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Rheological Characteristics of Olive Oil: A Statistical Approach

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Abstract: *In this study, a statistical analysis based on Response Surface Methodology (RSM) was employed to study the effect of temperature on the apparent viscosity of olive oil. The data generated from the response surface method showed that cubic effect of the temperature on the apparent viscosity was significant (p<0.05). Regression equation for the apparent viscosity was also established and found that the predicted values from the regression equation are in good agreement with the observed ones, implying that the regression equation could be used to predict and optimize the desired apparent viscosity. The obtained results also showed that a remarkable decrease in apparent viscosity with increasing temperature (from 10-50ºC) was observed.*

Keywords: rheology, viscosity, temperature, olive oil, statistical analysis.

1. INTRODUCTION

Olives is an important crop in the Mediterranean countries. The olive tree, which belongs to the olive family, grows in different regions of Mediterranean and semi Mediterranean climates and it produces the olive fruit from which oil can be extracted. Olive oil is a complex mixture of a number of compounds such as fatty acids, vitamins and phenolic compounds. This mixture of chemical compounds is believed to be able to provide good health benefits to the consumers [1-5]. For this reason, the olive has been a significant product for people of the Mediterranean countries for a long period of time. Among the factors that can affect the olive's shelf life are light and temperature. Heating or keeping the oil at high temperature will surely age it. This will make the oil rancid. Among important parameters in food industry, viscosity plays an important role. It is an important factor to determine the quality and stability of food system and to characterize the fluid texture [6].

Response Surface Methodology is an empirical modelization technique, which has been successfully used in different types of food all over the world [7-9]. It is considered as a combination of mathematical and statistical methods, which are suitable for designing experiments, building models, determining the influence of several factors, reducing the number of experimental runs required and finding optimum conditions for desirable responses [10-12]. In addition, RSM was designed for obtaining information in shorter time and at less cost [13].

The aim of this study is to examine the effect of temperature on the olive oil apparent viscosity by applying response surface methodology.

2. Materials and methods

The olive oil utilized in this work was obtained from a mill located in Sabratha city, west of Libya. All samples were kept in glass bottles in a dark place at room temperature until analysis.

2.1 Viscosity measurement

Rheological measurements were performed on a Brookfield Digital Viscometer, model DV-II + Pro, with an attached UL adapter. The viscosity was determined using 20 mL of the sample in each analysis and the shearing time was 15 second. Temperature was controlled using a water bath with precision of ±1ºC. Analysis was done in a temperature range of 10–50ºC.

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2.2 Statistical analysis:

The response surface graphs and regression analysis were carried out by using experimental data together with statistical software package Design Expert 6.0.6 (Stat-Ease Inc., Minneapolis, MN). One factor RSM experiment in Design Expert was applied in this work. Analysis of variance (ANOVA) was used for the evaluation of the statistical significance of the model. The experimental data along with the results are presented in Tables 1-3.

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Table 1: Design layout using the Design-Expert Version 6.0.6 software and experimental results.

Run	Std	Factor (Temperature)/(ºC)	Response (Viscosity)/(cP)
1	7	50.00	23.63
2	1	10.00	129.42
3	3	20.00	76.34
4	2	10.00	129
5	6	40.00	33.81
6	4	30.00	50.17
7	8	50.00	23
8	5	30.00	49.17

Table 2 : Response: Apparent viscosity. (Sequential Model Sum of Squares).

Source	Sum of Squares	DF	Mean Square	F-Value	Prob > F	
Mean	33093.93	1	33093.93			
Linear	12069.92	1	12069.92	59.37	0.0003	
Quadratic	1168.74	1	1168.74	114.67	0.0001	
<u>Cubic</u>	<u>48.23</u>	<u>1</u>	<u>48.23</u>	<u>70.78</u>	<u>0.0011</u>	<u>Suggested</u>
Residual	2.73	4	0.68			
Total	46383.54	8	5797.94			

Table 3: Lack of Fit Tests.

