Grasas y Aceites
Vol. 52. Fasc. 6 (2001), 369-372

Modeling of oil extraction from olive foot cake using hexane

By **Hocine Kadi*** and **Hocine Fellag**

Faculté des Sciences, Université Mouloud Mammeri, Tizi-Ouzou, 15 000, Algerie
e-mail: hokadi@yahoo.com

RESUMEN

Modelo matemático para la extracción de aceite de orujo usando hexano.

En este artículo se estudia la influencia de los factores que pueden tener efecto en la extracción del aceite de orujo usando hexano. Se propone a partir de los resultados experimentales, un modelo matemático para el cálculo del rendimiento de la extracción. Los tests estadísticos utilizados para obtener este modelo mostraron que el rendimiento no depende de la granulometría ni de la velocidad de agitación, pero si depende de la temperatura, del tiempo de contacto y de la relación líquido-sólido (L/S). También depende de las interacciones entre el tiempo y la temperatura y entre el tiempo y la relación L/S. La comparación entre los resultados experimentales y el modelo nos permite decir que dicho modelo es satisfactorio.

PALABRAS-CLAVE: Aceite de orujo - Extracción con hexano – Modelo matemático.

SUMMARY

Modeling of oil extraction from olive foot cake using hexane.

In this work, the influences of the factors, which can have effect on oil extraction from olive foot cake using hexane, are studied. From the experimental results, a mathematical model for calculation of the extraction yield is proposed. The statistical tests used to obtain this model show that the yield does not depend on the granulometry and the stirring rate, but depends on the temperature, the contact time and the liquid-solid ratio (L/S). Also, it depends on interactions between time and temperature and interactions between time and L/S. The comparison of the results given by the experiment and the model allows us to say that the model is satisfactory.

KEY-WORDS: Extraction with hexane – Mathematical model - Olive foot cake.

1. INTRODUCTION

The olive foot cake is a root of yield since we can recover the residual oil contained in it. In some studies (1) (2), there are attempts to improve the yield of this extraction always done using solvent. For a given foot cake and for the same solvent, this yield (ρ) can depend on the temperature (T), the ratio liquid-solid (L/S), the contact time (t), the stirring rate (Va) and the granulometry (d).

In this paper, we propose to carry out a parametric study on all the parameters which can have effect on the yield of the extracted oil from a given olive foot cake, the solvent being hexane. The statistical analysis (3) of the experimental results allows us to construct a mathematical model in order to forecast the value of the yield when the operative parameters are known.

2. EXPERIMENTAL

The used olive foot cake was taken from an oil mill equipped with a hydraulic press and a centrifuge. The initial moisture content of the sample was 44% which was reduced to 6,2% by drying at 80°C.

The raw material was ground. Granulometry was determined by screen analyses. We have used olive foot cake whose mean particle size was 0.5 mm to 1.25 mm; raw material without any grinding was also used.

The bath extraction was carried out in a beaker used as an open-air reactor. For each experience, a known amount of hexane (the solvent) was added to a 50 g sample of material. The mixture was stirred continuously. Only one stage of extraction is used.

The separation of solids from resultant miscella was performed by filtration in a Buchner funnel using a paper filter of medium porosity. The filtration was carried out under reduced pressure and the same conditions were used for all experiments. The miscella was distilled by means of a rotary evaporator.

Each yield value presented in this paper is the mean of three experimental results.

3. EXPERIMENTAL RESULTS AND MATHEMATICAL MODEL

The mathematical model proposed here is an empirical relation derived from experimental results. The procedure we used is as follows:

1. The analysis of variance (4) (5) (6) (ANOVA) of the results allows us to choose the parameters which have effect on the yield using independence tests.

2. Also, the ANOVA gives us transformations about interactions between parameters which have influence on this yield.
3. The model is fitted using multiple regression in such a way we take all the effects into account.

3.1. Influence of the parameters on the yield

We propose to test, using ANOVA, the independence between the yield and each parameter considered in this study. The table of ANOVA is presented one time only since the method used for computations is the same for all the parameters.

3.1.1. *Granulometry*

In Table I, we give values of yield in oil vs the granulometry (d) obtained in the experiments.

