

TABLE II
Analysis of Physically Refined Soybean Oil

	DEGUMMED & BLEACHED	STEAM REFINED
FFA, WT %	0.4	0.015
PEROXIDE, ME/KG	---	0.0
COLOR	3.6R/35Y	0.3R/5Y
FLAVOR	---	8.5

excellent. A flavor score of 8.5 (scale 1-10) was reported by a taste panel. They described the flavor as excellent as was flavor stability as measured by standard AOCS procedures.

The amount of bleaching clay as well as the steam usage and operating temperature of the steam refining deodorizer were at the same levels commonly used for deodorization in a chemical refining system. The high levels of clay reported in the recent symposium (8), which were a cause for the concern about economics of physical refining, were not required to make a satisfactory product. In addition it appeared the yield of product was somewhat greater than would be expected from chemical refining, but the run was too short to quantify this observation.

Whether physical refining can be used for soybean oil, regardless of quality, at this time is unknown. But now that extensive research is being done, commercial operations are beginning, and a considerable amount of interest in physical

refining is being generated, it can be assumed that processing techniques will be developed to handle crude soybean oil regardless of its quality.

The physical refining system has an inherent lower installed cost when compared to chemical refining, lower steam, water and power requirements, and eliminates soap-stock and its associated waste treatment problems. These facts will most certainly provide the impetus to bring physical refining of soybean oil to the forefront in the very near future.

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✿ Evaluation of Extractive Contact Units for Oil Extraction from Dehulled Sunflower Seed

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ABSTRACT

The efficiency of an extractive system generally depends on kinetic, thermodynamic and technological parameters. Each of these factors affects the extractive effectiveness, although the overall result depends on their interaction. In the present work this interaction is analyzed for oil extraction from dehulled sunflower seed. Experiments on laboratory scale were made to calculate the kinetic and thermodynamic parameters of the extraction. The behavior of the system in 2 typical contact units (mixer-settler and semicontinuous extractor) was studied and models assuming equilibrium conditions were formulated. The soundness of the models was checked by pilot-plant tests and good agreement was obtained as long as the residual oil concentration in the solid was higher than ca. 0.01 c_{s0} . At lower oil concentrations the contact time becomes the main factor of the process, and the extraction is much less affected by the extraction ratio and the composition of the liquid.

INTRODUCTION

This work is part of a more extensive research program that aims at developing a technology for producing edible proteins from sunflower seeds. The characteristics of this technology are: the near-total dehulling of the seed and the direct oil extraction (without prepressing) at a relatively low temperature. The apparatus normally used for the extraction of oil from seed (percolation) can be hardly used with this type of material for rheological reasons (1-3). Because of this, immersion systems must be taken into account. At the moment they are not very widespread

owing to their bulkiness and low efficiency (4-5).

In this work the possibility of improving the traditional immersion systems by affecting the rate limiting factors has been investigated.

EXPERIMENTAL PROCEDURES

Three groups of tests were performed.

Batch Laboratory Tests

Dehulled and flaked sunflower seeds were placed in contact with n-hexane in a 5 L stirred thermostatic vessel. The seeds had 3% residual hull (evaluated by direct weighing) and 60% lipids (petroleum ether extraction, according to AOCS method Ac 3-44). The kinetic of extraction was followed by measuring, as a function of time, the amount of oil in the liquid (spectrometric analysis at 232 nm) and in the solid (6-8). The tests were repeated by varying: seed/hexane extraction ratio (R') in the range of 0.1-0.0125 kg/L; extraction temperature (T) in the range 22-50 C; seed characteristics (humidity in the range 5-12%, flaking in the range of 0.1-0.5 mm).

