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Solvent Ekstraksiyon Yöntemi ve RSM İstatistik Yaklaşımı ile Beyazlatma Kili Temizleme Tüketimi

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Özet. Kil ağartmak terimi ile sofralık sıvı yağın arıtım aşamasında istenmeyen içerikleri ve boyaları emme kapasitesine sahip doğal veya etkinleştirilmiş durumdaki kil kastedilmektedir. Dolayısıyla, kil ağartmanın en önemli fonksiyonu yağ ürünlerinin görüntüsünü, tadını, kokusunu ve dayanıklılığını geliştirmektir. Hekzan, aseton ve methyl ethyl ketone bu çalışmada çözücü madde olarak ve RSM (Yüzeysel tepki metodolojisi) optimal parametreleri belirlemek için kullanılmıştır. Değişken parametreler kil oranı için çözücü (SCR) ve çıkarım zamanını içermektedir. Sonuçlar final yağ temizlemede methyl ethyl ketonenin %61.3 etkinlikle etkinlikleri %52.7 ve 59.1 olan hekzan ve asetona göre daha üstün olduğunu göstermektedir. En iyi labaratuar koşulları ve RSM kullanımı altında en yüksek çıkarım etkinliği ketone çözücüler (asetan ve methyl ethyl ketone) için 3 dakika 6 saniye çıkarım zamanı ile 5.97 ml/g ve hekzan için 24 dakika 30 saniye çıkarım zamanı ile 5.92 ml/g dır.

Anahtar Kelimeler: Temizleme, beyazlatma kili tüketimi, solvent ekstrasyonu, tepki yüzey metodolojisi (RSM), yağ saflaştırma

Cleaning Spent Bleaching Clay through Using Solvent Extraction Method and RSM Statistical Approach

Abstract. Bleaching clay refers to clays that in their natural or activated state have the capacity to absorb dyes and other remaining undesirable ingredients from edible oil during its purification processes. Thus, the most important function of bleaching clay is to improve the appearance, flavor, odor, and stability of the final oil product. Hexane, acetone, and methyl ethyl ketone were used as the solvents in this research, and RSM (response surface methodology) was employed for determining the optimal parameters. The variable parameters included the solvent to clay ratio (SCR) and the extraction time. Results showed methyl ethyl ketone with the final oil removal efficiency of 61.3% was superior to hexane and acetone, with efficiencies of 52.7 and 59.1%, respectively. Under the best laboratory conditions and using RSM, the highest extraction efficiency was 5.97 ml/g for the ketone solvents (acetone and methyl ethyl ketone) at the extraction time of 3 minutes and 6 seconds, and 5.92 ml/g for hexane at the extraction time of 24 minutes and 30 seconds.

Keywords: Cleaning, Spent Bleaching Clay, Solvent Extraction, Response Surface Methodology (RSM), Oil Purification

1. INTRODUCTION

Raw vegetable oils are purified to remove their impurities. One of the purification steps is the bleaching stage during which soap materials and other impurities remaining in the oil are removed by absorbents. Natural absorbent such as completely activated soil, and activated

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carbon are the main absorbent and are collectively called bleaching soil. These are usually bentonite clays from the montmorillonite group together with compact masses of crystalline aluminum silicates containing various amounts of alkali and transition metals [1]. After bleaching soil used in bleaching systems and is inactivated due to absorption of impurities and clogging of its pores, it will turns into spent bleaching soil (SBS), which is a flammable waste material that if not properly stored or disposed, it may cause fire. Depending on various factors such as the used processing technology and type of the oil, this waste usually contains 20 to 40% oils, fats, and pigments by weight [2]. In purifying vegetable oil, the quantity of used bleaching clay is 0.5-1% of the oil weight, with its exact amount depending on the type of the purification process and the used raw oil [3]. Disposal of SBS in landfills creates problems and hazards because it may cause fire due to the presence of oil in the clay, due to the potential leakage of fats into the water path, and due to the possibility of spontaneous combustion. Moreover, the oil in the SBS results in dangerous pollution, constitutes an economic loss, and damages the environment. Furthermore, disposal of SBS is turning into a potential problem in countries that produce this waste due to the rapid growth of industries with the concomitant increase in the rate of waste material production. Recovery of the oil from SBS substantially reduces the costs of SBS disposal, and it may be possible to reuse the recovered oil [4].

