Grasas y Aceites
Vol. 55. Fasc. 2 (2004), 129-134

Solvent extraction of jojoba oil from pre-pressed jojoba meal

By **Ferial A. Zaher*;Omayma S. El Kinawy*** and **Dalia E. El Haron****

*National Research Center, Fats and oil Department, Cairo, Egypt.

**Environics, Management of Environmental Systems,Dokki,Cairo,Egypt.

RESUMEN

Extracción con disolventes del aceite de jojoba a partir de harina prensada de jojoba.

Se ha estudiado el proceso de extracción con disolventes del aceite contenido en la harina residual del prensado mecánico de las semillas de jojoba. Como disolventes se han utilizado hexano técnico y éter de petróleo, habiéndose efectuado extracciones a temperaturas comprendidas entre 30º y 55ºC, con relaciones de disolvente a sólido, R, de entre 12 y 15 L/kg. Se han determinado las composiciones del extracto y del residuo sólido en el equilibrio y a partir de los datos de equilibrio, se ha estimado el coeficiente de partición o cociente de distribución, D, del aceite entre ambas fases. Asimismo, se ha determinado el número de etapas de extracción necesarias para conseguir un cierto grado de recuperación del aceite, utilizando diferentes relaciones entre hexano y harina.

También se han determinado las propiedades físicas y químicas del aceite de jojoba, incluyendo la composición química, el porcentaje de ácidos grasos, el índice de peróxidos, el punto de inflamación, el punto de ignición, el punto de deslizamiento, el índice de refracción y los índices de saponificación y de yodo. Se ha estudiado asimismo la estabilidad del aceite durante su almacenamiento a temperatura ambiente y durante el calentamiento.

PALABRAS-CLAVE: Aceite de jojoba - Extracción con disolventes - Lixiviación. Semilla de jojoba.

SUMMARY

Solvent extraction of jojoba oil from pre-pressed jojoba meal.

The solvent extraction process of jojoba oil from the meal cake obtained after the mechanical pressing of jojoba seeds was studied. Commercial hexane and petroleum ether were used as solvents and the extraction was carried out at temperatures ranging from 30 to 55 °C using solvent-to-solid ratios, R, between 2 and 15 L/kg. The equilibrium compositions of the solvent and solid phases were determined. Based on the equilibrium data, the partition coefficient or distribution ratio, D, of the oil between both phases was estimated. Also, the number of extraction stages necessary to achieve a certain degree of oil recovery has been determined using different hexane-to-meal ratios.

Jojoba oil was also tested for its physical and chemical properties including chemical composition, percentage fatty acid, peroxide value, flash point, fire point, pour point, refractive index, saponification and iodine values. The stability of jojoba oil during storage at room temperature and during heat treatment was also studied.

KEY-WORDS: Jojoba oil - Jojoba seed - Leaching - Solvent extraction.

1. INTRODUCTION

The Jojoba plant is unique among other plants in the sense that its seed contains a high percentage of oil, which ranges from 50-60 %. The oil is practically colorless and odorless and it is composed mainly of straight chain monoesters of C_{20} and C_{22} acids and alcohols with two double bonds. It is almost free of oil triglycerides which indicates that jojoba oil is different from all known seed oils since it is not a fat but a liquid wax.

The processing of seeds with a high oil content is usually carried out by mechanical pressing followed by solvent extraction. After mechanical pressing, the oil content of the final jojoba meal usually ranges from 20-24%. Solvent extraction of the oil from the pre-pressed meal can reduce the residual oil content in the meal to less than 1%. Several factors can affect the efficiency of the solvent extraction process such as the type of solvent used, extraction temperature, solvent-to-meal ratio and the number of extraction stages. In order to achieve a successful industrial solvent extraction process, it is important to perform the extraction process of the prepressed meal under optimum conditions (Wisniak, 1977; Miwa, 1980; National Research Council, 1985).

The aims of this work are to study the process of solvent extraction of jojoba oil from pre-pressed meal and to investigate the effect of different operating conditions on the percentage of recovered oil. The composition of solid and liquid phases at equilibrium will be determined which can be used to estimate the number of extraction stages necessary to achieve a certain percentage of oil recovery. Also, the extracted jojoba oil will be tested for its physical and chemical properties. This part is essential to evaluate the oil suitability for use in different industrial purposes.

2. EXPERIMENTAL

2.1. The Extraction Process

The pre-pressed jojoba meal used in this work was supplied by the Egyptian Company of Natural Oils, Cairo, Egypt. The oil content of the meal was determined by complete extraction using Soxhlet extractor and it was found to be 23%. Solvents used for oil extraction were commercial hexane and petroleum ether, which were obtained from El Nasr Company for Chemicals, Cairo, Egypt.

