A Study of Parameters Affecting the Solvent Extraction-Flocculation Process of Used Lubricating Oil

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ABSTRACT

The aim of this study was to investigate the effect of operating variables on, the percentage of removed sludge (PSR) obtained during re-refining of 15W-40 Al-Durra spent lubricant by solvent extraction-flocculation treatment method. Binary solvents were used such as, Heavy Naphtha (H.N.): MEK (N:MEK), H.N. : n-Butanol (N:n-But), and H.N. : Iso-Butanol (N:Iso:But). The studied variables were mixing speed (300-900, rpm), mixing time (15-60, min), and operating temperature (25-40,°C). This study showed that the studied operating variables have effects where, increasing the mixing time up to 45 min for H.N.: MEK, H.N.: n-Butanol and 30 min for H.N.: Iso-Butanol increased the PSR, after that percentage was decreased; increasing the mixing speed for all the studied solvents up to 700 rpm increased the PSR, after that the percentage was decreased, while increasing the operating temperature decreased the PSR for all the solvents. This study has resulted in reasonably accurate multivariate process correlation that relates the removed sludge percentage to the process variables. The determination coefficients (R^2) were significantly high and equal to 0.957, 0.974, and 0.944 for N:MEK, N:n-But, and N:Iso-Butl respectively. The best operating conditions resulted the maximum PSR were (30 min, 900 rpm, 30 °C), (45 min, 700 rpm, 35 °C), and (30 min, 700 rpm, 35 °C) for N:MEK, N:n-But, and N:Iso-Butanol respectively.

Key words: Solvent extraction-flocculation, Used oil re-refining, PSR.

دراسة العوامل المؤثرة على عملية الاستخلاص-التلبد بالمذيبات للزيوت المستهلك

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هذه الدراسة تتضمن دراسة تأثير الظروف التشغيلية على نسبة المواد المتكتلة المزالة خلال عملية معالجة زيت التزييت العراقي (15W-40) باستخدام طريقة الأستخلاص-التلبد بالمذيبات. لهذا الغرض أستخدمت مذيبات ثنائية و هي: النافثا الثقيلة : ميثايل ايثايل كيتون، النافثا الثقيلة : البيوتانول المستمر، بالاضافة الى النافثا الثقيلة : البيوتانول المتفرع. متغيرات العملية كانت سرعة الخلط (15- 60 دقيقة)، مدة الخلط (300-900 دورة)، و درجةحرارة (25-40 درجة مئوية). العملية اظهرت هذه الدراسة بأن الظروف التشغيلية المدروسة لها تأثير على العملية، حيث بزيادة مدة الخلط لغاية 45 دقيقة بالنسبة للنافثا الثقيلة و ميثايل ايثايل كيتون، النافثا الثقيلة و البيوتانول المستمر ازدادت نسبة المواد المتكتلة المزالة، و لغاية 30 دقيقة بالنسبة النافثا الثقيلة و البيوتانول المتفرع، بعدها انخفضت هذة النسبة، و بزيادة سرعة الخلط لغاية 700 دورة/دقيقة ازدادت نسبة المواد المتكتلة المزالة لجميع المذيبات، و بعدها انخفضت هذة النسبة، اما بالنسبة لدرجة الحرارة فأن زيادتها ادت الى انخفاض هذة النسبة لجميع المذيبات. هذة الدراسة انتجت معادلة رياضية تربط بين المواد الصلبة المزالة و متغيرات العملية، معامل الربط للمعادلة المقترحة كان ذو قيمة عالية بشكل ملحوظ، حيث كان 0.957، 0.974، و 0.944

بالنسبة الى النافثا الثقيلة : ميثايل ايثايل كيتون، النافثا الثقيلة : البيوتانول المستمر، و الى النافثا الثقيلة : البيوتانول المتفرع على الترتيب. افضل نسبة من الشوائب المتكتلة أزيلت عند، (30 د، 900 دورة، 30 درجة مئوية)، (45 د، 700 دورة، 35 درجة مئوية)، و (30 د، 700 دورة، 35 درجة مئوية) بالنسبة الى النافثا الثقيلة : ميثايل ايثايل كيتون، النافثا الثقيلة : البيوتانول المستمر، و الى النافثا الثقيلة : البيوتانول المتفرع على الترتيب .

