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BOX-BEHNKEN DESIGN OPTIMIZATION OF REMOVAL CRUDE OIL FROM WATEREMULSIONBY MOBIL COMPOSITION OF MATTER No. 41

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Abstract

The removal of crude oil pollutant from water emulsion was studied. Investigate of the potentiality Mobil Composition of Matter No. 41 (MCM-41 A) for removing crude oil from water emulsion. Batch experiments were carried out as a function of pH (4, 5 and 6), temperature(30, 40 and 50°C), different initial concentrations (50-200) ppm and weight of MCM-41 A(0.2-1 g). Effect of pH on crude oil remove by using MCM-41 Ashows the rate of crude oil remove was maximum atpH 5.The rate of crude oil remove was maximum attemperature 30° C and the percentage removal decreased with the increase in initial concentration. But uptake capacity increased with the increase initial concentration (49.8,96.8,144.2,190.3) mg/L. Turbidity decrease with the increasing weight of MCM-41 A. The value of turbidity was closely after ten hours and weight of MCM-41 A beyond(1g).The data obtained from the experiments conducted as per Design of Experiment (DOE), orthogonal second order design (Box-Behnken Design); response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing problems where several independent variables influence a dependent variable or response. The outcomes prove that this model can be used to optimize the removal of crude oil process in the presence of MCM-41 Awith R^2 81.24%.

Keywords: Batch Experiments, Crude Oil Removal, Mobil Composition Of Matter No. 41A,Orthogonal Second Order Design, Response Surface Methodology

Introduction

Oil spills, we usually think of oil tankers spilling their cargo in oceans or seas. However, oil spilled on land often reaches lakes, rivers, and wetlands, where it can also cause damage [1]. Lakes, rivers, and other inland bodies of water are called freshwater environments. When oil is spilled into an aquatic environment, it can harm organisms that live on or around the water surface and those that live under water [2]. Spilled oil can also damage parts of the food chain, including human food resources. The severity of the impact of an oil spill depends on a variety of factors, including characteristics of the oil itself. Natural conditions, such as water temperature and weather, also influence the behavior of oil in aquatic environments. Various types of habitats have differing sensitivities to oil spills as well[3].

Many researches had been forwarded towards organic sorbents for removing oil spills from the surface of salt water such as,Clays are the most popular materials, which are used as sorbents for oil spills. Clays such as kaolinite [4], bentonite [5]smectite[6] have been used. Also, removal of oil spills from salt water by magnesium, calcium carbonates and oxides [4] and hafnium oxide ceramics used to remove crude oil from crude oil-water emulsion [6].

Mobil Composition of Matter No. 41 (MCM-41) is a mesoporous material developed by the Mobil Oil Corporation. MCM-41 is part of a family of silicate and alum silicate solids.MCM-41 has a hierarchical structure that has, as its base, an ordered arrangement of cylindrical mesopores that range in diameter from 2nm to 6.5nm[7,8,9]. These independently adjustable mesopores form a unique, one-dimensional pore systemthat has sharp, well-defined pore distribution and large surface and pore volume[10].During the synthesis of MCM-41, surfactants (typically cetyltrimethylammonium bromide (CTAB)) are added to the synthesis solution. The surfactant initially forms rod-shaped micelles that eventually align together in hexagonal arrays. Silica species are added to cover the rods, and then calcination condenses the silanol groups, which bridges the silicon atoms with oxygen atoms. Ultimately, this organic template oxidizes and disappears, leaving behind fully-formed MCM-41[11]. Because there is no aluminum in the lattice framework, MCM-41 has no Bronsted acid centers and has an acidity analogous to amorphous alum silicates. The low level of cross-linking between silicate units and its thin walls mean MCM-41 is not hydrothermally stable.MCM-41 is widely used in catalytic cracking, separations, dye removal, and as an adsorbent[12] as seen in Table 1. Types Mobil Composition of Matter No. 41and Characterizations. Also, TEM Image of Material MCM-41 A as shown in Fig. 1.

The objective of this study to investigate of the potentiality MCM-41A for removing crude oil from wateremulsion.DOE method was used to optimize the parameters to remove the crude oil using the MCM-41 A.

MATERIALS AND METHODS

Mobil Composition of Matter No. 41 (MCM-41)

MCM-41 A supplied from ACS advance chemical supplier from market was used to remove crude oil.