The ANOVA of these results is given in Table II. It can be seen that the yield does not depend on this parameter. Indeed, the calculated F-value is less than Fisher table value $F_{4, 15, 0,.05}$= 3.06.

We can explain this independence as follows: the plant cells are sufficiently open to leave the oil by diffusion to solvent. The cake can then be used in the rough state, its grinding not being advised.

Table I
Yield in oil (%) vs the granulometry for different values of time

t (mn) / d (mm)	5	10	15	20
Raw material*	6.7	7.1	7.1	7.3
1.25	6.8	7.1	7.3	7.5
1.0	7.0	7.2	7.3	7.4
0.8	6.7	6.9	7.1	7.4
0.5	7.0	7.2	7.4	7.5

Conditions: T = 30 ºC, L/S = 2; Va = 1000 tours/mn
*raw material used without any grinding
mean of three replicates.

Table II
ANOVA when time varies

Source of Variation	Degrees of freedom	Sum of Squares	Mean of squares	F
Between Groups	4	0.19	0.0475	0.742
Within Groups	15	0.96	0.064	
Total	19	1.15		

3.1.2. *Stirring rate*

The ANOVA of the experimental results given in Table III shows that the granulometry has no effect on the yield. The calculated F is 0.069 but the Fisher table value is $F_{2, 9, 0.05}$ = 4.26.

This phenomenon can be explained in the following way: the oil is located in the plant cells, which are not very permeable. The advance migration of this oil through walls is very slow. It is governed by the diffusion through the membrane. Thus, even a vigorous stirring has small influence of the rate of transfer.

3.1.3. *Temperature*

The visual analysis of Table IV shows that the temperature has an effect on the yield. This conclusion is confirmed by independence tests which give F=7.872 with $F_{3, 4, 0.05}$ =6.59.

On can note that this dependence is foreseeable since the temperature increases the solubility and the easiness of diffusion of the solute by reducing the viscosity of the oil and the solvent.

The yield is a linear function of the temperature. For example, the regression of the yield and the temperature for t=10 mn and L/S=2 gives with a determination coefficient 83.33%, the following fitted model:

$$\rho = 0.053\ T + 5.48$$

Table III
Yield in oil (%) vs stirring rate for different values of temperature

T (^oC) / Va(rotations/mn)	25	30	35	40
600	7.0	7.2	7.4	7.6
800	6.9	7.0	7.3	7.7
1000	6.9	7.2	7.4	7.7

Conditions: t = 10 mn; L/S = 2; d = raw material without any grinding.

Table IV
Yield in oil (%) vs temperature and ratio L/S

L/S / T (^oC)	2	3	4
25	6.6	8.0	8.6
30	7.4	8.9	9.4
35	7.5	8.6	9.3

Conditions: t = 10 mn; Va = 1000 tours/mn; d = raw material without any grinding.

3.1.4. *Ratio liquid-solid L/S*

In Table V, we show the effect of L/S on the yield. Indeed, the value of F is 289.63 and the Fisher table value is $F_{2, 6, 0.05}$ =5.14.

The regression of the yield and L/S for t=5 mn and T=30° gives with a determination coefficient 93.33% the following fitted model

$$\rho = 3(L/S) + 26.32\ e^{(L/S)} + 3$$

3.1.5. *Contact time*

The ANOVA of the results given in Table VI shows, as planned, that there is dependence between the yield and the contact time.

The yield depends on the contact using a linear function. For T=30°C and L/S=2, we obtain with a determination coefficient 93.25%, the fitted model: $\rho = 0.044\ t + 6.65$

3.2. Interactions between influential parameters

We have proved that the yield depends on the contact time, the temperature and the ratio L/S. In the following, we propose to test the effect of interaction between these influential parameters on the yield in order to obtain a complete fitted model.

Table V
Yield in oil (%) vs contact time and ratio L/S

t (mn) / L/S	5	10	15
2	7.5	7.6	7.7
3	8.3	8.8	8.7
4	8.9	9.3	9.8

Conditions:T = 30ºC; Va = 1000 tours/mn; d = raw material without any grinding.

Table VI
Yield in oil (%) vs contact time and temperature

T (°C) / t (mn)	25	30	35
5	6.4	7.1	7.1
10	6.6	7.4	7.5
15	6.7	7.6	7.9

Conditions: L/S=2; Va = 1000 tours/mn; d = raw material without any grinding.