الكلمات الرئيسية: التمثيل الرياضي، الاستخلاص-التلبد بالمذيبات، معالجة زيت التزييت المستهلك، نسبة الشوائب الصلبة المزالة.

1. INTRODUCTION

The increment of energy demand caused the consumption of the lubricating oils to be increased, resulted in yearly increment in the used oil production, beside to disposal issues raised up **Aremu et al., 2015**. Spent lubricating oils have to be recycled due to the presence of highly toxic materials in the oil turning them to highly noxious and carcinogenic such as heavy metals and polycyclic aromatic hydrocarbons (PAHs) **Yang et al., 2013**. Approximately 9 times higher, the crude oil required to produce a specific amount of base oil than that from used oil **Ogbeide, 2010**.

Several techniques were proposed for used oils treatment **Speight, and Exall, 2014**. The eldest acid/clay treatment method produced acidic sludge which is toxic, harm the environment, and difficult to dispose. The acid free vacuum distillation/clay treatment considered an expensive process due to large amount of clay required; besides it produced low yield percentage and quality where, large portion of base oil lost in the clay and relatively high metal content. These two processes are out of service in most of developed countries, and replaced by solvent extraction **Hamad et al., 2005,** and hydrogenation processes **Almutairi et al., 2007**. Hydrogenation treatment method associated with the difficult to control hydrogenation step and the need of catalyst which need to be replaced frequently.

Solvent extraction was preferred over other treatment processes because it solved the problems associated with them and considered a much lower cost than other. Re-refining of used lubricants by solvent extraction-flocculation process mainly associated with two objectives, recovering of base oil and segregating of solid contaminants which are extremely pollutant for the environment. Solvent extraction is a simple process where, the used oil is mixed with an efficient solvent able to flocculate the impurities at an appropriate ratio. The employed solvent is then recovered and can be used again, **Elbashir et al., 2002**.

Statistical modeling of process could have a direct influence on evaluation of the process variables; also result in saving resources and time **Du et al., 2006**. A few attempts were carried out for modeling the treatment of waste lubricants by solvent extraction process each with constricts of its own, such that carried by **Gul et al. 2014**, and **Naqvi et al., 2014**.

The aims of this study are to investigate the effect of operation variables on the PSR for Iraqi used lubricating oil (15W-40) and present them in a mathematical correlation based on the data experimentally found.

2. EXPERIMENTAL WORK

2.1 Materials

The feed to be treated was Al-Duraa (15W-40) used lubricating oil with n-Butanol, iso-Butanol, MEK (analytical grade solvents) and Heavy Naphtha (produced from Al-Duraa Refinery). The properties of the feedstock are indicated in **Table 1.**

2.2 Procedure

In the beginning, determination of the optimum solvent to oil ratio or the critical clarifying ratio (CCR) was carried out by pretreating (settling and heating) the feed. Then the feed was mixed with solvent at different solvent to oil ratio (SOR, 2:1 to 6:1) at, 25±2 ^oC, 30 min, mixing time, and 500 rpm, mixing speed, then left for settling at room temperature for 24 hours. After settling time was finished, two layers were formed, the upper one was the extraction solvent and base oil, and the bottom liquid phase was heavier material (contaminants). After separation the upper layer, the lower layer weighted. The wet sludge then was washed with the same used solvent and then dried at 105 ^oC for 15 min to remove any remaining solvent and weighted. The percent oil losses (POL) and percentage of sludge removal (PSR) was calculated by using Eq. (1) and Eq. (2) respectively, **Durrani et al., 2012**:

$$POL = \frac{Wwet - Wdry}{Wo} \% \quad (1)$$

$$PSR = \frac{Wdry}{Wo} \% \quad (2)$$

Where:
Wo The initial amount of used oil.
Wwet The wetted sludge.
Wdry The dried sludge.

