

The Nondistillation Alcohol Extraction Process for Soybean Oil

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DEVELOPMENT of an ethanol extraction process for soybean oil which completely eliminates the distillation steps formerly required for the recovery of residual oil and byproducts has been achieved both in a continuous laboratory apparatus (3) and on a pilot-plant scale.² The elimination of these distillation steps and those necessary for rectification and dehydration removes the economic disadvantage occasioned by the high latent heat of ethanol (204 cal./gm.) as compared to hexane (80 cal./gm.). Theoretical calculations have shown an economic advantage in the use of ethanol in solvent extraction of soybean oil through lower energy requirements than when hexane is used. Consideration of improved products or additional byproducts which may be obtained is no longer fundamental.

It was the requirement of improved color in isolated soybean protein that led to the study of the effect of oil solvents (1, 2). This study demonstrated that the use of ethanol gave an improved color to the extracted flakes and isolated protein and that ethanol extraction also improved the flavor of the proteinaceous materials used for human consumption. Other advantages of the use of ethanol include the low, free fatty acid content of the oil (Table 2 of Ref. 1); the possibility of additional byproducts such as sterols, saponins, and sugars; greater industrial safety; the low toxicity of the solvent; and the possibility of preparing the solvent from agricultural residues.

The Alcohol Extraction Process

Alcoholic extraction of vegetable oils has always been intriguing because of the high temperature coefficient of solubility (6). On the basis of this large change in solubility as influenced by temperature, it is possible to extract the oil from the oil-bearing material with absolute alcohol at the boiling point. This is where complete miscibility of oil and solvent exists. After removing the miscella from the oil-bearing material it is cooled to about room temperature and practically all of the oil is recovered by phase separation. When using 95% ethyl alcohol as the oil solvent, it is advantageous to carry out the extraction under slight pressure in order to raise the temperature of the alcohol above 90°C. where complete miscibility is attained between the 95% alcohol and the oil. Obviously, the separation of the miscella from the oil-bearing material must also be carried out under pressure. Both 95% alcohol and absolute alcohol remove water from oil-bearing meal at the moisture content usually employed in solvent extraction and quickly become diluted to such an extent as to be of no further use as an oil solvent unless rectification or rectification and dehydration be used to remove the water.

To minimize this dilution of the alcohol the Japanese recommended (6) that the moisture content of the oil-bearing material be reduced. They did not recognize that an equilibrium point exists between the moisture content of the alcohol and that of the

beans and, therefore, they permitted the moisture content in their experiments to vary from 3% to 6%. It has been found (7) that when the moisture content of the oil-bearing soybean material is reduced below 3% no detectable solution of water by the alcohol takes place. In fact, under ordinary conditions of drying, the moisture content of the soybeans is often reduced well below 3% and an actual improvement takes place in the efficiency of alcohol as an oil solvent.

The Japanese, while referring in the literature cited as well as to English and American patents (4, 5, 8) regarding circulation and recirculation processes, never actually used a continuous recirculation process. Instead, their descriptions (5) refer to returning the alcohol to the same batch of material until the oil content is sufficiently reduced and then, after oil separation, removing the alcoholic solution to a still for recovery of residual oil and byproducts.

The Nondistillation Process

Elimination of the step requiring distillation of the alcohol to recover residual oil and byproducts was made possible by discovery at the Northern Regional Research Laboratory that, on cooling to 25°C. or lower, nonoil solubles separate from the alcohol to an extent dependent on the temperature of cooling. This effect has *not* been noted previously in the literature. (A patent application involving this step is on file in the Patent Office.) The Japanese specifically deny the separation of other solubles by stating (9) that "oil and oil alone" separates when the miscella is cooled to the extent required to remove the oil by their process. We have found that when the alcoholic miscella issuing from the extractor is cooled to 0°C. and the separated oil and nonoil solubles are allowed to settle, the supernatant alcoholic solvent will come to equilibrium after repeated reuse at a total-solubles content of 4.3% (after cooling and before addition of make-up solvent); and when the cooling is carried no lower than 20°C. the resulting total-solubles content of the supernatant alcoholic solution is no more than 5.5%. This value for the total-solubles content has been established by the reuse of the alcoholic solvent more than 85 times without distillation. The repetition was carried out in the continuous countercurrent extraction of soybeans in the laboratory extractor described in a recent article (3).

Use of the cooling step for the elimination of distillation obviously substitutes a step that involves only sensible heat for one that requires the latent heat of vaporization. Furthermore, most of the sensible heat transfer may be effected in heat exchangers since the cooled alcoholic solvent must be reheated for reuse in the extractor. An energy balance shows this alcohol process requires, theoretically, about seven-tenths as much energy as the hexane process. Specifically, the nondistillation process is calculated to require 776.2 pounds² of steam per ton of flaked

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² Beckel, A. C., Belter, P. A., and Deobald, H. J., Alcoholic Extraction of Soybeans; A Report of a Collaborative Study of the Effect of Pressure Conditions on the Nondistillation Vegetable Oil Extraction Process. A cooperative project between Allied Mills, Incorporated and the Northern Regional Research Laboratory.

