

Multilevel Factorial Design for Removing Lead from Used Lubricating Oil by Zeolite Na-A

Abdulsalam A. Matlob

Department of Medical Biochemical Analysis, Cihan University-Erbil, Kurdistan Region, Iraq

Abstract- The urgent requirements for saving the environment and the reusing of lubricating oil low cost have contributed to the need for green and a unique method for carrying out the optimization of chemical processes. In this study, a unique statistical technique called Multilevel Factorial Design and aluminosilicate material, zeolite Na-A, to optimize the removal process of lead from used lubricating oil has been applied and studied. Two variables with three levels have been used in the factorial design to obtain the experimental data. The two factors were the quantity of zeolite Na-A as adsorbent and the time of stirring. It turns out that the zeolite Na-A has the potential in removing lead ions from used lubricating oil. Lead ions concentration based on the results of Inductive Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) was lowered from 2.4 ppm in the untreated used oil to 1.3 ppm in the zeolite treated oil. The results showed the same main effect of the two factors on the removal of lead from used oil, as the concentration of the removed lead was directly proportional to the high level of both factors.

Keywords: Factorial design, Lubricating oil, Lead, Zeolite Na-A

I. INTRODUCTION

Lubricating oil is an organic mixture, consist of different macromolecules organic compounds with some additives. When the lubricating oil is used in the internal composition engine, degradation will have happened under the effect of a high temperature and many organic compounds will be formed such as carboxylic acids, carbonyl compounds, and some metals. Most heavy metals present in used lubricating oil are Pb, Al, Ni, and Fe, which are below detection in unused lubricants oil [1]. The presence of the metals could be attributed to oil additives degradation or as a result of engine or bearing wear [2]. Chemical wear and corrosion, may also produce soluble Metallo-organic species [3]. The lead content of the used motor oils is mainly caused by the use of tetra alkyl lead in the fuel [4]. According to international regulations, waste lubricants are considered hazardous substances and the use of these waste oils causes problems.

Fuscous has been spent to remove heavy metals from oil sludge [5], used lubricating oil [6,7, 8, 9], and Refinery effluents [10]. The common method used in removing the heavy metals process in its first stage includes dilution of oil by organic solvents like Hexane, acetone, 2-propanol. The second stage of the removing of heavy metals was adsorption from oils by using ions exchangers. Zeolites materials were the most affective ion exchanger has been used. It has the characteristics of being a solid crystal with a large surface area.

In this work, a statistical technique called Multilevel Factorial Design have been used. Factorial Design is a combination of mathematical and statistical techniques used for developing, improving, and optimizing processes. Usually contains three steps: (1) design and experiments; (2) response surface modeling through regression; (3) optimization. The objectives of this work are to remove cationic species especially lead as heavy metal from used lubricating oil by using zeolites Na-A, and to identify the effect of the two variables (oil\adsorbent w\w and period of stirring) on the adsorption process.

II. METHODOLOGY

- 1 gram of zeolite will add to different quantities of used lubricating oil (10, 20 and 30 ml) diluted up to 50 ml using organic solvent.
- The resulting mixture will stir magnetically for different period time (1, 2 and 3 h).
- The resulting material will filter and the filtrate will collect.
- The solvent will evaporate by heating at 50°C for 15 mins.
- The resulting material will test by ICP- OES before and after the addition of the adsorbent.

A. Experimental design

The details of the design and experiments that used in this work as follow:

Experimental design: Multilevel Factorial Design

Factors: 2

Total runs: 9

Number of levels: 3

The design of the experiment runs and the details of the two variables (oil\adsorbent w\w and period of stirring) are shown in table 2.1.

