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ORIGINAL RESEARCH PAPER

Recycling process of spent bleaching clay: Optimization by response surface methodology

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ABSTRACT: Oil refining is an inevitable step in production of edible and industrial oil. Bleaching is the most important process among the refining processes. Bleaching adsorption is the most common method and clay is the most widely used adsorbent in this method. Disposal of bleaching clay, as a waste from re-refining plants, makes many environmental problems and economic losses. In the current study, the effects of possible factors such as solvent to clay ratio, temperature, time, aggregation size and rotation speed of the stirrer (degree of mixing) on the efficiency of extracted lubricating oil were investigated by solvent extraction method. By conducting experiments at different reaction times and rotation speeds, it was concluded that the most important factor in obtaining the appropriate output was solvent to clay ratio. The tests conducted to investigate the effect of grain size on the efficiency indicated that agglomerates size did not have a positive effect on efficiency. Finally, for the solvent to clay ratios ranging from 2.48-9.53 ml/g and a time period ranging from 5 to 40 minutes, the main tests designed by the response surface methodology. The best efficiency was obtained at the highest level of solvent to clay ratio (9.53 ml/g) and at the time of 22.5 minutes that led to 88.60% oil extraction from the clay. The accuracy of the model output was estimated to be 96%.

KEYWORDS: *Bentonite; Bleaching clay; Methyl ethyl ketone (MEK); Used lubricating oil (ULO); Response surface methodology (RSM); Solvent extraction.*

INTRODUCTION

Motor oil constitutes the major part of the car oil, as well as 42% of the world's consumed lubricating oil (European Commission, 2001). Used lubricating oil (ULO) is among those difficult-to-handle hazardous wastes due to its toxicity and environmental consequences. The current production rate of ULO in Iran is approximately 300,000-350,000 ton/year. This amount of production emphasizes the significance of applying effective and practical methods to deal with this issue, especially in re-refining processes. In fact, the total cost and energy required to produce one liter of motor oil through refining is considerably less than that of crude oil. In return, it offers opportunities for remarkable raw material preservation and energy recovery. Acid/clay treatment, one of the earliest techniques in re-refining ULO, is the main process implemented in Iran. Waste oil has a great tendency to bind to clay particles (Pelitli *et al*., 2017). Considerable problems associated with this process are low production yield, incomplete removal of heavy metals especially lead, and excessive amount of acid sludge generated. Due to the lubricant boiling range and the oil spoiled during bleaching procedure, this process yields to a low order of efficiency (60-70%) and production of large amount of acid sludge (Jafari and Hassanpour, 2015). Disposal of this waste, in an environmentally acceptable manner, is difficult and expensive (Awaja and Pavel, 2006; Mortier *et al*.,

