Development of a Pilot-Plant Process for the Extraction of Soy Flakes with Aqueous Isopropyl Alcohol[1]

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ABSTRACT

Soy flakes were extracted with aqueous isopropyl alcohol (IPA) at 77 C in a Kennedy countercurrent continuous extractor at a retention time of 71 min. IPA concentration was varied from 85.0 to 90.5% w/w and included the 87.7% IPA-water azeotrope. Solvent to meal ratios were varied from 1.5 to 3.0. The oil-IPA miscella leaving the extractor was chilled and coalesced to yield an oil phase and an IPA phase. The IPA phase was recycled to the extractor without being distilled. Excess IPA was expressed from the defatted flakes, and this also was recycled to the extractor. IPA recovered by distillation in the evaporator-stripper and desolventizer-toaster accounted for less than 10% of the total. Refined deodorized oils from the IPA extraction process compared favorably with their hexane counterpart in color, peroxide value and phosphorous and free fatty acid contents. Desolventized meals from the IPA process compared favorably with their hexane-extracted counterpart in protein, ash and fiber content.

INTRODUCTION

In the extraction of oilseeds, hexane is the solvent used most extensively. Hexane, a hydrocarbon fraction derived from petroleum, has a boiling range of 63–69 C and is an excellent solvent for vegetable oils. Concerns about availability, safety and tighter emission restrictions have stimulated interest in alternatives to hexane as an extraction solvent. Isopropyl alcohol (IPA) is in search of a new market, because it is being replaced by cumene as a starting material for the production of acetone (1).

When we compare IPA to hexane we note some significant differences in properties and costs of the solvents (Table I). IPA costs more than hexane and requires about twice as much heat to vaporize. On the other hand, with its much higher flash point, IPA is safer than hexane. The NIOSH recommendation (2) for hexane exposure would, if adopted, reduce the maximum allowable concentration of hexane in the workplace.

Extraction of soybeans with ethanol was practiced in Manchuria during the 1930s and the process was evaluated at the Northern Regional Research Center in the mid-1940s (3). Low solubility of oil in 95% ethanol at ambient temperatures requires that the extraction be carried out under slight pressure to raise the temperature above 90 C where the oil and ethanol are completely miscible. Another disadvantage of ethanol is its tendency to remove moisture from the flakes unless they have been dried to below 3% moisture.

Isopropanol is a better solvent than ethanol for the extraction of oilseeds. It has been used to extract oil from cottonseeds (4, 5). Work done by previous investigators (3, 6) has shown potential energy savings when extracting oilseeds with aqueous alcohols because of the nondistillation recovery of oil. The purpose of this study was to develop a total pilot-plant process utilizing aqueous IPA for the production of finished oil and meal fractions from soybeans.

TABLE I

Some Significant Solvent Differences Between Hexane and Isopropyl Alcohol (IPA)

Item	Hexane	IPA azeotrope
Price per gallon (March, 1982)	$1.45	$1.95
Heat of vaporization (cal/g)	80	206
Flash point (C)	−14	18
Toxic level (ppm, PEL[a], TWA[b])		
OSHA	500	400
NIOSH recommendation	100	400

[a]Permissible exposure limit (PEL).
[b]Time-weighted average (TWA) work shift.

MATERIALS AND METHODS

Solubility of Soybean Oil (SBO) in IPA

Information in the literature was scanty on the solubility of SBO in the various aqueous IPA concentrations where we intended to conduct our investigations, so we generated solubility curves in an apparatus of our design. Quantities of SBO were mixed with aqueous IPA in the reservoir of the glass apparatus (Fig. 1). The mixture was heated to the boiling point, agitated, then cooled slowly ca. 10 C and equilibrated before assaying the IPA phase for oil content. The mixture was further cooled, equilibrated and assayed in ca. 10-degree increments until a solubility curve was generated for each of the three IPA concentrations (Fig. 2).

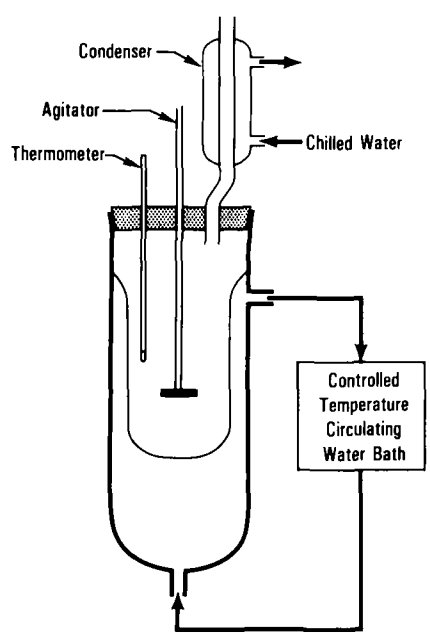

FIG. 1. Apparatus for the determination of the solubility of soybean oil in aqueous isopropyl alcohol.