In 1988, Kalam and Joshi regenerated SBE by using the hexane solvent for pre-purification and employing autoclave and heat in an aqueous environment [5]. Ng et al. (1997) first extracted the oil in SBE using solvent extraction, and then regenerated it by employing acid and heat treatments. In the same year, Al-zahrani and Alhamed used solvents, boil-off, and calcination as three methods of removing the oil from SBE, concluded that calcination without acid wash is sufficient to restore most of the clay activities, and that MEK was the best solvent for removing oil from clay [6]. In 2000, Al-zahrani and Alhamed used a Soxhlet extractor for conducting experiments with various solvents to classify them based on their solvent power and found MEK was the strongest solvent. They then classified the other solvents according to their efficiencies in removing oil from SBE and conducted experiments to remove oil from SBE using the 4 solvents acetone, MKE, hexane, and petroleum ether [7]. Waterman, Ager, King, et al. regenerated SBE by extraction using supercritical CO_2. In 2000, Tsai et al. used the pyrolysis process in a rotary furnace under inert atmosphere to regenerate thermally SBE that was used in an edible oil refinery [8]. In 2006, a study was conducted on using an alkaline pre-treatment method to remove oil from SBE in which bivariate linear regression analysis and RSM were employed to obtain the optimal NaOH solution to SBE ratio and the extraction time at boiling temperature. However, this method has not been used so far in solvent extraction for analyzing and interpreting results of experiments [9]. In another research conducted in a factory producing oil from date flesh and date seeds in 2010, solvent removal of SBE using hexane and the effect of the solvent to the solid matter ratio on oil removal were studied [10].

It can be concluded from previous studies that solvent extraction is the most common and the best method for oil removal from SBE. Experiments conducted on solvent extraction in the past used conventional methods and lacked designs and, therefore, mutual effects of parameters were not studied. In this research, the experiments were designed and mutual effects of variables were considered and, therefore, the most optimal results were found by conducting the least number of experiments. Effective factors in solvent extraction are the type of solvent, the extraction time, the extraction temperature, the solvent to clay ratio (SCR), and the degree of mixing [11, 12, 13, 14].

In this study, bivariate linear regression analysis and RSM were used for analyzing results. A model was then obtained for each solvent and the optimal conditions for achieving the highest efficiency were determined by optimizing the fitted model. Three solvents (hexane, MEK, and acetone) were used to study oil removal from SBS in the laboratory.

Indeed, the goals of this research were (1) employing solvent extraction for removing oil from SBS in order to regenerate the SBS and to compare the results obtained for the three above mentioned solvents, and (2) using RSM for optimizing the parameters that are effective in the solvent extraction process.

2. MATERIALS AND METHODOLOGY

Spent bleaching soil (SBS) that included undesirable remains of the edible oil refining process was obtained from the oil- bleaching unit of Behshahr Industrial Company. Solvent extraction was carried out using MEK, acetone, and hexane (produced by the German Company Merck), the first two from the ketone and the third from the hydrocarbon solvents categories. The Soxhlet experiments were conducted using MEK with high solubility, filter paper, a balance, a Soxhlet extractor, and a Bunsen burner together with a known quantity of SBE to separate the fats from the solid materials then a rotary evaporator was used to separate the oil and the solvent. Finally, the weight of oil was determined using a thermo-gravimetric method. Using the above mentioned solvents, and based on results of pre-experiments, the required experiments were designed by employing the RSM software. A complete description of the conducted experiments is presented through the next sections, as follow.

i) The Soxhlet experiment

Keeping SBS in a plastic bag at room temperature (25˚C), 5 g of SBS was placed in a Soxhlet extractor. MEK, which has high solubility, was a suitable solvent for performing a faster and more accurate Soxhlet experiment. Two hundred ml of MEK were poured into the round-bottom flask at the lower end of the Soxhlet extractor. The experiment was conducted under a hood due to flammability of MEK.

The solvent was siphoned to the heating flask after 30 minutes and the siphon cycle was repeated at 10-minute intervals. After 10 hours, the system was stopped and the heater and cooler were removed. The colorless solvent in the round bottom flask at lower end of the Soxhlet extractor was pale yellow at the end of the extraction process because of oil removal from the SBS and dissolving in the solvent. Then, the system was disconnected the flask at lower end of the extractor containing the mixture of oil and solvent was removed and transferred to the place for separating the oil from the solvent. Using this method, almost the entire oil within the SBS was removed.

ii) Oil extraction test

The mixture of oil and solvent was poured into the special flask, mounted in the rotary evaporator, and turned on. After 10 minutes, the solvent was evaporated under vacuum and collected in the side container, while the oil in the solvent remained in the original flask. To improve the accuracy of the experiment, the extractor was kept on for another 10 minutes to obtain oil to the highest volume. Then, evaporator was turned off and the flask containing the oil placed in a desiccator to cool without absorbing moisture. After an hour, the flask was removed from the desiccator and weighed. Obtaining the weight of the extracted oil, the weight of the flask was deducted from the 5-gram SBS sample and the percentage of oil in the representative SBS sample was calculated.

iii) Thermo-gravimetric experiment

The SBS sample, from which the oil was extracted, was removed from the Soxhlet extractor and mounted in an oven at 110 ˚C for an hour. The sample was then taken out of the desiccator and weighed to determine the weight of the extracted oil. This method was also used to calculate the weight of the sample oil. Finally, average of the two obtained values from the aforementioned tests (subsections ii and iii) was considered the oil content of the SBS.

iv) Solvent selection and experimental procedure

Normal hexane is a cheaper solvent and its extracted oil can be used for cooking. Out of hydrocarbon solvents family, hexane is used as the standard non-polar solvent in oil extraction [4]. In ketone group, MEK with its high solvency power, and acetone with its low molecular weight compared to other ketones were selected as the other two solvents in this research.

