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Short communication

Extraction of jojoba oil by pressing and leaching

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Abstract

Jojoba oil extraction by pressing alone, pressing followed by leaching, and leaching alone were investigated. The extraction process by first and second pressing followed by leaching gave about 50% by weight oil with reference to total seed, which is in agreement with what has been reported previously. The extraction by leaching process was carried out using different solvents. These solvents were; hexane, benzene, toluene, petroleum ether, chloroform, and isopropanol. Hexane, benzene, and petroleum ether gave the highest yield (all about 50% by weight oil with reference to total seed), but when cost is considered, petroleum ether is recommended as the best solvent to leach jojoba oil. The yield obtained in this work for leaching by hexane and benzene are 3–5% and about 10% for isopropanol more than those reported in the literature. Traces of solvent remained with the extracted oil after simple distillation followed by a second stage distillation via a Rotavapour apparatus. These traces slightly affected some of the oil properties such as pour point and flash point. ©2000 Elsevier Science S.A. All rights reserved.

1. Introduction

Jojoba (Simmondsia chinensis) is unique among plants in the fact that its product (seeds) contains about 50% by weight oil, which is more than twice the amount in soybeans and somewhat more than in most oilseed crops. The oil is composed mainly of straight chain monoesters in the range of C_{20} – C_{22} as alcohol and acids, with two double bonds, one at each side of the ester bond [1–3].

Interest in jojoba oil stems from its unusual properties that differ from all known seed oils. The complete absence of glycerin makes it a liquid wax, not fat. Jojoba oil has been evaluated for suitability in many applications such as cosmetics, pharmaceuticals, lubricants, food, electrical insulators, foam control agents, high-pressure lubricants, heating oil, plasticizers, fire retardants, and transformer oils plus others [1–3].

Different methods, similar to those applied to other oilseeds, have been used for extraction of jojoba oil from the seeds [3–6]. Those methods are mainly mechanical pressing, mechanical pressing followed by leaching (solvent extraction), or leaching only. Some of the oilseeds require pretreatment/preparation such as cleaning, dehulling, crush-

ing, flaking, cooking, etc. before the extraction process. Hexane is the solvent most commonly used in the leaching process because of its relatively low cost and low toxicity. Other organic solvents such as benzene, alcohol, chloroform are also used. Water as a solvent was evaluated to extract oil from soybean, but gave low yield and high potential of oil microbiological contamination [6–8].

Knoepfler et. al [9] used carbon tetrachloride, benzene, hexane, heptane, isopropyl alcohol and tetrachloroethylene to leach jojoba oil. They first cracked jojoba seeds into 8-12 pieces by passing them through corrugated crushing rolls. They were then chopped into flakes of 0.010 in. (0.25 mm) average thickness by passing them through a pair of smooth rolls. A Soxhlet extractor was used to extract the oil from the flakes. They found that carbon tetrachloride, benzene, hexane, and heptane extracted between 45-47% by weight (based on total seed). Spadaro et al. [10] investigated the conditions required for material-preparation and extraction to get efficient leaching of oil from jojoba seeds using a filtration-extraction process that was previously used for other oilseeds. The seeds were subjected to; flaking to different thickness (0.004–0.01 in.), cooking in a mixer type cooker, crisping by evaporative cooling for 20 min., slurrying the cooked material with solvent. The following filtration-extraction characteristics were studied; flake thickness, moisture content (which was varied between 5-20%) of flakes during cooking, rerolling of cooked