Source	Sum of Squares	DF	Mean Square	F-Value	Prob > F	
Linear	1218.91	3	406.30	1549.50	0.0001	
Quadratic	50.17	2	25.09	95.67	0.0019	
<u>Cubic</u>	<u>1.94</u>	<u>1</u>	<u>1.94</u>	<u>7.40</u>	<u>0.0726</u>	<u>Suggested</u>
Pure Error	0.79	3	0.26			

3. RESULTS AND DISCUSSION

3.1 ANOVA analysis and fitting of cubic model

Performance of test for significance on individual model coefficients and test for lack-of-fit is required to ensure a good model [14]. Generally, ranking of the significant factors is based on F-value or P-value (Prob. > F). Higher F-value (correspondingly lower P-value) indicates more significance of the corresponding coefficient. In this work, the ANOVA analysis for the apparent viscosity is presented in Table 4, the F-value for the model is 6499.02 (high value) and the corresponding P-value is very low (less than 0.0001), which suggests a significant model. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise for the response (apparent viscosity). Studying the precision of the model (Table 5) shows that the coefficient of determinations (R^2) values for apparent viscosity is 0.9998 (99.98%), which is very close to 1. This indicates that only about 0.02% of the total variation can not be explained by the model, which shows a good accuracy of the polynomial model. In addition, the predicted R^2 value of 0.9988 for apparent viscosity were found to be in an excellent agreement with the adjusted R^2 value which is 0.9996 for the response. Based on these observations, it can be observed that the regression model provides a good representation of the relationship between the variable and the response (apparent viscosity).

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Table 4: ANOVA Table for Response Surface Cubic Model (Response: Apparent Viscosity).

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	13286.89	3	4428.96	6499.02	< 0.0001	significant
A	*425.72*	*1*	*425.72*	*624.69*	*< 0.0001*	
A^2	*1168.74*	*1*	*1168.74*	*1715.00*	*< 0.0001*	
A^3	*48.23*	*1*	*48.23*	*70.78*	*0.0011*	
Residual	2.73	4	0.68			
Lack of Fit	*1.94*	*1*	*1.94*	*7.40*	*0.0726*	*not significant*
Pure Error	*0.79*	*3*	*0.26*			
Cor Total	13289.62	7				

Table 5: Model Statistics Summary

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS
Cubic	0.83	0.9998	0.9996	0.9988	16.22

Final Equation in Terms of Coded Factor:

$$\text{Apparent viscosity} = +49.09 - 39.06 * A + 27.08 * A^2 - 13.89 * A^3$$

where A is the temperarue.

3.2 Effects of parameters

The cubic response functions obtained with one-factor design is used to predict the apparent viscosity within the limits of the experimental factor. The normal probability plot of the residuals, the plot of the residuals versus the predicted response, and the predicted response versus actual values for the apparent viscosity are shown in Figure 1. Taking a close look at Figure 1(a) reveals that the residuals generally fall on a straight line indicating that errors are distributed normally. Figure 1 (b) shows that the residuals scatter randomly on the display, implying that the model proposed is adequate and there is no reason to suspect any violation of the independence or constant variance assumption [14]. In the graph of outlier T versus run numbers (Figure 1 c), all points fall within the limits (± 3.5) indicating reasonable data fit. Figure 1 (d) reveals that the predicted response values are in a good agreement with the actual ones in the range of the operating variable.

Figure 1 (e) shows the relationship between the response (viscosity) and variable (temperature). As it can be seen, temperature is significantly affecting the apparent viscosity i.e. the viscosity is highly dependent on temperature. An increase in temperature leads to a decrease in the apparent viscosity. Another point to be noted from the plot is that among all studied temperatures, the difference in viscosity between the two temperatures, 10ºC and 20ºC, is the largest.