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2010). Though this method is completely banned in developed countries, in developing countries such as Iran, it is the oldest and the most predominant method for treatment of used motor oil. It should be noted that acid/clay is accepted as an unacceptable method regarding environmental aspects (European Commission, 2001). Clay is suitable for bleaching or decolorizing applications (Komadel, 2016). In the bleaching process of used-oil refining, absorbents such as natural bleaching clay, activated bleaching clay, activated carbon and amorphous silicates are used (Ismadji, *et al.*, 2015). Mostly clay-bentonite type, which has a high bleaching potential, is used to remove the smell and the color of the product (Yildiz *et al*., 2004). Spent bleaching clay (SBC) which is the output of bleaching process, as a solid waste, contains 20-40% engine oil in its weight and it is spontaneously flammable (Beshara, 2014). In each cycle of oil treatment, the output SBC constitutes 10-15% of oil weight (Eliche-Quesadaa and Corpas-Iglesiasb, 2014; Tang *et al.*, 2015). There is no official report about the amount of SBC in used motor oil recycling industries in Iran. According to unofficial reports, this amount is approximately 300,000 tons per year in lubricating oil refining processes. There are several ways to regenerate oily clay. Among these ways, solvent extraction can be pointed out as one of the experiments carried out in this field (Al-Zahrani and Alhamed, 2000; Al-Zahrani and Daous, 2000; Foletto *et al.*, 2002; Nursulihatimarsyila *et al.*, 2004). In other cases, acid activation method has been used for clay recovery (Kheok and Lim, 1982; Yildiz *et al.*, 2004; Meesuk and Vorasith, 2006; Sarioglan *et al.*, 2010). In addition to these methods, others used thermal recovery along with solvent extraction (Al-Zahrani and Alhamed, 2002). Also, some researchers used acid activation method and thermal recovery together (Lee *et al.*, 2000; Boukerroui and Ouali, 2000; Wambu *et al.*, 2011). A few have applied pyrolysis technique - a kind of thermal recovery - to regenerate oily clay (Tsai *et al*., 2002) and some have used lye-extraction method (James *et al*., 2006; Al-Zahrani and Putra, 2013). Moreover, researchers have carried out various methods, examining each for a particular purpose to regenerate oily clay. Some researchers selected four safe solvents (methyl ethyl ketone, acetone, petroleum ether and n-hexane) from ten solvents and carried out solvent extraction tests on a clay containing corn oil (Mana *et al*., 2007; Geleel *et al*., 2003; Tsai *et al*., 2003). Results showed that methyl ethyl ketone (MEK) had the highest efficiency (77%) among them (Al-Zahrani and Alhamed, 2002). The optimum solvent to clay ratio (SCR) was 4-5, and mixing was conducted in a range of 150-200 rpm for 5 minutes. In a similar study, percentage of oil extraction for MEK was 72% at the best situation (Al-Zahrani and Daous, 2000). Using solvent extraction of clay in soybean oil bleaching industry, MEK was introduced as the best solution in this field (Foletto *et al*., 2002). Analyzing the extracted palm oil using different solvents (methanol, ethanol, petroleum ether, pentane, hexane, and heptane), revealed that the quality of all output oils was too low to be reused. Although the aforesaid management of SBC is attempted in edible oil industry, no particular case is mentioned for the output SBC from the motor refining industry (Lee *et al.*, 2010). All the previous studies have focused on the edible oil. All tests have been done using the traditional methods and no modeling or DOE (Design of Experiments) has been used except in one article (James *et al.*, 2006). Experiments in this study are focused on motor oil and designed based on solvent extraction method using response surface methodology (RSM). Extraction time (ET) and SCR are two important parameters studied in these experiments. Unlike edible oil, the obtained oil is capable to be returned to the cycle of oil production. The recovered clay can be used as filler in asphalt, bituminous mixture and cement industry. Otherwise, it can be retrieved in an appropriate percentage through further processing (Sangiorgi *et al.*, 2014; Liew *et al.*, 2016; Sangiorgi *et al.*, 2016). This study has been carried out in Environmental laboratory of K. N. Toosi University during 2015.

MATERIALS AND METHODS

The SBC samples were collected from a local ULO re-refining factory in Iran. They were collected from cake filters and then screened to obtain a uniform grain size distribution. The clay being used in this factory contains 20-40% oil in its weight, and it is completely black. The type of used bleaching soil is natural calcium bentonite clay. In the mentioned factory, the annual SBC production is about 3,700 ton/year, and 10-15% of the total product is light oil which has been neglected in this study. The MEK solvent used for extraction was obtained from MERCK Chemicals Company, Germany. All the tests were performed at atmospheric pressure and room temperature.