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flakes, and extraction temperature on mass velocity and extraction efficiency using commercial-grade hexane and heptane as solvents. Tests with heptane showed that increasing the extracting temperature from 80F (26.7°C) to 140 F (60°C) and/or increasing the moisture content up to 10%, increased the extraction efficiency by about 3%. Performing the extraction at 140 F and increasing the moisture content up to 20%, increased the extraction efficiency by less than 1%. Actually, their data shows no improvement with increasing the moisture content above 10%. For the 80 F extracting temperature, increasing the moisture content to 10, 15, and 20%, increased the efficiency by 2.6, 2.9, and 3.1%, respectively. They reported that the adequate mass velocity, defined as pounds of miscella filtrate per hour per square foot of filter area, for commercial application should be greater than 2000. For the 140 F extracting temperature, when the moisture content was increased to 10%, the mass velocity dropped to 1617. For the 80 F, the mass velocity dropped below 2000 when the moisture content was increased to 20%. Rerolling was found not required if the flakes are rolled initially to a thickness of 0.004 in.. Based on their results, they concluded that both heptane and hexane are suitable as solvents for commercial extraction of jojoba flakes as there are no significant differences in mass velocity and extraction efficiency obtained with the two solvents. However, they recommended using hexane rather than heptane, because it is more readily available, cheaper and has a lower boiling point, which facilitates its removal from the products.

They also conducted three experiments to determine the filtration-extraction characteristics of uncooked jojoba flakes. The results were erratic in that the mass velocity varied from about 200 and 1600 and extraction efficiency from 96.5 to 97.8. So they concluded that omission of the cooking step is not recommended in any commercial operations.

Rawles [11] reported on mechanical seeds grinding and pressing done at the Western Regional Research Center (WRRC), Albany, California, and at the San Carlos Apache Indian Reservation during 1972–1977. Many problems were encountered during the grinding operations that were tested. As the mill heated up, a sticky, mud-like meal formed that plugged the grinder. They were able to solve these problems by freezing the seed before grinding, but this is very expensive.

The effect of moisture and temperature on the efficient operation of a Rosedowns press, made in England, was investigated. The best results were obtained when operating between 175–190 F (80–88°C), and with 3–4% moisture content. The operation of the press required very careful monitoring to avoid loss of oil and plugging. An extraction rate of 38.2% was obtained, and the oil content remaining in the meal was 17–20%. A Hander press, made in Japan, was used for pressing at the San Carlos reservation. Experience with it indicated that, feeding 20% hulls by weight with the seed improved the extraction efficiency. Double pressings were necessary to bring the extraction rate up to 42% from

35–39% in a single press. Also preheating was required to get this extraction rate. The remaining meal, after double pressing, contained 9–10% oil. Ruiz et al. [12] also used the same model Hander press and concluded that moisture control during the feeding process was critical. Feeding with 4% moisture content, they were able to extract 80% of the original oil in one pass and up to 94% in two passes.

Miller et al. [13] reported on the mechanical rendering of jojoba oil by grinding and pressing the seeds. The dehulled seeds were grounded at room temperature by using 8 in. Bauer single-disk attrition mill modified by the addition of two L-shaped case wipers to the rotating disk to prevent plugging. Pressing was done by passing preheated feed (80–90 F) through a Rosedowns press. Pressing with about 4% moisture content gave 31.4% oil yield (based on total seed weight).

Spadaro and Lambou [14] investigated mechanical extraction and solvent-extraction of jojoba oil. The mechanical extraction (cold hydraulic pressing) was performed by cracking the seeds into 6-10 pieces, and then flaking to about 0.025 in. thick. The flakes were charged to a six-stack, pilot-plant model hydraulic press and pressed for 50 min at 4400 lb ram pressure. They collected 40 lb of oil from 130 lb of seed (30.8% oil). In the solvent-extraction, six solvents were used: carbon tetrachloride, benzene, isopropyl alcohol, heptane, hexane, and tetrachloroethylene. The process was carried out, after cracking and flaking the seeds, in a Soxhlet extractor. A total of 20-24 solvent passes were used. Extraction was done at the boiling point of the used solvent. Separation of oil from solvent was conducted under vacuum at 3-6 mm Hg for 2h. The stripped oil was dried in a vacuum oven at 105°C for 2h. They reported only the results of three of the solvents, which they considered to have the most potential. Their results as well as others' results are shown in Table 2.

Lanzani et al. [15] have reported that a wet process technology was applied to jojoba seeds to obtain oil and detoxified protein meal. The yield of oil was less than 20% by weight with reference to total seed.