Sampling

Crude oil samples were collected from the South of Iraq fields. It is a mixture of crude oils produced from nearly X- fields located at the Basrah fields. The tested crude oil according to the institute of petroleum (IP)test methods are listed in Table (2) (IP, 2001) [13,14].

Preparation of Crude Oil Stock Solution

The stock solution of crude oil was prepared by adding 1g of crude oil to double distilled autoclaved water and the volume was made up to 1000ml. The final concentration of the stock solution was 1000 ppm (1000 mg/L) and the stock solution was diluted to the required concentration for its use in the experiments. The stock solution was filter sterilized by passing it through a 0.25µM syringe filter.

pH

The studies of pH effect on crude oil removal was conducted by using analytical grade of 1 N hydrochloric acid (HCl) and 1 N Sulfuric acid (H_2SO_4) for 0.5litter of crude oil water concentration (100) ppm. Other parameters were kept constant, such as, temperature (30 ^oC), agitation speed (400 rpm) and dose MCM-41 A (1 g). The experiments were conducted with the jar test apparatus (PHIPPS & BIRD). pH measured using Orion pH meter. The pH was calibrated prior to analysis using standard buffer within the range of the pH to be measured Fig.2. Crude oil removal efficiency by using pH adjustment at pH (4,5 and 6) with HCl and H_2SO_4. There for pH was adjusted with 5 for all experiments by adding the 1N HCl[15].

Crude Oil Removal Studies

All removal experiments were performed in 250 ml Erlenmeyer flask containing crude oil at concentration ranging from 50 -200 ppm. Other parameters were kept constant, such as, temperature (30 ^oC), pH (5) agitation speed (400 rpm) and doseMCM-41 A (0.5 g), samples were withdrawn at regular time interval, centrifuged (10 ml for 3minutes) and analyzed for residual crude oil concentration. For each concentration triplicate experiments were performed under the same condition and mean value has been reported. Each experiment was carried out for until the residual concentration of crude oil in flask was found to saturate with time and amount of MCM-41 A it is below the detection limit.Crude oil removal percentage calculate by formula [16,17]:

Crude oil removal %=((Initial conc. of crude oil- Final conc. of crude oil)/Initial conc.of crude oil)× 100 (1)

Analytical Procedures

Measurements of crude oil were done by UV–vis spectrophotometer (Systronics**)** both in UV and visible range. The samples were centrifuged at approximately 5000 rpm for 25 min. The supernatant was used for crude oil determination. MCM-41 A attached to the walls of tubes was resuspended in distilled water and optical density of this suspension was measured against distilled water as reference at 600 nm using UV–vis double beam spectrophotometer. All the transfers were made in UV chamber, and glass wares and medium properly autoclaved. The batch experiments were repeated and the results were found reproducible within acceptable range [17,18].

Orthogonal Second Order Design (Box-Behnken Design)

Experimental design is the sequence of steps initially taken to ensure that the data will be obtained in such a way that its analysis will lead immediately to valid statistical inferences. The purpose of statistically designing an experiment is to collect the maximum amount of relevant information with a minimum expenditure of time and resources [19], and to find useful relationship between controllable variables and observed response. In the experimental design, the variables are usually called factors. The particular value of the variable is called the level of the factor. The combination of factors used in a particular experiment is called a treatment. The numerical result of a trial based on a given treatment is called the response corresponding to that treatment [20]. Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing problems where several independent variables influence a dependent variable or response, and the goal is to optimize this response. The independent variables are denoted by X_1, X_2, ……, X_k. It is assumed that these variables are continuous and controllable by the experimenter with negligible error.

The response which is the product (Y), is assumed to be a random variable. It is required to find the effect of pH (X_1), temperature (X_2) and initial concentration(X_3) that maximize the yield of the removal process. The observed response (Y) may be expressed as a function of the levels of pH, temperature and initial concentration as below:

$$Y_b = f(X_1, X_2, X_3) + Error \tag{2}$$

In most RSM problems, the form of the relation between the response and the independent variables is unknown. Thus, the first step in RSM is to find a suitable approximation for f (X_1 , X_2 , X_3). Usually, a low order polynomial in some region of the independent variables is employed. If the response is well- modeled by a linear function of the independent variables, then the approximating function is the first order model;

$$Y_b = B_o + B_1X_1 + B_2X_2 + \ldots\ldots\ldots\ldots + B_k X_k + Error \tag{3}$$

