TABLE IV

Simple Correlation Coefficients between Fatty Acids and Alcohols and Percent Oil in the Seed of 1156 Samples of Native Jojoba Plants

	Fatty acid					Fatty alcohol				Oil
	16:0	18:1	20:1	22:1	24:1	18:1	20:1	22:1	24:1	(wt %)
Fatty acid										
16:0		0.75[a]	-0.24	-0.31[a]	-0.53[a]	0.64[a]	0.55[a]	-0.54[a]	-0.56[a]	-0.13
18:1			-0.21	-0.49[a]	-0.64[a]	0.54[a]	0.75[a]	-0.77[a]	-0.70[a]	-0.11
20:1				-0.71[a]	-0.33[a]	-0.05	0.30	-0.30	-0.34[a]	0.16
22:1					0.61[a]	-0.33[a]	0.78[a]	0.79[a]	0.74[a]	-0.04
24:1						-0.47[a]	-0.70[a]	0.70[a]	0.75[a]	-0.03
Fatty alcohol										
18:1							0.49[a]	-0.55[a]	-0.50[a]	-0.04
20:1								-0.95[a]	-0.87[a]	-0.03
22:1									0.84[a]	0.05
24:1										0.01

[a]Significant at 5% level of probability.

20:1, which is the major oil constitutent, correlates significantly only with longer chain acids and alcohols suggests that synthesis of this fatty acid does not involve fatty acids 16:0 and 18:1 as in animal systems (3,4).

The results presented indicate that in a large population of jojoba seed samples from diverse habitats, there was extensive variability in oil content but low variability in chemical composition. Compositional uniformity is a major asset in terms of the industrial application of this oil. No correlation was found between oil content of the seed and seed yield per plant. A strong correlation was found between oil content of the seed and seed size.

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Extraction of Tallow from Meat Meals: I. Assays in Bench Scale

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ABSTRACT

A laboratory study on solvent extraction of tallow from meat meals is described for both a stirred batch system and a flooded percolation system. The stirred batch system data, interpreted by standard diffusion concepts, show that the extraction process proceeds through a somewhat complex mechanism, which involves at least two types of extractable fat. The results for the percolation system, which show the effect of different operating variables, indicate good yields are possible with a substantially low solvent requirement.

INTRODUCTION

Meat, or meat and bone meal are obtained by high temperature rendering of meat and bone scrap and other residues that normally contain a high fat level. Coagulation of proteins and almost total elimination of water occurs during the rendering step, yielding a semisolid product (a mixture of melted fat and solids) which, after being drained and pressed, is milled, constituting the product known commercially as "meat meal" (1).

This product is marketed on the basis of its protein level—typically ranging between 40 and 55%—to be used in animal and poultry feeds. Other important components of the product are mineral salts, water and fat (tallow); the tallow is present in quantities of 10-20% in most cases. This relatively high residual fat level in the meal impairs its conservation capability because of possible rancidity and degradation. The total or partial extraction of fat from the product, besides minimizing those problems, results in additional advantages: (a) the protein level is increased, which makes commercialization more flexible; (b) the tallow obtained from the product is sold for an economic value that is not apparent in the original product; and (c) if the reduction of the fat content is carried out before the milling step, the efficiency of this operation is notably increased, providing a potential augment in the production rate.

On this basis, it seemed relevant to do a bench-scale study on the solvent extraction of tallow from meat meal, for the purpose of developing a simple method that could fit the requirements of many rural packing installations (such as those typical of southern Brazil) of low and discontinuous productions, where the possibility of grouping or associating is scarce. In choosing the method, the possi-

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bility of obtaining high extraction yields with a low solvent consumption was considered most important. From the possible alternatives, fixed bed percolation best fulfilled the requirements.

The experimental study is present here for meat meals of different origin. Studies with a laboratory percolation system have been presented in the literature for oleaginous materials (2,3). Extraction of fat from meat offal with trichloroethylene has been reported (4) but the solvent-to-solid ratio was rather high.

In this paper we will present the results of assays in a batch stirred system, which were needed to clarify certain effects that could be masked in the percolation system.

