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Extractive/oxidative desulfurization optimization of diesel feed stock by ionic liquid utilization/thermodynamic analysis

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ABSTRACT

The current research paper focuses on the physicochemical optimization's parameters for the Microwave Assisted Extractive/Oxidative Desulfurization (MAEOD) of a real Algerian diesel. The oxidative desulfurization was carried out using vanadyl acetyl acetonate as catalyst, hydrogen peroxide (30%) as oxidant, and an acid Bronsted Ionic Liquid (IL) in situ synthesized as extractor. The utilized optimization method is consistent with the full factorial experiment. The engaged parameters are the masse ratio (catalyst/oil), the volume ratio (IL/oil), and the reaction time. Quantification of total sulfur in samples was realized by X-ray fluorescence, and a regression model is formed. The most important model parameters' interaction effects are examined, and the ISO-response curves are outlined in order to simulate the optimal conditions. The optimum yield (81.77%) was achieved during a time radiation of 70 s with a catalyst/oil ratio of 0.4% and IL/oil volume ratio of 11%, with correlation 99.21.

Keywords: Optimization; Oxidative desulfurization; Microwaves; Ionic liquid.

1 INTRODUCTION

Limiting sulfur content to ultralow levels has recently been one of the most important fuel regulations with the goal of decreasing the environmental and health consequences because of the acid rains and the air pollution associated with the transportation fuels' combustion, which forms SOx. It is looking ahead to have zero-emission in the forthcoming future based on (Aguiar et al., 2014 & Moussa et al., 2014) as a result of the increased attention, which has been directed toward efficient and novel desulfurization technologies (Su et al. 2019).

Recently, there has been a better interest in examining some alternate energy sources in the transportation and energy sectors (Afzal et al., 2019).

As it can be accomplished without the need for Hydrogen and also at low pressure and temperature values, Oxidative Desulfurization (ODS) is considered a better approach than Hydrogen Desulfurization (HDS) and, as a result, one of the encouraging technologies (Meman et al., 2014).

In ODS approach, the temperature value should be under a sensitive control, and as this is reachable with using microwave energy, this can be one of the key reasons for spreading this approach and getting the researchers' attention. This energy has a frequency ranging from 0.3 GHz to 300 GHz, with electromagnetic spectrum being between radiofrequency and infrared radiation and wavelengths between 1 mm and 1 m (Ma et al., 2014). Some effective methods of nitrogen and sulfur compounds removal, using ILs from real and model fuels, were presented by a study done by Zolotareva et al. (2019). A review study about "Ionic Liquids for Desulphurization" was recently carried out by S. Abdullah et al. (2018). It concluded that further research is needed in the field of extractive desulphurization process using selective ILs.

Because of their attractive chemical and physical properties, that is, no flammability, thermal and chemical stability, and low vapor pressures, Ionic Liquids (ILs) have recently attracted the scientific community's attention. The latter is considered a stellar option to substitute volatile organic solvents for extra clean and green technologies (Nie et al., 2013; Martinez-Magadan et al., 2012). It is well known that the ILs can not remove a high amount of sulfur with the absence of oxidant. Nevertheless, high oxidant/sulfur rations should be applied for sulfur removal if there is no catalyst. Many types of the latter, which are able to remove high sulfur at low oxidant/sulfur ratios, molybdenum, and iron catalysts, have been examined by Mota et al. (2012), Zhu et al. (2011), Lü et al. (2014), and Jiang et al. (2014), respectively. A prepared functional "[Bmim] Cl/$CoCl_2$ ionic liquid" with potassium monopersulfate (PMS) was prepared to eliminate benzothiophene from octane. [Bmim] Cl/$CoCl_2$, PMS dosage, reaction time, and temperature for desulfurization were examined based on response surface methodology and real experiment. The order of four experimental conditions followed the temperature > time > [Bmim] Cl/$CoCl_2$ dosage > PMS solution dosage (Y. Zhang et al., 2018).