Oil content mass balance

The oil content determination was done on a 10 g sample, and the results were averaged. For this purpose, the Soxhlet extractor with a 500-ml flask was used. A 10-g sample of SBC was extracted with 150 ml of boiling MEK and the extraction process was stopped after 40 siphons. Then, the oil was separated from the solvent by a rotary vacuum evaporator and subsequently dried in an oven at 110˚C to remove any possible moisture. Additionally, to establish a mass balance for the oil loss of clay, the final sample saturated with solvent was dried in an oven at 110˚C. Eventually, the cooled samples (oil and clay) were weighed, and the results were compared. The oil obtained in rotary process and the clay weight loss due to oil extraction was 3.62 g and 3.36 g, respectively. Considering 10-g primary sample, the mean oil content of 35% was determined for further calculations. For the extraction experiments, different samples of equal weights of 10 g of SBC were extracted by MEK organic solvent in order to investigate the effective factors. In all experiments, first, 10-g samples of bentonite with specific amounts of MEK, namely 2,4, 6 and 8 ml/g with respect to the amount of clay (SCR), were mixed on a magnetic stirrer for 10, 20 and 30 minutes. The experiments were performed in 400-ml beakers to avoid splashing of the mixture. The 10-g samples were placed in the beaker and the required amount of solvent was added to each one. The mixture was stirred by a magnetic stirrer with the speeds of 100, 200, 400 and 500 rpm. Eventually, after mixing and separating the solid phase (clay and residual oil and the trapped MEK) and liquid (oil and MEK) by filter paper, solid phase was heated at 100°C temperature for 1 hour in an oven. After evaporation of trapped MEK in clay, the final sample was weighted and the differential weight of the primary sample (10 g) and the secondary sample showed the amount of extracted oil. Finally, efficiency of the solvent extraction process was obtained for each test based on Eq. 1.

$$Efficiancy\ (\%) = \frac{W_E}{W_S} \times 100\ \% = \frac{3.5 - oil\ removal}{3.5} \times 100\% \quad (1)$$

Where, W_E and W_S are the weights of oil extracted in the experiment and in the Soxhlet extractor, respectively.

Pre-experiments

As a procedure to find the effective parameters and their optimum ranges of extraction, the traditional method of one-factor-at-a-time was employed as pre-experiments. In this method, a variable is changed whereas the others are kept constant. This process is repeated for all the variables and in each case the optimum value of the variable is determined. Even though this procedure is assumed to be weak in considering the interaction of variables, it is used widely as a primary evaluation for the determination of the optimum variation ranges (Al-Zahrani and Alhamed, 2000; Al-Zahrani and Daous, 2000; Al-Zahrani and Putra, 2013).

Clay grain size

One of the influential factors during adsorption is particle size (Mittal *et al*., 2009; Joshaghani *et al*., 2014; Khamparia, and Jaspal, 2016). Two series of tests were conducted with SCRs of 2 and 4 ml/g to study the effect of grain size on the efficiency. The results are presented in Tables 1 and 2. As it is observed, efficiency of the non-grained samples is higher. Therefore, grain size is not required to achieve a better efficiency.

Table 1: Efficiency of grained samples in SCR of 2

Grain size condition	Efficiency (%)	Oil loss (g)	Secondary weight (g)	Primary weight (g)
Random (mixed)	38.41	1.48	9.53	11.01
< 2mm	35.75	1.25	8.74	9.99
> 2mm	36.92	1.30	8.76	10.06

Table 2: Efficiency of grained samples in SCR of 4

Grain size condition	Efficiency (%)	Oil loss (g)	Secondary weight (g)	Primary weight (g)
Random (mixed)	65.65	2.30	7.71	10.01
< 2mm	55.55	1.95	8.08	10.03
> 2mm	64.44	2.26	7.76	10.02

Stirring speed

Since the tests with mixing speeds of 100 and 200 rpm (low speed) did not result in an appropriate efficiency, the remained pre-experiments were only carried out in the mixing rates of 400 and 500 rpm.

SCR and extraction time

In order to select a proper range of SCR, a number of pre-tests were conducted in different SCRs from 2 to 8 (2, 4, 6 and 8) with mixing speeds of 400 and 500 rpm. The obtained efficiencies are shown in Table 3. As it is shown, efficiency is enhanced by increasing SCR, while it does not change in constant SCR. As a result, SCR is expected to be the most effective parameter in main tests, as well. Notably the maximum efficiency was achieved in 20 minutes.

Design of experiments by RSM method

RSM is an effective statistical application that helps in analyzing the interaction between different parameters, such as time, cost and process variability percentage. Hence, providing an optimal global solution would result in overall refinement of the process (Khamparia and Jaspal, 2017).