The objectives of this work are: (i) to study oil extraction from jojoba seeds grown in Jordan by pressing alone without pretreatment, pressing followed by leaching with hexane, and leaching with different solvents, (ii) to investigate the chemical and physical properties of the pressed oil and the oil leached by hexane, and (iii) to find out if there are any differences between them.

2. Equipment and experimental procedures

2.1. Pressing only

To obtain the jojoba oil by pressing, a manually operated hydraulic press type (P/N, 15.011), made by SPECAC Limited (UK), with variable load (0–15) metric tons was used. For each run, about 80 g of whole jojoba seeds were

placed in a cylindrical container (6.52 cm inside diameter), then were subjected to the press load. The amount of oil collected was considered as the weight of oil extracted. A second pressing was done on some samples by removing it from the cylindrical container after the first press, breaking the disk–like residue and then putting it again in the cylindrical container and subjecting it to a second pressing. Both the first and second pressing were done at room temperature.

2.2. Pressing followed by leaching

In this case, the jojoba seeds were subjected to first and second pressing as mentioned above, then the sample was taken out of the cylindrical container and crushed to <1.0 mm average size. The crushed sample was leached by hexane using Soxhlet extractor. The total amount of oil extracted is the sum of that obtained by first and second pressing and from the leaching step.

2.3. Leaching only

A Soxhlet extractor was employed for the leaching experiment. For each run, 30 g of crushed jojoba seeds was charged into the Soxhlet extractor and 150 ml of organic solvent was used. The solvents used were; hexane, petroleum ether, isopropanol, benzene, toluene, and chloroform, which were laboratory grade. Leaching was carried out at the boiling point of each solvent until a clear liquid was obtained from the jojoba, which indicated complete leaching of the leachable oil. The Soxhlet extraction process took about 18 h. The extracted phase (oil and solvent) was then distilled in two stages to separate the oil and solvent. The first stage was a simple distillation followed by a second stage, which was a Rotavapour apparatus. A vacuum pump was attached to the Rotavapour apparatus to ensure complete removal of the solvent. The oil produced by this method was compared with the pure pressed oil by measuring their properties.

3. Results and discussion

First, the amount of oil extracted by pressing was found as a function of the press load. Fig. 1 shows the oil yield (by weight with reference to total seed) with pressure. The amount extracted increases exponentially as the pressure increases, but as the pressure is increased to $35.4 \,\mathrm{MPa}$, it starts to level off indicating that this is about the required load to get the maximum amount extracted by pressing. This type of data is needed for the mechanical design of any press to be used in large-scale production. Further pressing runs in this study were subjected to $35.4 \,\mathrm{MPa}$ pressure. The experimental data was fitted to a third polynomial to give the following equation with $R^2 = 0.987$.

% oil =
$$0.0009 P^3 - 0.0761 P^2 + 2.7086 P$$

where P is the pressure.

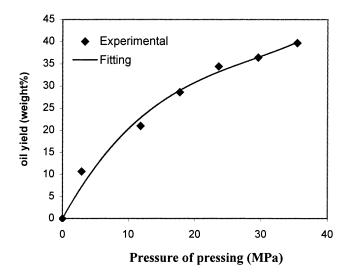


Fig. 1. Jojoba oil yield (wt.% with reference to total seed) as a function of press load.

The results obtained from first and second pressing and followed by leaching using hexane as a solvent are shown in Table 1. The first pressing gave about 35.4%, while the second pressing gave about 8.4% by weight oil. Both presses were subjected to 35.4 MPa pressure. Leaching process gave about 6.7% by weight oil. As can be seen, there is a slight difference in the percentage of oil recovered for the different trails. This is expected, because there will be a slight difference in the amount of oil present in each seed and the seeds have different sizes. Table 2 shows the results of pressing obtained in this work and those reported in the literature, which are in agreement.

3.1. Leaching process

The leaching process depends mainly on the chemical structure of the solvent and the kind of solute that will be extracted from solid material. This probably follows the principle 'like dissolves like'. This does not mean that both the solute and the solvent have to be chemically similar but rather have similar functional groups. Solvents are usually classified by the number of functional groups present in their molecules, which affect the interaction of either or both types of physical and chemical interactions between the solute and the solvent [16].