The overall objective of this project work is to investigate the use of nonconventional, other than Hydrodesulfurization (HDS) process, chemical methods for the desulfurization of fuel oils. In general, the chemical methods should be more selective, inexpensive, efficient, and regenerable to justify further development of work. These methods will include chemical oxidation, solvent extraction, adsorption, and the use of a novel laser-based photo-oxidation process. The authors, here, report a simple procedure for desulfurization of model fuel oil by chemical oxidation and extraction with ionic liquids. So, the principal idea behind the current research is to examine the optimum circumstances to reach a maximum desulfurization level of real diesel feedstock using a combination of microwave radiations and catalytic activity of (VO $(acac)_2$) in an acid Bronsted ionic liquid, synthesized under microwave radiations.

2 MATERIALS AND METHODS

2.1 Materials

Vanadyl acetylacetonate (VO $(acac)_2$) catalyst was synthesized with respect to a method reported by Woolins et al. (2014) and purified by recrystallization from $CHCl_3$ (Gekhman et al., 1998). Real diesel fuel with a total S-content of 450 ppm is supplied by Algiers Refinery. Chemicals were purchased from different companies and used as received. Chloroacetic acid, hydrogen peroxide, and pyridine (30 %, v/ v) were delivered from "Sigma Aldrich." All solvents, $CaCl_2$, and sulfuric acid were purchased from "Panreac Company."

2.2 IL synthesis and characterization

The acid Bronsted ionic liquid [CH2COOHPy] [HSO4] was made regarding the "two-step synthesis method." At the start, 1.2 mol of Pyridine and 1.0 mol of Chloroacetic Acid were placed in a refluxed round bottom flask and mixed in a magnetic stirrer, and the mixture is heated intermittently in a perforated household MW oven (Brandt SE2612S-900W) at 150 W (1 min irradiation with 10 min mixing) until a white precipitate is formed.

In order to get [$CH_2COOHPy$] Cl, which then was dried using vacuum, the precipitate was firstly filtered off and then washed with dichloromethane three times.

After that:

1- A "3-neck refluxed round" bottom flask is used.
2- Equimolar quantities of "Sulfuric acid" (96%) were dropped, gradually, to [$CH_2COOHPy$]Cl at the same conditions of the microwaves to obtain [$CH_2COOHPy$][HSO_4].
3- Sodium hydroxide solution was then used to absorb the by-product HCl.
4- Once the generated HCl was not sensed by Ammonia solution, the [$CH_2COOHPy$][HSO_4] needed to be dried at vacuum, and we used a desiccator to store it.

The structure of IL has been identified by ^{1}H NMR and ^{13}C NMR (Bruker 300 MHz) and infrared spectra (Bruker Alpha-T). The density of IL was determined by "Pycnometer method," and "Rotational Rheometer (RS600, Thermo, USA)" was operated to find out its viscosity, where the results were as follows:

"Density: 1.75 g/ml (30 °C); viscosity: 0.30 Pas (30 °C).), infrared spectrum (KBr, γ/cm^{-1}): 3396, 2924, 1735, 1638, 1490, 1229, 1063, 839, 585, 641, 455. ^{1} H NMR (300 MHz, D_2O): 5.21 (s, 2H), 7.79(t, 2H), 8.30 (t, 1H), 8.49 (d, 2H); ^{13C} NMR (300 MHz, D_2O): 58.66, 126.22, 143.64, 145.04, 166.13."

2.3 Extraction–Oxidation Desulfurization

The experiments of MAEODS were carried out using a 50 mL of a "round-bottom flask." The 1.2 mL of ionic liquid, 20 mL of model oil, 40mg of VO $(acac)_2$, and H_2O_2 mixture was stirred vigorously under MW conditions for 3.5 min as shown in Figure 1. Two layers formed. The upper oil phase was separated, washed, dried, and analyzed. The lower IL/catalyst phase was reused without regeneration.