Semicontinuous Pilot-Plant Tests

This system consisted of a number of extraction units (N) (600 L volume each) connected in series cyclically and fed with solvent for a time (t). Specifically, the connection allowed the isolation of the loading and discharging unit

OIL EXTRACTION FROM SUNFLOWER SEED

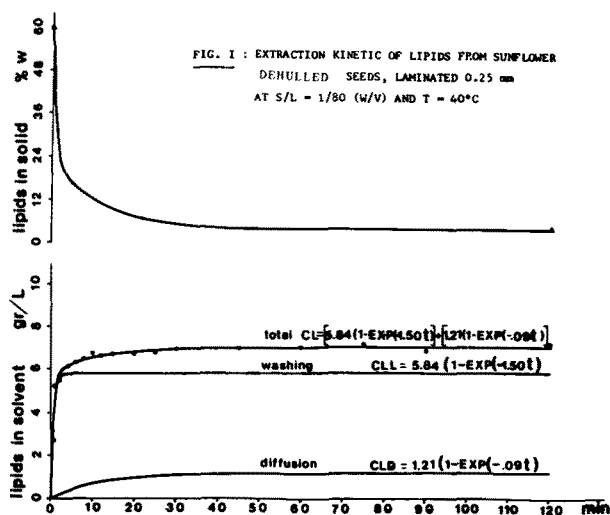


FIG. 1. Oil extraction kinetic from dehulled sunflower seeds—flaking, 0.25 mm; extraction ratio, $S/L = 1/80$ (W/V); temperature, 40 C.

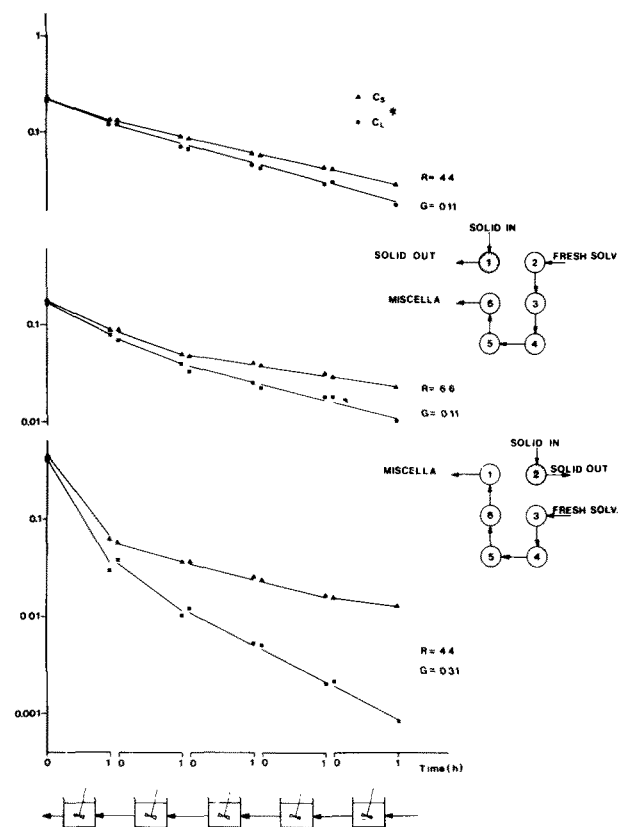


FIG. 2. Oil concentration in the solid and liquid (c_s , c_L^* , kg oil/kg residue) during a semicontinuous extraction for different extraction ratio (R , kg hexane/kg residue) and vessels filling ratio (G , kg residue/(kg residue + kg hexane)).

from the others under extraction, and the shifting of the solvent input/output points from one unit to the next, along the extraction battery (Fig. 2).

The main characteristic of the system is that the solid, while remaining stationary in each unit, undergoes $(N-1)$ solvent extractions with a gradual decline in oil concentration. The last contact occurred between practically exhausted solid and fresh solvent. When the system is at steady state, the oil concentration profile in the solid and

liquid were the same as would be obtained in a continuous countercurrent system. The extraction process was studied by measuring, as indicated above, the concentration of oil in the liquid (c_L) and in the solid (c_s) in each vessel as a function of time at steady state. The tests were repeated by varying: extraction ratio (R) in the range 4-7 (kg solvent/kg extraction residue), vessel filling ratio (G) in the range 0.11-0.31 kg residue/(kg solvent + kg residue), extraction temperature (T) in the range 20-55 C, cycle time (t) in the range of 0.8-1.2 hr.

Continuous Mixer-Settler Pilot-Plant Tests

The system consisted of a series of countercurrent solid-liquid contact units (capacity ca. 1000 L each). The extraction process was studied by measuring, at steady state, (c_L) and the (c_s) in each stage, and the amount of the solvent (i) (kg solvent/kg residue imbibing the solid transferred from one stage to the next). The solid-liquid separation was carried out by a decanter centrifuge (5000 RPM; 230 mm diameter). The tests were repeated by varying R in the range 3-15 and number of stages ($N = 2$; $N = 3$).