Table I
Percentage of jojoba oil recovery using commercial hexane and petroleum ether at different solvent-to-meal ratios, R

R	% Oil Recovery		Difference,% V1-V2
	Hexane, V1	Petroleum Ether, V2	
2	65.65	62.22	3.43
3	71.14	67.70	3.44
5	81.10	78.26	2.84
9	94.22	91.28	2.94
15	94.28	92.29	1.99

The batch-wise extraction process of jojoba oil was conducted by mechanical stirring of the meal-solvent mixture at 220 rpm for 1 hr. The solvent/meal ratio ranged between 2 and 15 L/kg. The mixture was left to stand for 30 minutes until the two phases were separated. The composition of both the extract and solid phase at equilibrium were then determined. The total percentage of oil recovered has also been estimated in each case. The above procedure has been carried out at room temperatures (≈ 30°C), 45°C and 55°C.

2.2. Testing of the Physical and Chemical Properties of Jojoba Oil

The fatty acid composition of jojoba oil was determined using gas liquid chromatographic analysis of the oil ethyl esters. Modification of the oil to its ethyl esters was made using 2% H_2SO_4 as catalyst in the presence of dry ethyl alcohol in excess. The chromatographic analysis was made using Hewlett Packard Model 6890 Chromatograph. A capillary column 30 m length and 530 μm inner diameter packed with Apiezon® was used. Detector temperature was 280°C, injection temperature was 300°C and the column temperature was programmed from 100 to 240°C at 15°C/min.

The oil has also been tested for several other characteristics including percentage of fatty acid, saponification number, iodine value and peroxide value using Standard Tentative Methods of Analysis (AOCS, 1991). The dynamic viscosity of jojoba oil as well as flash, fire and pour points have also been determined. The dynamic viscosity in centipoise (cP), has been measured using Brookfield Viscometer (spindle 18) whereas flash, fire and pour points have been determined according to ASTM standard methods D92-85, D 287-82 and D 97-85 (ASTM, 1985), respectively.

3. RESULTS AND DISCUSSION

The results of this work were used to study the effect of the different process variables including the solvent type, solvent-to-meal ratio and extraction temperature on the percentage of oil to be recovered from pre-pressed jojoba meal. The percentage of oil recovery (%) is calculated as $(w_1/w_2)x100$ where w_1 is the amount of oil obtained by a single batch solvent extraction and w_2 is the total oil that can be obtained after complete extraction.

3.1. Effect of solvent type and solvent-to solid ratio, R

Table I lists the percentage of oil that can be recovered from the extraction process using petroleum ether (60-80°C) and commercial hexane at different solvent-to-solid ratios, R. The listed results show that commercial hexane, which is cheaper than petroleum ether, is more efficient as an extracting solvent. The estimated percentage of oil recovered by oil extraction using solvent-to-solid ratio 2 or 3 is 3.4 % higher than that using petroleum ether.

The effect of solvent-to-solid ratio, R (L/kg) on the amount of oil that can be recovered from jojoba meal if the extraction process is carried out in a single or in two successive stages of extraction is shown in Figure 1. It is clear that the amount of oil obtained from solvent extraction increases almost 22% by extraction in two successive stages rather than in one stage. Recovery of about 78% oil can be achieved using 4 L of solvent for each kg of solid in two successive stages, while this ratio should be increased to nine if the extraction process is carried out in a single stage.

Based on the equilibrium composition of the overflow and underflow streams (solvent and solid phases), the distribution ratio or partition coefficient

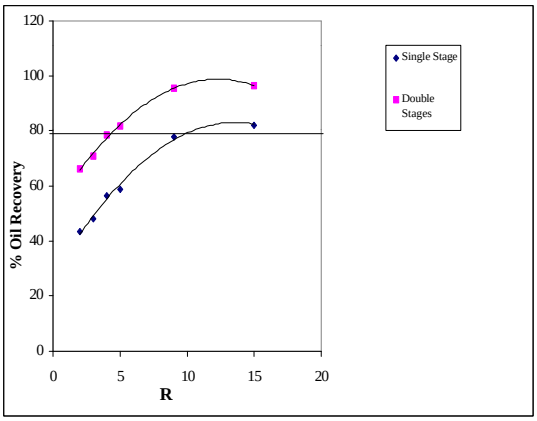

Figure 1
Percentage of oil recovery related to R using single and double stages.