The studied solvents were binary such as, N:MEK, N:n-But and N:Iso-But; at (20:80), (30:70), and (40:60) ratio of solvent to solvent were investigated. The optimum solvent to used oil ratio which is called the critical clarifying ratio (CCR) was calculated graphically as described by **Elbashir et al., 2002,** for the best binary system composition.

The operation variables were mixing time (15-60, min), mixing speed (300-900, rpm), and operating temperature (25-40, ^oC); by using adjustable hot plate magnetic stirrer (Heidolph®) which are able to mix up to 1400 rpm and heat up to 100 ^oC. The effect of these variables was investigated at the CCR of solvent to used oil.

3. RESULTS AND DISCUSSIONS

The experimental findings of the best solvent to solvent composition resulted that, the best binary solvent composition was 30:70 for all of the investigated solvents, as indicated in **Table 2**. By comparing the obtained values of the base oil yield, POL, and PSR of the three studied composition of binary solvent, the best binary was selected. 30:70 solvent to solvent ratio resulted in high base oil yield, low POL, and high PSR. At this ratio of solvent to solvent, the CCRs were obtained graphically and were equal to 3.4, 3.8, and 3.7 for N:nBut, N:iso-But, and N:MEK respectively, as indicated in **Fig. 1**. The obtained CCR does not necessary remove the maximum sludge, **Nimir*et al*., 1997.**

The effect of mixing time on sludge removal for the used solvents was shown in **Fig. 2**. For N:MEK, the PSR value increased with increasing the mixing time up to 45 min, this behavior can be attributed to: with increasing the mixing period the contacting between the solvent and used oil, as well as the contact between the impurities with each other would increase, as a result more of the impurities. Beyond 45 min the PSR decreased, this behavior could be attributed to the flocculated impurities which would be dissociated after 45 min of mixing time. For N:n-But, at 15 min of mixing the PSR was higher than 30 min, after 15 min of mixing the two layers (extracted and raffinate) were not completely separated, that way the PSR was higher than at 30 min, as the POL was higher than at 30 min. After 30 min the PSR increased due to the more mixing time, the more of impurities were flocculated as well as the more of oil lost.

For N:Iso-But, increasing the mixing time up to 30 min increased the PSR, this can be explained as, the increment of mixing time increased the contacting time, and more of impurities flocculated. Increasing the mixing time more up to 45 min made the flocculated impurities to dissociate and the PSR decreased; after 45 min, the increment in the PSR value may be related to that the dispersed impurities before 45 min re-flocculated again. The difference between N:n-But and N:Iso-But can be attributed to the atoms configuration difference between n-Butanol and Iso-Butanol, **Filho et al., 2010**. These results were in agreement with the findings of **Hussein et al., 2014**,

Fig. 3 shows the effect of mixing speed on the sludge removal. As the mixing speed was increased up to 700 rpm, the sludge removal was increased due to increasing of contacting frequency of particles and each other. After 700 rpm high mixing frequency caused to dissociate and/or disperse of solid contaminants, thus the sludge removal percentage was decreased, these results were in agreement with the findings of **Hussein et al., 2014**,

The operating temperature has effect, but not as mixing time and mixing speed as shown in **Fig. 4**. At low temperature (25-30 ^oC) the sludge removal was increased, as the temperature increment would increase the affinity as a result the PSR increased; further increment of temperature would increase affinity, as well as the solubility of used oil in solvent **Durrani et al., 2012,** for this reason the percentage of sludge removal was decreased with increasing temperature. These results are in corresponding with the finding of **Durrani et al., 2012**, **Hussein et al., 2014**, and **Aremu et al., 2015**.