RESULTS

The oil extraction kinetic from dehulled flaked sunflower seed, for one of the batch laboratory tests is shown in Figure 1. Two distinct phases can be identified: an initial phase with a high material transfer velocity, and a second phase characterized by a substantial reduction of this velocity and a slow approach to equilibrium conditions.

The oil concentration measured at a steady state in each of the 5 stages of the semicontinuous pilot plant are shown in Figure 2. The oil concentration in the solid was expressed as (c_s) (kg oil/kg residue), and the oil concentration in the liquid as (c_L^*). (c_L^*) is the concentration of oil in the solid at equilibrium with a liquid at concentration (c_L) ($c_L^* = c_L/\alpha$), and (α) is the equilibrium constant evaluated from the results of the laboratory tests ($\alpha \approx 1.3$ kg residue/kg solvent). We observed that in the first phase of the extraction, the system works, in every stage and at all times, in equilibrium conditions ($c_s \approx c_L^*$). In the final phase of the extraction a departure from these conditions was observed, specially when G and R increased.

The residual oil concentrations in the exhausted solid in the mixer-settler pilot-plant tests are shown in Figure 3. The measurements were made at steady state for different values of the extraction ratio and number of stages. Also in Figure 3, the theoretical curves, calculated by assuming equilibrium conditions in each stage, are reported. For both $N = 2$ and $N = 3$ we observed a good agreement with the model as long as c_s/c_{s0} is greater than ca. 0.01.

DISCUSSION

The results obtained in the present work indicate that the behavior of the system is characterized by a complex interaction among kinetic parameters, the equilibrium constants and the process parameters (extraction ratio, contact time, degree of stirring, number of extractors, extraction system and imbibition coefficient).

For the optimum design of the system, a sensitivity analysis is required to show the advantage inherent in the variation of a parameter when the others are fixed at a base value. This analysis is very simple for a continuous mixer-settler system if we assume equilibrium conditions in the stages, linearity of the equilibrium relation and constant imbibition coefficient (Fig. 4). These hypotheses are quite well satisfied when c_s/c_{s0} is higher than 0.01 (Fig. 3).

The sensitivity of the system indicates (Fig. 5) that the

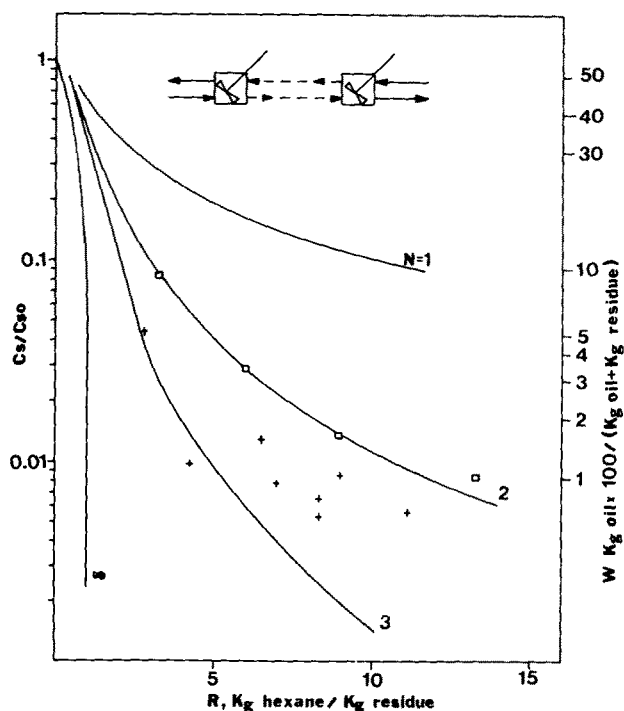


FIG. 3. Residual oil concentration as a function of the extraction ratio and the number of stages for mixer-settler extraction. Theoretical curves calculated from relationships indicated in Figure 4; experimental results for $N = 2$ (\square) and $N = 3$ ($+$).