MATERIALS

Meat meals from various industries that used different raw materials were tested. Typification of these samples is listed in Table I. The solvent was commercial hexane, purchased from Petrobras, and was distilled before use.

ANALYTICAL METHODS

Water and total protein content were determined by standard methods. Fat content in the meal was determined by AOAC Method No. 7.045 (5) using hexane as solvent. The precision for this method is estimated at 2% of the fat content. For the determination of fat content in micelles, the following technique was used: a known amount of micelle (usually between 5 and 30 g) is diluted with hexane up to 100 ml. This is allowed to settle for 2 hr, after which an aliquot is taken (10 or 25 ml) and evaporated to constant weight in an aluminum dish in a vacuum oven at 100 C. The probable error using this method is estimated at 1% of the fat content.

Stirred Batch System

In order to ascertain the effect of the solvent-to-solid ratio and particle size in the absence of external diffusion or fluidodynamic complicating effects, a series of extraction tests were conducted in a stirred batch system at room temperature (28-31 C).

Two very different types of meal samples, 2 and 5 (Table I), were classified by size fractionation. Table II shows the characteristics of the fractions obtained. In this Table, it can be observed that a segregation of fat occurs in the classified fractions from each sample. This was to be expected for sample 5, since the fragments of bones with less-than-average fat content are more resistent to the milling operation and thereby accumulate in the coarser fractions; however, the same effect was observed in sample 2, which had a homogeneous appearance and practically no bones.

EXPERIMENTAL PROCEDURE

Samples (20-60 g) from each size range (Table II) were added to a given volume of hexane (100-150 ml) in a 250 ml volumetric flask and were allowed to extract for 6 hr in a laboratory shaker. Four runs were conducted simultaneously. Evaporation of hexane was kept below 0.4 g by loosely covering the flasks. During the runs, aliquots (5 or 10 ml) were taken and centrifuged to eliminate vestiges of solids and were quantitatively transferred to weighed aluminum dishes. The fat content was determined by drying the sample under vacuum at 100 C.

Percolation System

The percolation system is shown schematically in Figure 1. It consists of a device to control the solvent feed and a thermotated fixed bed column. Several possible modes

TABLE I

Characteristics of the Meat Meals Tested

Sample	Source	Water (%)	Fat (%)	Protein (%)	Solid density (g/ml)	
					Initial	Extracted
1	Swine meat meal	3.7	26.1	51.4	1.32	1.36
2	Swine cracklings meal	7.0	15.2	71.7	–	–
3	Swine bone meal	12.2	10.2	25.2	1.72	1.87
4	Swine meat & bone meal	3.8	17.7	49.5	1.46	1.55
5	Bovine meat & bone meal	7.5	14.5	41.0	1.46	1.55
6	Swine meat & bone meal	–	14.2	63.9	–	–

TABLE II

Effect of Particle Size from Samples 2 and 5: Stirred Batch System

Fraction designation	Size range (mm)	Water (%)	Fat (%)	
			(a)	(b)
2-I	0.5 - 1.0	7.4	15.9	16.3
2-II	1.0 - 2.0	7.2	13.8	14.0
2-III	2.0 - 3.3	6.6	12.1	12.1
2-IV	3.3 - 4.0	6.3	11.5	11.4
2-V	4.0 - 4.8	6.7	10.7	11.1
5-I	0.0 - 0.5	6.9	15.8	15.8
5-II	0.5 - 1.0	6.9	14.0	13.7
5-III	1.0 - 2.0	7.2	11.0	10.8
5-IV	2.0 - 3.3	7.0	10.9	10.5

(a) Data from Soxhlet extraction with hexane for 6 hr.

(b) Data obtained by extrapolation of the values of f to infinite time (see text).