The optimized operating conditions were found to be as, (30 min, 900 rpm, 30 ^oC) for N:MEK, (45 min, 700 rpm, 35 ^oC) for N:n-But, and (30 min, 700 rpm, 35 ^oC), for N:Iso-But. The results indicated in **Table 3**, have been obtained at the optimized operating conditions. Decreasing specific gravity value indicates that aromatic compounds and solids have been removed, and lowering of ash content value indicates that dust or oxidation products, or arise from the additives that contain metals have been reduced **Speight and Exall, 2014**. Low value of viscosity index (VI) means that the non-metallic polymeric VI improver, **Kamal and Khan, 2009**. The decrement in the value of the used oil contaminants was in agreement with the finding of, **Kamal and Khan, 2009**, and **Mahmood, 2015**.

4. CORELATION ANALYSIS

The percentage of sludge removal during the extraction-flocculation process nonlinearly changed with the operation variables as indicated in **Fig. 2, 3,** and **4**. It has been indicated by **Naqvi et al. 2014**, that cubic polynomial function efficiently describes such a relation, so a cubic polynomial equation was suggested. The suggested equation variables were eliminated by backward technique. The good fit indicator to be considered was the determination coefficient. The coefficients of determination R^2 were calculated by Eq. (3), **Naqvi et al., 2010**.

$$R^2 = \frac{SS_{regression}}{SS_{Total}} \quad (3)$$

Where:

R^2 Coefficient of determination.

SS Sum of square.

The best possible polynomial process correlation resulted contains the smallest number of PVs (predicted values) that best describes the variation of the sludge yield against process variables x in a statistically significant manner with high confidence level was the following:

$$\boldsymbol{y = A_o x_1 + A_1 x_1^2 + A_2 x_1^3 + A_3 x_2 + A_4 x_2^2 + A_5 x_1 x_2 + A_6 x_2 x_3 + A_7 x_1^2 x_2^3} \quad (4)$$

Where:

y Percentage of Sludge Removal.

x_1 Mixing Speed.

x_2 Mixing Time (min).

x_3 Operating Temperature (^oC).

A Constant.

Eq. (4) was constrained to the following constraints, $300 \leq x1 \leq 900$ rpm, $15 \leq x2 \leq 60$ min, and $25 \leq x3 \leq 40$ ^oC. All of the results concerning the suggested correlation were obtained by Statistica Software Program. The detailed equation for each of the studied binary solvent can be found in **Table 4**.

The statistical details of the process correlation equation obtained by *Statistica software program* can be found in **Table 5** and **Fig. 5**. In the analysis of variance **Table 5**, the variability amount (8.005657, 33.48974, and 55.27092) is obviously larger than the residual error amount (0.719098, 1.75763, and 6.56869) for N:MEK, N:n-But, and N:iso-but respectively. The significant difference is sufficient to confidently deny the null hypothesis. Besides the P value is less than 0.05; that is, no MLR relationship exists between (y) and the retained PVs **Naqvi et al. 2014**. The R^2 value was equal to 0.957, 0.974, and 0.944 for N:MEK, N:n-But, and N:iso-but respectively indicates that more than 95.7%, 97.4%, and 94.4% of the variation in the calibration response data has been absorbed by the process correlation. These are strong evidences, ensuring that in new circumstances (within the range of the process variable constraints) the correlation will have good predictive capability for percentage of sludge removal.

5. CONCLUSIONS

Solvent extraction-flocculation process operation variables have a direct influence on the percentage of sludge removal. But by using N:MEK, N:n-But, and N:Iso, the specific gravity significantly reduced to less than the virgin base oil value, the TAN value have been recovered to the same value of the virgin base oil, and the ash content have been reduced by 50%. The studied variables were efficiently represent by a mathematical correlation, which could have a direct impact on the economic considerations as it was used to determine the optimum operating conditions, thus save the time and resources.