Starting the experiments, three 200-ml beakers were carefully washed using a detergent and distilled water. After they were dried, 15 g of the SBS were poured into each of the beakers. The next stage was the preparation of the solvents. Considering the SCR selected for each experiment, the determined quantity of the solvent (hexane, MEK, or acetone) was added to the SBS within the beaker. It is to be mentioned that each experiment was conducted using one solvent, one SCR, and one specific mixing time. During the experiments of cleaning the SBS by the solvents, a magnetic mixer (RH basic 12 IKAMAG) was used to mix the SBS with the solvent for maintaining a homogeneous mixture.

The experiments were conducted at room temperature with no extra heat. A constant speed of the mixer (200 rpm) was used in all experiments. Immediately after each experiment was completed, the mixture was taken out, the SBS inside the filter paper was put inside a crucible and heated at 110 ˚C for 90 minutes, and it was then put in a desiccator. After an hour, the sample was taken out of the desiccator and weighed. Considering the weight of the filter paper, the initial weight of the SBS (15 g for each sample), and the final weight of the cleaned SBS, the efficiency of the SBS cleaning by each of the solvents (with the related SCR and extraction time) was obtained. Equation 1 was used to obtain the efficiency through the percentage of oil removal from the SBS, as follow.

$$R = \frac{Oil_{total} - Oil_{removal}}{Oil_{total}} \tag{1}$$

In eq.1, Oil $_{removal}$ is the quantity of oil extracted from the 15-g SBS sample in the solvent extraction and Oil $_{total}$ the total oil content of the SBS determined in the Soxhlet experiment (3.3 g in the 15-g sample). All materials used in experiments were weighed using a model MP-300G digital balance with the accuracy of 1 mg made by the AND Company.

2.1. Designing the main experiments

Utilizing the results of the pre-experiments, the main experiments of the research were designed. The estimated suitable range for the SCR variable was 1-6 ml/g and that for the optimal extraction time was 2-25 minutes. The RSM (response surface methodology), which is a collection of new methods in designing experiments, was used in this research to optimize the studied parameters and, finally, to select the optimal point. Considering previous research, the parameters of extraction time and solvent to clay ratio (SCR) were studied as the effective factors in RSM. In total 33 experiments were conducted using hexane, MEK, and acetone as solvents (11 experiments for each solvent) resulting the efficiency of the solvents. MINITAB was then employed to analyze laboratory data, and the quadratic polynomial model in equation 2 was adapted to the laboratory data to optimize the process.

$$Y = \beta_{\circ} + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i} \sum_{j} \beta_{ij} X_i X_j \tag{2}$$

While the parameters are as follow, Y the result predicted by the model, β_e a constant coefficient, β_i the linear coefficients, β_{ii} the quadratic coefficients, β_{ij} the interaction coefficients, and X_i and X_j the coded values of the tested variables.

3. ANALYSIS AND INTERPRETATION OF THE RESULTS

In this section, the accurate oil content of the SBS was determined and the efficiency of each of the solvents was calculated. Finally, using statistical methods and the RSM, a model was obtained for oil removal efficiency in solvent extraction by each of the three solvents (hexane, MEK, and acetone) and the validity of each model was evaluated as follows.

3.1. Results of the Soxhlet experiment

The Soxhlet experiment was conducted under suitable laboratory conditions under a hood using 10 g of the SBS and 200 ml of MEK. A rotary evaporator was then used to separate the oil and solvent of the mixture collected in the laboratory flask. The oil separated from the solvent was poured in a flask that had been weighed in advance. The flask was weighed and the weight of the oil was found to be 2.16 g (21.6% of the SBE by weight). The oil content of the 15- gram sample for the main experiments was considered as 3.3 g.

3.2. Modeling the cleaning process

In this section, the cleaning process of SBS impregnated with oil using the solvents hexane, MEK, and acetone was modeled employing optimal ranges of the effective parameters. The response surface methodology (RSM) was employed to narrow these ranges and to study their effects and determine even the smallest variations. This modeling was performed for all three solvents and a model suitable for each one was obtained. Each experiment yielded results that included the efficiency of oil removal. Finding the optimal values of factors influencing removal of oil from SBS, the experiments were conducted using the solvent extraction method, considering the two parameters of SCR and duration of the experiment as the effective parameters. Taking into account results of previous studies and pre-experiments, the experiments were run with SCRs in range of 1-6 and extraction times within 2-22 minutes.