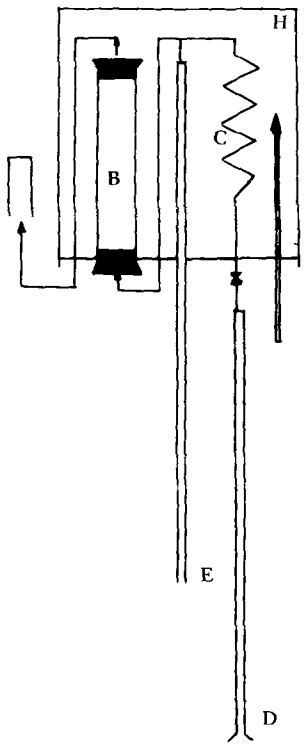

FIG. 1. Bench scale extraction apparatus.

of operation (6) were considered (upward and downward circulation, flooded and conventional operation) in preliminary tests; the conclusion was that downward, flooded percolation was the most satisfactory since it yielded a higher extraction degree and was easily performed. A flooded percolator, as used here, is not to be confused with the immersion extractor (6,7), where the particles become dispersed and agitated and have a higher solvent-to-solid ratio. Instead, it resembles a chromatographic column in the sense that a sharp solvent front is achieved, allowing higher extraction efficiency through minimal axial mixing.

The meat meals to be extracted with this system were previously milled in a No. 3 Wiley Standard Knife Mill through a 2 mm mesh.

The extraction column (B) shown in Fig. 1, 3.1 cm id and 15.0 cm high, was loaded stepwise with the solids to allow a compacting of the bed to the desired bed density until the available volume (ca. 100 ml) was reached. The final amount of solids (75-110 g) was obtained by weight difference. The column was placed in the thermostatic bath (H) and left for 1 hr to reach thermal equilibrium at the operational temperature (55 ± 1 C). Hexane was fed from buret (D) through preheater (G). Buret (E) was used to purge the air liberated by the solvent. The time to fill the column with solvent, measured from the start of hexane feeding to the exit of the first drop of micelle, was between 0.5 and 1 hr for all runs. At that moment, the hexane volumetric rate was adjusted so that 20 ml of micelle could be collected per hour. Operation was continued until the micelles became very diluted (less than 1% fat), giving a total extraction time of between 5 and 6 hours.

When the run was terminated, the bed was drained by applying a slight overpressure and the exiting micelle was collected. The solvent retention was determined by weight difference. The solid was desolventized by oven drying at 100 C for 1 hr and the residual fat content was determined. Finally, the collected micelles, including those from the draining step, were weighed and analyzed.

RESULTS AND DISCUSSION

Stirred Batch System

For this experiment, the degree of extraction was measured through the variable X, defined as,

$$X = f/f_{max}, \quad [I]$$

$$\text{where } f = 100\,\frac{(V_{h,i}/v_{h,i})\, m_{g,i} + \sum_{j=1}^{j=i-1} m_{g,j}}{m_s}, \quad [II]$$

$$\text{and } V_{h,i} = V_{h,i-1} - v_{h,i-1} \quad [III]$$

$$v_{h,i} = v_i - (m_{g,i}/d_g) \quad [IV]$$

The meaning of the symbols is as follows: V_h = volume of hexane in the flask before taking the sample, ml; v_h = volume of hexane in the sample, ml; v = total volume of sample, ml; m_g = fat content in the sample, g; m_s = initial mass of solid sample, g; d_g = density of fat = 0.91 g/ml; i,j = subscripts indicating the sample number; f = fat content (hypothetical) dissolved at a given time, relative to the initial mass of solid sample, %; f_{max} = limiting value of f at infinite time, %.

Equation II merits further explanation: first, the cumulative mass of fat in the preceding samples had to be included (second term in the numerator), since the volume of the samples taken was substantial with respect to the initial volume of solution. Second, the first term in the numerator implicitly contains the assumption that all the hexane present in the flask is associated with the interparticle micelle concentration. Actually, part of the solvent belongs to the internal micelle of a higher, unknown concentration. This is why f is called a hypothetical quantity. However, it is clearly proportional to the real quantity, and becomes equal to this at infinite time -f_{max}-, thus corresponding to the percentage of fat initially present in the solid sample.

X varies between 0 and 1 and represents the fraction of equilibrium attained at a given time. The function X vs time (t) can be theoretically predicted by simple diffusion theory for spherical particles into an infinite medium (8) as:

$$X = 1 - \frac{6}{\pi^2}\left[\exp(-A) + \frac{1}{4}\exp(-4A) + \frac{1}{9}\exp(-9A) + \ldots\right] \quad [V]$$

where,

$A = \pi^2\, Dt/r^2$ and $D = D_{ef}/e_p$

In this equation, e_p = porosity of particle; D = diffusivity; D_{ef} = effective diffusivity; and r = radius of particle.