[1]Presented at the 73rd AOCS annual meeting, Toronto, 1982.
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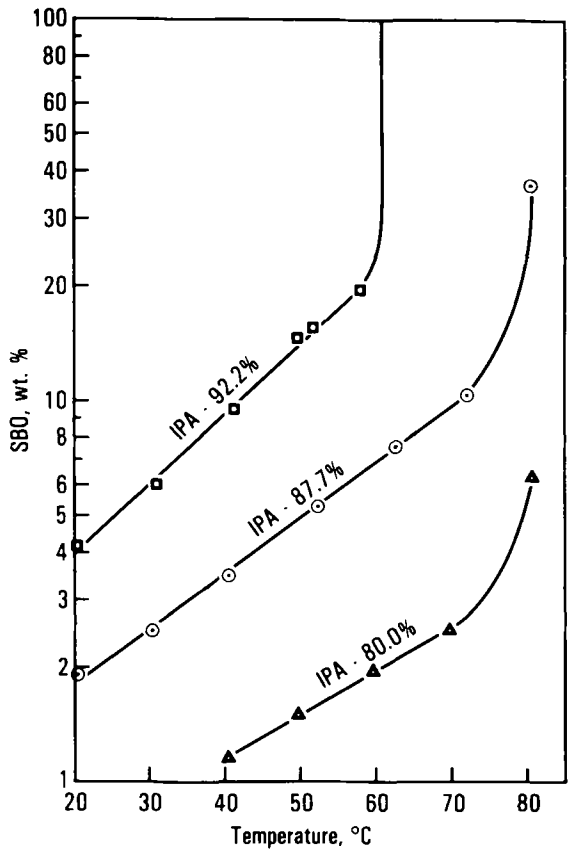

FIG. 2. Solubility of soybean oil in isopropyl alcohol.

Although the method was completely different, the results are in general agreement with the earlier work of Rao and coworkers (7).

Experimental Design

Based on the solubility curves for SBO in IPA, it was calculated that with the extractor operating slightly below the boiling point (bp) at 77 C, a maximum oil concentration in the miscella of 15% would be obtained with the azeotrope (87.7% w/w IPA). Based on an estimated oil content of 22% in flakes, a solvent to meal ratio of 1.5:1 was calculated to be minimal and therefore, a 2:1 ratio should be adequate. It was further calculated from extrapolation of the solubility curve data that IPA concentrations below 85% would not be adequate for good oil recovery at this same solvent to meal ratio. On the upper side of the azeotrope the choices were unrestricted, and 90.5% IPA was chosen simply to achieve a balance on either side of the azeotrope. Four experiments were run in which these three concentrations of IPA were tested against hexane, all at a solvent to meal ratio of 2:1 and a retention time of 71 min. The IPA extractions were made at 77 C, and because of its lower bp, the hexane extraction was made at 60 C.

In a fifth experiment, 85.0% IPA was tested at a higher solvent to meal ratio (3:1) because the 2:1 ratio was projected to be borderline. Because of its greater solubility potential for SBO, 90.5% IPA was also tested at a lower (1.5:1) solvent to meal ratio in a final experiment.

Milling and Flaking

Beeson variety seed-grade soybeans were cracked by passage through 15-cm diameter corrugated rolls set for 0.19 cm clearance. As the cracked beans passed onto the double screen shaker, the hulls were removed by an aspirator and collected. The larger pieces of cracked soybeans passing over the top screen (0.48-cm perforated round hole) of the double screen shaker were recycled into the cracking process. Bottom screen was 14-mesh black wire screen. The dehulled beans (meats) retained on the bottom screen passed to the tempering conveyor, where they were heated to 74 C by indirect steam. Sparge steam was used as rquired to maintain the moisture at 10–11%. Tempered meats were flaked through 30.5-cm diameter smooth rolls to a thickness of ca. 0.025 cm (8).

