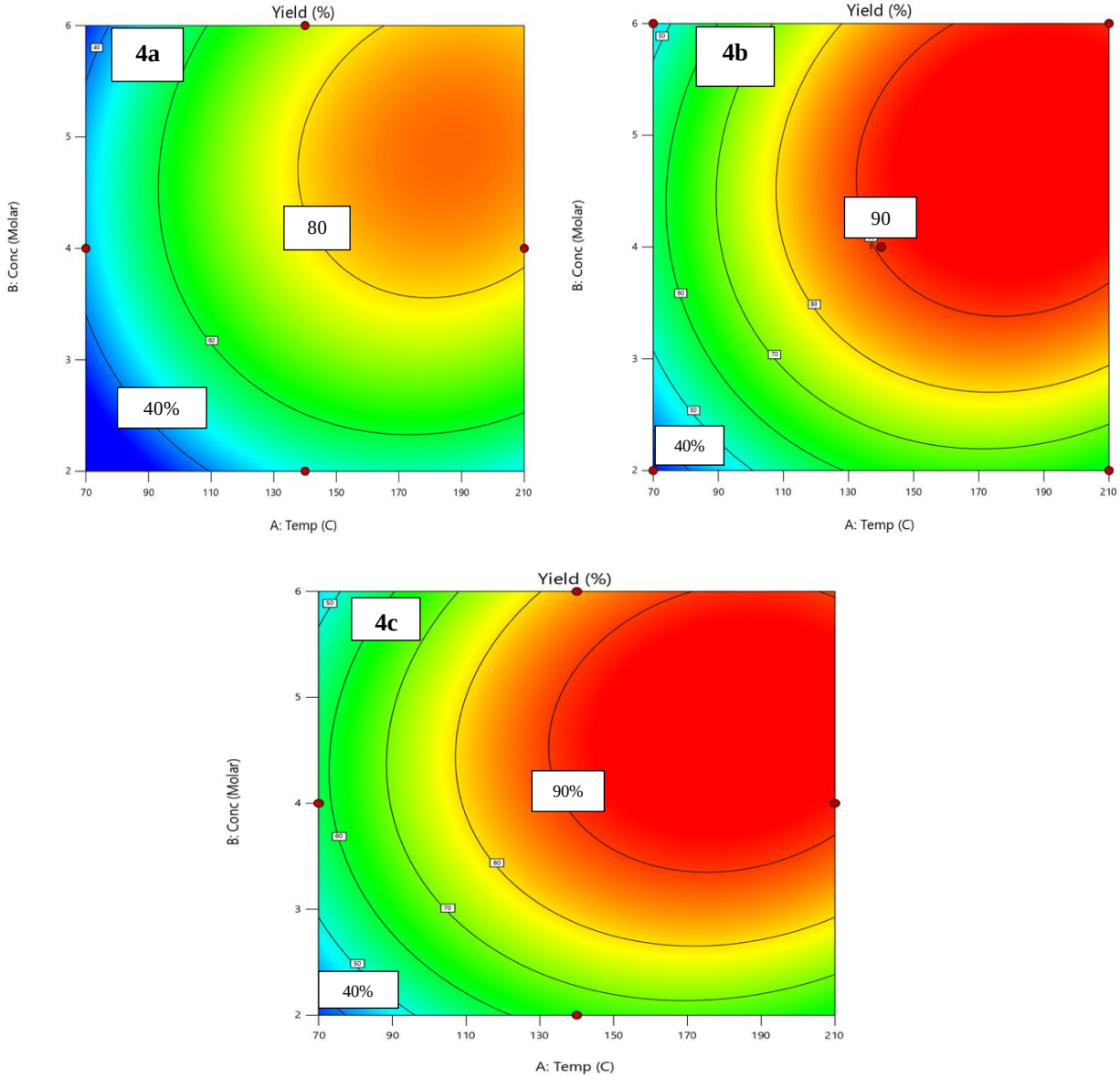

Figure 4: Effect of the Temperature and Acid Concentration on Alum's Yield (a, 60 min - b, 90 min - c, 120 min).

b) Effect of the Temperature and Time on AS Yield

Figure 5 (a, b, c) displays the effect of temperate and time on the AS yield at different acid concentrations; minimum concentration (a, 2M), average concentration (b, 4 M), and maximum concentration (c, 6M). At minimum value of acid concentration (figure 5a), increasing temperature increases the yield but the maximum yield can be obtained is 60% at 140°C and 90min. At average value of acid concentration, the yield reached 90 % at about 140°C and 90 min.