In order to obtain f_{max} for each solid sample, a plot of f vs 1/t was drawn (Fig. 2), and the f values were extrapolated to the origin (t = ∞), with the condition that equation (V) predicts dX/d(1/t) = 0 at (1/t) = 0. The values thus obtained are presented in Table II, last column. They show a close agreement with those from the Soxhlet analysis, with hexane as the solvent (Table II, column a), and where used to calculate X.

Effect of the Solvent-to-Solid Ratio, S

Three assays were conducted in parallel on fraction 2-V (Table II); S ranged between 1.09 and 4.90. The results, shown in Table III, indicate the extraction process is independent of S and is therefore independent of the

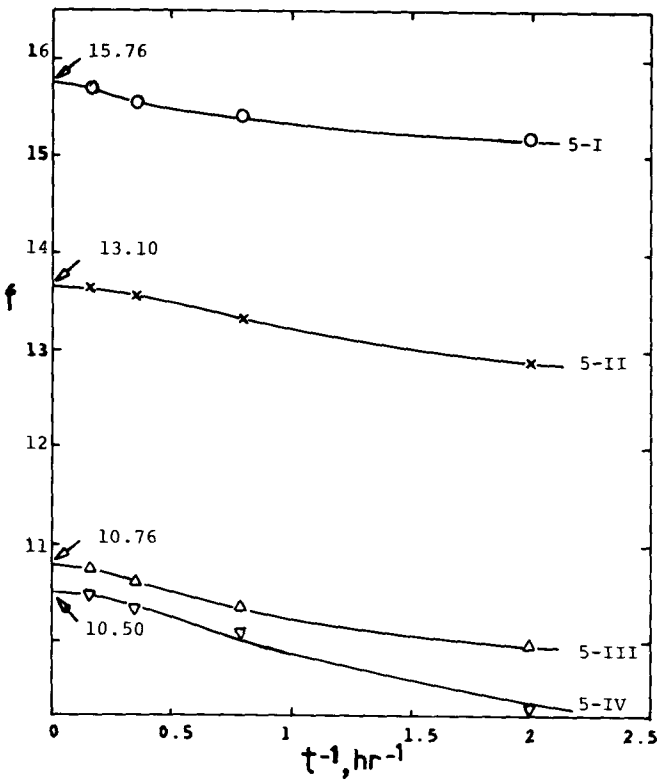

FIG. 2. Determination of f_{max} for sample 5.

micellar concentration in the range 0-10%. A similar conclusion is reported in the literature for the extraction of soybeans (9,10).

Effect of Particle Size

This is shown for the different fractions of samples 2 and 5 in Figures 3 and 4. The curves are internally consistent, which is apparent from the low dispersion of the data. However, the shapes of the curves are different from those predicted by equation V for each particle size (not shown here). Thus, for fraction 5-IV (Fig. 4), the best correlation with equation V yielded a diffusion coefficient D_{ef}/e_p = 5.2 x 10^{-6} cm^2/sec with a standard deviation of 9%, which is much higher than the experimental error.

The experimental curves present a much longer tail than that expected from equation V for all fractions. This can be interpreted as a decrease of the effective diffusion as extraction proceeds, which would not be attributable to a variation of diffusion with concentration, since the average concentration of the micelle within the particles decreases and therefore the diffusion should increase with extraction time; this is an opposite effect compared to the one observed.

These data suggest an improved model of the process

TABLE III

Extraction Yields vs Time for Different Values of S: Fraction 2-IV

S g/g	Time (h) 0.51	1.5	3.0	Final micelle concentration (%)
1.09	0.84	0.94	0.94	9.2
1.63	0.84	0.94	0.98	—
4.90	0.87	0.94	0.98	2.2