Extraction

The flow sheet for the extraction of soy flakes with IPA is shown in Figure 3. The oil was extracted from the soy flakes in a 20-stage Kennedy (9) countercurrent continuous extractor (Fig. 4) with commercial grade hexane or with commercial grade 99% IPA diluted with water as dictated by the experiment. The solvent was preheated and entered near one end of the extractor; because the extractor is built at a slight incline, the solvent overflowed into the next and subsequent stages and continued moving in that direction until it left as a full miscella at the opposite end.

The flakes were fed into the opposite end of the extractor by a metered feeder and were picked up by slotted paddlewheel flights that rotated slowly on a variable drive shaft. This advanced them to the next and subsequent stages, moving them through the extractor, where they were picked up by a dragline on the opposite end and given a final drain on an inclined slotted bar screen before being discharged from the extractor. Extractor temperature was contolled at 77 ± 2 C (60 ± 2 C when extracting with hexane) by a circulating water bath. Retention time in the extractor was 71 min.

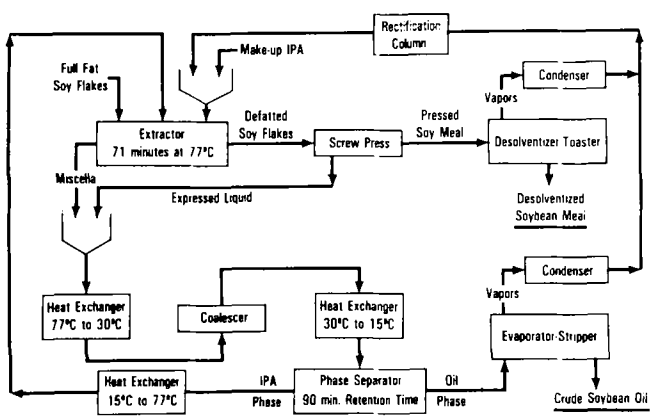

FIG. 3. Flow sheet for the extraction of soy flakes with isopropyl alcohol (IPA).

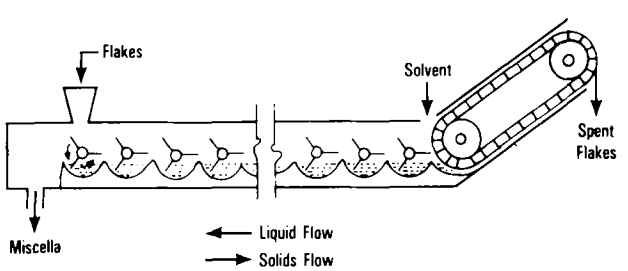

FIG. 4. Kennedy 20-stage countercurrent extractor.

The mode of operation was as follows: 1st day—extract soy flakes with fresh IPA to fill the system and build up an inventory of IPA phase; 2nd day—extract with continuously recycled IPA phase plus make-up IPA.

Phase Separation

The IPA miscella leaving the extractor was passed through a heat exchanger and cooled from 77 C to 30 C, where the solubility of SBO in IPA was considerably diminished and an oil-IPA emulsion was formed. The emulsion was next passed through a coalescer (10) that increased the size of the oil droplets, and the separation into an oil phase an an IPA phase was initiated. The emulsion was passed through a second heat exchanger and cooled to 15 C before entering the phase separator, causing more oil to separate out. The phase separator was a 15-cm diameter glass column 3.1 m high, the feed entering at 0.77 m off the bottom. IPA phase was continuously drawn off the top of the column and recycled to the extractor. Oil phase was drawn off the bottom of the column and collected in a storage tank. Retention time in the phase separator was ca. 90 min.

Evaporator-Stripper

Oil phase was pumped from its storage tank into the evaporator section of an Artisan evaporator-stripper (ES). The feed was heated and the low boiling components were vaporized. The liquid vapor mixture leaving the evaporator entered the vapor disengaging section through a tangential inlet. The oil stream flowed down through the stripper section, which reduced the volatile content even further by supplying additional heat to the oil as it flowed from surface to surface in a thin film. To reduce the solvent content of the oil below the equilibrium value, sparge steam was introduced at the base of the stripper section and flowed upward, countercurrent to the flow of the oil. The stripper was operated at 300 mm Hg.

Screw Pressing

The IPA-extracted flakes retained considerably more solvent than their hexane counterpart. By various squeezing and pressing techniques, the flakes could be forced to release a significant portion of the solvent. However, a continuous method was sought which had sufficient capacity to process the flakes leaving the extractor and at the same time to recycle the expressed liquid to the extractor. A Fuji Bunka continuous screw press met these requirements and performed satisfactorily.