Table 1: experiment runs, oil\adsorbent w\w, and period of stirring

Run Order	Oil\Adsorbent w\w	Time of stirring\h
1	20	1
2	10	1
3	30	3
4	10	3
5	20	2
6	10	2
7	20	3
8	30	1
9	30	2

B. Potential application of zeolite Na-A

According to the international regulations, waste lubricants are considered hazardous substances and cause environmental problems. When lubricating oil is used in the internal composition engine, degradation will take place under the effect of the high temperature, and many organic compounds will be formed such as carboxylic acids and carbonyl compounds and some metals. Heavy metals present in used lubricating oil are Pb, Al, Ni and Fe, which are below detection in unused lubricant oil [11]. The presence of metals could be attributed to oil additives or to the wearing of engine or bearings [12]. Chemical wear, corrosion, may produce also soluble metallo-organic species [13]. The lead content of the used motor oils is mainly caused by the tetraalkyl lead formed in the fuel [14]. In this study synthesized Na-A zeolite was used as adsorbent to test the amount of lead ions that can be removed from used lubricating oil using the factorial design.

design variables which adopted in this study were quantity of zeolite Na-A as adsorbent and time of stirring. The response in this study is the concentration of lead. The used motor oil was obtained from a proton car at 5000 km engine service. A 2^2 factorial experiment was used; it considers two levels (the base) for each of two factors (the superscript) producing $2^2 = 4$ factorial points. The original concentration of lead in the oil was 2.4 ppm based on the results of ICP- OES using the method of ASTM 5185.

III. RESULTS AND DISCUSSION

In this research, responses are measured at all combinations of the experimental factor levels. Table 2.2 shows the factorial design and the concentrations of adsorbed lead in ppm of all set of variables. It can be concluded from this study that lead was partially removed from used oil.

Table 2: Experimental design and the concentration of adsorbed lead in ppm by zeolite Na-A

Runs	Zeolite(g)	Time of stirring(min)	Pb adsorbed(ppm)	Pb adsorbed (%)
1	1	30	0.6	25
2	2	30	0.9	37.5
3	2	15	0.7	29.1
4	1	15	0.4	16.6

A. Main effect and interaction of the two factors on the removal of lead from waste oil

The main effect plot in Figure 3.1 indicates that the two factors used, stirring time and the quantity of zeolite Na-A, have similar effects on the removing of lead from used oil. For both factors, concentration of removed lead increases as moving from the low level to the high level of the factor. As the increasing of stirring time allows increasing the contact time of absorbent with the used oil. This leads to more removal of lead from the oil.

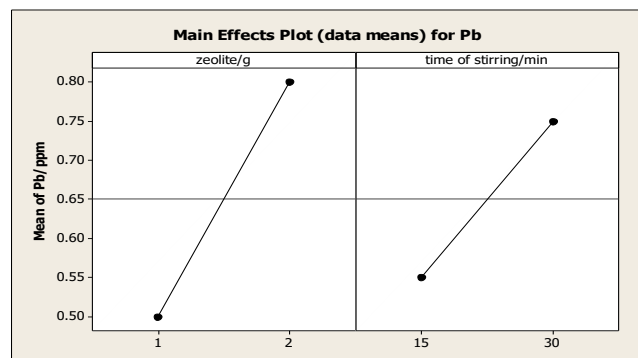


Figure 1. Main effect plot of zeolite (g) and time of stirring (min)

However, the interaction plot in Figure 3.2 shows that there is no such effect on the adsorption of lead on zeolite Na-A as the two lines in the interaction plot, which belong to the two factors, are parallel. That means there is no difference in response value of changing one factor depending on the levels of another factor.