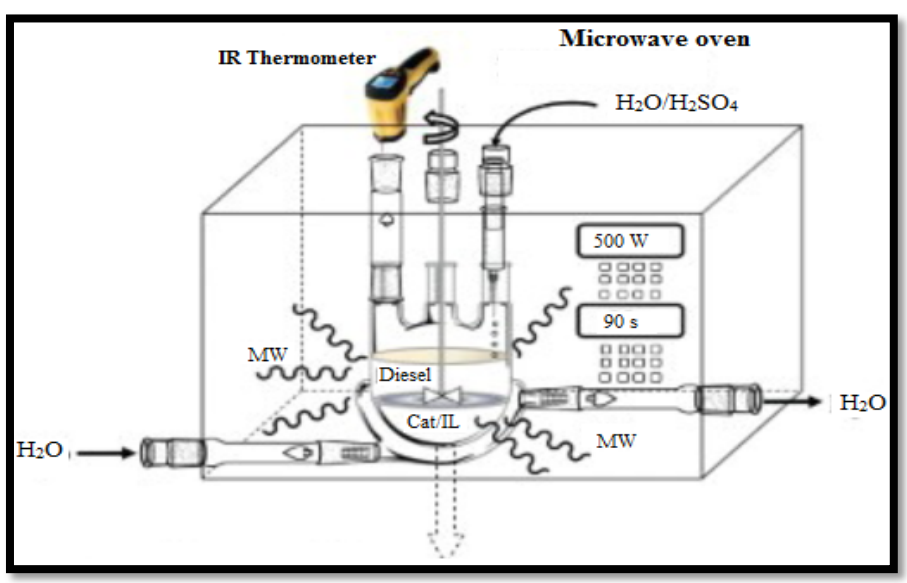

Figure 1. The experimental setting.

The "desulfurization yield (R %)" is computed using equation (1):

$$R\% = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

where C_0 and C_t represent the initial and at time (t) (after treatment) sulfur concentration (ppm), respectively.

2.4 Sulfur analysis

The total concentration of sulfur in diesel samples was analyzed by X-ray fluorescence using un "X-ray fluorescence spectrometer (OXFORD Lab X-3500)" according to the standard method (ASTM 2622). Using what is known as "six liquid calibration standards (obtained from AccuStandard)" and at a range from 0 to 1000 ppm concentration ratio, the spectrometer calibration was carried out.

2.5. Design of experiments

Understanding the procedure of optimization in depth can lay the groundwork for correct and suitable optimization with lower possible cost (Daabo et al., 2018-2020). The term "fitness" in nature can be characterized as the most robust strategy. From this point, the idea of optimized Extractive/Oxidative Desulfurization began and is presented in this paper. With the aim of optimizing the related parameters as well as analyzing their interaction, a complete "factorial design" was operated in this research. The latter is a "first-degree polynomial mathematical model" and a "statistical technique." It is beneficial and suitable for "modeling and analyzing the problems," in which their "response of interest" is affected by a number of variables (Montgomery, 2001). These variables are as follows:

1- "Volume reaction (IL/oil)", X_1.
2- "Masse ratio (catalyst/oil)", X_2.
3- "Reaction time", X_3.

The above parameters as well as their upper and lower values, that is, ranges, were selected based on the preliminary experiments and literature data and. The mathematical model of the experimental results is given as follows:

$$Y = a_0 + \sum_{i=1}^{n} a_i X_i + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} a_{ij} X_i X_j + \sum_{i=1}^{n-2} \sum_{j=i+1}^{n-1} \sum_{k=j+1}^{n} a_{ijk} X_i X_i X_k + \sum_{i=1}^{n-3} \sum_{j=i+1}^{n\_2} \sum_{k=j+1}^{n-1} \sum_{l=k+1}^{n} q_{ijkl} X_i X_j X_k X_l \quad (2)$$

where the predicted response (desulfurization yield) is represented by Y, a_0, a_i $a_{i\text{-}l}$, are the constant coefficient, the linear coefficients, and the interaction coefficients, respectively. Similarly, X_i, X_j, X_k and X_l are the "coded values" of the desulfurization conditions variables.

3 RESULTS AND DISCUSSİONS

3.1 Optimum Conditions

Using the "full factorial experiment" at two various levels, where the lower level was coded with value of (-1) and the upper one with (+1), the current analysis has been carried on, as given in Table 1.

Table 1. Conditions of experimental design.