This displays the studied parameters interaction and the high effect of acid concentration on the reaction. In figure (5 c) increasing the temperature and time increasing the yield as the concentration of acid is maximum, but in this case the increasing concentration above 4 molar has no effect.

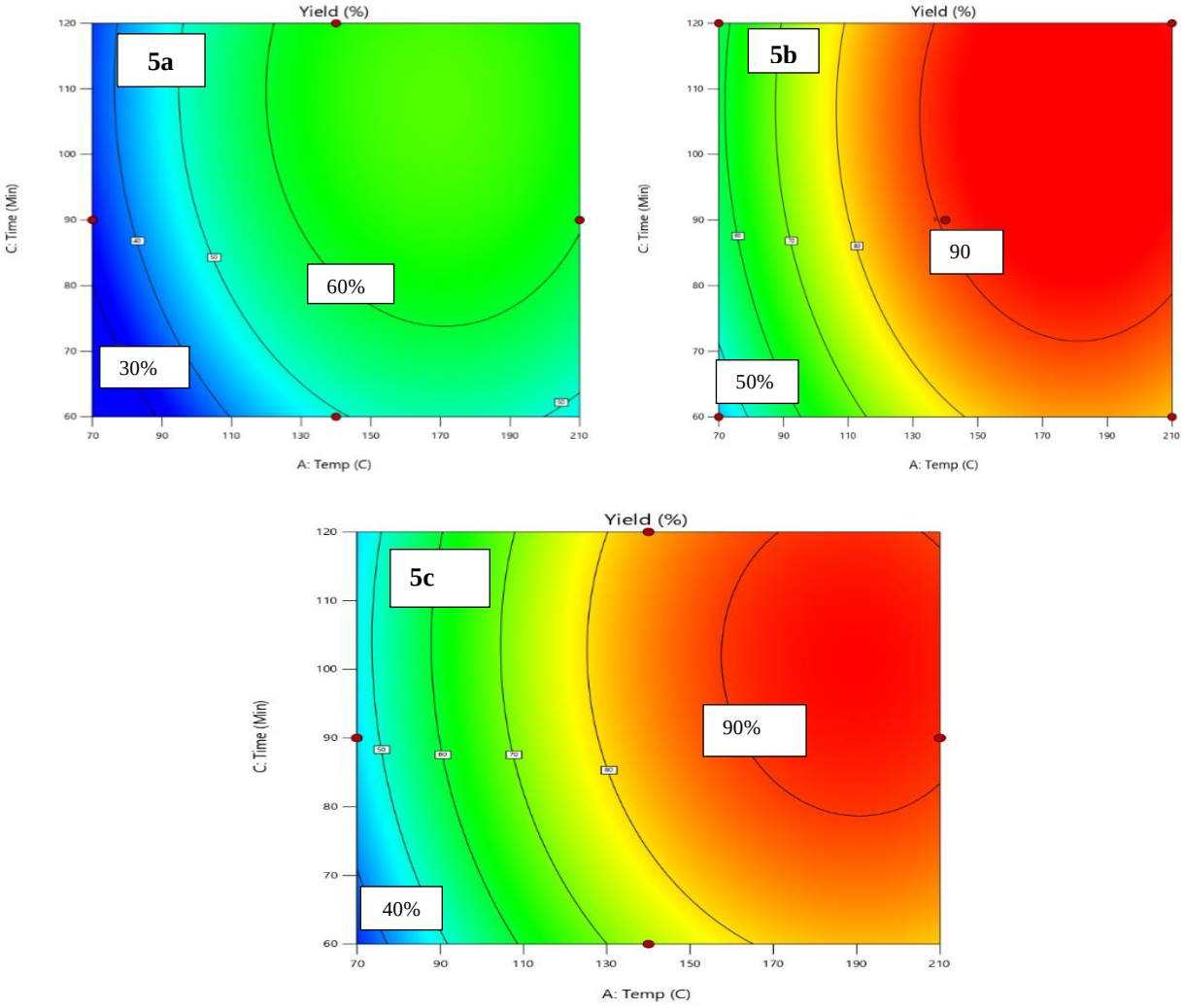

Figure 5: Effect of the Temperature and Time on Alum's Yield (a, 2M, - b, 4 M - c, 6M).