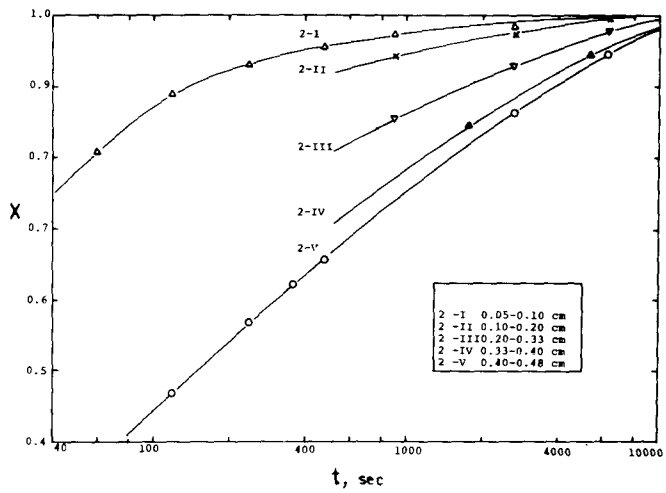

FIG. 3. Effect of particle size on the rate of extraction for sample 2.

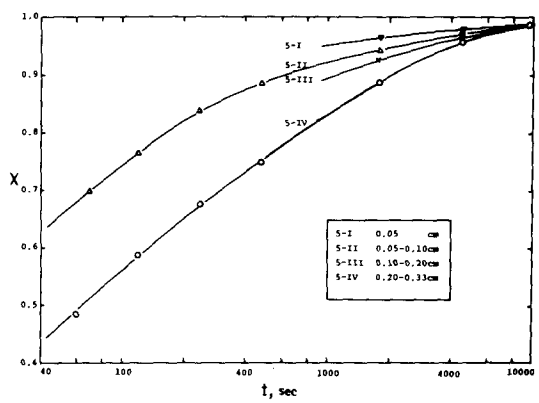

FIG. 4. Effect of particle size on the rate of extraction for sample 5.

would consider at least two different types of fat liaison to the solid: (a) fat contained in the pores of the solid, extractable by a normal diffusion process, and (b) fat more strongly held to the solid, probably bound to the protein matrix or associated with a microporous structure with lower accessibility to the solvent, thus yielding the tailing effect that can be related to a lower diffusion coefficient for this fraction. That b more correctly describes the true situation was verified by fitting the experimental data to a model, assuming 75% of the fat to be of the type a and 25% to be of the type b, diffusing independently and in a parallel fashion. This approach yielded diffusion coefficients D_{ef}/e_p = 14 x 10^{-6} and 0.27 x 10^{-6}, respectively, with a standard deviation of only 3%.

Even more flexible models could be proposed, such as one that considers another fraction of the fat to be contained in large voids or irregularities on the surface of the solid, which is readily dissolved by the solvent and does not contribute to the internal diffusion process. However, this would be beyond the scope of this work, since the purpose of this discussion is to show the extraction process under study is not simple. Nevertheless, this agrees with the findings of other authors (7,11) concerning the extraction of oil-bearing materials.

With respect to the extraction time, the figures show that, even for the longest particle size (fraction 2-V) the degree of extraction is over 90% after 1 hr at ambient temperatures (ca. 30 C). These assays were not repeated at higher temperatures, but the extraction time is expected to be substantially reduced.

It is notable that the behavior of the two samples, 2 and 5, is similar in spite of appearing inherently different. The similarity does not extend to the final period of extraction, where sample 5 shows a stronger retention of fat (longer tail). This departure is unimportant, however, for practical or industrial purposes, where the main objective is to reduce the initial fat content to a final value of 2 or 3% and therefore is of little relevance to the behavior of extraction for values of X over 90%.

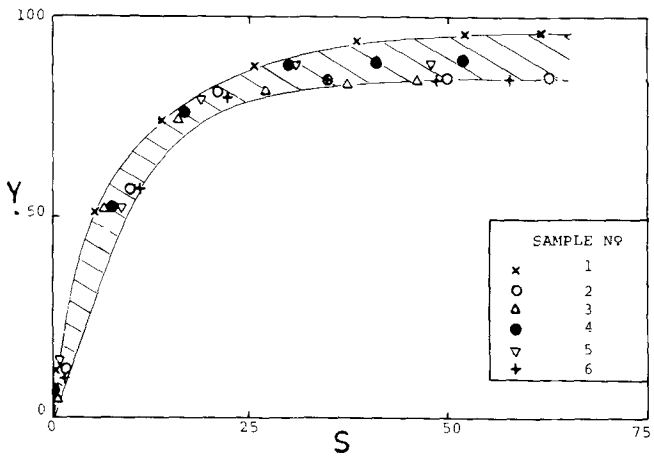

FIG. 5. Extraction yield vs solvent consumption for different meat meals (see Table I).