The two most significant factors, i.e. SCR and ET, and their ranges were selected. Table 4 shows the coded values and variables used in the experiments. Based on Eq. 1, the efficiency parameter was selected for the response. The design of experiments includes 22 runs (lower and upper limits of variables), 2×2 runs at extreme points and four replicates at the design center resulting in 12 experiments. The mathematical model of the response (Y) was obtained by applying a quadratic model to the variables (X) in Eq. 2, which is appropriate for prediction of the response in the experimental range (Montgomery, 2009; Myers and Montgomery, 2009).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i}^{k} \sum_{j}^{k} \beta_{ij} X_i X_j \quad (2)$$

Where, Y is the response, β_0 is the constant, β_i, β_{ii} and β_{ij} are the coefficients of linear, quadratic and interaction effects, respectively. X_i is the value of the factor i and k is the number of variables. In this study, the design of experiments and data analysis were performed using central composite design (CCD). Use of CCD enabled to study the most efficient parameters affecting the process and cooperated in evaluating the possible interactions between the two parameters during the process (Khamparia and Jaspal, 2017). The final results were analyzed by Minitab Software through analysis of variance (ANOVA). The capability of the model in fitting was evaluated by coefficients of determination ($R^2$and adjusted R^2). The selected set of variables was chosen based on the probability value (P-value) with 95% confidence level. The prediction capability of the model was studied at R^2 prediction coefficient which was calculated by predicted residual error sum of squares (Sabour and Amiri, 2017; Antony, 2003; Bezerrab *et al.*, 2008). The response surfaces and contour plots, depicted by Minitab, were used to demonstrate the interaction of variables. In the main experiments, tests were carried out with non-graded samples, at 400 rpm speed of stirrer and at room temperature (25°C).

Table 3: Efficiencies obtained in different SCRs and ETs

SCR	ET (min)	Stirring speed (rpm)	Efficiency (%)
2	10	400	31.31
2	10	500	26.73
2	20	400	29.58
2	20	500	30.10
4	10	400	65.65
4	10	500	66.18
4	20	400	66.62
4	20	500	69.90
6	10	400	73.83
6	20	400	74.36
6	20	500	81.74
6	30	500	71.29
8	10	400	83.83

RESULTS AND DISCUSSION

Among twelve main tests presented in Table 5, the best output (efficiency) corresponds to the highest SCR in 22.5 min extraction time. Improving the SCR, oil recovery efficiency is also increased. The increase of the efficiency also appears in the final color of the

Table 4: Coded levels and variables in the main experiments

Variables (unit)	Symbol	Coded values				
		-1.414	-1	0	+1	+1.414
SCR (ml/g)	X_1	2.48	3.5	6	8.5	9.53
ET (min)	X_2	5	10	22.5	35	40

clay, in a way that in tests with high efficiencies, clay color changes from black to light gray. As can be seen, in similar tests in the central point, the obtained efficiencies are very close to each other, showing the high accuracy of the performed tests. An important point is the considerable effect of SCR compared to ET.

Conducting the main tests and obtaining the test results, the experiments have been investigated by Minitab software. Then, Eq. 3 is derived considering significant terms. As it is observed, the coefficient of SCR in this equation is much greater than other coefficients, showing the high effect of this parameter on the final response. On the other hand, the negative coefficient of the extraction time shows that longer extraction (after 22.5 minutes) has no desirable effect on the final efficiency.

$$\text{Efficiency (\%)} = 75.37 + 11.27x_1 - 1.14x_2 - 2.00\,x_1^2 - 1.65x_2^2 + 0.17\,x_1x_2 \quad (3)$$