c) Effect of the Acid Concentration and Time on AS Yield

Figure 6 (a, b, c) illustrates the effect of acid concentration and time on the AS yield at different temperatures; minimum (a, 70°C), average (b, 140°C), and maximum (c, 210°C).In Figure 6a, at minimum temperature increasing acid concentration, and time increases the yield but, the maximum value is about 50% even at maximum time and acid concentration, which indicates the high effect of temperature on the yield. At average value of temperature, Figure 6b, the yield reached 90% at about 4.5 M and 85 min. At maximum temperature, Figure6c, 90% yield is obtained at about 4.2 M and 78min.

The results discussed in contour figures confirms that; the studied parameters effect on each other, and the strong effect of temperature and concentration on the yield.

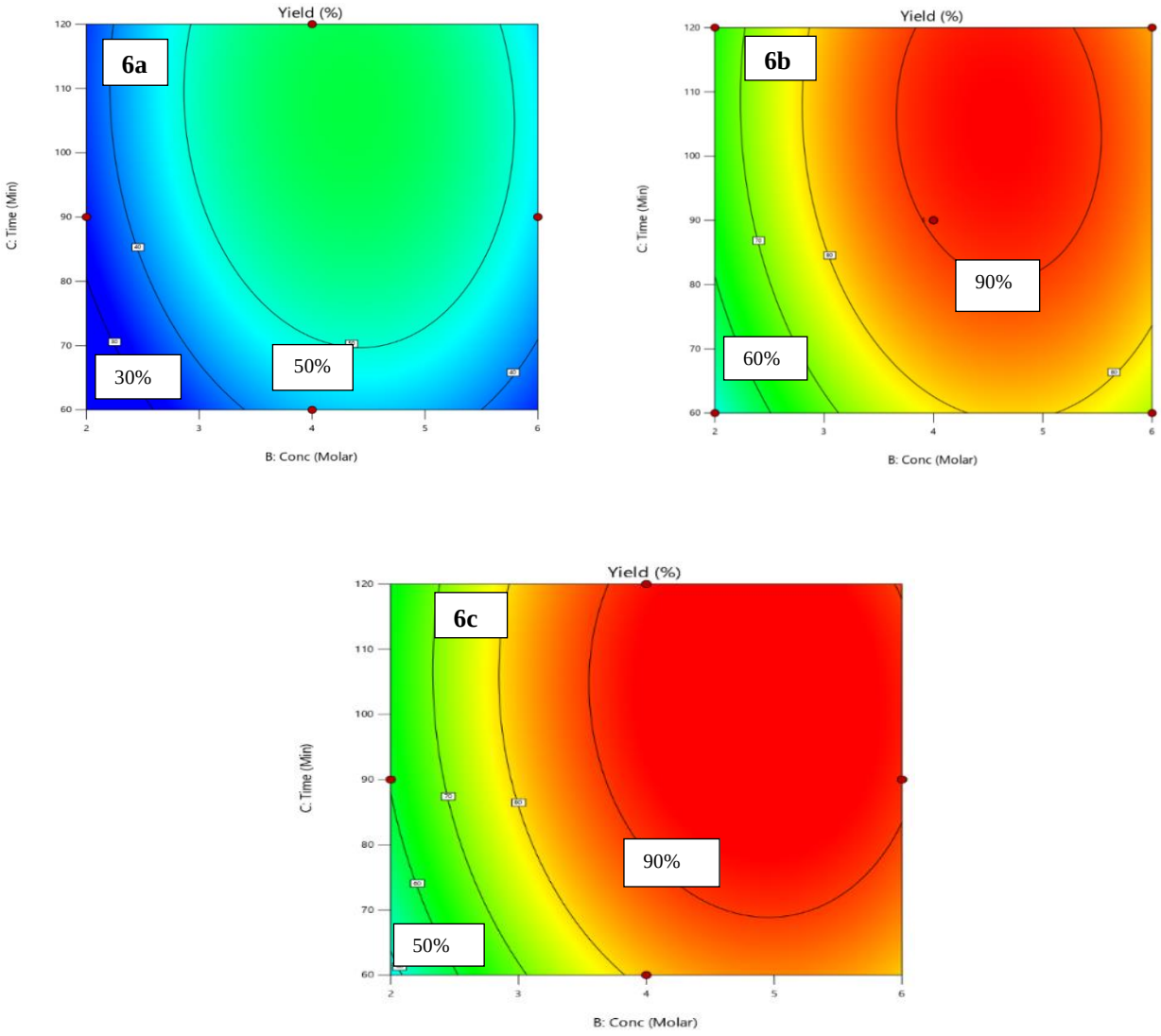

Figure 6: Effect of the Acid Concentration and Time on AS Yield (a, 70^{o}C - b, 140^{o}C - c, 210^{o}C).

3.2.3 Selecting the Optimal Conditions