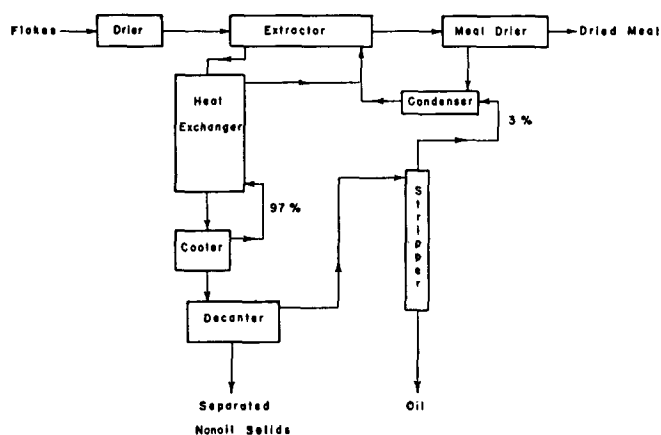


FIG. 1.

soybeans while the hexane process is variously reported to require from 1,100 to 1,600 pounds of steam per ton of flaked soybeans. A reduction of the size of the heat exchange unit (Figure 1) from 100% heat recovery to 85% heat recovery would reduce investment costs and make the thereby increased energy cost of the alcohol process approximately equal to that of the hexane process.

The complete nondistillation oil extraction process is schematically diagrammed in Figure 1. The oil-bearing meal is fed into the drier, passes through the extractor and meal desolventizer to be discharged as dry, extracted meal. The hot alcoholic solution from the extractor passes through the heat exchanger into the cooler in which separation of the alcoholic solvent and the oil and solids takes place. About 97% of the alcoholic solvent entering the cooler is freed of all but 4.5% to 5.5% total solubles, depending on the temperature of the cooler, and is returned through the heat exchanger directly to the extractor. The oil and solids from the cooler are discharged into the separation unit consisting of a settler or decanter with a solids discharge and with provision for the oil to pass to the stripper for removal of the alcohol remaining in the oil. It is returned to the recycled alcohol. This alcohol is about 3% of the solvent in the cooler and about 7% on the weight of the oil. We have found that nearly all of this alcohol is removed when the oil is heated at 100°C. under 20 inches of vacuum. The remaining alcohol, which is about 0.02% of the oil, is removed in the first water-wash step of the refining process, and the monetary value

of the alcohol is much less than the labor charge on the energy required for recovery.

The results of a typical run and materials balance, when the laboratory equipment mentioned earlier (3) was used, are given in Table 1. The operation was carried out on a batch of dehulled soybean flakes which analyzed 19.5% oil, 6.51% nitrogen, and 2.52% moisture. The ethanol solvent has been through the extraction cycle 75 times without rectification and had a cloud point of 20.5°C. Throughout the run temperature conditions were adjusted to give the solvent the same cloud point.

TABLE 1.

Materials Balance Data for a Typical Operation With 95% Ethanol.*
Temperature of Extraction 78°C.

Materials	Weight	Per cent
	grams	
Total solubles in ethanol at start of operation.....	5.5
Moisture content of flakes entering extractor.....	2.52
Flakes, moisture present, entering extractor.....	7724
Flakes, moisture free, entering extractor.....	7530
Extracted flakes, 8.67% moisture.....	6596
Extracted flakes, moisture free.....	6017
Oil content of extracted flakes.....	1.02
Recovered oil solvent free.....	1411
Total solubles in solvent at end of run.....	5.50
Meal recovery, per cent of original flakes.....	79.9
Oil recovery, per cent of original flakes.....	18.7
Foos recovery, per cent of original flakes (phosphatides, sugars, and other alcohol soluble materials).....	1.4

* The utility of the process using 95% ethanol as the solvent under 8 pounds' pressure has been demonstrated on a pilot-plant scale.

Summary

A new vegetable oil extraction process has been developed, with alcohol as the oil solvent. The process requires *no distillation* to recover the oil or the solvent characteristics of the alcohol. This has been demonstrated by reuse of the solvent more than 85 times. The theoretical energy requirement of the process is about three-fourths that of the hexane process.

REFERENCES

1. Beckel, A. C., Belter, P. A., and Smith, A. K., Oil and Soap (Current issue).
2. Beckel, A. C., and Smith, A. K., Food Indus. 16, 616 and 664 (Aug., 1944).
3. Beckel, A. C., Belter, P. A., and Smith, A. K., Ind. Eng. Chem., Anal. Ed. 18, 56 (1946).
4. British Patent 336,273 (1930).
5. British Patent 336,274 (1930).
6. Okatome, Sajiro, Contemporary Manchuria 1 (3), 83-101 (1936).
7. Singer, P. A., and Deobald, H. A., Allied Mills, Inc., Peoria, Ill. Private communication.
8. U. S. Patent 1,892,366 (1930).
9. U. S. Patent 1,892,360 (1930).