

ure 3, STP continues to have excellent soil-removal effect. Others (3) have shown that sodium is adsorbed on both glass and quartz though the extent for the latter substrate is less by about 75%. Such a degree of lowering with STP was not found, suggesting either that the same adsorption sites which adsorbed sodium were not involved or that others are active with STP and this soil. This leaves only a competition for polar adsorption sites between STP and the tristearin, and whichever was adsorbed first would have considerable influence on the ease of replacement by the other. The fact that oily soil is more readily removed from a STP-treated substrate and that STP can almost completely remove tristearin from the fat-treated substrate suggests the greater polarity of the STP system, as might be expected.

The mechanism of soil removal for solvent system removal of tristearin (1) from glass consists either of dissolution of coherent soil or a "stripping" or preferential displacement process for removal of adherent (adsorbed monolayer) soil. Since STP is neither a solvent nor a surfactant, neither dissolution nor micelle solubilization is involved for this system in coherent soil removal. Instead it appears that the upper soil layer may be removed in part by emulsification, but more likely that both coherent and adherent soil are removed nearly simultaneously by the "stripping" or preferential displacement mechanism. The lower layer is desorbed and, along with it, coherent soil. In support of this hypothesis is the fact that the continuous soil film of Figure 3 is removed with much more difficulty (time and concentration increase) than the spotted soil application. For the latter type of soil application, the potential area for penetration and contact is obviously much greater.

*Implications.* Use of STP in dishwashing compositions has generally been based upon its effectiveness as a sequestant type of water softener. While it certainly has these characteristics, these data show its much broader utility. Sequestant type of softeners are the only ones found to pretreat glass surfaces so that subsequent soiling is less retentive. Soiling prevention occurs through pretreatment, but, more important, once a soiled glass (not pretreated) has been cleaned with a composition containing appreciable amounts of STP, the effect upon subsequent soiling is that of a soil-preventive pretreatment. STP not only softens water without precipitation, it is an effective detergent for an oily soil and, when used for washing, imparts a soil-resistant surface to the glass.

*Additional Factors Requiring Investigation.* This report is the first of several, for factors which must be investigated are: effect of other builders, builder combinations, builder-surfactant combinations, adsorption-desorption isotherms, other soils, and other substrates.

### Summary

The mechanism of radio-tagged tristearin removal from a glass substrate by sodium tripolyphosphate is primarily one of preferential displacement. Tristearin removal by STP appears to be a competition for primary polar adsorption sites: being the more polar, STP displaces the soil. Continuous soil films are more slowly removed than spotty soil films because initially fewer accessible adsorption sites are available for displacement attack by STP. It is believed that emulsification of these heavy films occurs initially by the "stripping" or preferential displacement by STP of the adsorbed monolayer at a site, followed by a rolling up of coherent soil along with the desorbed monolayer, resulting in a degree of emulsification.

In addition to its sequestant water-softening action, previously considered its main function, and its detergent effectiveness, another very important feature has been discovered. Tripolyphosphate (and sequestant type of anion) adsorbs on a glass surface and reduces the tenacity of subsequent fatty resoiling. This may account to a considerable degree for the demonstrated high practical cleansing effectiveness of the compound.

Adsorption of STP is not by a base exchange mechanism for quartz also displays the same effect as glass. It seems apparent that the same type of adsorption sites exists on both glass and quartz surfaces though those on quartz suggest either a stronger adsorption of a higher energy level or a larger number of adsorption sites.

These data demonstrate the relatively high detergent efficiency of the polyphosphates for this soil/substrate system. Sodium metasilicate and sodium carbonate fall considerably lower in soil-removal value.

### REFERENCES

1. Anderson, R. M., Satanek, J., and Harris, J. C., *J. Am. Oil Chemists' Soc.*, **36**, 286-294 (1959).
2. Hensley, J. W., *J. Am. Cer. Soc.*, **34**, 188 (1951).
3. Hensley, J. W., Long, A. O., and Willard, J. E., *J. Am. Chem. Soc.*, **70**, 3146 (1948).
4. Hensley, J. W., Long, A. O., and Willard, J. E., *Ind. Eng. Chem.*, **41**, 1415 (1949).
5. Willard, J. E., *J. Phys. Chem.*, **57**, 129 (1953).