If there is a curvature in the system, the polynomial of higher degree, such as the second order model must be used;

$$Y_b = B_\circ + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} {X_i}^2 + \sum_{i<j}^{k} \sum_{j}^{k} B_{ij} X_i X_j \tag{4}$$

Almost all RSM problems utilize one or both of these approximation polynomials. The RSM analysis then done in terms of the fitted surface. If the fitted surface is an adequate approximation of f (X_1, X_2, X_3) parameters and actual value are listed in Table 3. The analysis of the fitted surface will be approximately equivalent to the analysis of the actual system [16].The coded, real variables and response of experiments for crude oil are listed in Table 4.

In order to define the relationship in between the operating parameters and responses of the model, second degree equation in Eq. (5) was used.

$$Y_b = B_\circ + B_1X_1 + B_2X_2 + B_3X_3 + B_{11}{X_1}^2 + B_{22}{X_2}^2 + B_{33}{X_3}^2 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{23}X_2X_3 \quad (5)$$

$B_\circ$ indicates the constant of the model, B_i indicates the linear coefficients, B_{ii} indicates the quadratic coefficients, B_{ij}indicates the interaction coefficients.

The regression coefficients are determined as follows [19]:

$$B_i = \frac{\sum_1^{10} X_{iu}Y_u}{\sum_1^{15} {X_{iu}}^2}$$

$$B_{ij} = \frac{\sum_1^{8} X_{iu}X_{ju}Y_u}{\sum_1^{15} (X_{iu}X_{ju})^2}$$

$$B_{ii} = \frac{\sum_1^{15} {X_{iu}}^2 Y_u}{\sum_1^{15} (X_{iu}')^2}$$

$$(X_{iu}')^2 = {X_{iu}}^2 - \frac{\sum_1^{15} {X_{iu}}^2}{N} \quad (6)$$

RESULTS AND DISCUSSION

Effect of pH on crude oil removal percentage

The experimental were done under the conditions of constant temperature (30 °C), constant time (1h), MCM-41 A amount (0.5g), agitation speed (400 rpm) and initial concentrations were kept at 50 ppm for crude oil. While different pH (4, 5 and 6). The experimental data are presented in Fig. 3. It was found that the removal of crude oil decrease with the increase in pH.It may be due to the environment changed to close to the isoelectric point (iep), the result in zeta potential was nearly zero, and then the demulsification was occurred. The separation of crude oil droplet could be observed obviously when pH was lower than 6[21,22]. This phenomenon can be demonstrated as follows: MCM-41 A has active sites with negative charge while the crude oil has active sites with a positive charge at low pH. The increase in pH causes increase in the concentration of hydroxyl ion (OH^-) negative charge which increases the hindrance for diffusion of crude oil and MCM-41 A charge and thus reduces the removal percent of crude oil. Therefore, the initial pH= 5 is the best value and will be selected as for the next experiments to removal crude oil from waste water by usingMCM-41 A. This agree with a researchers have been reported the pH could affect the oil emulsion in the wastewater to enhance the grease and oil removal efficiency, the pH of wastewater should be adjusted to be low[15].

Effect of temperature on crude oil removal percentage

Effect of temperature on removal of crude oil is studied by conducting different sets of experiments at different temperatures (30, 40and 50°C), while the other operational parameters were kept constant at initial concentration (50ppm), pH (5)agitation speed (400 rpm), amountMCM-41 A (0.5g) and contact time (1h). Fig.4.Shows that the removal percentage decreasing with temperature increase that be attributed to the possible damage to active sites in MCM-41 A at higher temperatures.Increasing removal at 50°C could be attribute to the types of removal where the quantity ofmaterial physically MCM-41 A increases as the temperature decreases, while the quantity of crude oil on surface increases with increased temperature. The same behavior has been proven by researches concluded the optimum temperature range of the used materials to remove oilspills was (20 - 40°C) indicating that the removal process depends on the temperature[3].