For this, we need a two-way ANOVA. As an example, we present in Table VII the experimental results of the yield vs the contact time and the temperature.

In Table VIII, the corresponding ANOVA of contact time-temperature interaction and the yield is proposed.

Also, we can show, using the same procedure that the yield depends on the contact time-L/S interaction.

3.3. Mathematical model

To obtain the fitted mathematical model of the yield, the multiple regression analysis is used assuming the following conditions:

1. when the values of contact time and L/S are fixed, the yield is a linear function of the temperature;
2. when the values of temperature and L/S are fixed, the yield is a linear function of the contact time;
3. when the values of temperature and the contact time are fixed, the yield vs L/S is a function of the form :

$$\rho = \alpha(L/S) + \beta e^{(L/S)} + \gamma$$

where α, β and γ are constants.

Table VII
Yield in oil (%) vs contact time and temperature

t (mn) / T (°C)	5	10	15
25	6.39 6.48	6.52 6.64	7.10 7.10
30	7.08 7.15	7.32 7.40	7.50 7.50
35	7.08 7.10	7.50 7.40	7.92 7.86

Conditions: L/S=2; Va = 1000 tours/mn; d = raw material without any grinding.

Table VIII
Two-Way ANOVA of interaction

Source	Degrees of Freedom	Sum of Squares	Mean of Squares	F
Mean	1	919.347		
Rows	2	1.899	0.949	82.231
Columns	2	0.910	0.455	39.413
Cells (interaction)	4	0.286	0.072	6.204
Within groups	9	0.104	0.012	
Total	18	922.546		

Contact time - temperature and the yield. $F_{4, 9, 0.05}$ =3.63.

4. The yield depends on the interaction contact time-temperature and the interaction contact time-L/S only.

The Stepwise regression gives the following model:

$$\rho = 3.65 - 0.25\ t + 1.64\ L/S - 0.047\ e^{L/S} + 0.007\ t.T + 0.049\ t.L/S$$

with the determination coefficient R^2 =97.12 %.

The chi-square goodness of fit test applied to the model shows that the residuals are distributed according to a normal distribution N $(0,\sigma^2)$ with mean 0 and standard deviation $\sigma = 0.18$.

Therefore, one can affirm that our model is valid.

4. CONCLUSIONS

Our investigations allow us to affirm that the yield of extracted oil in the case of traditional pomace-olive does not depend on neither the granulometry nor the stirring rate.

However, it depends on the temperature, the contact time and the liquid-solid L/S ratio. The interactions between time and temperature and between time and L/S have influence on this yield.

Our fitted model,

$$\rho = 3.65 - 0.25\ t + 1.64\ L/S - 0.047\ e^{L/S} + 0.007\ t.T + 0.049\ t.L/S$$

pass successfully the statistical goodness of fit test and the relative error between theoretical and experimental results is less than 5%.

NOMENCLATURE

d: granulometry (mm)
L/S: liquid-solid ratio (cm^3/g)
T: temperature (^oC)
t: contact time (mn)
Va: stirring rate (rotations/minute)
ρ: extracted oil yield (%)

REFERENCES

1. Kmieciak, S., Kadi, H., Meziane, S. and Moussaoui, R. (1991). Oil extraction from olive foot cake with acidic hexane. *Grasas y Aceites*, **42**, 46-50.
2. Hensarling , T.P. and Jack, T.J. (1983). Solvent extraction of lipids from soybeans with acidic hexane. *J. Am. Oil Chemist's Soc.* **60**, 783-784.
3. Thomas, D. and Murphy, Jr. (1977). Design and analysis of industrial experiments. *Chemical Engineering*, 168-182.
4. Draper , N. and Smith, H. (1981). Applied Regression Analysis. 2nd Ed.- John Wiley & Sons Eds.
5. Lehmann, E.L. (1959). Testing Statistical Hypothesis. John Wiley & Sons Eds.
6. Mood, A.M., Graybill, F.A. and Boes, D.C. (1974). Introduction to the theory of Statistics. Third Ed.- McGraw-Hill Eds.

Recibido: Diciembre 2000
Aceptado: Junio 2001