Holdup and Extraction Characteristics of Jojoba Meal

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Holdup and drainage characteristics have been determined for three jojoba meals of different nature and size distribution, using hexane and isopropanol as solvents. Density and viscosity properties of the oil-solvent solutions have been measured. The experimental information should be of value in the design of solvent extraction equipment for jojoba nuts.

Jojoba *(Simmondsia chinensis* Link) is a desert shrub that grows wild in southern Arizona, northwestern Mexico and neighboring areas. It tolerates extreme daily temperature fluctuations and grows well under soil and moisture conditions not suitable for agricultural crops. Jojoba is a woody evergreen shrub which is commonly 2-3 ft high, easily recognized by its thick, leathery, bluish-green leaves and dark brown nutlike fruit. The male and female flowers are borne on separate plants, the number of female and male plants being about equal in number. Extensive studies are being conducted to adapt the shrub to intensive plantation culture. Interest in jojoba stems from the unusual properties of the oil that can be extracted from its seeds. Jojoba is unique among plants in that the nuts it produces contain about 50% by weight of a practically odorless, colorless oil composed mainly of the straight chain monoesters of the C_{20} and C_{22} alcohols and acids, with two double bonds, one at each side of the ester bond. The almost complete absence of glycerin indicates that jojoba differs radically from all known seed oil. It is not a fat, but a liquid wax.

Three publications of the National Academy of Sciences (1-3) have concluded, among other things, that jojoba oil can duplicate sperm oil performance, can be used as a substitute for the complete range of its uses, and has a myriad of possibilities not attainable with sperm whale oil.

The growing enthusiasm for the commercial future ofjojoba oil and its derivatives is generated by its many potential uses.

Many publications have discussed the possibilities for economic development of the oil and its chemical and industrial potential (1-5). The oil has been evaluated for its suitability for sulfurization to produce lubricants, lubricant additives and rubber-like factices for use in the manufacture of linoleum and printing inks. It offers excellent possibilities as a lubricant at high temperatures and pressures. The oil has a high dielectric constant, which may make it suitable for electrical applications. It may also be useful as an ingredient in the manufacture of carbon paper, stencils, pharmaceuticals, cosmetics, insecticide carrier, thermosetting compositions for use in the formation of a magnetizable film, dental prosthetic materials and hypocaloric foods. The oil can easily be hydrogenated to form an extremely hard white wax with a high melting point and properties competitive with beeswax and candelilla, carnauba and spermaceti waxes. Jojoba oil is

unique as a source of C_{20} and C_{22} straight-chain alcohols, which could prove valuable in the manufacture of detergents, wetting agents, dibasic acids, long chain ethers, hydroxy ethers and sulfated products.

A byproduct of jojoba seeds is the meal remaining after the oil has been pressed and extracted. This material constitutes about 50% of the seed, and plans for commercial processing must take into account the handling of large amounts of meal and its potential uses. The possibility of using the meal for liquid fuels has already been considered (6,7). Much effort has been devoted to the possibility of usingjojoba meal as an ingredient in feeds for domestic animals (8).

Even after the most efficient pressing, jojoba meal will retain an appreciable amount of absorbed oil, usually 3 to 8% by weight. Liquid solvent extraction is usually used to recover the larger part of this oil.

Extraction always involves two steps: (i) contact of the solvent with the solid to be treated so as to transfer the solute to the solvent, and (ii) separation or washing of the solution from the residual solid. These two stages may take place in the same piece of equipment or in different ones. In a real extraction process it is impossible to completely separate the liquid phase from the solid. As a consequence, the flows that will result from each extraction stage will consist of a liquid phase (that may or not contain suspended solids) and a marc formed by the inert solid and the adhered solution. An equilibrium stage usually is defined as one in which the liquid phase has the same composition as the solution adhered to the solids.

Liquid always adheres to the solids which must be washed to prevent either the loss of solution if the soluble constituent is the desired material, or the contamination of the solids if the solids are the desired material. In most solid-liquid extractions a single saturated solution is obtained as a product because of the desire to obtain a high recovery of the solute, or to obtain the inert solids relatively free from the solute.

In the solvent extraction process for recovery of oil from jojoba meal, information is needed for predicting the flow rate of solvent through the meal, extraction rate of the oil, and the holdup of solvent and solution in the extraction bed. The amount of holdup of the solution allows calculation of the number of stages needed in the extraction battery, and the amount of solvent holdup determines the quantity of heat necessary for desolventizing the meal. Holdup is defined as the weight of total solution remaining in the extraction **bed** per unit weight of oil-free and moisture-free meal. This variable is normally inversely proportional to the mass velocity with which the liquid leaves the bed, and proportional to μ/ρ^2 where μ is the viscosity of the solution and p its density.