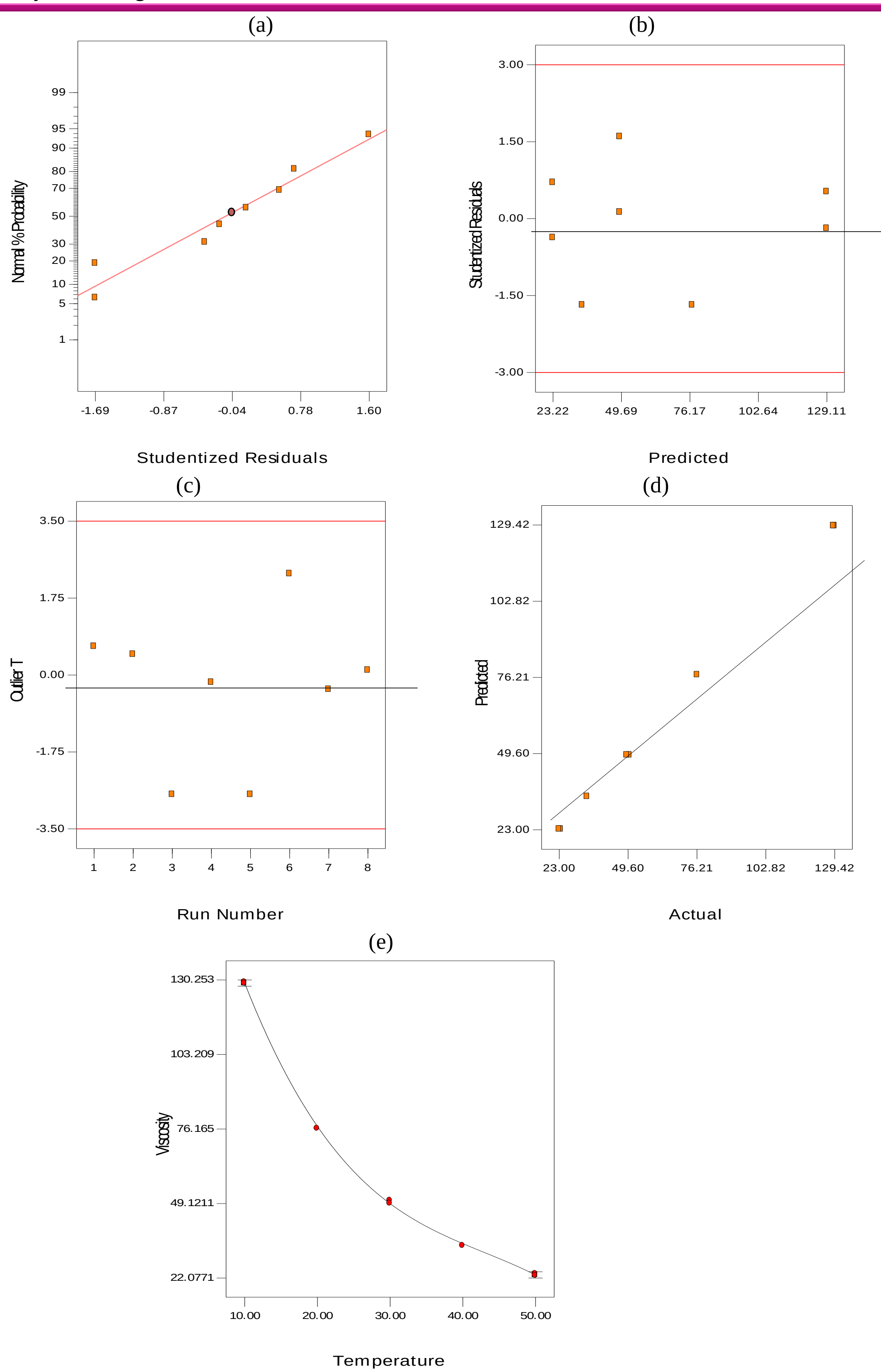

Fig. 1. Normal probability plot of residuals, (a), plot of the residuals versus the predicted response, (b), plot of outlier T, (c), plot of predicted response versus actual values, (d) and plot of response (viscosity) versus variable (temperature), (e).

4. CONCLUSION

RSM was employed to study the effect of temperature on the apparent viscosity of olive oil using One-Factor Design. It was observed that temperature is significantly affecting the apparent viscosity ($p < 0.05$). The regression equation derived in this study was found to be suitable for finding optimum conditions for the desired apparent viscosity of the oil within the condition range applied in this study.

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