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## Direct Extraction of Jojoba Seed<sup>1</sup>

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JOJOBA SEED contains about 50% of a liquid wax, the composition of which is unique in that it is one of the few agricultural products known to have such

high proportions of C<sub>20</sub> and C<sub>22</sub> acids and alcohols combined as esters. This liquid wax is generally referred to as an "oil" because of its liquid state at room temperature. In several reviews and technical publications (1, 8, 10, 11) it has been shown that both the oil and the products derived from the oil have

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numerous potential uses, thus establishing the potential value of jojoba oil as a valuable raw material for industrial use.

Many papers, such as those listed by Daugherty *et al.* (1), have been published on the technical aspects of jojoba oil and its products as well as on the agronomy of the jojoba plant. However, to the knowledge of the authors, no information has been published that describes the application of a practical extraction method or the conditions necessary for preparing the seed for extraction.

The use of various solvents, such as benzene and hexane, have been mentioned by some investigators for obtaining jojoba oil for experimental work. Extraction methods and conditions were not described. In most cases it was simply stated that "conventional pressing and solvent-extraction methods can be used" with no further details.

A study on the effects of six solvents for the extraction of jojoba seed conducted by Knoepfler *et al.* (9) showed that hexane and heptane were as good as or better than other solvents tested for extraction of raw flakes in laboratory Soxhlet apparatus.

The purpose of this paper is to present bench-scale data showing the material-preparation and extraction conditions required for the efficient recovery of oil from jojoba seed by the recently commercialized filtration-extraction process (7). This process consists essentially of a) cooking oilseed flakes at lower temperatures and higher moisture contents than normally used in cooking for hydraulic- and screw-pressing operations, b) crisping the cooked material by evaporative cooling, c) slurrying material with a miscella filtrate, and d) filtering the slurry and countercurrently washing the cake on a rotary vacuum filter. The concentrated miscella and solvent-extracted meal are sent to conventional oil-recovery and desolventizer systems, respectively, for separation of solvent from oil and meal. Although this process has proven to be versatile in its application to various oil-bearing materials (2, 6, 7), including high oil-content seed, the inherent characteristics of each specific oil-bearing material necessitate the determination of a set of optimum processing conditions.

### Experimental

**Material and Equipment.** The jojoba seeds used in this investigation were obtained in 1957 from the Boyce-Thompson Southwestern Arboretum, Superior, Ariz. The nut-like jojoba seeds are about the size and shape of large coffee beans and have a thin, dark-brown outer layer over a white perisperm. The seeds, after decortication, contained 47.9% lipids and 4.1% moisture. During the interim between experiments and prior to preparation, the seeds were stored at 32°F., and, before processing, they were allowed to warm to room temperature. The extraction solvents used were commercial-grade hexane and heptane.

The equipment used for preparation of the jojoba seed for extraction included Allis-Chalmers pilot-plant, single-pass cracking and flaking rolls, and the bench-scale cooking apparatus described in an earlier publication (3). This cooker was designed (4) to make possible the accurate maintenance of desired cooking temperatures and to permit the quantitative addition and recovery of all material components. With this apparatus conditions similar to those used in commercial plants for cooking oilseeds can be

studied on a much reduced scale. To evaluate the filtration-extraction characteristics of the prepared materials, a bench-scale apparatus previously described by Graci *et al.* (5) was used. It consists of a stainless steel funnel with a removable filter, 5.25 in. in diameter. With its auxiliary units the apparatus can be operated to obtain data which are directly applicable to the commercial-scale process.

**Procedure.** The general experimental procedure consisted of flaking the seed to the desired thickness, cooking in the mixer type of cooker, crisping in open trays for about 20 min., slurrying the cooked material with solvent, and determining the filtration-extraction characteristics of the material. Experiments were designed to investigate the effect of flake thickness, moisture content of flakes during cooking, rerolling of cooked flakes, and extraction temperature on mass velocity and extraction efficiency, using hexane and heptane as solvents.

The following cooking procedure was used. The mixer type of cooker was preheated to about 180°F., and the flakes were charged to the cooker. When the flake temperature reached 180°F., the predetermined quantity of water (5 to 20%) was sprayed on the flakes through a nozzle in a 2-min. period. Temperature of the moistened flakes was then increased to and maintained at 210–220°F. for 30 min. For the first 15 min. of cooking the moisture content of the material was kept constant by refluxing, then reduced gradually to a predetermined level during the last 15 min. of cooking. The materials were crisped immediately after cooking by evaporative cooling at ambient temperature. Crisping decreases the moisture content of the cooked flakes by 1 to 2%. The cooked material was then extracted "as is" or after rerolling through smooth rolls set at 0.003 in.