Desolventizing-Toasting

The pilot plant desolventizer-toaster (DT) is a cylindrical, jacketed 316 stainless-steel vessel with bottom-driven variable speed sweep as described by Moulton et al. (11).

Iron-constantan thermocouples were located in the unit to measure the meal temperature, vapor temperature and jacket steam temperature. Best flake depth for the DT was found to be 23–25 cm. This represented about a 32-kg charge, dry basis. A sweep speed of 33 rpm effectively mixed the flakes during sparge, and 60 rpm was best for meal drying. When desolventizing-toasting hexane-extracted flakes (12–15), sparge steam was introduced at 7.03 kg/cm^2 until the meal temperature reached 100 C, then the meal was dried out for 35 min with 2.8 kg/cm^2 steam on the jacket.

The procedure had to be varied somewhat when desolventizing IPA-extracted flakes (16). First, for effective IPA removal and inactivation of trypsin inhibitor (TI) and urease, it was necessary to add back water to make up for a portion of the solvent hold-up removed from the flakes in the screw press. Secondly, the meal was heated with 2.8 kg/cm^2 jacket steam to 85 C, which drove off the bulk of the IPA, and then sparged with low pressure steam (1.4 kg/cm^2) for 5 min. This was followed by a 10-min dryout with jacket steam (2.8 kg/cm^2).

Oil Refining

Alkali refining, bleaching and deodorization were carried out on 1-kg samples in bench-scale apparatus. Deodorization was carried out in the four-unit glass deodorizer described previously (17).

Analytical Methods

Moisture, crude fat, ash, crude fiber, protein, urease, nitrogen solubility index, phosphorus, peroxide value, iron and copper, water, neutral oil and free fatty acid were determined by official AOCS methods (18). Trypsin inhibitor was assayed by the procedure of Hamerstrand et al. (19). Residual alcohol on the DT meals was run by the method of Dupuy et al. (20). Lipoxygenase was determined by the method of Smith (21). Residual oil were analyzed by the method of Black et al. (22).

RESULTS AND DISCUSSION

With the extractor operating in the continuous recycle mode, changes in characteristics of the three main streams surrounding the extractor were monitored. The three streams were defatted meal, IPA phase and oil phase.

Defatted Meal

Changes in defatted meal during the continuous recycle period are shown in Figure 5. The IPA content of the defatted meal ranged from 36 to 40% near the end of the continuous recycle period, the lowest value being obtained with 85.0% IPA. The water content of meals extracted with

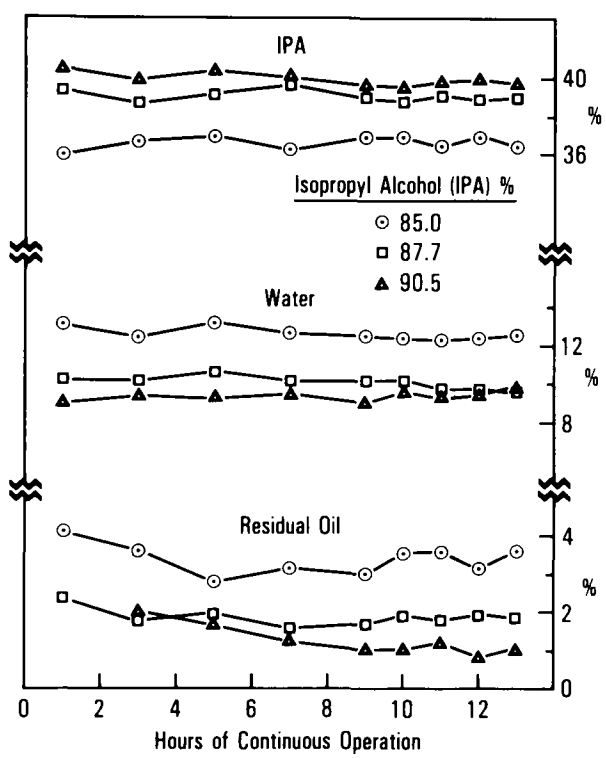

FIG. 5. Characteristics of defatted meal during continuous recycle operation.

87.7% IPA was consistent with the moisture (10%) of the incoming flakes, indicating that there was little, if any, water migration in either direction between flakes and solvent. However, when meals were extracted with 85.0% IPA, there was clear evidence of water migration from solvent to meal; the meal was discharged from the extractor at ca. 12.5% water. When extracting with 90.5% IPA, migration of water from meal to solvent was evident near the beginning of the continuous recycle period; however, this was diminished somewhat near the end. The residual oil values ranged from near 1 to 3.5% as equilibrium was being approached—the lowest value being obtained with 90.5% IPA.