Results of the designed experiments, which are presented in Table 1, showed that the maximum percentage of oil removal from the SBS for the solvent hexane (46.3%) was achieved with SCR of 5.41 and extraction time of 21 minutes and 47 seconds. The percentages for acetone and MEK were 54.8% and 58.4% with SCR of 5.41 and extraction time of 6 minutes and 14 seconds.

Table 1. Tests results in accordance with experiment designs

No.	Variable		MEK	Hex	Ace
	SCR (ml/gr)	Time (min)			
1	2.59	6.22	35.9	40.6	42
2	5.41	6.22	58.4	45	54.8
3	2.59	21.78	44.6	40.1	40.6
4	5.41	21.78*	55.8	46.3	54.5
5	6	14	57.5	46	53.6
6	2	14	33.6	39.5	28.6
7	4	25	53.45	45	54
8	4	3	49.8	41.5	51.6
9	4	14	54.6	36.8	47.78
10	4	14	51.9	35.3	47.5
11	4	14	49.2	34.6	46.6
*21.78 mean 21 minutes and 47 seconds.					

Considering regression coefficients obtained from data analysis, a quadratic polynomial model was obtained for oil removal efficiency of the 3 solvents, as follow.

Hexane Solvent:

$$\text{Oil Removal}_{\text{Hexane}} = (35.57) + (2.47)x_1 + (0.72)x_2 + (3.59)x_1^2 + (3.84)x_2^2 + (0.45)x_1 \cdot x_2 \quad (3)$$

Acetone Solvent:

$$\text{Oil Removal}_{\text{Acetone}} = (47.29) + (7.76)x_1 + (0.21)x_2 - (2.84)x_1^2 + (3.01)x_2^2 + (0.28)x_1 \cdot x_2 \quad (4)$$

Methyl Ethyl Ketone Solvent:

$$\text{Oil Removal}_{\text{MEK}} = (51.90) + (8.44)x_1 + (1.41)x_2 - (3.15)x_1^2 - (0.12)x_2^2 - (2.83)x_1 \cdot x_2 \quad (5)$$

In all of the above mentioned equations the parameters are as follow; Y the percentage oil removal, X_1 the coded SCR, and X_2 the coded extraction time. In Figures 1, 2, and 3 the actual observed values are plotted versus obtained predicted from the models. All three figures indicate quite well conformity of the 2 sets of values for 3 solvents.

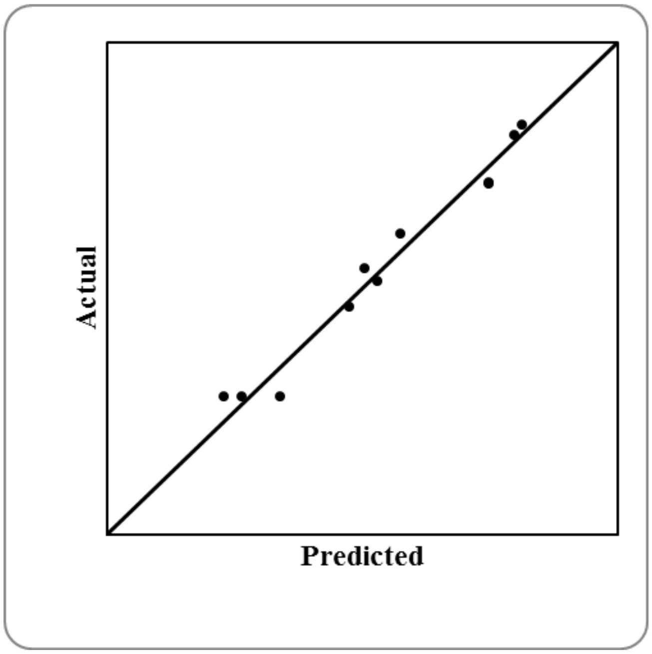

Figure 1: Predicted versus actual values for Hexane solvent extraction efficiency