According to the above principles, jojoba oil extraction depends on the kind of organic solvent and its structure. Jojoba oil is a nonpolar ester compound with a very long straight chain structure. Therefore, any solvent that has a similar structure (and nonpolar compound) will leach more oil. Table 3 shows the yield of different solvents used in this work and those reported in the literature. Solvents' properties and their cost [17] are also shown in Table 3. This work results of the jojoba oil yield are the average of four runs. Hexane, petroleum ether, and benzene gave a high percent of yield because they are nonpolar hydrocarbon compounds. Chloroform, which is polar compound, leached a lower per-

Table 1 Yield of jojoba oil after first and second pressing followed by leaching process using hexane.

No. of trial	Mass of seeds, g	Mass of oil after first pressing, g (% oil)	Mass of oil after second pressing, g (% oil)	Mass of Leached oil, g (% oil)	Total oil, g (% oil)
1	90.0	34.1 (37.9)	8.0 (8.9)	4.3 (4.8)	46.4 (51.5)
2	90.0	31.4 (34.9)	7.1 (7.9)	7.3 (8.1)	45.8 (50.9)
3	90.0	30.3 (33.7)	7.7 (8.6)	6.3 (7.0)	44.3 (49.2)
Average	90.0	31.9 (35.4)	7.6 (8.4)	6.0 (6.7)	45.5 (50.6)
SD		1.95	0.46	1.53	1.08

Table 2 Pressing results

Source	% oil ^a	Type of press	No. of pressing	Pretreatment or treatment processes
This work	35.4	hydraulic (manually operated)	single	none
	43.8	hydraulic (manually operated)	double	breaking the disk-like residue from first press
Rawles [11]	38.2	Rosedowns press	single	preheating, hulls and moisture contents were adjusted
	35-39	Hander press	single	preheating, hulls and moisture contents were adjusted
	40-42	Hander press	double	preheating, hulls and moisture contents were adjusted
	43	Hander press	triple	preheating, hulls and moisture contents were adjusted
Ruiz et al. [12]	80–94 ^b	Expeller Hander EX-100	single	flaking, preheating, hulls contents were adjusted, moisture content = 4%
	50-70 ^b	Expeller Hander EX-100	single	flaking, preheating, hulls contents were adjusted, moisture content = 6%
Spadaro and Lambou [14]	30.8	hydraulic press (pilot-plant)	single	preheating, cracking, flaking
Miller et al. [13]	31.4	Rosedowns press	single	grinding, preheating, moisture content = 4%

a Based on total seed weight.

Table 3

A comparison between the organic solvents used in the leaching process

Solvent	Structure	Refractive index (25°C)	Boiling point (°C)	Specific gravity (G/ml)	Color No. of leached oil (ASTM)	Cost of the solvent (\$/L)	% Leached oil	
							This work	Literature values
Hexane	C_6H_{14}	1.3723	68.7	0.670	0.5	25.0	52	48.8 [14], 46.9 [9]
Petroleum ether	a mixture of	1.3787	60-100	0.656	L 2.0	11.2	50	_
(ligroin)	hydrocarbon ^a							
Benzene	C_6H_6	1.4972	80.1	0.874	L 1.5	28.0	49.3	45.5 [9]
Chloroform	CHCl ₃	1.4459	61.2	1.490	L 2.0	25.8	32.5	_
Isopropanol	CH ₃ HCOHCH ₃	1.3772	82.2	0.785	1.5	19.0	45	36.1 [14], 35.1 [9]
Toluene	$C_6H_5CH_3$	1.4941	110.5	0.867	3.5	17.0	44.8	_
Carbon tetrachloride	CCl ₄	_	76.5	1.584	_	_	_	45.7 [9]
Tetrachloroethylene	C_2Cl_4	_	121.1	1.620	_	_	_	42.3 [9]
Heptane	C ₇ H ₁₆	_	98.4	0.684	-	_	-	48.1 [14], 46.5 [9]

^a Mainly hexane and heptane.

centage of jojoba oil than the above solvents. Isopropanol and toluene leached about the same amount. The yield obtained in this work for hexane, benzene, and isopropanol are higher than those reported in the literature. This could be due to the solvents' purity and the time of leaching. The results obtained in this work for hexane, petroleum ether, and benzene show that these solvents are able to get the same (or slightly more in the case of hexane) the amount obtained by first and second pressing followed by leaching.