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Table 1. The properties of the Al-Daura oils.

No.	Specification	Base oil	Virgin oil	Used oil
1	Specific gravity@ 60/60 °F	0.8822	0.8854	0.8933
2	Viscosity, cSt, @ 40°C	65.811	113.6	117.02
3	Viscosity, cSt, @ 100°C	8.656	14.994	15.134
4	Viscosity index	100.6	136.9	134.4
5	Ash wt.%	0.002	0.002	1.051
6	Total acid number (TAN) mg/g.	5.928	2.903	3.584
7	Water wt.%	Nil	Nil	Nil

Table 2. Experimental results of binary solvents at different composition.

		N:MEK			N:n-But			N:Iso-But		
	SOR	Yield	POL	PSR	Yield	POL	PSR	Yield	POL	PSR
40:60	2:1	88.45	13.40	5.57	76.1	24.93	3.83	74.25	21.29	7.11
	3:1	89.71	10.28	3.91	83.7	22.06	3.51	78.27	17.39	3.57
	4:1	95.27	8.825	3.70	86.4	10.44	2.41	78.40	12.81	2.16
	5:1	95.04	5.037	2.99	89.2	7.657	0.58	79.45	10.85	1.13
	6:1	95.52	2.164	2.20	91.3	4.827	0.21	80.52	10.74	0.71

30:70	2:1	92.04	7.62	4.12	89.43	11.65	3.54	92.30	9.19	6.49
	3:1	92.13	7.24	2.95	90.55	9.11	1.00	94.22	9.11	1.41
	4:1	92.57	3.50	1.75	90.60	7.57	0.96	98.38	6.03	0.75
	5:1	93.62	2.75	1.42	91.72	6.16	0.54	98.96	5.62	0.74
	6:1	95.75	2.08	1.04	94.96	4.70	0.12	99.79	5.70	0.46
20:80	2:1	85.48	20.52	1.33	88.56	6.03	4.24	80.19	16.35	6.70
	3:1	88.97	15.70	0.92	88.47	4.37	0.96	92.55	11.03	5.20
	4:1	89.09	4.79	1.00	88.68	4.29	0.71	94.30	8.07	4.12
	5:1	89.68	4.04	0.71	89.76	3.87	0.69	94.63	6.82	3.99
	6:1	90.67	3.37	0.37	91.14	3.33	0.33	95.50	6.45	3.91

Table 3. The Properties of used oil and recovered base oil at the optimized conditions.

Properties	Used oil	Recovered base oil		
		N:MEK	N:n-But	N:Iso-But
Specific gravity at 15°C g/cm^3	0.8934	0.8785	0.8802	0.8803
Viscosity at 40 °C ,cSt	117.02	26.293	44.893	26.982
Viscosity at 100 °C, cSt	15.134	5.048	7.197	5.096
Viscosity index (VI)	134.4	120.5	121.2	118.2
Ash content (wt%)	1.0511	0.8051	0.6070	0.5767
Water Content	Nil	Nil	Nil	Nil
Total acid number (mg KOH/g oil)	3.5839	1.9702	2.8542	2.1786
Yield (%)	-	86.25	87.75	88.88
POL (%)	-	4.54	8.46	3.62
PSR (%)	-	2.54	5.63	6.12

Table 4. The binary solvent mathematical correlations.