IPA Phase

Changes in the IPA phase during the continuous recycle period are shown in Figure 6. The oil content of the IPA phase ranged from 3 to 4.5% near the end of the continuous recycle period, the lower value being obtained with 85.0% IPA. The nonvolatile matter (oil free) showed some scatter near the beginning of the continuous recycle period but then tended to converge on a value of 2 ± 0.2% for all three IPA concentrations.

Oil Phase

Changes in the oil phase during the continuous cycle of IPA are shown in Figure 7. The volatile matter in the oil phase ranged from 11 to 13% near the end of the continuous recycle period, the lowest value being obtained with 85.0% IPA. The peroxide values were a little higher at the beginning of the continuous recycle period but tended to converge on a value of 3 ± 1 meq/kg. Exceptionally low (0.1%) free fatty acid (FFA) values were obtained when extracting with 85.0% IPA. Although slightly higher values (ca. 0.3%) were obtained with the other two IPA concentrations, these values would still be on the low side when compared to the hexane extraction (0.84%). Phosphorus values started out low for all three alcohols at the beginning of the continuous recycle period, then tended to level out at 200—500 ppm near the end of the period. The lowest value was obtained with 85.0% IPA and the highest with 90.5% IPA.

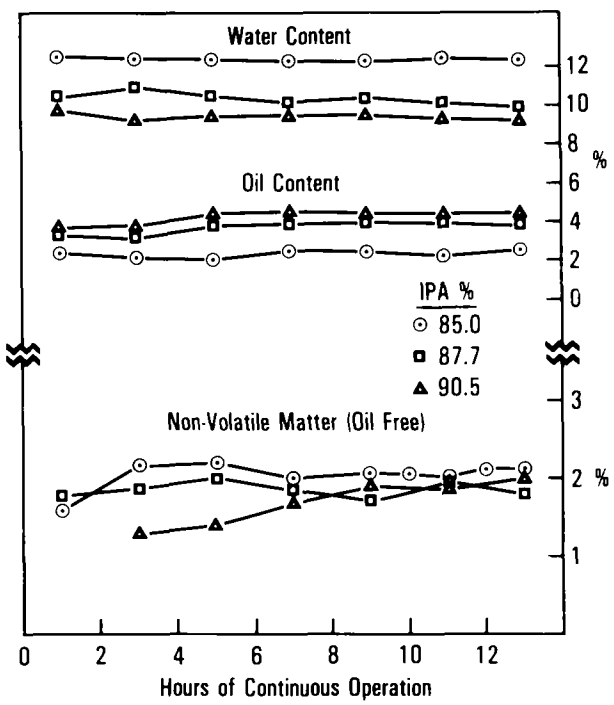

FIG. 6. Characteristics of IPA phase during continuous recycle operation.

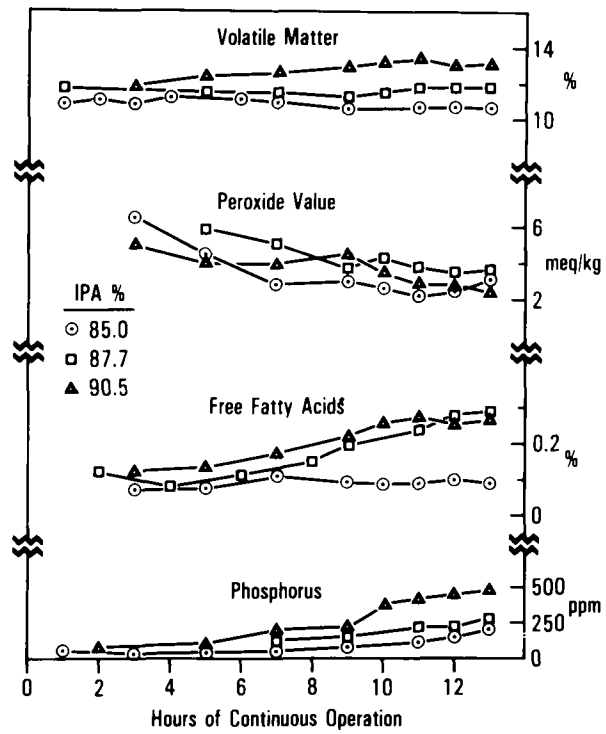

FIG. 7. Characteristics of oil phase during continuous recycle operation.