Analysis of the proposed model is presented in Table 6. P-values of the equation terms are given in the right column. The lower the P-value term is, the more influence on the response of the output is seen. As shown in Table 6, all the P-values are less than the amounts specified as proper limits (i.e., when P-value <0.05) and this indicates the high effect of X_1, X_2, X_{12} and X_{22} terms on the final efficiency. Consequently, in the obtained equation, β_{12} coefficient was low and had a slight effect on the output efficiency, but the effect of other terms was more significant. For the lack of fit, P-value of greater than 0.05 (here 0.498) shows the adequacy of the model (Sabour and Amiri, 2017). Referring to Table 6, the P-value for $F_0 = 167.66$ is remarkably less than 0.05 (i.e., <0.0001), implying the importance of the terms within the model. Furthermore, the F-statistics derived from testing linear or quadratic terms contribution (391.62 and 18.36, respectively) indicates that both of them participate significantly in the model. The ANOVA results of Table 6 present the high R^2 (99.29%) and adjusted R^2 (98.70%), ensuring the quadratic model to be acceptably adjusted to the experimental data (Wu *et al.*, 2010). Hence when R^2 and adjusted R^2 have a considerable difference, there is a chance of insignificant terms being included in the model, therefore such insignificant terms must be eliminated (Myers and Montgomery, 2009). As far as the values are close together in the study, one can prove that the above possibility is invalid. Proximity of the three values reported in Table 6 indicates the high accuracy of the fitted model.

Table 5: The design values, experimental variables and observed responses

Number	Variables				Response
	X_1		X_2		Y
	Coded	Value	Coded	Value	%
1	-1	3.5	-1	10	54.91
2	+1	8.5	-1	10	86.97
3	-1	3.5	+1	35	49.86
4	+1	8.5	+1	35	83.31
5	-1.414	2.48	0	22.5	44.77
6	+1.414	9.53	0	22.5	88.60
7	0	6	-1.414	5	69.58
8	0	6	+1.414	40	66.64
9	0	6	0	22.5	73.07
10	0	6	0	22.5	75.77
11	0	6	0	22.5	75.79
12	0	6	0	22.5	76.86

Table 6: Analysis of variance for the proposed model

Source of variation		Sum of squares	Degree of freedom	Mean square	F_0	P-Value
Regression		2196.77	5	439.35	167.66	<0.001
SSR (β1, β2, | β0)		(2052.43)	(2)	(1026.22)	(391.62)	< 0.001
SSR (β11, β22, β12, | β0, β1, β2)		(144.34)	(3)	(48.11)	(18.36)	0.001
Residual		15.72	6	2.62		
Lack of Fit		7.88	3	2.63	1.01	0.498
Pure Error		7.84	3	2.61		
Total		2212.49	11			
R^2= 0.993	Adj. R^2= 0.987	Pred. R^2 = 0.968	PRESS = 69.9858			

On the other hand, comparison of the observed experimented values and the model-predicted ones in the plot shows that there is an acceptable agreement between the observed data and the values derived from the model (Figs. 1 and 2).

Evaluation of the model efficiency

The amount of extracted oil increases by increasing the SCR. This means that efficiency improvement continues by increase of SCR until the last test. However, in high values of SCR, the rate of efficiency enhancement decreases and it is expected that by continuing the tests (enhancing solution saturation of the solvent, MEK), the resulting output reaches a constant level. It seems that efficiency increase continues until the solvent (MEK) covers almost the whole surface of clay particles. Afterwards, SCR increase has no considerable effect on final efficiency. Furthermore, SCR increase up to covering the whole surface of all particles is not an economical treatment, knowing that an acceptable efficiency occurs when the increasing rate starts to decrease. This means there is no need to add excessive MEK. Fig. 3 indicates the contour plot of the model predicted by the software. The X_1 axis is the coded SCR and the X_2 axis shows the coded ET. As Fig. 3 depicts, nearly half of the zone is constituted by more than %75 efficiency. In addition, it can be seen that the best possible time duration for solvent extraction process in this domain of experiments (5-40 minutes) is about 20 minutes. The results show that by increasing the amount of the ET (by a certain amount), the process helps the clay particles to be more separated in a constant SCR. This separation causes greater clay particles area to be exposed to the solvent to improve oil adsorption and extraction by the solvent. From this time, with an increase in extraction time, the soil particles stuck together again, making some amount of oil trapped