Solvent	Correlation	R^2
N:MEK	$y = 0.0396\, x_1 - 5.98E - 5\, x_1^2 + 4.5E - 8\, x_1^3 - 1.0578\, x_2 + 0.0282\, x_2^2 + 4.44E - 4\, x_1 x_2 - 0.002\, x_2 x_3 - 1.025E - 9\, A_7 x_1^2 x_2^3$	0.957
N:n-But	$y = -0.0911\, x_1 + 0.0003\, x_1^2 - 1.5E - 7\, x_1^3 + 0.261\, x_2 + 0.013\, x_2^2 - 0.0012\, x_1 x_2 + 0.0016\, x_2 x_3 - 5.13E - 10\, x_1^2 x_2^3$	0.974
N:iso-But	$y = -0.1871\, x_1 + 0.0004\, x_1^2 - 2.82E - 7\, x_1^3 + 3.8279\, x_2 - 0.0771\, x_2^2 - 0.0018\, x_1 x_2 - 0.0072\, x_2 x_3 + 2.58E - 9\, x_1^2 x_2^3$	0.944

Table 5. Analyses of variance and goodness of fit of the correlation with using binary solvents.

Heavy Naphtha : MEK (N:MEK)				
	SS	DF	MS	P-value
Regression	64.04525	8.00	8.005657	0.0169
Residual	2.87639	4.00	0.719098	
Total	66.92164	12.00		
Heavy Naphtha : n-But (N:n-But)				
	SS	DF	MS	P-value
Regression	267.9179	8.00	33.48974	0.0062
Residual	7.0305	4.00	1.75763	
Total	274.9485	12.00		
Heavy Naphtha : iso-But (N:iso-But)				
	SS	DF	MS	P-value
Regression	442.1674	8.00	55.27092	0.0281
Residual	26.2748	4.00	6.56869	
Total	468.4421	12.00		

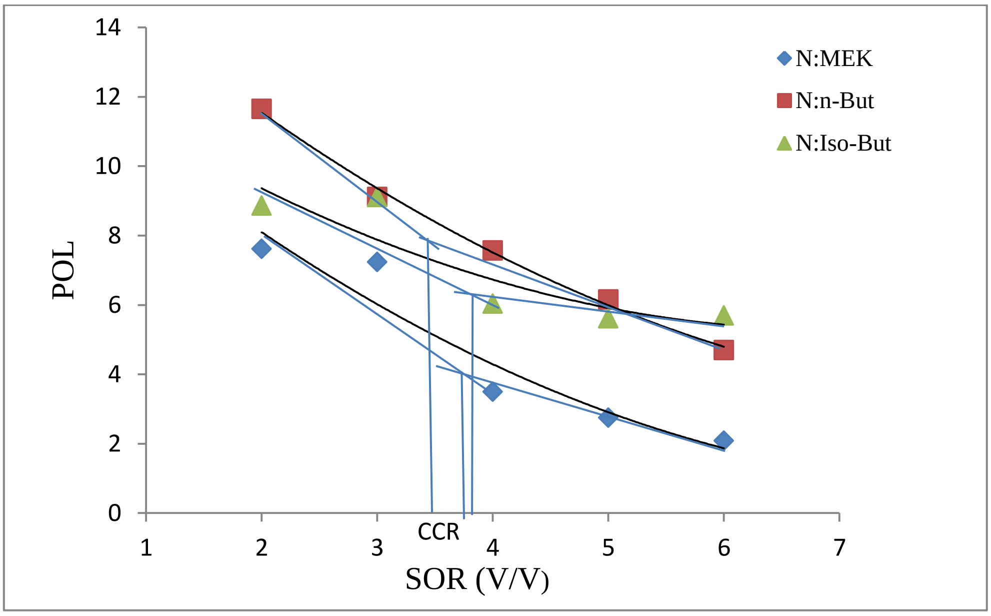

Figure 1. Graphical determination of the critical clarifying ratio (CCR) based on percentage of oil loss (POL).