The prepared materials were slurried with an equal weight of solvent either at room temperature or at elevated temperatures (120°F. for hexane and 140°F. for heptane). The slurry was hand-stirred to simulate the mixing-conveyor action of the commercial slurry mixer.

In determining the filtration-extraction characteristics of the prepared materials, using the bench-scale apparatus, certain conditions were standardized on the basis of previous experience. They were as follows: 30-min. slurrying time, three solvent washes, 2-in. cake thickness, 60 × 60 mesh filter screen, vacuum of 4 in. of mercury, and a 10-sec. drain period after addition of the final wash. As the slurry was poured on the filter unit of the bench-scale apparatus, air blow-back was used to maintain a homogenous mixture. By means of a 3-way valve, the air was cut off and vacuum was applied. Wash solvents were added when the slurry liquid disappeared from the surface of the cake. Filtration time began with the application of vacuum and included a 10-sec. drain period after the final wash liquid disappeared from the surface. The weight of the slurry and wash filtrates and the filtration times were used to calculate mass velocity, which is defined as pounds of miscella filtrate per hour per square foot of filter area. A mass velocity value of 2,000 is considered adequate for commercial application.

### Results and Discussion

Table I shows the experimental data obtained when heptane was used as the extraction solvent. Flake thickness for these experiments ranged from 0.008 to

TABLE I  
Filtration-Extraction Data.<sup>a</sup> Extraction Solvent—Heptane

Experiment No. ....	1	2	3	4	5	6	7	8
Maximum moisture during cooking, %.....	5.35	5.35	10.0	10.0	15.0	15.0	20.0	20.0
Moisture of flakes to extractor, %.....	3.27	3.02	5.78	5.44	7.28	7.62	8.39	8.33
Extracting temperature, °F.....	80	140	80	140	80	140	80	140
Mass velocity, lbs./hr./sq. ft.....	3,219	3,227	3,077	1,617	2,729	1,091	1,564	962
Extracted meal (solvent-free)								
Moisture, %.....	7.7	7.9	6.4	5.5	7.6	7.7	7.2	7.3
Residual lipids, %.....	4.55	1.98	2.28	1.16	1.99	1.22	1.70	1.10
Extraction efficiency, %.....	95.1	98.0	97.7	98.8	98.0	98.8	98.2	98.8

<sup>a</sup> Test conditions: material wt., 450 g.; solvent-to-meats ratio, 1 to 1; slurring time, 30 min.; washes, three; cake thickness, 2 in.; and filter screen, 60 mesh.

0.010 in. The two main variables were the moisture content of the flakes during the initial cooking period and the extracting and washing temperature. In the first two experiments the flakes were cooked "as is" with no added moisture. As cooking moisture of flakes was increased to 10%, the extraction efficiency increased rapidly with a corresponding decrease in residual lipids. This was true for both the 80° and 140°F. extraction temperatures. No improvement in extraction efficiency was attained for the 140° extraction when the material was cooked with moisture contents above 10%. For the 80° temperature a slight increase in extraction efficiency was attained with an increase in cooking moistures to 20%. In all cases extraction efficiency was greater when the higher extraction temperature was used.

Increasing the moisture content of flakes during cooking to 20% resulted in a decrease in mass velocities for both extraction temperatures. The decrease in mass velocity was more rapid initially for the higher extraction temperature. At 10% cooking moisture the mass velocity dropped below 2,000. Except for the experiments in which the cooking moisture contents were 5.35%, the over-all mass velocities were greater when the 80° extraction temperatures were used.

If heptane were to be considered as an extraction solvent for jojoba seed in commercial operations, it appears that either a cooking moisture content of about 10% and extraction temperature of about 140°F. or a cooking moisture of 15-18% and extraction temperature of 80°F. would be used. The choice of conditions would be a compromise between attaining adequate mass velocity and a desirable extraction efficiency.

The data for the filtration-extraction tests in which hexane was used as the extraction solvent are shown in Table II. Preliminary experiments had shown that high extraction efficiencies (low residual lipids) could be obtained when the particle size of the seed was decreased. Consequently flake thicknesses of 0.007 and 0.004 were investigated as well as the effect of rerolling the cooked, crisped flakes prior to extraction. Except for one experiment (No. 2) the

extraction temperature used was 120°F., the extraction temperature employed in most commercial solvent-extraction plants.