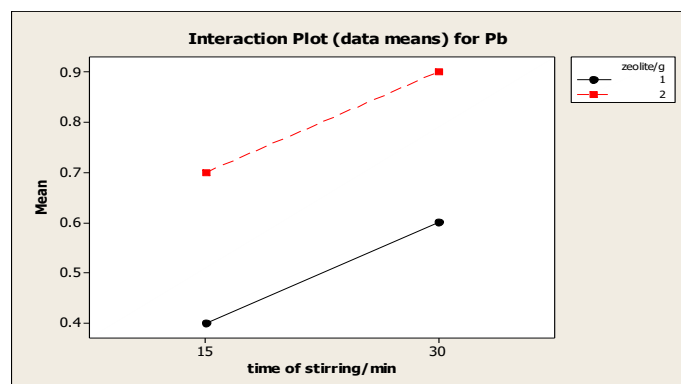


Figure 2. Interaction plot of zeolite (g) and time of stirring (min)

B. Contour plot and surface plot

Figures 3.3 and 3.4 represent the contour plot and the 3-D surface plot of removed Pb vs. zeolite quantity and time of stirring respectively. Both the contour and the surface plots show increase in Pb removed as both zeolite Na-A quantity and time of stirring increased. However, the surface plot (Figure 3.3) illustrates that the amount of Na-A zeolite has a greater effect on removing Pb compared to the duration of stirring.

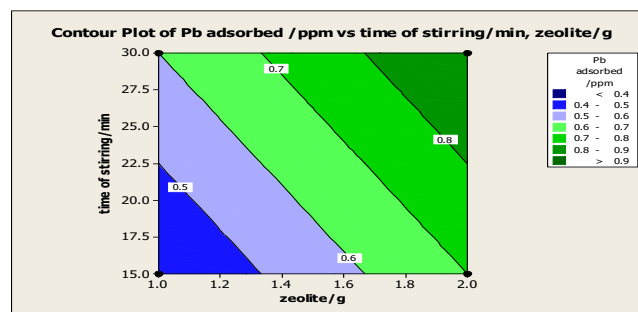


Figure 3. Contour plot of Pb (ppm) vs. time of stirring (min) and quantity zeolite Na-A (g)

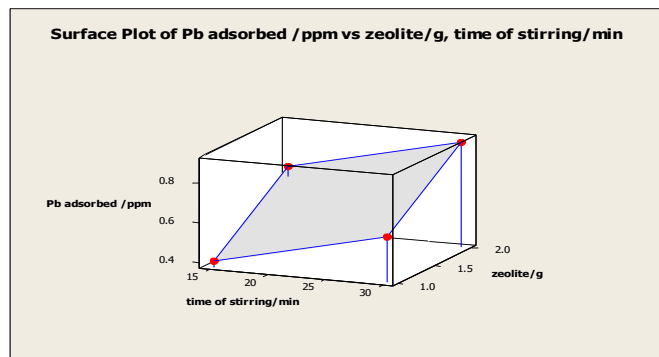


Figure 4. Surface plot of Pb (ppm) vs. time of stirring (min) and quantity zeolite Na-A (g)

C. Zeolite Na-A in the removal of lead from used lubricating oil

Zeolite Na-A was used to remove lead ions from used lubricating oil. Lead was removed partially when different amount of zeolite Na-A as adsorbent accompanied by different time of stirring were used. Lead ions concentration was lowered from 2.4 ppm in the untreated used oil to 1.3 ppm in the zeolite treated oil. This result was achieved when 10 g of used oil was stirred for 30 minutes in the presence of 2 grams of zeolite Na-A. Both factors, zeolite Na-A quantity and time of stirring, are significant and have a similar effect on the removal of lead ions. The removal of lead ions increased in used lubricating oil as the two factors (mass of zeolite and stirring time) increased.

CONCLUSION

This research was conducted to evaluate the removing of lead from used lubricating oil by zeolite Na-A using factorial design. Lead was removed partially when different amount of zeolite Na-A as adsorbent accompanied by different time of stirring were used. Lead ions concentration was lowered from 2.4 ppm in the untreated used oil to 1.3 ppm in the zeolite treated oil. This result was achieved when 10 g of used oil was stirred for 30 minutes in the presence of 2 grams of zeolite Na-A. Both factors, zeolite Na-A quantity and time of stirring, are significant and have a similar effect on the removal of lead ions. The removal of lead ions increased in used lubricating oil as the two factors (mass of zeolite and stirring time) increased.

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