To determine the optimal conditions for obtaining a value for a yield, this is done by using the RSM program by numerical optimization by choosing the maximum value for the yield, bearing in mind that the parameters affecting the reaction must be within its range.

As shown in Figure 7, a predicted maximum yield of 97.3062 % was obtained using a 5.3 molar acid concentration, temperature of reaction is 180℃-, and 92-min time of reaction. To confirm the validity of these results, 5 runs were performed at the aforementioned conditions.

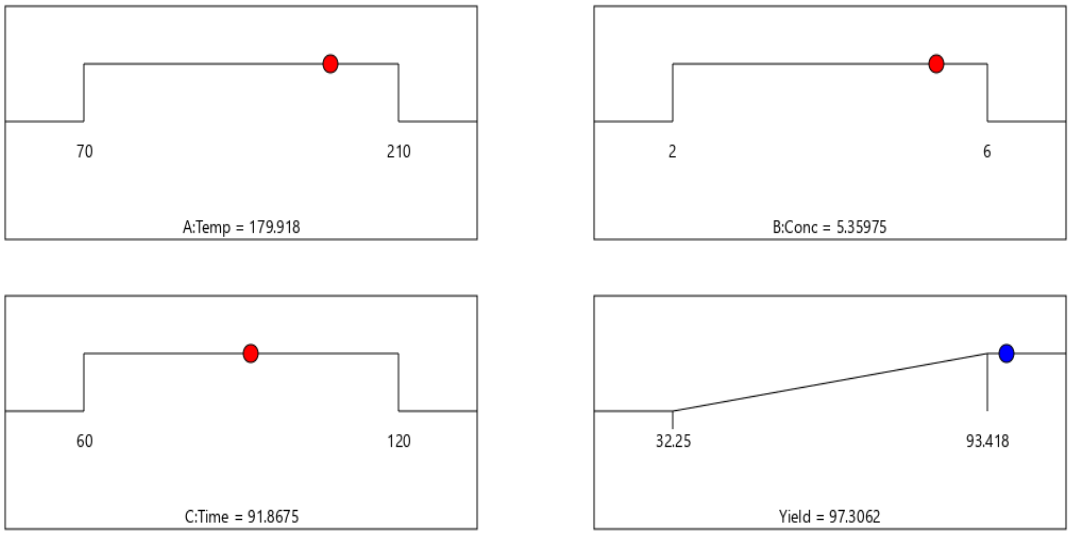

Figure 7: Optimal conditions by numerical optimization

3.3 Characteristics of Alum

Table 6 shows some of the measured properties of the produced samples compared to the commercial samples. There is agreement between the produced samples and the commercial samples. The tested properties of the optimum sample show perfect characteristics compared to Egyptian standards.

Table 6: Characteristics of alum[34].