Effect of Initial Concentration on crude oil removal percentage

Effect of initial concentration on crude oil was investigated by conducting different sets of experiments at different initial concentrations (50-200) ppm and contact time (2-10) hours. Other parameters were kept constant, such as, temperature (30 °C), pH (5) agitation speed (400 rpm) and dose MCM-41 A (0.5 g).The removal versus time were ploted Fig. 5.It was found that the percentage removal decreased with the increase in initial concentration of the crude oil . But the uptake capacity increased with the increase in initial concentration (49.8, 96.8, 144 and 190.2)ppm respectivliy .The higher initial removal concentration provided higher driving force to overcome mass transfer resistances of the crude oil from the aqueous to the solid phase resulting in higher probability of collision between crude oil and active sites in MCM-41A .This result improves the fact that the removal percent of crude oil is stronglydependent on the initial concentration of crude oil[3,17].

Effect of Weight HMM and Time on Turbidity Removals

Oakton instruments WD-35635-05Eutech TN-100 turbidity meter 0 to 1000NTU used to find turbidity removal.The removal of crude oil increases with increasing contact time before equilibrium is reached.Turbidity removals by different weight MCM-41 A (0.2-1) g in function of time (2-10) hours were kept constant at initial concentration (150 ppm), at temperature (30°C), pH (5) and agitation speed (400 rpm) is illustrated in Fig. 6. It could be noted that when the turbidity of water emulsion was less than 14 NTU then the crude oil was less than 140 mg/L, so it was still some crude oil emulsified in water even the crude oil removal was over 93.33%.With the reason of residual oil remained and problem of the combine sediment and float of floc, the coagulation with normally used as a coagulant aid was also studied to enhance crude oil removal efficiency. The result was corresponded the turbidity decrease with the increasing weight of MCM-41 A as showed in Figure 6. The value of turbidity was closely after ten hours and weight of MCM-41 A beyond 1g. The turbidity value varies with the colloid concentration in water, it could be used as the indicator of crude oil residual in water [23,15,24].

Orthogonal Second Order Design (Box-Behnken Design)

This research methodology is termedas DOE that attempts to extract maximum information with minimum number of experiments. The data were analyzed byusing Minitab 16, a total of fifteen runs were designed based on BBD response surface methodology (RSM) and conducted to examine the effects of pH, temperature and initial concertation. The empirical outcomes were later used to develop an equation based on quadratic model in order to establish the correlation between independent and dependent variables. The independent variables in this study are pH (X_1), Temp. (°C) (X_2) and initial concertation (ppm) (X_3), whereas the predicted response of removal efficiency is designated as Y[16,20]

The equation was validated confirmatory experiments were conducted for verification based on the predicted optimal parameter values. The equation of BBD model, obtained for crude oil removal from water emulsion byMCM-41 A, is given in Eq. (7).

$$Y=76.2- 22.6 X_1 - 27.3 X_2 + 11.0 X_3 + 28.3 X_1*X_1 + 19.3 X_2*X_2 + 6.2 X_3*X_3+ 8.7 X_1*X_2+ 1.4 X_1*X_3- 61.9 X_2*X_3 \quad (7)$$

It is being seen from Eq.7and Table 5the positive state of the coefficients indicates the obvious effect of the relevant parameters, and the negative state of the coefficients indicates the aggressive effectof the relevant parameters. The regression model is adequate with 81.24%.

The data estimated by the assistance of the model and the experimental data are in matching as seen from Fig.7and Table 4. The model versus actual value plot approximates along a straight line implying that the second-order regression model was satisfactory with R^2 99.56 %.

CONCLUSION

The results indicated that MCM-41 A had proved to be an efficient for the removal of crude oil from water

emulsion with low concentration. Optimal conditions in terms of pH and temperature 5, 30 ^{o}C respectively.

50 ppm initial concentration crude oil has been found better removal efficiency, while increase in the concentration of the crude oil in the medium, the decreases which owes to the toxic MCM-41 A. Turbidity decrease with the increasing weight of MCM-41 A.A statistical model was developed to describe the efficiency of crude oil removal by using the BBD response surface methodology (RSM).The predicted model explains good correlation between the values of experimental and predicted responses.