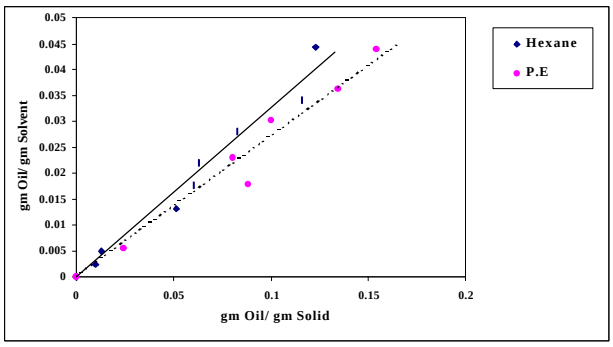

Figure 2
Distribution coefficient, D of jojoba oil using different solvents.

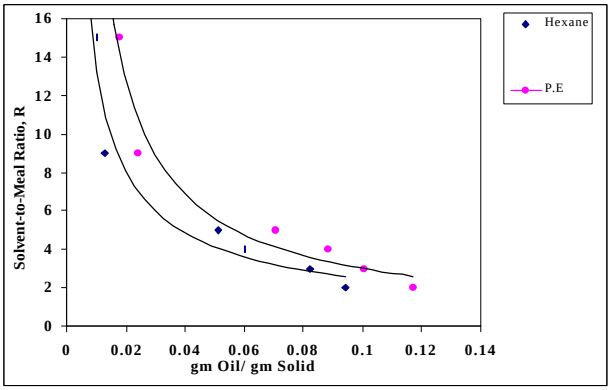

Figure 3
Effect of solvent-to-solid ratio on the residual oil content in the underflow.

D, of jojoba oil between solvent and meal (Thobani and Levente, 1997; Coulson and Richardson, 1985) was predicted. This parameter equals the slope of the straight line in Figure 2 which fits the relationship between the composition of solvent phase, g oil/g solvent and that of solid phase, g oil/g solid. It was found to be 0.3255 and 0.2727 when using commercial hexane and petroleum ether, respectively. This result indicates the greater efficiency of commercial hexane as an extracting solvent compared to petroleum ether. Also, the effect of the solvent-to-solid ratios, R, on the residual oil content in the solid phase after solvent extraction is shown in Figure 3.

It was found that the unrecovered oil (residual oil) in the meal decreases gradually by increasing the solvent-to-solid ratio, R, but its effect becomes almost negligible by increasing this ratio over nine.

3.2. Effect of the number of extraction stages

The results of jojoba oil extraction using commercial hexane at equilibrium have been also used to establish the right angled triangular equilibrium diagram as shown in Figure 4. This equilibrium diagram has been then used to estimate the number of extraction stages necessary to achieve a certain degree of jojoba oil recovery from the pre-pressed meal using different values of R (Coulson and Richardson, 1985). The results of these estimations have been used to show the effect of the number of extraction stages on the percentage of the oil obtained (Figure 5). It is clear that the effect of the number of extraction stages becomes more pronounced as the solvent to solid ratio, R decreases.

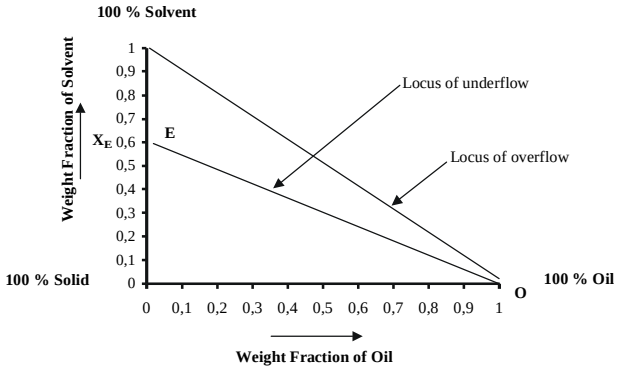

Figure 4
Right-angled triangular diagram at equilibrium.

3.3. Effect of the extraction temperature

The results of jojoba oil extraction process at 45°C and 55°C are compared to those at room temperature (≈ 30° C) in Table II. It can be stated that there is no significant change in the percentage of jojoba oil to be recovered from the meal if the extraction is carried out at temperatures higher than 30°C.

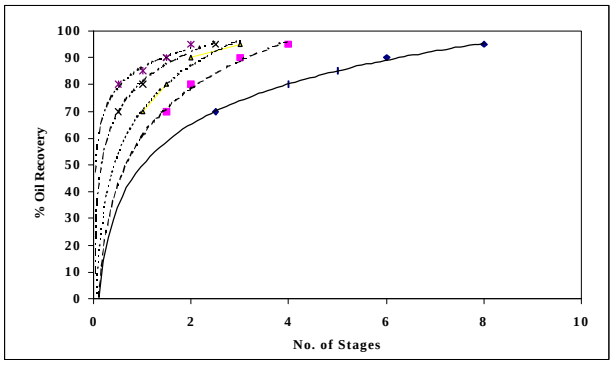

Figure 5
Effect of solvent-to-solid ratio, R and the number of stages on the percentage of oil separation.