For the usual percolation extraction scheme, using fixed or moving beds, the number of equilibrium stages is primarily a function of the holdup of solution throughout the system. Holdup of solutions by the solids undergoing extraction is one of the fundamental properties of the solids-oil-solvent system, and sufficient data to define this property quantitatively

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must be known before the extraction equipment may be designed.

It has been postulated that the rate-controlling factor in the solvent extraction of seed flakes is probably the internal resistance of the flakes to the molecular diffusion of solvent and oil (9), so that the extraction rate should be in indirect proportion to the flake thickness. Othmer and Agarwal (10) have found that the extraction rate is proportional to the 3.97 power of the flake thickness and to the 3.5 power of the residual oil.

Graphs of hold-up during draining of meal beds have been reported by Smith (11) and Karnovsky (12) for beds of soybean and cottonseed flakes. Kocatas and Cornell (13) have reported on the holdup of hexane by soybean flakes, while Keane and Smith(14) have studied the holdup and drainage times for soybean and cottonseed flakes, and presscakes from flaxseeds, peanuts and sesame seeds.

MATERIALS AND EQUIPMENT

Two different meals were tested. Meal I was in the form

FIG. 1. Drainage of hexane-jojoba oil solutions from meal I.

FIG. 2. Drainage of isopropanol-jojoba oil solutions from meal I.

of amorphous, granular porous particles, showing a large internal surface. Its size distribution was as follows: 68.6% wt less than 3 mm; 24.3% between 3-5 mm, and 7.1% between 5-8 mm. Meal II was in the form of thin compressed flakes, with size distribution of 5% wt under 5 mm, 77% from 5-8 mm, and 18% larger than 8 mm. To compare both meals, a fraction (meal IIa) was separated by sieving from meal II to contain 72% wt of particles of less than 3 mm, 20% between 3-5 mm, and 8% between 5-8 mm. All meals contained 6.5% humidity and were thoroughly extracted with hexane before use.

The experimental equipment consisted of a covered sintered glass filter (150 μ) 10 cm high and 9 cm in diameter, connected through a valve to a drain collector and a glass condenser. The apparatus was supported on an analytical balance so that readings could be taken at different times without disturbing the flow process. The meal was added to the filter and then enough of a solution of known composition was added to flood the bed, while the drain valve was kept closed. Experimental data showed that after two days the solid particles were thoroughly wet. The bottom valve was then opened and the times required to drain a certain amount of solution were recorded. The process was continued until no further changes in weight were observed. The procedure was repeated for solutions of different concentration and different solvents. Material balances indicated that no significant losses of solvent took place.

RESULTS

Figures 1 and 2 and Tables 1 and 2 show the experimental results obtained. Inspection of the data shows that there are significant differences in the holdup and drainage characteristics, depending on the solvent and the nature and particle size distribution of the meal. In the drain process described here there is a strong relationship between gravitational and capillary forces. For these reasons, the density and viscosity of the different solutions were measured in an attempt to find a partial explanation to the experimental results (Table 3). It is seen that solutions of jojoba in isopropanol have a higher viscosity and density than those of jojoba oil in hexane. We can expect that higher viscosities will lead to higher holdups, and this assumption is substantiated by the data.

TABLE 1

Holdup of Solutions of Jojoba Oil in Hexane and Isopropanol, Meal Ia

 a Average of 3 runs.

TABLE 2

Holdup of Jojoba Oil-Hexane Solutions by Meal II and Meal IIa^a

Jojoba oil (wt $\%$)	Holdup, kg solution/kg inert	
	Meal II	Meal IIa
0	0.413	0.333
20	0.466	0.410
40	0.480	0.432
50	0.493	0.450
60	0.521	0.462
80	0.546	0.480
100	0.550	0.490

aAverage of 3 runs.

TABLE 3

Density and Viscosity of Jojoba Oil Solutions in Hexane and Isopropanol at 25 C

The experimental evidence can be summarized as follows:

- For the same meal, a solvent that produces more viscous solutions will have larger holdups and lower rates of extraction. Higher holdup values are indicative of low bed permeability.
- For meal of the same nature and the same solvent, the meal with the smaller particles has a lower holdup.
- For meals of different particle form, flakes show lower holdups than granular particles.

Following the analysis of Kocatas and Cornell (13) a modified holdup, H", defined as H μ/ρ^2 where μ is the viscosity and ρ the density, was plotted against the viscosity of the solution (Fig. 3). It is seen that the data for both solvents fall on the same straight line. Other solvents are being tested to see if this is a general fact.

The above information can be used for calculating the number of ideal stages using standard unit operation methods (15). Assume it is necessary to extract 1000 kg ofjojoba meal containing 10% of oil, using 1500 kg of recovered solvent mixed with 10 kg oil, and that the final meal should contain 10 kg of oil. The number of ideal stages is:

FIG. 3. **Variation of modified** holdup H* with viscosity.

Efficiencies of real extractors are about 50% of the theoretical value, so the actual number of stages probably will at least double.

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