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NOMENCLATURES

Table 1

Symbol	**Description**	**Units**
B_i,B_ii,B_ij	Regression Coefficients	-
Error	Random experimental error	-
MCM-41 A	Mobil composition of matter	g
i	Initial value	-
k	Total number of independent variables	-
rpm	Rotation per minute	Cycle/min.
0	Null points	-
X	Coded variable	-
X_1	First coded variable	_
X_2	Second coded variable	$°C$
X_3	Third coded variable	mg/L
X_K	Coded variable number K	-
Y_b	Calculated value of concentration	mg/L
Y_u	Observed value	mg/L

Figure 1: TEM Image of ACS Material MCM-41 (Type A)a-100 nm, b-50nm and c-0.5 nm[10]

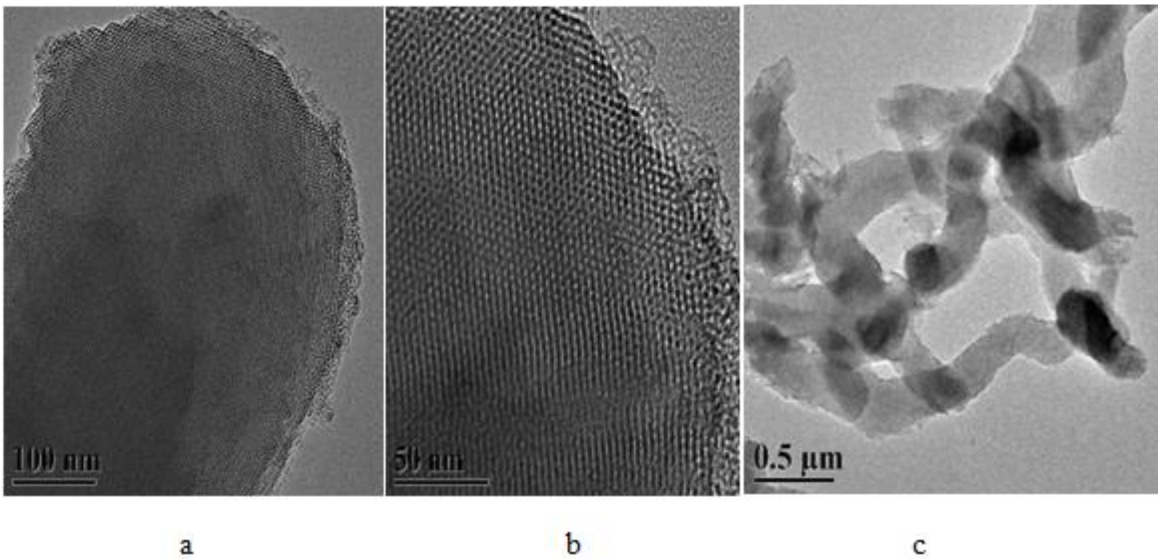

Figure 2: Crude oil removal efficiency by using pH adjustment at pH (4,5 and 6) with HCl and H_2SO_4

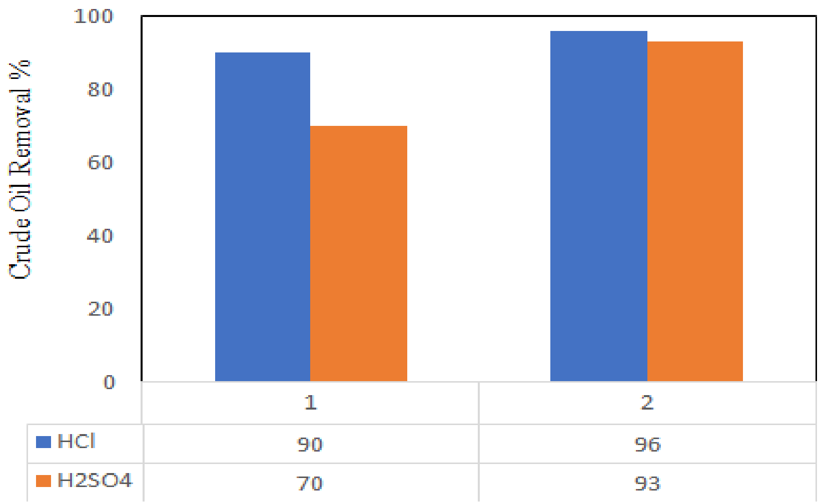

Figure 3: Effect of pH on crude oil removal percentage T=30 °C, time =1h, MCM-41 A amount (0.5g), agitation speed (400 rpm) and initial concentrations for crude oil = 50 ppm. While different pH (4, 5 and 6).