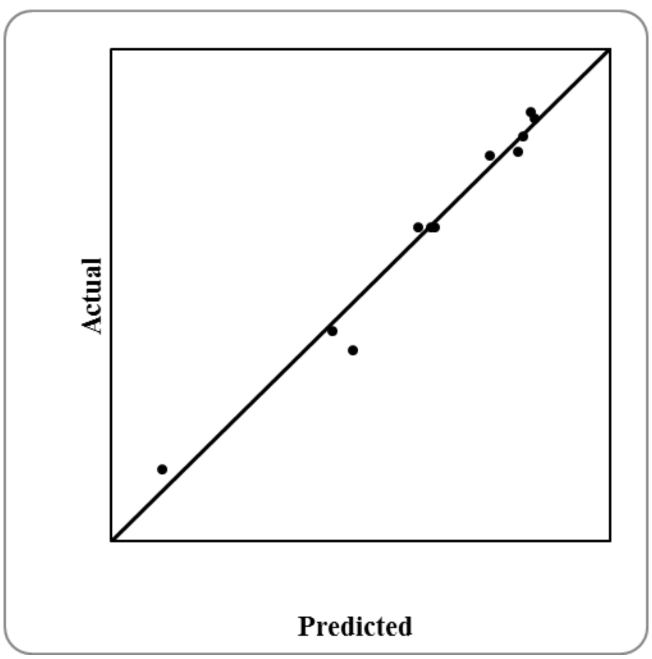

Figure 2: Predicted versus actual values for Acetone solvent extraction efficiency

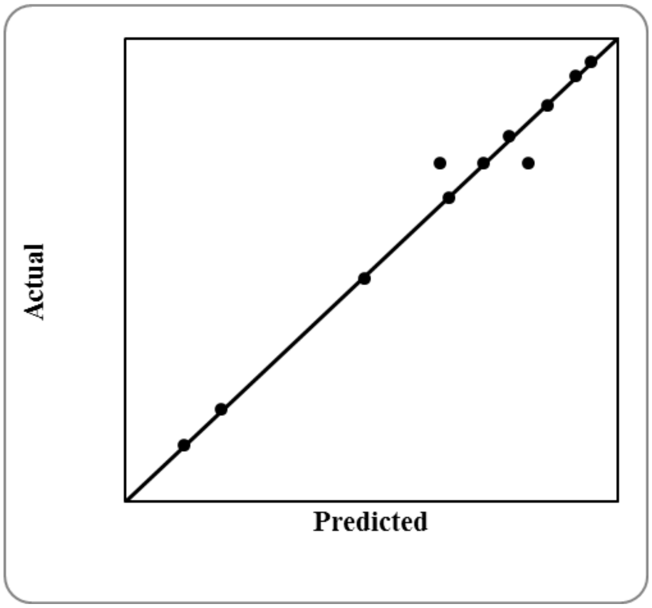

Figure 3: Predicted versus actual values for MEK solvent extraction efficiency

3.3. Evaluation of the validity of the models

For all three solvents, the probability of the regression equation was less than 0.05 (providing the confidence level of 95%), which showed the quadratic polynomial model matched the laboratory data quite well. Moreover, the F values obtained for the hexane, acetone, and MEK solvents were 35.46, 81.01, and 46.07, respectively, which are obviously higher than the F value of the table (3.45 for $\alpha = 0.05$). Therefore, the fitness of the models was confirmed. Using results of the experiments, along with the help of MINITAB, the ANOVA tables for the 3 solvents (Table 2-4) were obtained.

Table 2: ANOVA for Hexane solvent

$y = (35.57) + (2.47)x_1 + (0.72)x_2 + (3.59)x_1^2 + (3.84)x_2^2 + (0.45)x_1 \cdot x_2$					
Source of Variation	**Sum of Squares**	**DF**	**Mean Square**	**F_0**	**P-Value**
Regression	174.72	5	34.94	35.46	< 0.001
SS_R (β_1, β_2, | β_0)	53.10	-2	26.55	26.94	0.002
SS_R (β_{11}, β_{22}, β_{12}, | β_0, β_1, β_2)	121.62	-3	40.54	41.14	< 0.001
Residual	4.93	5	0.99		
Lack of Fit	2.40	3	0.80	0.63	0.66
Pure Error	2.53	2	1.26		
Total	179.64	10			
R^2 = 97.26%	Adj. R^2 =94.51%	PRESS = 22.7532	Pred. R^2 = 87.33%		

Table 3: ANOVA for Acetone solvent

$y = (47.29) + (7.76)x_1 + (0.21)x_2 - (2.84)x_1^2 + (3.01)x_2^2 + (0.28)x_1 \cdot x_2$					
Source of Variation	**Sum of Squares**	**DF**	**Mean Square**	**F_0**	**P-Value**
Regression	651.70	5	130.34	81.01	0
SS_R (β_1, β_2, | β_0)	514.59	-2	257.30	159.91	0
SS_R (β_{11}, β_{22}, β_{12}, | β_0, β_1, β_2)	137.11	-3	45.70	28.40	< 0.001
Residual	8.05	5	1.61		
Lack of Fit	7.29	3	2.43	6.39	0.138
Pure Error	0.76	2	0.38		
Total	659.74	10			
R^2 = 98.78%	Adj. R^2 =97.56%	PRESS =53.5124	Pred. R^2 = 91.89%		