Table II
Effect of temperature on oil extraction using commercial hexane

Temperature, °C	% Recovery
30	70.77
45	69.7
55	70.2

3.4. Properties of Jojoba Oil

3.4.1. *Fatty Acid Composition*

The fatty acid composition of jojoba oil as estimated by gas-liquid chromatography is listed in Table III. According to the listed results, jojoba oil consists mainly of $C_{20:1}$ (70.7 %), $C_{22:1}$ (14.1 %), palmitic acid (1.6 %), oleic acid (11.2 %) and $C_{24:1}$ (1.6 %). These results are in agreement with those reported in the literature (Wisniak, 1977; Miwa, 1980; National Research Council, 1985; Tonnet and Dunstone, 1984).

Table III
Chemical composition of jojoba oil

Fatty Acid	% Composition
$C_{16:0}$	1.60
$C_{16:1}$	0.10
$C_{18:0}$	0.14
$C_{18:1}$	11.20
$C_{20:0}$	0.20
$C_{20:1}$	70.70
$C_{22:0}$	0.30
$C_{22:1}$	14.10
$C_{24:0}$	0.02
$C_{24:1}$	1.64

3.4.2. *Chemical and Physical Properties*

Several chemical and physical properties of jojoba oil have been determined and are listed in Table IV. These properties include refractive index, viscosity, saponification number, iodine value, percentage of fatty acid, flash point, fire point and pour point. According to these results**,** the saponification number of jojoba oil was 88-89 mg KOH/g oil. This value is relatively low compared to those of other vegetable oils such as rapeseed, castor and soybean, which have saponification values equal to 175, 187 and 193 mg KOH/g oil, respectively. This suggests higher stability of jojoba oil. This also indicates that jojoba oil is polar and this property imparts rust protection, antifoaming and oily characteristics.

Table IV
Characterization of jojoba oil

Property	Pressed Jojoba oil	Extracted Jojoba Oil
Refractive index	1.4533	1.4565
%Fatty Acid, %FA	1.975	0.3675
Peroxide Value, meq/kg	0	0
Saponification Value, mgKOH/gm oil	89	88
Iodine Value, gm/100gm fat	82	84
Flash point, °C	286	234
Fire point, °C	344	290
Pour point, °C	9	12

Table V
Effect of extraction method on chemical stability of jojoba oil at 30 °C

Method / Time,weeks	% FA[a]		P.V[b]	
	Solvent	Pressing	Solvent	Pressing
0	1.975	0.3675	0	0
1	2.05	0.3716	0	0
2	2.06	0.378	0	0
3	2.08	0.3805	5.5	0
7	2.12	0.3850	8.5	0

[a] % FA: Percentage free fatty acid.
[b] P.V : Peroxide value, meq/kg

Table VI
Effect of temperature on the development of free fatty acid of extracted jojoba oil

Temperature / Time,weeks	60 °C	110 °C
	% FA	
1	0.4301	0.4402
2	0.4206	1.044
3	0.4267	1.408

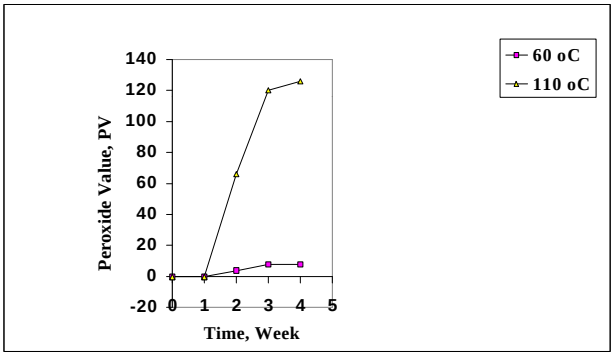

Figure 6
Effect of the temperature on oxidation rate of extracted jojoba oil.

The estimated iodine value of jojoba oil was 82, which is much lower than that of other oils such as soybean and cottonseed oils, which have iodine values of about 132 and 107, respectively, which indicates a higher melting point. Like other vegetable oils, the iodine value of jojoba can be reduced by saturation of the double bonds by catalytic hydrogenation. In turn, the melting point can increase to a limit that allows its use as a substitute for beeswax and carnubawax.