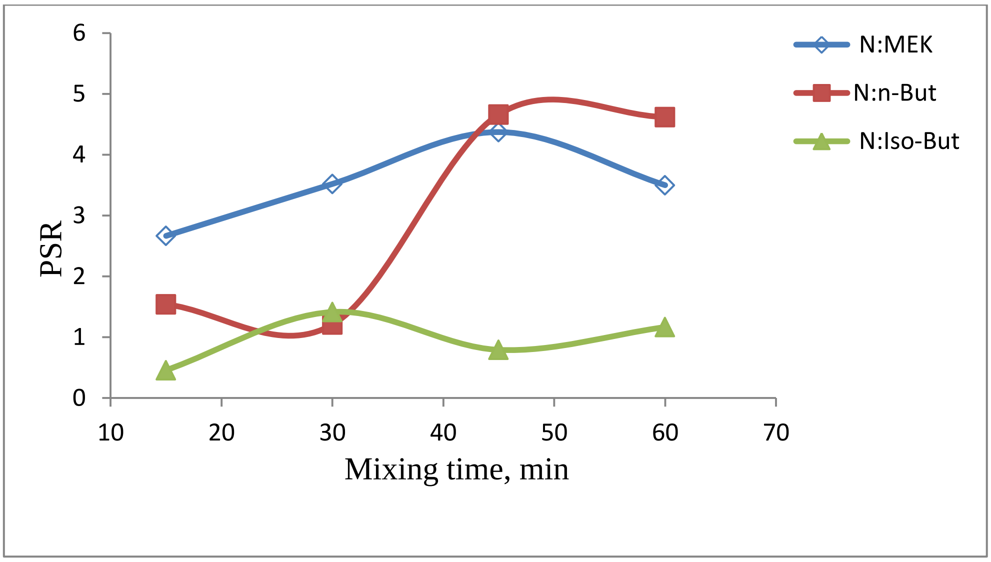

Figure 2. The effect of mixing time on the percentage of sludge removal (PSR).

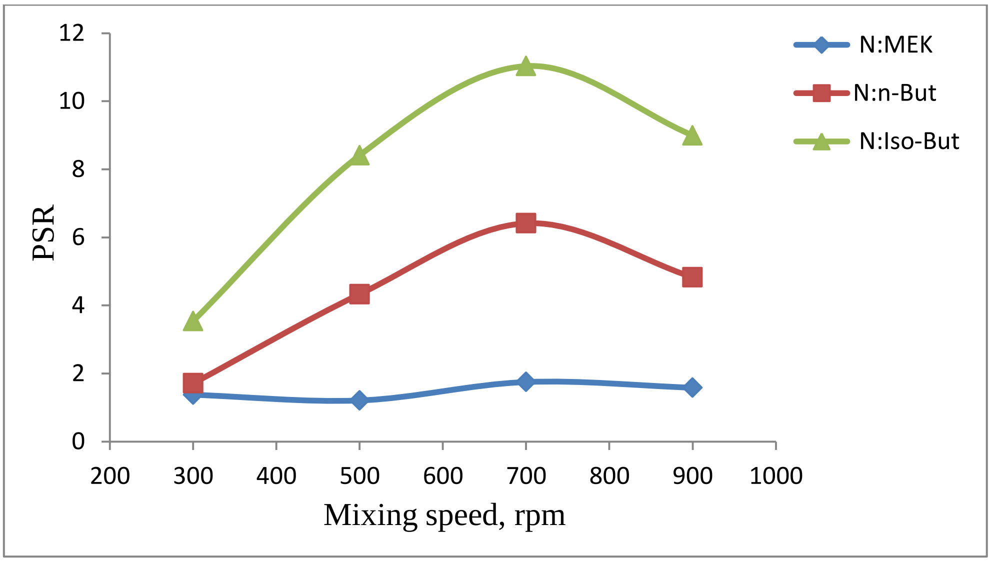

Figure 3. The effect of mixing speed on the percentage of sludge removal (PSR).