Table 4: ANOVA for methyl ethyl ketone solvent

$y = (51.9000) + (8.44)x_1 + (1.41)x_2 - (3.15)x_1^2 - (0.12)x_2^2 - (2.83)x_1 \cdot x_2$					
Source of Variation	**Sum of Squares**	**DF**	**Mean Square**	**F_0**	**P-Value**
Regression	677.52	5	135.50	46.07	0
SS_R (β_1, β_2, | β_0)	585.38	-2	292.69	99.52	0
SS_R (β_{11}, β_{22}, β_{12}, | β_0, β_1, β_2)	93.14	-3	31.05	10.56	<0.001
Residual	14.71	5	2.94		
Lack of Fit	0.13	3	0.04	0.01	0.999
Pure Error	14.58	2	7.29		
Total	692.23	10			
R^2 =97.88%	Adj. R^2 =95.75%		PRESS = 33.7050	Pred. R^2 =95.13%	

As shown in the ANOVA table of each model, the p-values for all three models were less than 0.05, which shows the high conformity of the models.

The regression coefficient R^2, expresses the accuracy of the regression and indicates the relationship between laboratory data and predicted results, with high regression coefficients close to 1, which is remarkably desired. The regression coefficients for the solvents used in the experiments were as follows in Table 5.

Table 5: Regression coefficient for three solvents

Solvent	**R^2**	**Adj. R^2**	**Pred. R^2**
Hexane	97.26%	94.51%	87.33%
Acetone	98.78%	97.56%	91.89%
MEK	97.88%	95.75%	95.13%

These regression coefficients indicate that the laboratory data and the predicted results matched very well and the fitted models for all three solvents enjoyed very high conformity.

3.4. Efficiency of solvents in oil extraction

Efficiency of the 3 solvents and their comparison are presented in this section, as follow.

i) Hexane

Hexane is one of the hydrocarbon solvents and, hence, it behaved differently from acetone and MEK in the experiments and yielded results that varied from those of the other two solvents, which belong to the ketone family. Figure 4 shows contour and RSM diagrams of the SCR and extraction time effects on efficiency of oil removal from the SBS. These two parameters had optimal values in the range of 1-6 for SCR (in ml/g) and within 2-22 minute for extraction time.

As shown in the figure, oil removal efficiencies at average SCR and extraction time values were lower compared to those at higher values of these two parameters. The reduced efficiency near the center of the diagram indicates the interaction of these two parameters. Explaining the behavior of hexane in the model, it is a hydrocarbon solvent with high molecular weight compared to the other two solvents. The high weight cause a relatively low ability in extracting oil, while its oil removal ability is relatively good in the zones that the interactions of the two parameters are at low levels (corners of the RSM diagram).

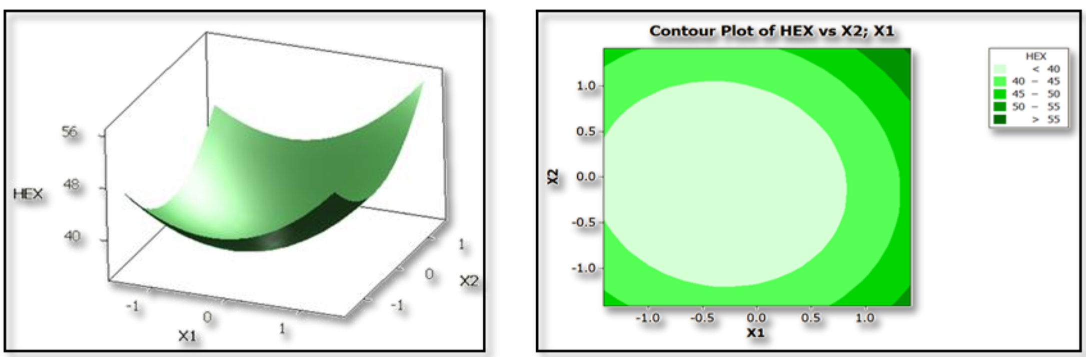

Figure 4: Contour and RSM diagrams of the SCR and extraction time on efficiency of oil removal by Hexane solvent

ii) MEK

MEK, a solvent of the ketone family with lower molecular weight than hexane, had similar results to acetone (which is another solvent of the ketone family), and because of its relatively low molecular weight had the highest oil extraction efficiency under laboratory conditions. As shown in the diagrams in Figure 5 for MEK, the two parameters had considerable mutual effects on oil removal from the SBS. Oil extraction efficiency improved when SCR values increased to a certain level, beyond which further increases in the value of

SCR did not considerably change the efficiency. The interaction of the two parameters showed that oil extraction efficiency was low at low SCR(X_1) and extraction time (X_2) values, but it improved with increases in SCR values and under the mutual effects of the two effective parameters.