Property (unit)	Aluminum Sulphate	
	Measured value	Egyptian standard
Density at 25 ºC (g/ml)	2.36	1.62 – 2.67
pH	3.1	2.9 – 3.5
Property (unit)	Potassium Aluminum Sulphate	
	Measured value	Egyptian standard
Density at 25 ºC (g/ml)	1.7	1.72
pH	3	3 – 3.5

Infrared spectroscopy is often used to characterize solid-state catalysts to identify both the organic and inorganic surface functional groups.

Figure 8: shows the spectrum of $KAl(SO_4)_2 \cdot 12H_2O$, (commercial alum (a) and prepared alum(b)), the result of Fourier transform infrared spectroscopy (FTIR) was used within the range (400–4000 cm^{-1}), The PAS sample (b) is very close to the commercial alum (a) and the peaks at different wavenumber almost the same.

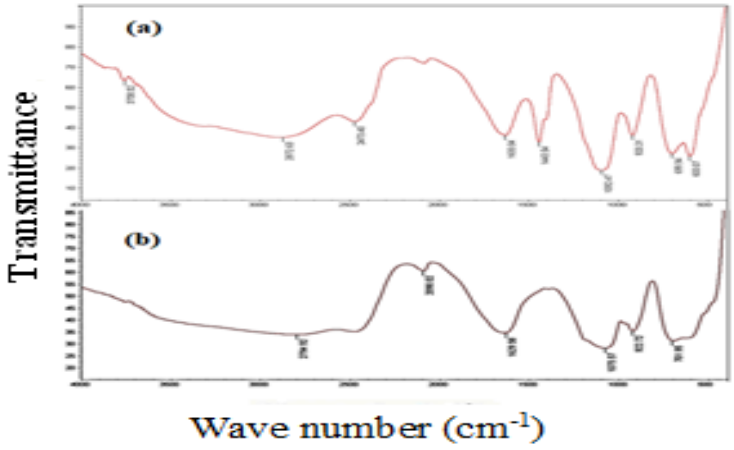

Figure 8: FTIR for commercial alum (a) and prepared alum(b).

Figure 8(b) The peaks appearing at 1629 cm^{-1} indicate O-H bending of H_2O. A short peak at at 2090.02 cm^{-1} can be attributed to the H_2O overtone band though the band at 1443.54 cm^{-1} in commercial alum is possibly due to the S-O bond vibrations of sulfate[35].
Peaks at 1075.57 cm^{-1} is due to the vibration of stretching of S═O group, peaks at 920 cm^{-1} and 701 cm^{-1} are due to the stretching vibrations of S─O and Al─O bonds. Peaks in the region of 750 - 400 cm^{-1} indicated the vibration of Al–O [36, 37]

3.4 Result of using alum as coagulant in wastewater treatment
Sample of produced alum is used in treatment of synthetize wastewater under the same conditions, which are (alum dose 250 PPM), (pH 6.5) and (time 20 min) to compare between them and to know the quality and efficiency of the produced alum. Produced alum has efficiency of removal 77.55% of total suspended solids and commercial alum has efficiency of removal 74.69%.

4. Conclusions
- Alum is a coagulant used in water treatment, rapid industrialization is contributing to water pollution, that is why cheaper and efficient techniques are needed.
- Alum is produced from various raw materials such as bauxite, kaolin and aluminum dross.
- Response surface methodology (Box – Behnken method) is used to predict the optimum conditions that affect the reactions. The optimum conditions were time (92 min), temperature (180 C) and acid concentration (5.3 molar).The results were assessed by using five runs and the standard deviation was 0.89.
- A predicted maximum yield of 97.3062 % was obtained on using a 5.3 molar acid concentration, a 180°C and a 92 minute.A mean percentage ofyield for 5 runs is 94.592 % with a standard deviation = 0.89, and the percent error did not exceed 2.49%.
- Produced alum has efficiency of removal 77.55% of total suspended solids and commercial alum has efficiency of removal 74.69%
- FTIR chart results show symmetrical results with the standard alum.

Conflicts of interest

There are no conflicts to declare.

Formatting of funding sources

There are no funding sources.

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