Degumming

The oil phase being discharged from the phase separator near the end of the continuous cycle had phosphorus contents of 480, 190 and 130 ppm for 90.5%, 87.7% and 85.0% IPA concentrations, respectively. After only a short time in storage, a self-induced degumming was observed. The oil phase was generally held a day or two before stripping the solvent. The desolventized crude oils had phosphorus contents ranging from 5 to 14 ppm, which indicated that the oil phases had almost completely degummed themselves in storage in spite of the wide variation in phosphorus contents at the time they were put in storage. If the oil phase was centrifuged, perhaps the gums could be recovered before storage.

Crude Desolventized Oils

In general, the crude desolventized oils obtained with the IPA extractions were superior to the hexane-extracted oils. Free fatty acids were much lower for the IPA-extracted oils and exceptionally low (0.06%) for the 85.0% IPA extraction (Table II). The hexane-extracted oil had a phosphorus content of 425 ppm, which could be considered fairly typical, and the low phosphorus value with the IPA-extracted oils are again evident. The neutral oil values were higher for the IPA-extracted oils, indicating a higher degree of purity.

Refined Deodorized Oils

The refined deodorized oils from the IPA extraction process compared favorably with their hexane counterpart in

TABLE II

Characteristics of Crude and Refined Oils

	I 85.0 IPA	II 87.7 IPA	III 90.5 IPA	IV Hexane
Crude desolventized oils				
Total volatile matter (%)	0.13	0.35	0.28	0.55
Water (%)	0.07	0.15	0.12	0.19
Free fatty acids (%)	0.06	0.23	0.18	0.84
Peroxide value (meq/kg)	2.0	2.2	2.2	2.0
Phosphorus (ppm)	14	11	5	425
Neutral oil (%)	99.6	99.6	99.0	97.8
Refined deodorized oils				
Water (%)	0.04	0.04	0.04	0.05
Free fatty acids (%)	0.06	0.06	0.06	0.06
Peroxide value (meq/kg)	0.0	0.0	0.0	0.0
Lovibond color (Yellow)	9	10	10	15
Lovibond color (Red)	0.2	0.2	0.3	0.2
Copper (ppm)	0.003	0.003	0.003	0.004
Phosphorus (ppm)	<1	<1	<1	<1
Iron (ppm)	0.04	0.04	0.03	0.04

TABLE III

Characteristics of Defatted Meals Discharging from Extractor Near End of Continuous Recycle Periods

Experimental conditions			Meal characteristics			
Solvent	Solvent/meal ratio	Temperature (C)	TVM[a] (%)	Oil[d] (%)	NSI[b] (%)	TI[c] (% inactivated)
85.0% IPA	2:1	77	48.0	3.4	13	57
87.7% IPA	2:1	77	48.9	1.9	25	44
90.5% IPA	2:1	77	49.3	1.0	32	28
Hexane	2:1	60	38.9	0.9	58	10
85.0% IPA	3:1	77	49.1	2.9	14	51
90.5% IPA	1.5:1	77	50.8	2.1	29	31

[a]Total volatile matter.
[b]Nitrogen solubility index.
[c]Trypsin inhibitor.
[d]As is basis.

color, peroxide value, and phosphorus and free fatty acid content (Table II).

Characteristics of Defatted Meals at Extractor Exit

Comparisons were made of some significantly characteristics of defatted meals obtained in the extractions (2:1 solvent to meal ratio) that tested the three alcohol concentrations against hexane (Table III). The IPA-extracted meals held ca. 25% more volatile matter than did the hexane meal. The residual oil content was nearly equal for 90.5% IPA and hexane. Nitrogen solubility index (NSI) is a measure of the amount of protein denaturation, the higher number showing the least amount of denaturation. The flakes fed to the extractor had a NSI of ca. 58%, so the denaturation with hexane was negligible. However, with the alcohol extraction, a trend was evident of increasing amounts of denaturation as the alcohols become more aqueous, similar to the observation of Smith and coworkers (23). Trypsin inhibitor inactivation followed the same trend, with only 10% inactivation obtained with hexane and increasing inactivation with the more aqueous alcohols.

Increasing the solvent to meal ratio from 2:1 to 3:1 for the 85.0% IPA extraction reduced the oil content of the meal from 3.4% to 2.9%. However, it should be pointed out that screw pressing the meals reduced the oil content by another one-third. Decreasing the solvent to meal ratio from 2:1 to 1.5:1 with the 90.5% IPA extraction increased the oil content of the meal from 1.0 to 2.1%.