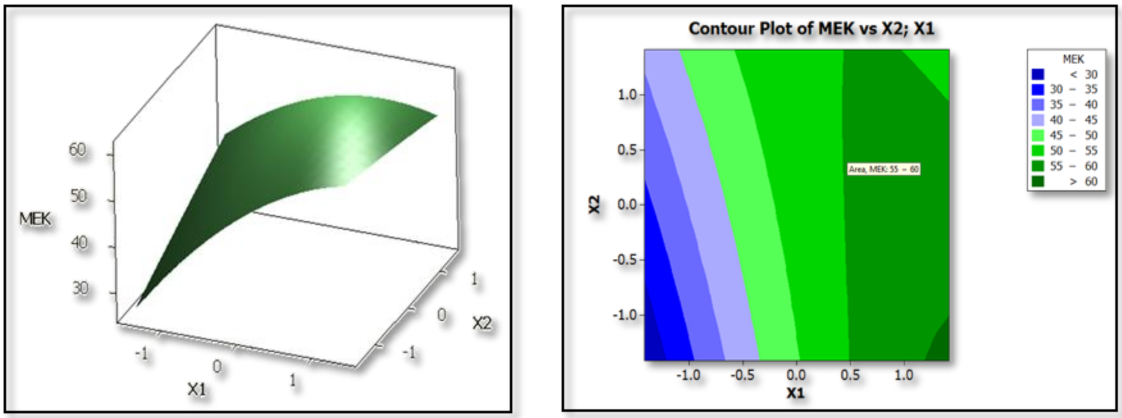

Figure 5: Contour and RSM diagrams of the SCR and extraction time on efficiency of oil removal by MEK solvent

iii) Acetone

Acetone is a solvent of the ketone family with molecular weight lower compared to those of the other two solvents that were used. Figure 6 shows the contour and RSM diagrams of the effects of SCR and extraction time on oil removal from the SBS by acetone. When SCR (X_1) values increased from 2 to 6, there was an ascending trend in oil removal efficiency up to the SCR value of 6, at which it reached a constant level and did not increase further. This point is in the optimal SCR range used in the experiment with acetone.

In general, efficiency of acetone in oil extraction was higher than that of hexane. Moreover, as Figure 6 depicts, the RSM diagram is saddle-shaped and the efficiency of acetone in oil extraction reached its maximum at two symmetrical ranges; i.e. when SCR had a relatively constant value at the two extraction times that were symmetrical with respect to the center (one at low extraction time and the other at high extraction time), the efficiencies were relatively the same and at its maximum level.

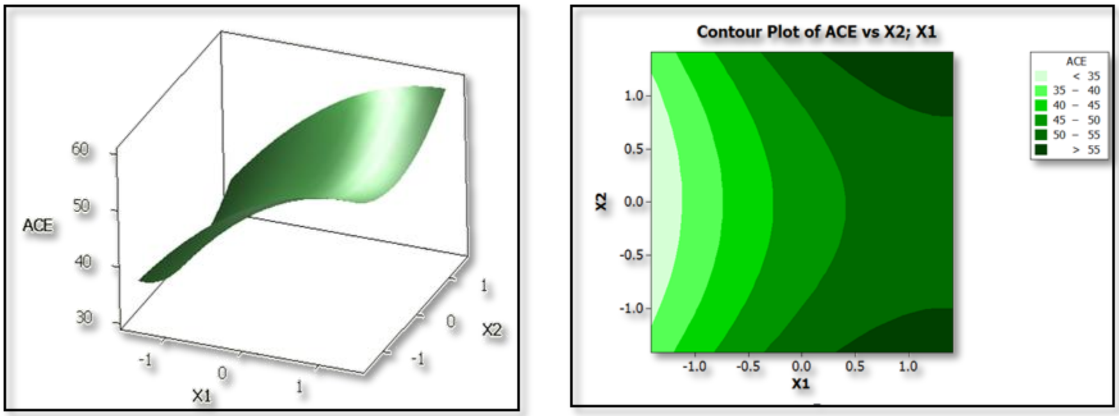

Figure 6: Contour and RSM diagrams of the SCR and extraction time on efficiency of oil removal by Acetone solvent

4. Comparison of the solvents efficiencies

Figure 7 compares results of experiments that were conducted using the three solvents. The horizontal axis represents the numbers of the experiment and the vertical axis represents the oil extraction efficiencies. As shown in the figure, the three solvents behaved rather similarly under similar conditions. This shows that except for the type of solvent, operational parameters decreased or increased oil recovery efficiency equally for all three solvents. Another noteworthy point, shown in the diagram, was the great similarity between the acetone and MEK behavior. This can be explained by the fact that both of them belong to the ketone family, while hexane is from the hydrocarbon family and behaved differently from the other two solvents. In fact, nature of the solvents has the most severe effect on its efficiency in oil extraction.