The effects of cooking with maximum moisture contents of 10, 15, and 20% are illustrated in the first four experiments (Table II) in which the flakes used were 0.007 in. thick and cooked materials were rerolled. Extraction efficiency varied only from 98.1 to 98.6 and residual lipids from about 1.3 to 1.7. Mass velocities however decreased with an increase in cooking moisture contents of 10, 15, and 20% for the 120°F. extraction temperatures as follows: 3078, 2498, and 1782, respectively, (Expts. 1, 3, and 4). The first two values are well within the range of practical operations; the 1,782 mass velocity approaches the desired minimum of 2,000. Experiment No. 2 was comparable to No. 3 except that the extraction temperature was 80°F. Extraction efficiency and mass velocity did not appear to be significantly affected by extraction temperature.

In an effort to eliminate rerolling, tests were conducted, as shown in experiments 5 through 8 with flakes 0.004 in. in thickness. Materials cooked with 10 and 15% moisture contents resulted in mass velocities above 4,500 and extraction efficiencies of 98.0%. For comparison purposes the cooked meats in experiment 7 were rerolled. Although extraction efficiency was increased to 99% (less than 1% residual lipids), the mass velocity decreased to 1,450 apparently due to the smaller particle size of the rerolled meats. In Experiment 8 the mass velocity was purposely decreased by decreasing the vacuum to about 1 in. of mercury, thereby increasing the contact time of the solvent during filtration. Extraction efficiency was increased to 98.3%.

Using hexane as the extraction solvent, cooking moisture contents of 10 and 15% with reduction to 6 to 7% prior to extraction are adequate to obtain mass velocities and extraction efficiencies suitable for commercial application. Rerolling is not required if flakes are rolled initially to a thickness of 0.004 in.

When optimum seed preparation and extraction conditions are used, both heptane and hexane are considered suitable as solvents for commercial ex-

TABLE II  
Filtration-Extraction Data.<sup>a</sup> Extraction Solvent—Hexane

Experiment No. ....	0.007				0.004			
	1	2	3	4	5	6	7	8
Raw flake thickness, inches.....								
Rerolling.....	yes	yes	yes	yes	no	no	yes	no
Maximum moisture during cooking, %.....	10.0	15.0	15.0	20.0	10.0	15.0	15.0	15.0
Moisture of flakes to extractor, %.....	3.7	6.4	6.4	7.3	6.7	6.2	5.7	6.8
Extracting temperature, °F.....	120	80	120	120	120	120	120	120
Mass velocity, lbs./hr./sq. ft. <sup>b</sup> .....	3,080	2,890	2,500	1,780	6,170	4,520	1,450	2,710 <sup>b</sup>
Extracted meal (solvent-free)								
Moisture, %.....	5.8	7.5	7.0	5.5	5.6	5.8	5.5	5.8
Residual lipids, %.....	1.27	1.55	1.69	1.34	1.78	1.96	0.92	1.58
Extraction efficiency, %.....	98.6	98.3	98.1	98.5	98.0	98.0	99.0	98.3

<sup>a</sup> Test conditions: material wt., 450 g.; solvents-to-meats ratio, 1.5 to 1; slurring time, 30 min.; washes, three; cake thickness, 2 in.; and filter screen, 60 mesh.

<sup>b</sup> Mass velocity reduced by reducing vacuum.

traction of jojoba flakes as there are no significant differences in mass velocity and extraction efficiency obtained with the two solvents. However hexane is the preferred solvent because it is more readily available, is lower in cost, and has a lower boiling point, which facilitates its removal from the products.

Three experiments were conducted to determine the filtration-extraction characteristics of uncooked jojoba flakes. Results were erratic in that mass velocities varied from about 200 to 1,600 and extraction efficiencies from 96.5 to 97.8 (residual lipids 1.9 to 3.2%). Use of uncooked flakes is not recommended for commercial filtration extraction operations.

### Summary

Data for the application of the versatile filtration-extraction process to jojoba seed on a bench-scale has been presented. Based on experience with other oil-seeds, there should be good correlation between the bench-scale and its commercial application. Moisture contents of the material during cooking were optimum at 10 and 15%. Mass velocities in excess of 2,000 and extraction efficiencies of over 98% were obtained. These results are considered suitable for commercial application. Hexane is recommended over heptane as the extraction solvent. The use of uncooked flakes is not considered feasible for large-scale application.