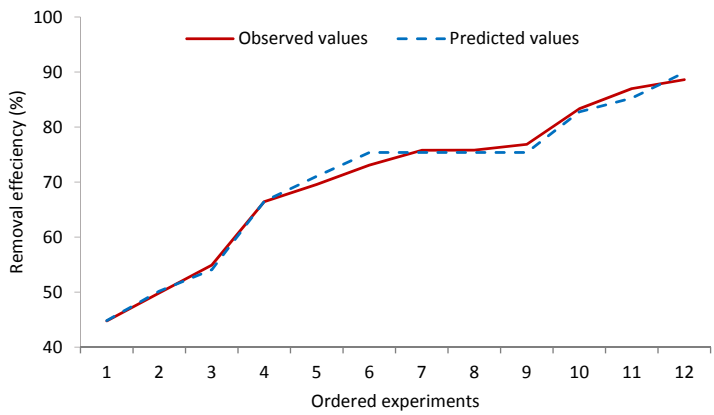

Fig. 1: Comparison of the observed experimented values and the model-predicted ones

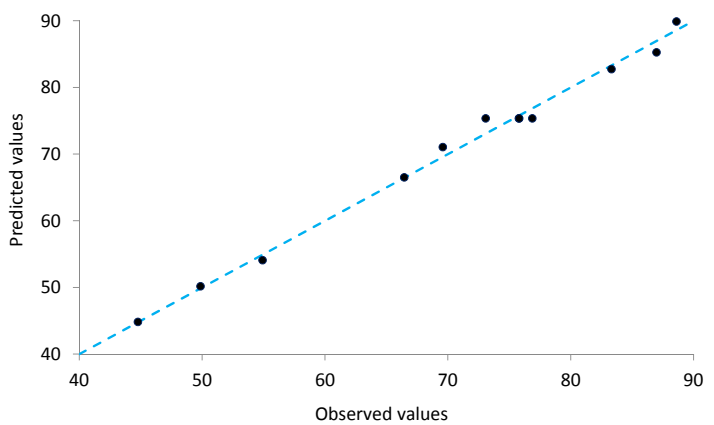

Fig. 2: Predicted values vs. experimental values plot for extraction efficiency

in. This trapped oil is neither mixed with MEK, nor extracted by the solvent anymore.

Fig. 4 illustrates the response of this model. In order to have a better understanding, Fig. 4 is presented in two directions. In both, the vertical axis is the percentage of the efficiency, and the X_1 and X_2 axes are the SCR and the ET, respectively. As can be seen, the highest efficiency is obtained by the maximum SCR (9.53 ml/g) and the average ET (22.5 min).

Verification of the model

Four experiments with efficiencies of more than 85% were selected based on the proposed model. These four points were the best samples of optimized experiments. The real values (not-coded) of parameters for these tests are given in Table 7.

Finally, by using the results obtained from Table 7, two experiments were carried out. Then efficiencies of experiments and the obtained contour graph were