Parameters	Designation	Level (−)	Level (+)
Volume ratio IL/oil (%)	X_1	9	11
Masse ratio catalyst/oil (%)	X_2	0.4	0.6
Reaction time (s)	X_3	70	110

3.2 Planning of Desulfurization Reactions

As is clear, eight reactions can be achieved using four parameters for any "experimental design." The latter has been carried out based on the "Matrix of Experience" shown in Table 2 that uses three parameters under investigations, mentioned earlier. So, 23 (= 8) will result in reactions and requires the realization of 8 desulfurization reactions. The results of desulfurization are given in the last column.

Table 2. Experimental design matrix for diesel oxidative desulfurization.

Test n°	X1 (v,%)	X2 (wt,%)	X3(t)	Desulfurization yield (%)
1	-1	-1	-1	71.54
2	+1	-1	-1	81.77
3	-1	+1	-1	74.12
4	+1	+1	- 1	77.46
5	-1	-1	+1	75.9**0**
6	+1	-1	+1	79.12
7	-1	+1	+1	78.79
8	+1	+1	+1	75.90

3.3 Development of Regression Model

Based on Yates method (Atikler et al., 1997), which results in model that has eight unknown variables and equations, the current work has been carried out with the aim of finding out the effect of both the main variables and their interactions. The mentioned model is given using the "Matrix product" where the relevant data were statistically preserved using the software "MODDE 6.0." Equation (3) shows its mathematical model:

$$Y = 76.9373 + 1.64125X_1 - 0.161249X_2 + 0.69875X_3 - 1.52876X_1X_2 - 1.55875X_1X_3 + 0.0787482X_2X_3 \quad (3)$$

Using the "Ascendant regression method, forward technique" (Boehm et al., 1994), the influence of each individual parameter has been figured out. The highlighted technique results in "submodels," in which different criteria can be utilized to govern their choice. It is well known that the value of "the correlation coefficient (R^2)" can be one of the most important methods that can evaluate the suitability degree (Gürses et al., 2002). Having said that, the value of "the correlation coefficient (R^2)" can be increased monotonically when new variables are introduced, that is, less or more correlated with respect to the old ones. So, to solve this issue, "the higher adjusted R^2_{adj} value" was selected.

The "parameter significance level" was set to p=0.05, where the outcome of programmed "tests model" is given in Table 3, which shows that the highest influence parameter, with a 0.000366019 probability, was the volume ratio (LI/oil) (X_1).

Table 3. The influence of the selected parameters and their interactions.

Model		
Y	Coeff	P
Constant	76.9373	$8.41282.\ 10^{-13}$
X_1	1.64125	0.000366019
X_2	-0.161249	0.440601
X_3	0.69875	0.0150875
X_1*X_2	-1.52876	0.000511185
X_1*X_3	-1.55875	0.000466655

As seen in Table 3, the masse ratio (catalyst/oil) is not an influent parameter for the MAEODS (P=0.440601). As the standards of choice of "influent parameters" should not be only mathematical, this parameter has been maintained. The best "statistical indicators" of the model parameters are shown in Table 4, while the general equation, that is, equation 3, becomes

$$Y = 76.9373 + 1.64125X_1 - 0.161249X_2 + 0.69875X_3 - 1.52876X_1X_2 - 1.55875X_1X_3 \quad (4)$$

Table 4. Adsorption yield of variances (ANOVA).

Desulfurization yield (Y)	Dl	SS	Variance (MS)	F	P	SD
Total	11	65178.1	5925.28			
Constant	1	65112.8	65112.8			
Measured response of corrected average $\left(Y_i - \bar{Y}\right)$	10	65.2813	6.52812			2.55502
Corrected model of the average $\left(Y_i' - \bar{Y}\right)$	5	63.7977	12.7595	43.0036	0.00	3.57205
Residues	5	1.48354	0.296708			0.544709

By completing the modeling, variance analysis (ANOVA) contributes to obtaining more data about the investigated parameters (Goupy et al., 2006), where both pertinence verification and postulated model quality evaluation were the main concern of this step. Similar to the previous table, Table 5 presented the results obtained about the "parameters and their interactions on the adsorption yield." The achieved results of R^2, R^2_{adj}, the predictive ability given the coefficient Q^2, and the probability P, that is, 0.979, 0.958, 0.888, and close to 0, respectively, indicated the ability of the statistical tests used in the variances analysis to verify the model. After modeling, the "analysis of the residues" (Goupy et al., 2006) was utilized in order to obtain the information included in the responses. Therefore, the way that residues were dispersed, with respect to the "responses predicted by the model," can be figured out.