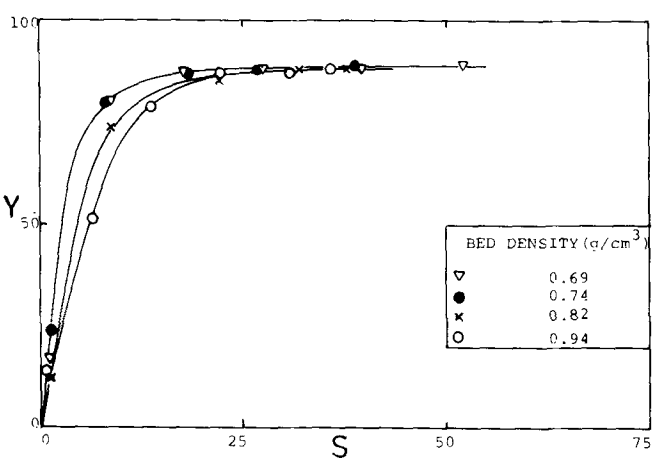

FIG. 6. Effect of bed compaction on extraction yield for sample 5.

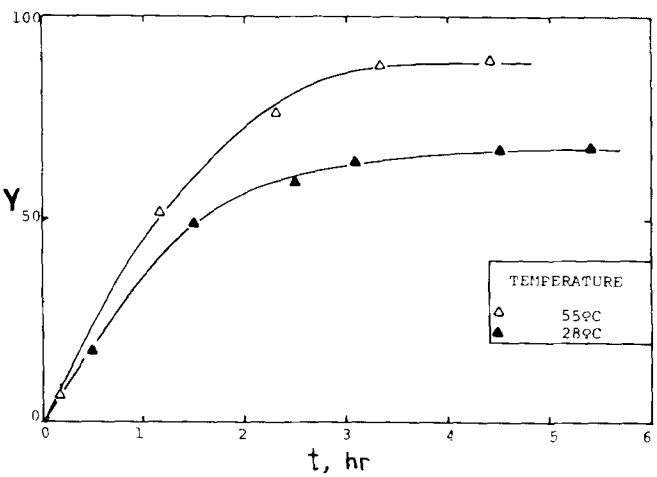

FIG. 7. Effect of temperature on extraction yield for sample 4.

Percolation System

The results are presented as Y vs S plots. Y is defined as the percentage of fat extracted in the micelles compared to the initial fat content in the meal and represents the extraction yield. S is the percentage of cumulative mass of solvent compared to the initial mass of solid and is a measure of the spent solvent. The total solvent consumption is given by S + R). R is the percentage retention of solvent by solid at the end of the operation, relative to the initial mass of solid.

Figure 5 shows the results obtained for the different meat meals listed in Table I. The shape of the curves is similar for all cases, yielding a degree of extraction over 80% for S = 25% and a total solvent consumption on the order of 53%. No appreciable differences are apparent among the meals up to values of X of ca. 75%; deviations are observed only for extraction degrees over 85%. This is consistent with the behavior already reported for the stirred batch system and also with those reported in the literature (7,11) for oily substances of vegetable origin.

The effect of bed compaction on X was studied on sample 5 where the bed density varied between 0.69 and 0.94 g/cm^3. The results are shown in Figure 6. There was no marked effect for X > 80%. This is because the rate of diffusional flux within the particles is very slow and rate-controlling during this step of the process, which agrees with the effects observed in the stirred batch system. On the other hand, the differences which are apparent for X < 80% and which become more marked with increased bed density probably reflect a reduction in the particle surface available to the solvent.

The effect of temperature on extraction yield was studied using sample 4. For this experiment, the results are presented as a function of time (Fig. 7), to show the behavior in the time scale that was typical of all runs.

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