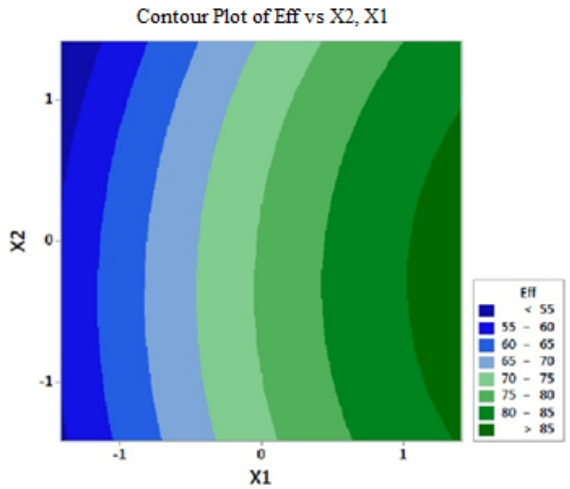

Fig. 3: Contour plot of efficiency in terms of X1 and X2

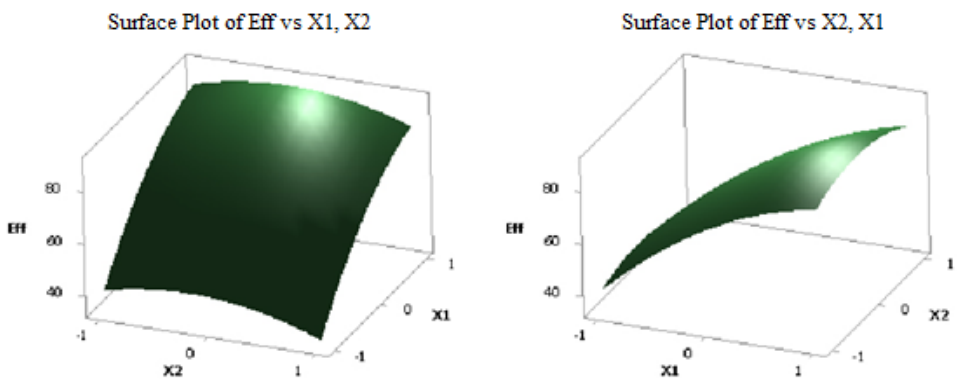

Fig. 4: Surface plot of efficiency

Table 7: Four verification tests for the fitted model

Sample	SCR		ET		Efficiency (%)
	Coded	Value (mg/l)	Coded	Value (min)	
1	1.388	9.46	-0.245	19.47	87.28
2	1.040	8.6	-0.246	19.46	85.07
3	1.274	9.18	0.262	25.74	86.13
4	1.284	9.20	-0.853	11.94	86.13

Table 8: Comparison of the efficiencies of experiments in the fitted model

Sample	Optimum Condition		Efficiency (%)		Error (%)
	SCR (mg/l)	ET (min)	Observed	Predicted	
1	9.46	19.47	87.29	87.11	0.18
2	8.6	19.46	85.07	85.16	-0.09

compared in Table 8. Due to the proximity of both results and parameters, the two cases with minimum and maximum efficiencies - with less than 2% difference – were sufficient. Considering the very low error calculated, the model verified successfully.

CONCLUSION

The SBC tested samples contained 20–40% used lubricating oil. The purpose of this study was to assess the efficiency of oil recovery through utilizing samples and solvent extraction method. Tests were designed by RSM, while SCR and ET were the two selected effective parameters in main experiments. Based on the obtained results, SCR was the most effective parameter, since 88.60% of oil is extracted in SCR of 9.53 ml/g in 22.5 minutes. Statistical analysis indicates that all the three parameters of R^2 have an appropriate proximity with the amounts of 99.29%, 98.70% and 96.84%, respectively. Hence, the predictability of the model is exquisite. Besides, verification of the model was approved through carrying out two tests on the fitted model, providing reliability of it.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

ABBREVIATIONS

RSM	*Response surface methodology*
ULO	*Used lubricating oil*
SBC	*Spent bleaching clay*
DOE	*Design of experiments*
ET	*Extraction time*
SCR	*Solvent to clay ratio*
MEK	*Methyl ethyl ketone*
min	*Minute*
rpm	*Revolutions per minute*
Eff	*Efficiency*
Eq	*Equation*
P-value	*Calculated probability*
ANOVA	*Analysis of variance*
ml/g	*Milliliter per gram*
X or x	*Coded value of variable*
Y or y	*Response*
SSR	*Sum Square Regression*
R^2	*Coefficient of determination*
Adj. R^2	*Adjusted coefficient of determination*
Pred. R^2	*Prediction coefficient of determination*
PRESS	*Predicted residual sum of squares*
F	*F-value*
Fig.	*Figure*
k	*Number of variables*
β_0	*Intercept constant in response equation*
β_i	*First-order regression coefficient*
β_{ii}	*Second-order regression coefficient*
β_{ij}	*Interaction between factors i and j*

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