Based on Figure 2, the computed desulfurization values were distributed in a way that does not give a clear tendency to those points. So, the mentioned information was considered not that important for those specific data, and the model was considered appropriate.

The relative importance of different "significant parameters" is presented in Figure 3. The main outcome from this figure was that it highlighted that, compared to other parameters, X_2 is the only one that has "negative effect on the desulfurization reaction."

Figure 4 indicates a considerable interaction concerning X_1 and X_3, while X_1 is kept at its extreme levels (-1) or (+1), and the desulfurization yield decreases or increases, respectively.

The desulfurization variation highlights that when X_1 is preserved at its low level, it rises with increasing X_3,, but when X_1 is maintained at its high level, it decreases with rising X_3, so when X_1 is kept at its high level (+1), and X_3 is kept at its low point, the yield is better.

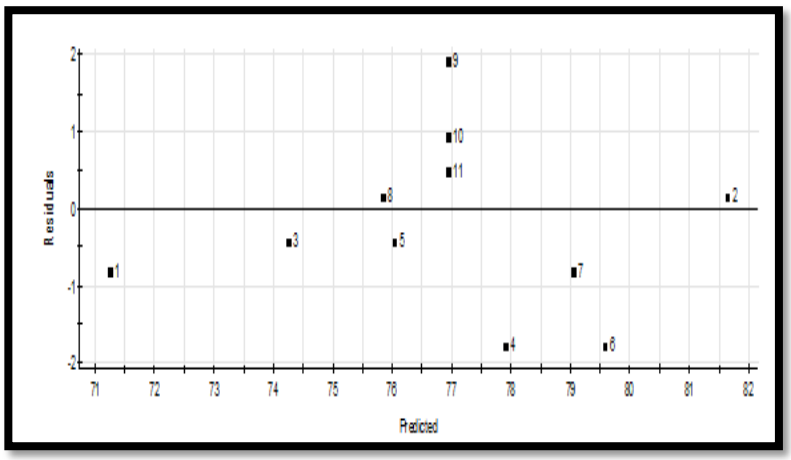

Figure 2. Response residuals achieved by the current study.

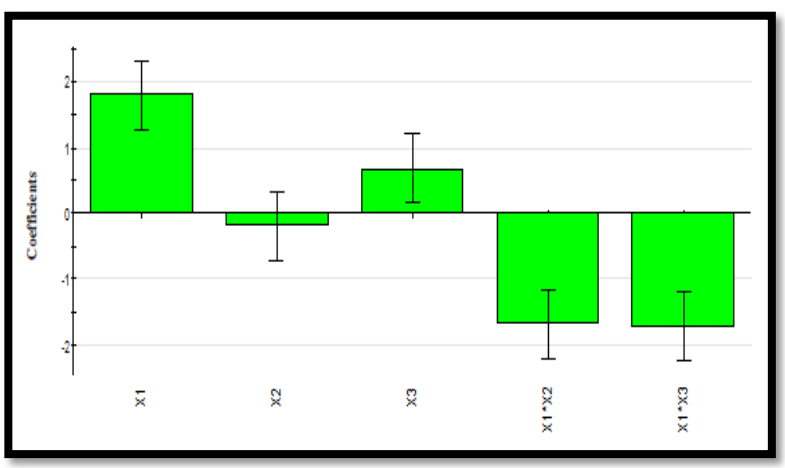

Figure 3. The influence of both the parameters and the interactions of the simplified model.