Residual Alcohol in DT Meals

The pressed soymeal from the screw press contained ca. 20% volatile matter, of which about half was water. When desolventizing extracted meal with heat alone, the residual alcohols in the DT meals averaged 2000–8000 ppm. Heating to 85–95 C, which drove off the bulk of the alcohol, followed by a 2-min sparge gave residual alcohol values of 900–1400 ppm. Increasing the sparge time to 5 min reulted in residual alcohol values of 300–700 ppm. When a portion of the water removed in the screw press was added back to the meal to increase the moisture from ca. 10 to 13%, heating up to 85–95 C, followed by a 5-min sparge, gave residual alcohol values of 100–300 ppm, which is an acceptable range. However, by adding water to increase moisture level to 18%, the same procedure reduced the residual alcohol in the meal to 50–150 ppm.

DT Meal

Defatted meal from extractions with the three concentra-

TABLE IV

Characteristics of Desolventized Soybean Meal

Component (dry basis)	IPA concentration 85.0	87.7	90.5	Hexane
Residual alcohol (ppm)	70	59	43	–
Trypsin inhibitor (mg/g)	1.7	1.3	2.0	1.3
Urease (pH increase)	0	0.19	0.13	0
Nitrogen solubility index (%)	4.8	4.7	5.2	8.4
Ash (%)	6.6	7.1	7.8	6.6
Oil (%)	2.6	1.1	0.7	1.3[a]
Fiber (%)	3.7	3.8	3.3	3.6
Protein (%)	58.9	59.9	59.3	59.6

[a]Hexane-extracted meal was not screw-pressed.

tions of IPA were processed via the revised procedure (i.e., water added back to 18%, Δ 85–95 C, sparge 5 min, dryout 10 min). These are compared to conventionally processed hexane-extracted meal (Table IV). Reidual alcohol was 70 ppm or less for the three alcohol-extracted meals. Trypsin inhibitor values were 2 mg/g of meal or less for all four meals, which translates to more than 90% TI inactivation.

IPA Balance

An IPA balance around the extractor for a 2:1 solvent to meal ratio split 30% with defatted meal and 70% with miscella (Fig. 8). Of the 70% with the miscella entering the phase separator, 68% was recycled to the extractor and only 2% was vaporized in the ES. Of the 30% leaving the extractor with the defatted meal and entering the expeller, 25% was returned to the extractor as expressed liquid and only 5% had to be vaporized in the DT.

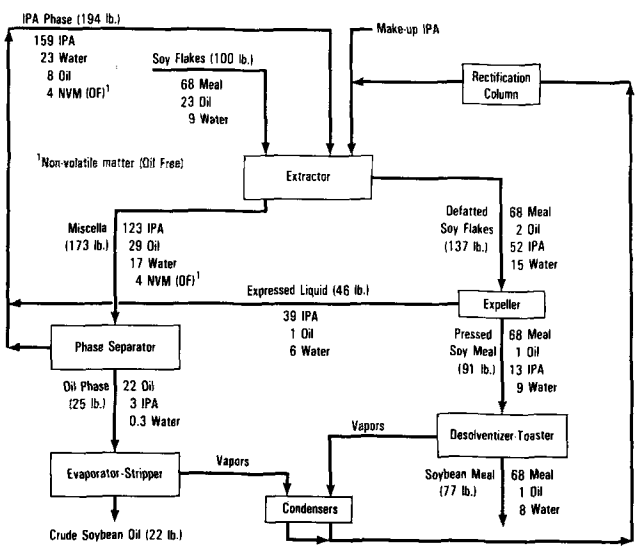

FIG. 8. Continuous countercurrent extraction of soy flakes with 87.7% isopropyl alcohol at a 2:1 solvent to meal ratio with continuous recycle of IPA phase.

Comparison of the Three Concentrations of IPA

In the oil phase near the end of the continuous recycle periods, the free fatty acids, phosphorus and total volatile matter were lowest when extracting with 85.0% IPA. The lowest peroxide value was obtained with 90.5% IPA.

In the defatted meal being discharged from the extractor near the end of the continuous recycle periods, the least amount of IPA hold-up and the greatest amount of TI inactivation were obtained with 85.0% IPA. The lowest residual oil was obtained with 90.5% IPA and the least amount of water migration with 87.7% IPA.