The results listed in Table IV also show that the flash, fire and pour points of jojoba oil were 234°C, 290°C and 12°C for solvent extracted oil,whereas these values were 286°C, 344°C and 9°C for the oil obtained by pressing. This difference may be attributed to the presence of traces of hexane in the oil obtained by the solvent extraction method. These two oils are also different in their free fatty acid content.

The percentage of free fatty acid of solvent extracted oil was 1.975 compared to 0.3675 of the pressed oil. This may be attributed to the fact that jojoba oil remaining in press cake after mechanical pressing degrades quickly with a resulting increase in free fatty acid content (Whittaker, 1996).

The measured dynamic viscosity of jojoba oil at 25°C and 40°C was about 33.3 and 20.7 centipoise (cP), respectively. These values are lower than those of some other vegetable oils such as sunflower and soybean oils with viscosities of 64 and 55 cP, respectively.

3.5. Chemical stability of jojoba oil

3.5.1. *Stability during storage at room temperature*

The effect of storage of jojoba oil at room temperature on the development of free fatty acids as well as peroxides is shown in Table V. It is clear

that the hydrolisis of the oil triglycerides required to yield free fatty acids during storage is almost negligible. The results have also proven that jojoba oil is one of the oils which is stable against deteriorative oxidation. The oil obtained by mechanical pressing resists oxidation over a storage period of seven weeks. However, the oil obtained by solvent extraction was less stable as its peroxide value was increased to 8.5 meq/kg oil during the same storage period. It may be that the extraction process of the oil using solvent enhances the extraction of some components from the seed which have pro-oxidant activity.

3.5.2. *Stability against deterioration by over heat treatment*

The rate of jojoba oil oxidation and that of triglyceride hydrolysis at 60°C and 110°C can be understood from the results listed in Table VI and graphically represented in Figure 6. The development of free fatty acids by the hydrolysis of triglyceride seems to be very slow whereby the percentage of fatty acids increases to 1.4 % only after a period of three weeks at 110°C. However, oil oxidation occurs at a faster rate whereby the peroxide value was increased to 7 and 125 meq/kg oil after four weeks at 60°C and 110°C, respectively .

REFERENCES

Abu Arabi, M.K., Allawzi, M.A., Al Zoubi, H.S. and Tamimi, A. (2000). Extraction of Jojoba Oil by pressing and leaching. Chem. Eng. J. **76**, 61-65 .

AOCS (1991). Official Methods and Recommended Practices of the American Oil Chemists' Society, 4 th Ed., AOCS Press, Champaign IL.

ASTM (1985). Book of Standards, American Society for Testing and Materials, Philadelphia PA.

Chung, H.; Kim, T.W.; Kwon, M.; Kwon, I.C. and Jeong, S.Y.(2001). Oil components modulate physical characteristics and function of the natural oil emulsions as drug or gene delivering system. J. Controlled Release **71**, 339-350.

Coulson, J.M. and Richardson, J.F. (1985). Chemical Engineering, Vol. 2, 4th Ed., Pergamon Press, Oxford.

Miwa, T. K. (1980). Jojoba: fundamental and applied research, Vol. 1, Jojoba Plantation Products Inc., Los Angeles CA, 8-32.

National Research Council (1985). Jojoba: New Crop for Arid Lands, New Raw Material for Industry, National Academy Press, Washington DC, 38-62

Swern, D. (1996). Bailey' s Industrial Oil and Fats Products, 5 th Ed., Vol. 4, J. Wiley & Sons, New York.

Tandy, D.C. (1988). Proceedings of the Seventh International Conference on Jojoba and Its Uses, American Oil Chemists' Society, Champaign IL, 151-164 .

Thobani, M. and Levente, L. (1997). Two Phase Solvent Extraction of Canola. J. Amer. Oil Chemists' Soc. **74** (3) 207-214.

Tonnet, M.L. and Dunstone, R.L. (1984). A rapid micro method for the quantitative analysis of Jojoba wax and its components, J. Amer. Oil Chemists' Soc. **61** (6) 1061- 1064.

Verbiscar, A.J. and Banigan , T. F. (1982). Jojoba Seed Meal as an Animal Feed, in the Final Report on NSF Grant No. AER 76-23895, June 25, California.

Whittaker, C.A. (1996). Analytical Methods for Evaluating the Quality and Purity of Jojoba Oil, in Proceedings of the 9th International Conference on Jojoba Oil and Its Uses, 121- 125. L.H. Princen and G. Rossi (Ed.). Association for the advancement of industrial crops.

Wisniak, J. (1977). Prog. Chem. Fats Other Lipids in Jojoba Oil and Derivatives, **15**, 167-218.

Recibido: Octubre 2002
Aceptado: Julio 2003