SINGLE STAGE



$$\frac{(C_s)_i}{(C_s)_o} = \frac{1 + i\alpha}{1 + R\alpha} \quad \text{with } R = L/S$$

TWO STAGES



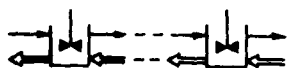
$$\frac{(C_s)_i}{(C_s)_o} = \frac{(1 + i\alpha)^2}{\alpha R(\alpha R + 1) + 1 + i\alpha}$$

THREE STAGES



$$\frac{(C_s)_i}{(C_s)_o} = \frac{(1 + i\alpha)^3 (R\alpha)^{-1}}{R\alpha(R\alpha + 1) + (1 + i\alpha)^2 + (1 + i\alpha)R\alpha}$$

INFINITE STAGES



$$\frac{(C_s)_i}{(C_s)_o} = \frac{1 - \alpha R}{1 + i\alpha}$$

FIG. 4. Performance relations for mixer-settler extraction.

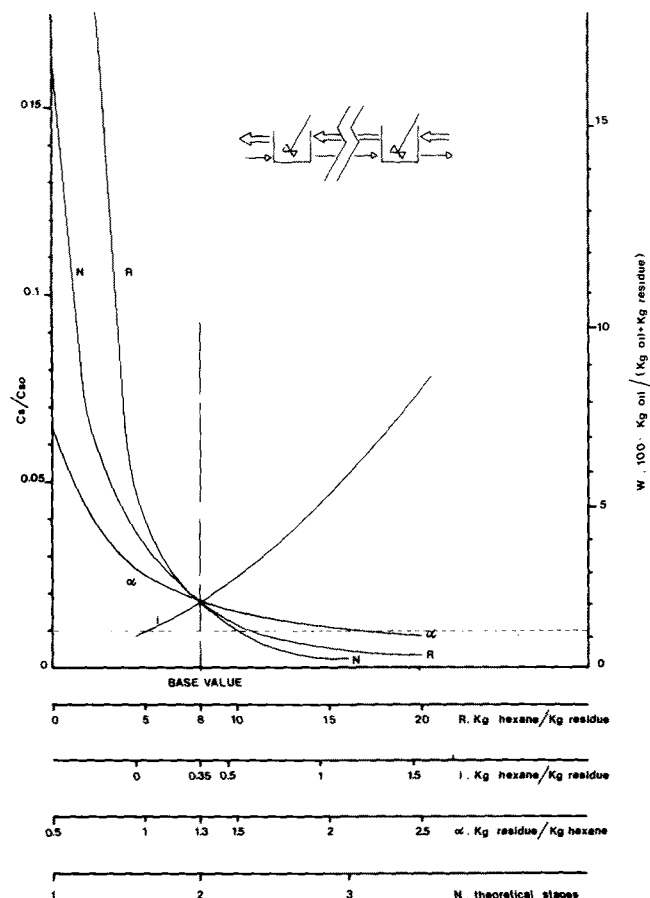


FIG. 5. Sensitivity analysis of the extraction efficiency to the main parameters of the system (mixer-settler).

advantage of an increase in the number of the stages becomes unimportant after 2 or 3 units. In the same way, an increase in R beyond 8 or in α beyond 1.5 produces less marked reduction in c_s/c_{s0} . Outside the range of validity of the model (c_s/c_{s0} less than 0.01), a still lower sensitivity of the system to parameters, e.g., R , N and α , is likely to occur whereas the main effect will arise from the residence time in the extractors. A sensitivity analysis of the semicontinuous system can be deduced directly from the oil-extraction kinetics measured for different values of G and R (Fig. 2).

An increase of G from 0.11 to 0.31 or an increase of R from 4.4 to 6.6 noticeably improves the extraction efficiency (the final value of c_s decreases from 0.033 to 0.014 for the increase of G and to 0.025 for the increase of R) because of the improved solvent replacement in each stage. At high values of R , and mainly at high values of G , the departure from equilibrium is considerable. In these conditions the reduction of c_s vs time is not improved by the reduction of oil concentration in the liquid. This result agrees with the observations of others who reported that the oil concentration in the miscella had very little effect on the extraction at low c_s value (9,10).

A simple kinetic model was presented in (6) to simulate the sharp reduction of the mass transfer rate during the extraction with 2 diffusion coefficients: (a) a higher one (washing coefficient) $K_w = 1-2 \text{ min}^{-1}$; (b) a lower one (diffusion coefficient), $K_D = 0.01-0.02 \text{ min}^{-1}$ in the conditions studied. This model simulates quite well the results obtained in the laboratory tests (Fig. 1).