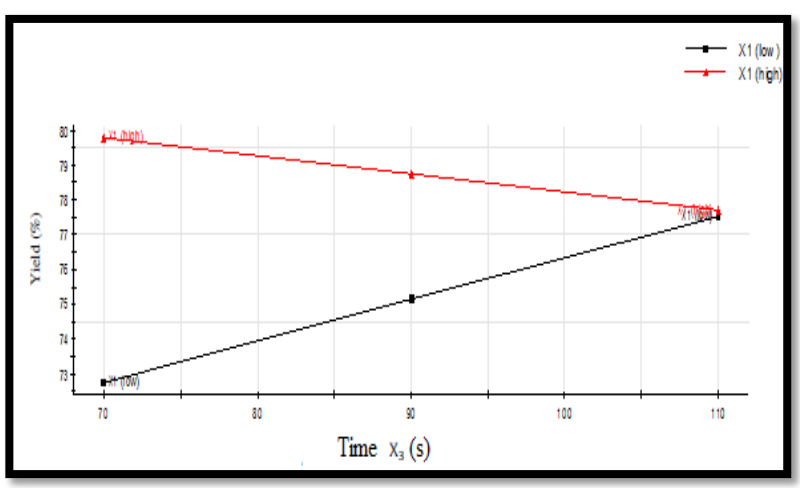

Figure 4. The influence of interaction X1 *X3.

3.4 Investigation of the Optimum Zone

To simulate the optimum conditions for the diesel fuel desulfurization, the "mathematical model" given in Equation (4) can be applied. To attain such a target, the "method of iso-response curves" may be a means. Definitely, equation (4) is adapted to another arrangement, equation (5). As key parameters X_i and X_j were nominated, the other variables were arranged at their optimum levels. As a first step, the most effective factor, that is, the parameter that can highly influence the desulfurization yield (X_1 and X_3) was nominated.

Then, those factors were fixed at their optimum levels, so X2 turns into the "key variable."

$$X_j = \frac{a + b \cdot X_i + Y}{c + d \cdot X_i} \tag{5}$$

The X_i= f (X_j) curves were charted for each constant yield (Y), where Table 3 highlighted that tests 2, 6, and 7 gave a desulfurization yield more than 78%. These "experimental conditions" were utilized in the current work of "iso-response yield curves," and the other two situations, which were highlighted previously, were studied as below.

Figure 5 shows that increasing X_1 increases desulfurization yield when reaction time is low, but when increasing reaction duration, it is possible to achieve a percent of 79. Figure 6 indicates that one cannot go beyond 79% sulfur elimination at 110 s and 9 v.% of IL the conditions of the test no. 7 when X_1 is maintained at its low level (-1). That can be explained by the effect of the interaction X_1*X_3 as shown in Figure 3. When X_2 is maintained at its high level as shown in Figure 5, it can be seen that the parameters X_1 and X_3 rise together with the desulfurization yield until reaching a desulfurization yield of 79% for a volume ratio (IL/oil) of 11% (test no. 6). For the same volume ratio at a minimal time reaction 70 s, we can reach a desulfurization yield higher than 81% (test no. 2).