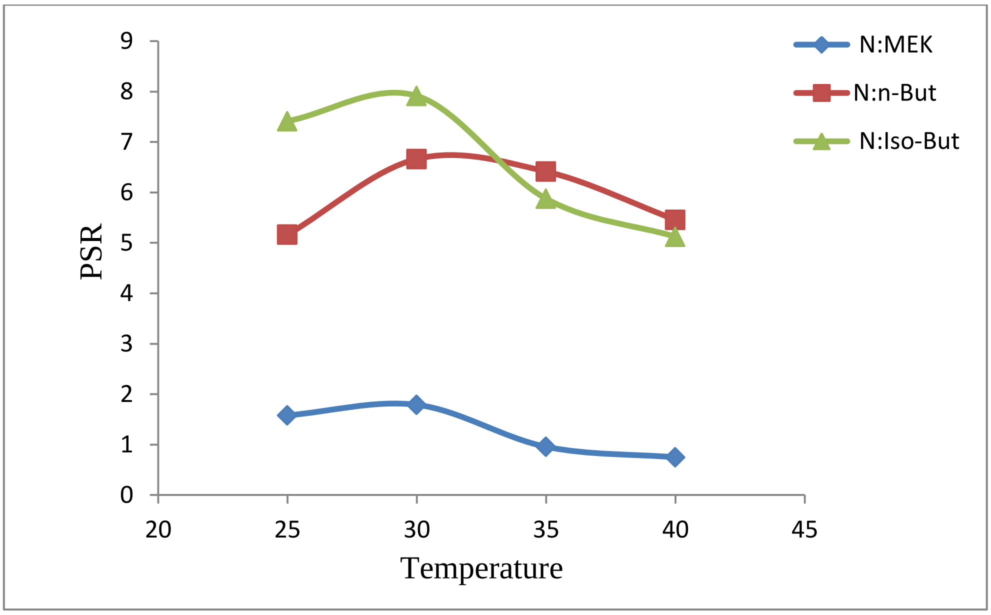

Figure 4. The effect of operating temperature on the percentage of sludge removal (PSR).

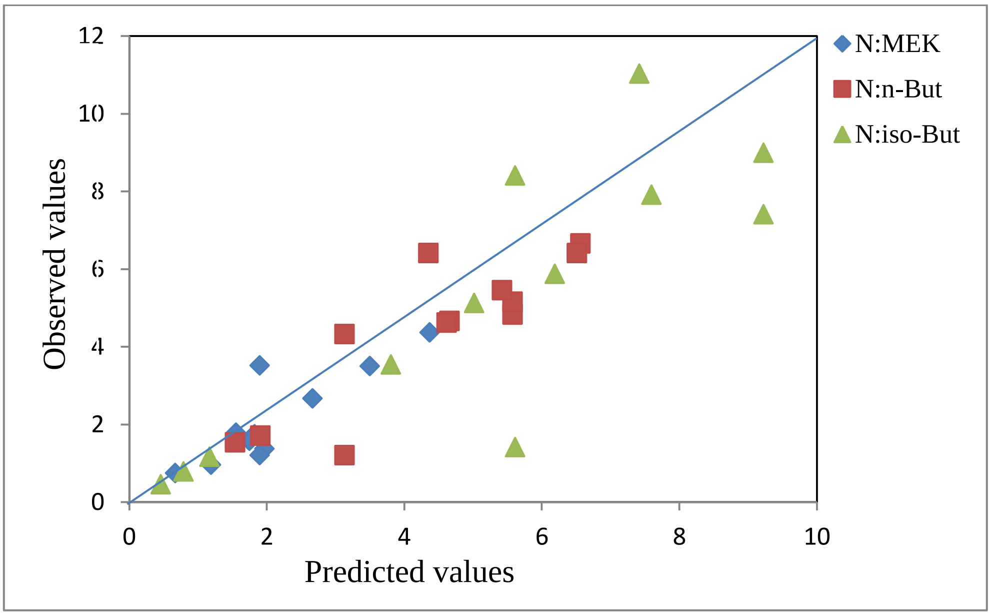

Fig 5. The observed versus predicted values of the PSR.