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### REFERENCES

1. Daugherty, P. M., Sineath, H. H., and Wastler, T. A., Georgia Inst. Technol. Eng. Expt. Sta. Bull. No. 17, 36 pp. (1953).
2. D'Aquin, E. L., Spadaro, J. J., Graci, A. V. Jr., Eaves, P. H., Molaison, L. J., Knoepfler, N. B., Crovetto, A. J., Gardner, H. K., and Vix, H. L. E., J. Am. Oil Chemists' Soc., 31, 606-613 (1954).
3. Eaves, P. H., Molaison, L. J., and Spadaro, J. J., Ind. Eng. Chem., 48 (10), 45A-46A (1954).
4. Eaves, P. H., Molaison, L. J., Pollard, E. F., and Spadaro, J. J., J. Am. Oil Chemists' Soc., 34, 156-159 (1957).
5. Graci, A. V. Jr., Spadaro, J. J., Paredes, M. L., D'Aquin, E. L., and Vix, H. L. E., J. Am. Oil Chemists' Soc., 32, 129-131 (1955).
6. Graci, A. V. Jr., D'Aquin, E. L., Paredes, M. L., and Vix, H. L. E., J. Am. Oil Chemists' Soc., 33, 645-650 (1956).
7. Haines, H. W. Jr., Perry, G. C., and Gastrock, E. A., Ind. Eng. Chem., 49, 920-929 (1957).
8. Jones, M. A., and Knoepfler, N. B., Chemurgic Dig., 16 (1), 5-6, 11 (1957).
9. Knoepfler, N. B., McCourtney, E. J., Molaison, L. J., and Spadaro, J. J., J. Am. Oil Chemists' Soc., 36, 644-648 (1959).
10. Knoepfler, N. B., and Vix, H. L. E., J. Agri. Food Chem., 6, 118-121 (1958).
11. Molaison, L. J., O'Connor, R. T., and Spadaro, J. J., J. Am. Oil Chemists' Soc., 36, 379-382 (1959).

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## A Simple Method for Evaluation of Heat Treatment of Soybean Meal

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EXTRACTED SOYBEAN MEAL is heat-treated in order to destroy heat-labile, antinutritional factors which impair the nutritional value of the meal. Proper heat treatment is capable of destroying toxic or undesirable factors in soybean oil meal, such as trypsin inhibitor (1, 2, 3), hemagglutinin (4, 5), saponin, a goitrogenic substance (6), an anticoagulant factor, a diuretic principle, and lipoxidase. The heating must not be applied too long or at too high a temperature since serious damage may occur to the quality of the meal. Essential components may be destroyed with a concomitant decrease in the biological value (7, 8, 9, 10).

The most widely used *in vitro* methods for testing efficiency of soybean meal toasting include measurement of urease activity (11), destruction of trypsin inhibitor, solubility of protein (in water or mild alkali), fluorescence (12, 13), ability of meal to absorb dyes containing a phthalein group (14, 15, 16), availability and extent of destruction of amino acids during the heating process or decrease in free basic amino groups in soybean meal (17, 18, 19). A collaborative study of the urease method (20) showed that inadequately heated meals gave a positive test, but the method should be considered as having limited value for testing proper toasting and of no value

in detecting overheating (21, 22). Evans and St. John (23) followed changes in solubility of soybean proteins during progressive increases in severity of moist heat treatment. Simon and Melnick (24) showed that reduction in protein solubility were generally correlated to biological improvement of soybeans during heating. Smith *et al.* (25) studied the effect of the age of soybeans, time and temperature of extraction, steam treatment, pH, and presence of wetting agent on the peptization of soybean meals. Balloun *et al.* (22) correlated the results of several *in vitro* laboratory tests with the nutritional value of soybean meals when fed to chicks. A determination of fluorescence was considered adequate for testing overheating of solvent-extracted meals but was of little value in detecting overheated expeller meals.

During the manufacture of soybean meal a quick control is essential, and only simple methods which enable an immediate decision to be made are of value. On the other hand, the information which can be expected from such methods is, of course, limited. No single physical or chemical (and probably not even biological) test can provide an evaluation significant in respect to all possible applications and uses to which the meal may be put. The value of the meal depends—in addition to severity and length of heat treatment—on the method of preparation of the soybean meal (solvent, hydraulic, or expeller) and

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