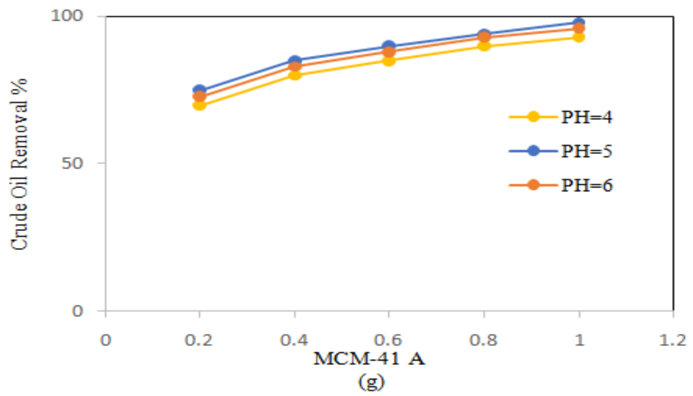

Figure 4: Effect of temperatures on crude oil removal percentage pH =5, time =1h, MCM-41 Aamount MCM-41 (0.5g), agitation speed and initial concentrations for crude oil = 50 ppm. While different temperature (30, 40 and 50°C)

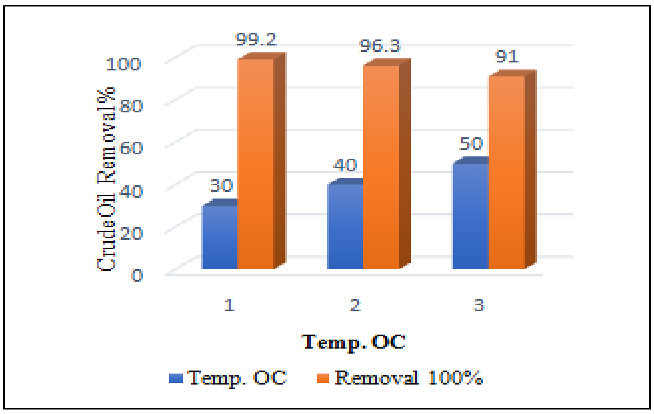

Figure 5: Effect of Initial Concentrationon crude oil removal percentage pH =5, temperature =30°C, MCM-41 A amount (0.5g), agitation speed (400 rpm). While different initial concentrations for crude oil (50-200) ppm and time (2-10) hours

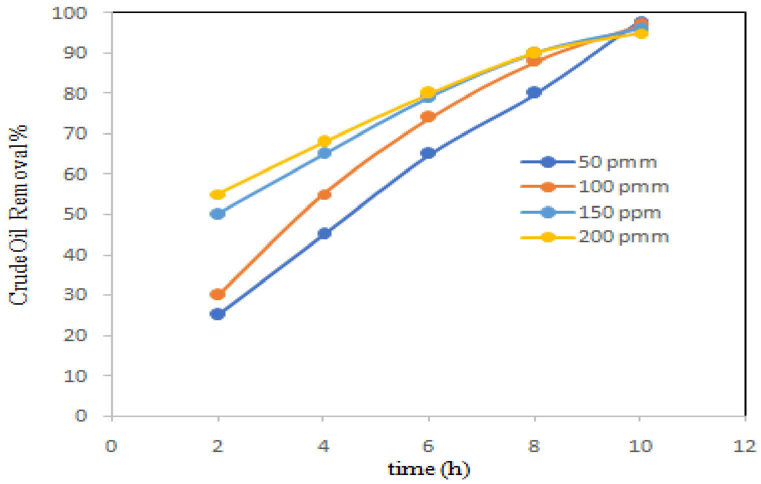

Figure 6:Turbidity removals by different weight MCM-41 A (0.2-1) g in function of time (2-10) hours were kept constant at initial concentration (150 ppm), at temperature (30°C), pH (5) and agitation speed (400 rpm).