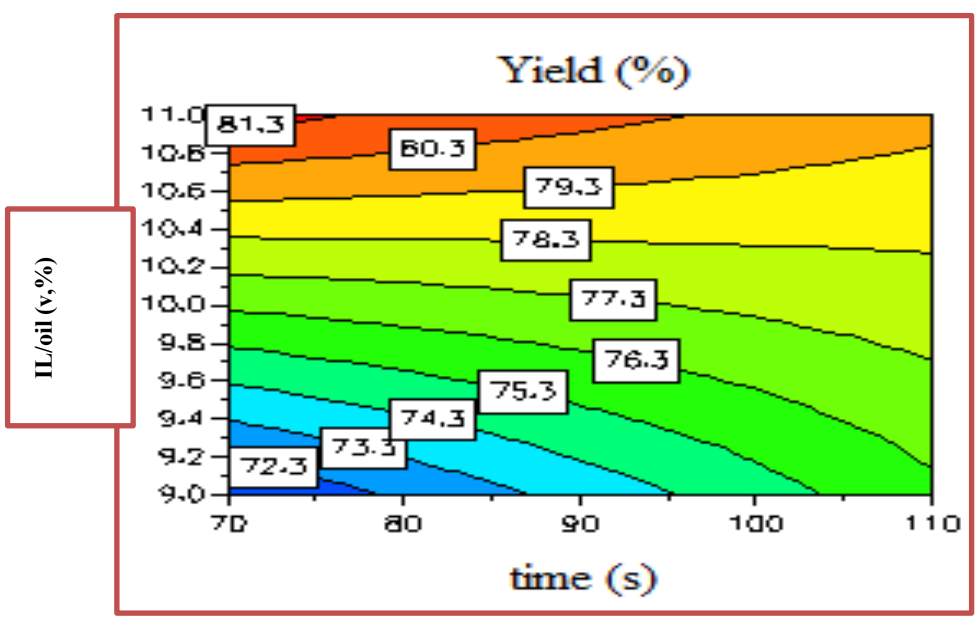

Figure 5. Evolution of (v, %) (IL/oil) according to time reaction.

After determining the theoretical optimal conditions, we performed experiments with these conditions and found an experimental optimal yield of 81.13. The correlation is 99.21%, which is shown in table 5.

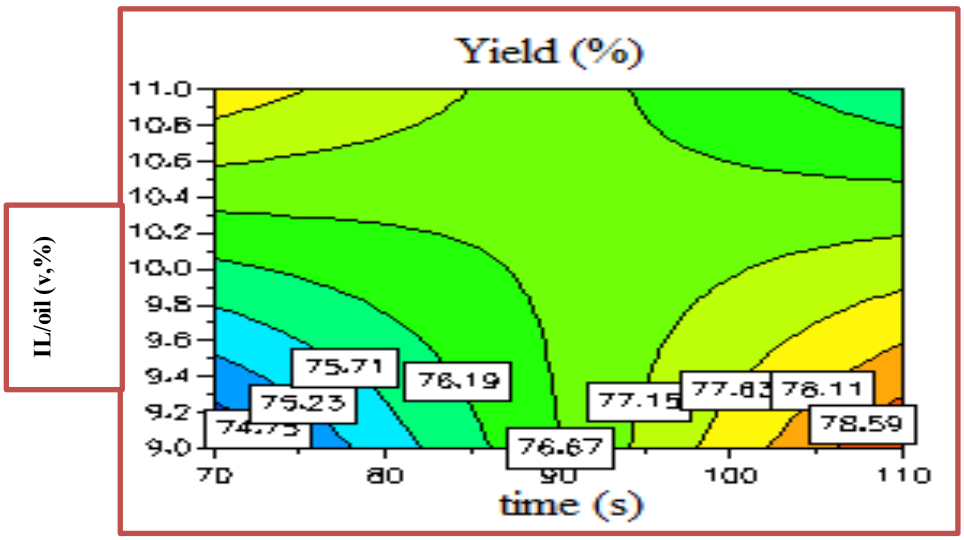

Figure 6. Evolution of (v, %) (IL/oil) according to X_2= -1, time reaction X_2=+1.

Table 5. Optimal conditions for MAEOD.

Optimal condition			Optimal yield	
X_1	X_2	X_3	R_{exp} (%)	R_{the} (%)
11	0.4	70	81.77	81.13

4 CONCLUSION

A model that shows the effect of all investigated factors as well as their interactions has been established based on the experimental outcome with respect to the "full factorial design."

Based on the variance analysis, the statistical experiments not only allow identifying further data on the "simplified model," but also validate their strength. The circumstances of the microwaves assisted extraction/oxidation desulfurization have been experimented. The "experimental design" has been operated to go into the details a "regression model" for the process parameters analysis by spotting the influential parameters. The optimum yield (81.77%) was achieved during a time radiation of 70 s, with a catalyst/oil ratio of 0.4% and IL/oil volume ratio of 11%; nonetheless, the results that were obtained using "iso-response curves" demonstrate that the analyses were carried out with 81.33% "confidence level." The interaction consequences among the parameters should be taken into concern. Using "full factorial experiment" application, the simulation and determination the optimal conditions are possible.

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