The results obtained in the last phase of the extraction in the pilot-plant tests can also be simulated by such a

model by defining c_{sD} , the "diffusive" oil concentration present in the solid (6), as $c_s = c_L^* + (c_{sD} - c_L^*) \exp(-K_D t)$. In fact, when K_D is low enough (even lower than the values obtained in [6]), c_s is a function of the time almost unaffected by the concentration of the liquid c_L^* .

Actually, a more rigorous model that took into account the oil concentration gradients within the solid particles and the different transport properties of the chemical species involved (lipids and phospholipids) could allow a closer fitting of the experimental results.

The results of the present investigation indicate that the rate-limiting step of the extraction is the removal of the last portion (diffusive) of the oil of the seed. This step could be improved by increasing the residence time, or the temperature in the last phase of the extraction. So that satisfactory extraction efficiencies (residual oil less than 1.5% with extraction ratios R in the range 4-5) could be attained even with very simple immersors (mixer-settler or semicontinuous extractor).

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✿ Almost Complete Dehulling of High Oil Sunflower Seed

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ABSTRACT

An almost complete dehulling (hull residue lower than 3%) of sunflower seeds, before oil extraction, reduces to a minimum both the transfer of pigments from hulls to the flour and the content of fiber in the finished product. In this paper some results of our work on the dehulling of high-oil seeds with an air-jet impact huller are presented. The effectiveness of dehulling has been evaluated as a function of characteristics of the seed (variety, moisture and so forth) and of operative parameters (impact velocity, etc.). The optical analysis of the impact of the seeds on the target was made by means of high-speed cinematography (about 8000 frames/sec) to have a better view of the phenomenon and to measure the parameters of energy involved. The use of proper seed momentum, which is a function of the characteristic of the seed, can allow selective hull breaking with minimum kernel breakage. Almost complete hull-free kernels from high-oil sunflower seeds were obtained by means of a continuous dehuller-separator pilot plant.

INTRODUCTION

The scope of this work was to determine the optimum conditions for the production of almost complete hull-free kernels from sunflower seeds. This work is part of a more extensive research program aimed to develop a technology for the production of food-grade sunflower flour. For producing food-grade flour, an almost complete dehulling (residual hulls less than 2-3%) is required to minimize the transfer of pigments from the hulls to the flour and the fiber content in the finished product (1-3).

Partial dehulling of the seed, up to about 10-12% of residual hulls, is quite common in the oil industry (4-7) and presents the following advantages: decreased volume of product to defat; decreased erosion of the presses; better quality oil (lower wax content); better quality of the extracted flour (increase in the protein content).

Industrial applications of complete dehulling of the seeds are unknown. An important contribution on this subject was reported by The Food Protein Research and Development Center, Texas A & M University, which carried out research with the aim of producing hull-free kernels (3). The results of this study, and those of other researchers (8-11) show the technical difficulties and the low yields occurring in the dehulling of high-oil sunflower seed. To try to overcome these difficulties, research mainly devoted to the dehulling of high-oil seed with an air-jet impact huller, has been carried out in the present work. The effectiveness of dehulling, in a once-through laboratory device, and in a continuous dehuller-separator pilot plant, has been measured as a function of the characteristics of the seed (variety, moisture and so forth) and of the operative parameters (impact velocity and so forth).

EXPERIMENTAL PROCEDURES

Laboratory Tests

Once-through dehulling tests have been performed using a laboratory air-jet impact huller manufactured by Hydro-mecanique et Frottements (HF) (12,13). This huller consists of a seed-sucking section (Ventury effect), an acceleration tube and a metal target tilted at an angle to the axis of the tube (Fig. 1). Proper air (Q) and seed flow rates (W) are fed to the huller for a certain time (t). The outgoing product is collected and the following fractions are separated by hand and analyzed by direct weighing: percentage of hulls (H); percentage of whole or broken kernels; percentage of fines (F) (broken kernels with diameter less than 2 mm); percentage of partially dehulled seeds and percentage of unde-hulled seeds.

The dehulling efficiency (E), is defined as the percentage of dehulled seed and is evaluated, as $E = H \times r$, r being the