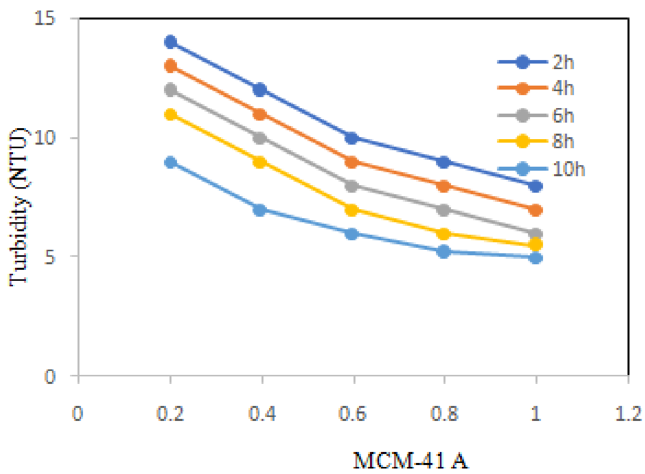

Figure 7: The theory values vs. the experiment value

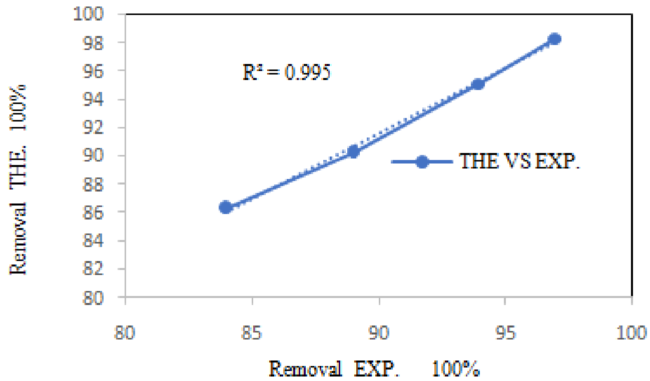

Table 1: Types Mobil Composition of Matter No. 41and Characterizations [10]

Type	**A**	**B**
Grade	Reagent Grade	Industrial Grade

Appearance	White Powder	White Powder
Particle Size	100-1000 nm	200-1000 nm
Average Pore Diameter	3.4 nm	3.5-4.0 nm
BET surface area (m^2/g)	>850	>800
Pore Volume(cm^3/g)	≥0.75	≥0.75
Na_2O	≤0.5%	≤0.5%

Table 2:Physical properties of crude oil

Test	**Units**	**Test method**	**Results**
Density at 15°C	Kg /L	IP 160	0.86
Water	%Volume	IP 74	.4
Sediment	% mass	IP 53	NIL
Pour point	°C	IP 15	-4
Salt	% mass	IP 77	0.005
Viscosity Redwood at 37.8°C Sec.	Sec.	IP 212	32
Sulfur	% mass	IP 336	1.5

Table 3: Parameters and actual value

Parameter level	**Low**	**Center**		**High**
	-1	0		+1
Parameters	Symbol	Actual Value		
pH	X_1	4	5	6
Temp. (°C)	X_2	30	40	50
C_i (ppm)	X_3	50	100	150

Table 4:The coded real variables and response of experiments for crude oil.

No.	**X_1**	**X_2**	**X_3**	**x_1=pH**	**x_2=Temp. (°C)**	**x_3= initial concentration (ppm)**	**Y_u**
1	0	1	1	4	30	100	98.0
2	0	-1	1	5	30	100	144.0
3	0	0	0	4	50	100	75.0
4	0	-1	-1	5	50	100	126.0
5	-1	-1	0	4	50	50	45.5
6	0	0	0	5	40	50	90.0
7	1	-1	0	4	40	100	16.5
8	0	0	0	5	40	100	63.5
9	0	1	-1	5	40	50	135.0
10	-1	1	0	5	30	50	146.0
11	1	0	-1	5	50	100	32.0

12	1	0	1	5	50	100	63.0
13	1	1	0	5	50	150	106.5
14	-1	0	1	5	50	150	147.0
15	-1	0	-1	5	50	150	147.0

Table 5:Coded coefficients

Term	**Coef**	**SE Coef**	**T-Value**	**P-Value**
Constant	76.2	34.0	2.24	0.075
X1	-22.6	20.8	-1.08	0.328
X2	-27.3	20.8	-1.31	0.247
X3	11.0	20.8	0.53	0.620
X1*X1	28.3	30.7	0.92	0.399
X2*X2	19.3	30.7	0.63	0.557
X3*X3	6.2	30.7	0.20	0.849
X1*X2	8.7	29.5	0.30	0.778
X1*X3	1.4	29.5	0.05	0.965
X2*X3	-61.9	29.5	-2.10	0.090