From the microscopic point of view, methyl ethyl ketone ($CH_3CH_2COCH_3$) and acetone (CH_3COCH_3) have higher solvent extraction efficiencies than hexane ($CH_3C4H_8CH_3$), with the molecular weight of 86.18) due to their lower molecular weight. Moreover, the covalent bonds between carbon and oxygen atoms in MEK and acetone cause them to form more stable compounds with soybean edible oil (which contains aromatic compounds) compared to hexane, in which there are single bonds between carbon and hydrogen atoms. This fact elaborates the effect of solidity and stability of the molecular structure of solvent in its removal efficiency.

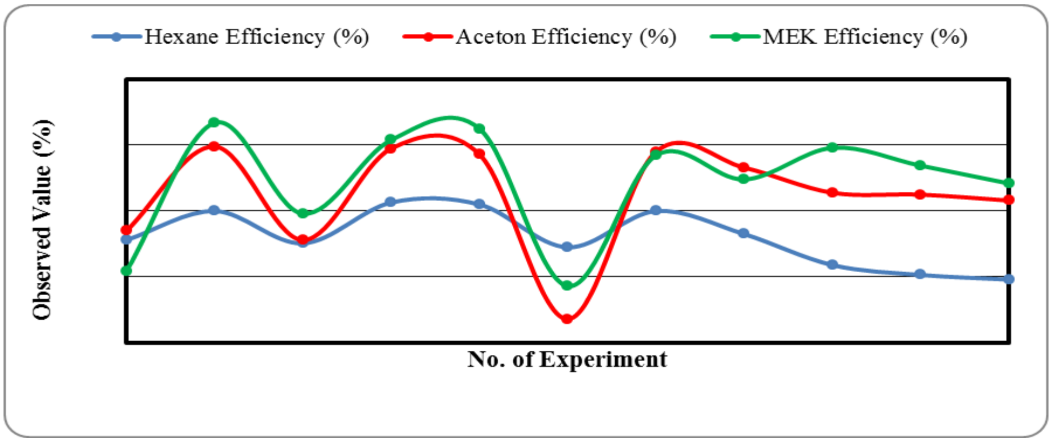

Figure 7: Extraction efficiencies for the three solvents

5. Optimal efficiency of solvents and verification of the models

One of the main goals of this research was to determine the optimal conditions for oil extraction from SBS that was generated in purification of edible oil. For this purpose, by means of MINITAB the fitted model was optimized to obtain the optimal conditions for extraction of oil from SBS by the three solvents. These conditions are listed in Table 6. Furthermore, after the optimal conditions were found for each solvent, another experiment was conducted using the optimal conditions estimated by the model in order to verify the model.

Table 6: The optimal conditions for the solvent and fitted model test

Solvent	Predicted (%)	Actual (%)	Optimal conditions		Error (%)
			Time (min)	SCR	
Hexane	54.2	52.7	24.5*	5.92	2.846
Acetone (1)	58.6	59.5	24.89	5.97	1.522
Acetone (2)	58.1	59.1	3.1	5.97	1.692
MEK	60.9	61.3	3.1	5.97	0.652

*24.5 mean 24 minutes and 30 seconds.

As the table shows, there were two optimal boundaries in oil extraction at maximum efficiency for acetone (case 1 and 2). At extraction times of 24 minutes and 54 seconds and 3 minutes and 6 seconds, with equal SCR, extraction efficiencies were very close to each other. Therefore, it is obvious that the best and most optimal extraction time for oil extraction by acetone is 3 minutes and 6 seconds. Having the low solubility power for Hexane, the relatively long extraction time of 24 minutes and 30 seconds is needed to achieve maximum efficiency. The highest efficiency was obtained at the extraction time of 3 minutes and 6 seconds for MEK, with better and more optimal efficiency results compared to those of the other two solvents.

4. CONCLUSIONS

After optimization of the fitted models by the software, the optimal values of the parameters were obtained for the three solvents hexane, acetone, and MEK. The highest efficiency belonged to MEK (61.3%) and the lowest (52.7%) with a long extraction time to hexane. Utilizing RSM and designed experiments improved process efficiency, reduced changes in response, decreased extraction time, and lowered costs of i) materials used ii) repeating experiments, and iii) the required workforce. Results of the experiments, and comparison of the efficiencies of the three solvents, revealed that MEK with the efficiency of 60.9%, SCR of 5.97, and extraction time of 3 minutes and 6 seconds had the highest efficiency in oil removal. Therefore, MEK was the best solvent in this research because it had the highest efficiency in oil removal, within very short period of time (3.1′).

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