If each of the factors were weighted equally (which they are probably not), the 85.0% IPA would appear to be more desirable than the other two alcohols. However, the 85.0% IPA will require a higher solvent to meal ratio than the other two, and there is the problem of moisture migration that probably could be minimized by feeding higher moisture flakes to the extractor. Realistically, however, the vapors coming off the evaporator-stripper and desolventizer-toaster would probably require rectification and it would be rather difficult to run a process where the extracting solvent was maintained on either side of the azeotrope. Finally, then, the IPA process has been examined in detail and the following conclusions have been reached: the IPA extraction process produces a high quality crude oil which requires less refining. IPA is safer and less toxic. Although IPA has a higher heat of vaporization, only a small fraction of the total IPA in the system requires vaporization and an energy saving should result. Because of the significant amount of TI inactivation achieved with the hot aqueous alcohols, the DT cycle is considerably shorter producing a high quality meal. The process can be retrofitted to existing plants.

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An Emulsion Method for the Sensory Evaluation of Edible Oils[1]

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ABSTRACT

The flavor intensity of soybean oils was evaluated in emulsions stabilized with gum acacia. A 10-point scale was used with a blank to establish the bland end of the scale and a standard diacetyl solution to establish a point near midscale. Tasting oils in emulsion gave significantly different scores than tasting oil directly. Evaluation in emulsion decreased panel error for poor quality oils but not for very bland oils. At least six samples could be tasted in emulsion without casusing panel fatigue or reducing accuracy. The concentration of oil in the emulsion could be adjusted to increase sensitivity to weak flavors or improve the evaluation of intensely flavored oils. Soybean oils containing various amounts of linolenic acid were evaluated by the emulsion method, and those with lesser amounts of linolenic acid were shown to be more stable. A gas chromatographic total volatile method was shown to correlate fairly well with sensory evaluation of oils tasted in emulsions under conditions where both flavors scores and total volatiles changed significantly with time.

INTRODUCTION

The traditional method for the evaluation of oil flavor grew out of research at the Northern Regional Research Laboratory of the United States Department of Agriculture (1-4), A 10-point scale was developed on which a completely bland oil scored 10. To avoid taste carry-over and fatigue, it was recommended that only two samples at a time be tasted and that the samples not be swallowed. Samples were to be smelled first and tasted in order of increasing odor.

The search for objective tests that might correlate with sensory evaluation of fats and oils has been pursued with considerably more diligence than the improvement of sensory evaluation, but satisfactory objective tests have been elusive (5-8). Recently, gas chromatographic measurement of volatiles from fats and oils has been explored, and good correlations between total volatiles and flavor evaluations have been claimed (9-22).

This paper presents a method for the evaluation of the flavor of fats and oils in emulsions stabilized by gum acacia. Correlations between the flavors of oils tasted in this way and a total volatile test are given.

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METHODS

Water for the emulsions was obtained by adding 100 mg $CaCO_3$, 35 mg $MgCO_3$, 10 mg NaCl, and 10 mg Na_2SO_4 per liter of deionized distilled water and gently carbonating with stirring to dissolve the carbonates as bicarbonates. The diacetyl standard emulsion was made by blending 2 g gum acacia powder, 300 mL of water, and 3 mL of diacetyl solution (10 μL diacetyl in 25 mL mineral oil prepared fresh daily) for 1 min. The blank was made by blending 2 g of gum acacia in 400 mL of water for 20 sec. Sample emulsions were made by blending 2 g of gum acacia, 6 mL of sample oil, and 400 mL of water for 1 min. All blending was at the highest speed of the blender (Osterizer pulsematic 10, Milwaukee, WI) in a glass blender jar.

Oil samples were presented in plastic 30-mL cups, emulsions and blank in 90-mL paper cups. All samples were coded with 3-digit random numbers. Red lighting was used to mask color differences in samples. Judging was done in individual booths which provided a quiet, comfortable atmosphere.

A 10-member panel was trained by presenting them with soybean oils and emulsions that had a range of qualities and flavor intensities. After initially scoring the samples, the panel discussed and retasted them to allow the judges to train themselves to rank samples according to the expected degree of oxidation. Efforts were made to keep the panel motivated and interested by providing treats, discussing experimental results, and soliciting suggestions.

Panel members were given the following intructions: "You will evaluate oil-in-water emulsions for flavor intensity using a 1-10 scale. The emulsion marked standard is the same intensity every day and should represent a fixed point on the scale for you. The blank has a score of 10. Score each sample using the flavor intensity of the standard as a reference and the information on the scale describing its end points. Smell the samples before tasting, taste the most oxidized samples last and do not swallow anything". Response was made by marking numbers on a 10-number scale. The number 10 was indicated as bland and tasteless and the number 1 as extremely intense. Rinsing the mouth