Petroleum ether is the best solvent used in this research since its cost is relatively low and it leached a high percentage of oil, while hexane gave the highest yield but its cost is relatively high. Benzene gave the same yield as that of petroleum ether, but its cost is the highest one.

Toluene and isopropanol can be ranked third with regard to the amount of oil leached. Toluene has a high boiling point so it needs more heat to vaporize in any distillation recovery process compared to the other solvents. The oil leached by toluene has different color number, as shown in Table 3, from those obtained by other solvents which probably means that toluene may extract materials from the seeds other than the oil, such as sugar, pigments, etc. Chloroform leached the least amount of oil, also its cost is relatively high. Additionally, chloroform has high specific gravity that resulted in the flotation of the jojoba meal during the leaching process. Overall, petroleum ether can be classified as the best solvent based on the foregoing discussion.

^b This is % extracted from original oil, the whole oil content in the seeds is not reported.

Table 4 A comparison between the properties of pressed and leached oil by hexane

Characteristics (ASTM No.)	Observed value of pressed jojoba oil (literature value)	Observed value of leached jojoba oil oil
Flash point -open cup, °C		
(D 92)	275 (295)	267.0
Aniline point, °C (D 611)	52.9 (N/A)	52.7
Pour point, °C (D 97)	$8.0 (9.0)^a$	6.0
TAN, mg KOH/g (D 974)	0.36 (N/A)	2.89
TBN, mg KOH/g (D 2896)	1.0 (N/A)	1.0
Ash content, wt.% (D 482)	$0.0 (0.0)^a$	0.0
Color number (D 1500)	1.0 (1.5)	0.5
Refractive index (D 1218)	1.4593 (1.465)	1.4600
Viscosity, Cst (D 446)		
at 40°C	24.75 (24.92) ^a	26.16
at 100°C	6.43 (6.43) ^a	6.57
Viscosity index (D 2270)	233 (233) ^a	233

^a These values were taken from reference [18], while the other literature values were taken from reference [2].

Jojoba oil produced from the leaching process by organic solvents is different from that produced by the pressing process since the leached oil is not pure (i.e., some of the organic solvents do not separate completely from oil during distillation process). In Table 4, the properties of jojoba oil leached by hexane and the pressed oil are shown. The pour point of the leached oil is 2°C less than that of the pressed oil meaning that some long chain paraffin's were extracted less by hexane, and therefore the pour point of the leached oil would be reduced. Flash point is another indicator of the presence of traces of hexane because the flash point of hexane is much less than that of jojoba oil. Thus, traces of hexane will reduce the flash point of the leached oil compared to that of the pressed oil. The other properties did not significantly change. Further treatment to remove these traces from the oil is needed, if it is to be used in cosmetics, pharmaceuticals or any other products that may come in contact with living tissues.

4. Conclusions

The yield of jojoba oil (with reference to total seed) that can be obtained by pressing is about 44% by weight and by first and second pressing followed by leaching is about 50% by weight. The pressure required in the hydraulic press is about 35.4 MPa to obtain the maximum amount by pressing. The six organic solvents used in the leaching process leached different amounts depending on the type of solvent (polar or nonpolar) and its structure. Hexane leached the highest amount of oil followed by Petroleum ether, benzene, isopropanol, toluene, and then chloroform. When cost

is considered as a parameter, petroleum ether is the best solvent to be used in the leaching process. Further investigation is needed to determine the percentage of solvent recovery and how much losses take place to be able to really decide which is the best solvent. Traces of solvents remained with the oil after simple distillation followed by a second stage distillation via a Rotavapour apparatus. These traces affect some of the oil properties.

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