# Design of Oilseed Extractors. II. Multicomponent Extraction

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**Extraction of carbohydrates from white flakes with aqueous ethanol is used to illustrate some generalizations about extraction of any multicomponent solute. Since commercial design must be based on a laboratory simulation, a preferred simulation is presented.**

Oil in seeds may be the only solute in a product of nature which, from the standpoint of solvent extraction, is a single component very soluble in the solvent. Other possible solutes in seeds, such as carbohydrates or phosphatides (1), are effectively multicomponent, because their components have considerably different solubilities in a commercial solvent such as aqueous ethanol.

Practically all of the technology used in the continuous tonnage extraction of particulates with volatile, flammable solvents originated in the oilseed industry. Even now, most such plants extract oilseeds. The only other applications that come to mind are extraction of pyrethrum, extraction of montan wax from lignite and washing low molecular weight polymer from polyethylene. Applications of solvent extraction surely will increase, and engineers will look to the oilseed experience for guidance.

A case in point is extraction of bitumen from tar sand. When petroleum shortages recur, mined tar sand will be one of the first sources of bitumen to be exploited. Bitumen is now recovered from tar sand by floating with hot water; but extraction with hydrocarbons is better. Bitumen has many components; the use of benzene and toluene, the only solvents in which all of the components are soluble, is no longer permitted. Nevertheless, naphthenic hydrocarbons in which asphaltenes are hardly soluble extract practically all of the asphaltenes when the extraction is countercurrent. The reason will be apparent from the experience with carbohydrates extraction from soybeans described in this article.

## SPC BY CARBOHYDRATES EXTRACTION

To make soy protein concentrate (SPC), well-dehulled, full-fat flakes (2) or white (defatted) flakes are extracted with aqueous ethanol or isopropanol to dissolve enough carbohydrate so that the dry residue contains at least 70% protein. This well exemplifies solvent extraction of a multicomponent solute, because the carbohydrates in soybeans vary considerably in their solubility.

A typical dry-basis material balance for the production of SPC from soybeans by extraction with aqueous ethanol is given in Table 1.

The loss of 1.2 parts of protein from white flakes reflects that any aqueous alcohol solution which extracts enough carbohydrate also will extract some protein. The alcohol concentration must be as high as possible to minimize protein extraction. In practice, the alcohol concentration is 60–70 wt %; at higher concentrations, not enough carbohydrate goes into solution. At least 55% (16/29) of the carbohydrates and phosphatides in soybeans must be extracted to make SPC; at least 54.1% (15.7/29) extracted from white flakes.

To measure carbohydrates solubility a typical experiment is that reported by Circle and Whitney (3). In a 250 ml bottle, 7.1 g of white flakes were shaken for one hr with 100 ml (90 g) of 60 wt% ethanol. The resulting filtered solution contained 0.1% protein and 1.6% carbohydrates and phosphatides. Assuming, since they did not give the analysis, that the white flakes contained 35.7% carbohydrates and phosphatides (consistent with the material balance) and 10% water, then the sample contained 2.53 g of carbohydrates and phosphatides. The filtered solution contained 1.46 g (90 × 1.6/98.3) of carbohydrates and phosphatides; 57.9% of those in the sample. This is hardly a promising basis for a commercial countercurrent process in which at least 54.1% must be extracted into a miscella whose concentration would practically have to be considerably higher than 1.6%. Yet, SPC can be made by extraction with 60% ethanol. The same reference reported countercurrent extraction of white flakes that produced a miscella whose carbohydrate concentration was 4.63%.

Clearly, more carbohydrate is extracted in a countercurrent process than seems possible based on the apparent solubility in aqueous ethanol. The carbohydrate miscella formed in countercurrent contacting in turn dissolves carbohydrates not soluble in the "solvent." The dissolving power of a solution of the carbohydrates soluble in aqueous ethanol may increase with their concentration.

The following ideas about the extraction of a multicomponent solute whose components are chemically similar can be surmised:

- There is little purpose in extensive experiments to determine apparent solubility, which is a function of the components in solution and their concentration, and of the components still undissolved.
- There is no purpose in laboratory batch extractions with solvent to determine rate of extraction.
- The best laboratory experiment is one which simulates as closely as possible a proposed commercial countercurrent extraction.

TABLE 1

**Material Balance (Dry Basis) for the Extraction of Oil from Soybeans to Make White Flakes, and the Extraction of Carbohydrates from White Flakes to Make SPC[a]**

| | Soybeans (kg) | White flakes (kg) | SPC (kg) |
|---|---|---|---|
| Oil | 21 | 0.6 | 0.6 |
| Ash and fiber | 10 | 3.1 | 3.1 |
| Protein | 40 | 40.0 | 38.8 (70%) |
| Carbohydrates and phosphatides | 29 | 28.7 | 13.0 |
| | 100 | 72.4 | 55.5 |

[a]Basis: 100 kg dry soybeans.

- Since miscella is a better solvent for some components than the "solvent" is, it may be advantageous to make the initial extraction cocurrent instead of countercurrent.
- Since solvent power tends to increase with miscella concentration, there is, for any given amount of solute to be extracted, an optimum miscella concentration and corresponding optimum solvent ratio.
- Since the "solvent" is not necessarily the best solvent for the solute, it may likely be better not to distill the miscella, but instead to recycle to the extractor mother liquor from the controlled crystallization from the miscella of net dissolved solute.
- Since the miscella may be saturated or nearly saturated with many solute components, it can be expected that extraction will be slow. Extraction of glycerides from soybeans takes 10 min; extraction of carbohydrates to make SPC requires one hr.

## LABORATORY SIMULATION OF COUNTERCURRENT EXTRACTION

If the particulate solids form a percolating bed, then it is simple in the laboratory to simulate the performance of a percolation extractor. A batch is supported as a bed by a screen at the bottom of a vertical, jacketed glass tube open at the top. A succession of miscellas of decreasing concentrations, the amount of each corresponding to the amount of miscella collected in a stage compartment and pumped to a manifold by a stage pump, is poured through the flooded bed. To establish correct concentrations of the miscellas, a first batch is extracted with fresh solvent only, and the miscellas draining from the bed are collected in successive measured cuts. The first cut, equivalent to full miscella, is discarded; the other cuts are percolated in succession through a second batch, followed by an amount of fresh solvent whose ratio to flakes in the batch is the same as the ratio of solvent to solids fed in the continuous process being simulated. After several batches of flakes are so treated, the concentrations of the miscella cuts reach a steady state characteristic of the operation of a percolation extractor.

In such an experiment, 1300-g batches of white (defatted) flakes containing 50% protein and 10% water were extracted at a solvent ratio of 4:1 with 58.7 wt % ethanol at a temperature of 130 F. Miscellas were collected in flasks numbered 1 (full miscella) through 5 (weakest miscella). The flakes were first slurried in the contents of flask 2 to swell them; then the slurry was introduced into the tube to form a bed. The first liquid to drain from the forming bed was recycled to the top of the bed, just as in a commercial extractor. After solvent addition was complete, the flakes in the bed were completely drained before they were dumped, then pressed between pads in a hydraulic press. Flakes retention time was 60 min. Expressed liquid was put into flask 4. The amounts and the steady state concentrations of the miscellas are listed in Table 2.

TABLE 2

**Amounts in Each Flask and Steady State Concentrations of the Miscellas**

| Flask # | Miscella in flask (g) | Miscella conc. (% diss. solids) |
|---|---|---|
| 1 (Final miscella) | 4400 | 7.14 |
| 2 | 7800 | 4.54 |
| 3 | 6400 | 3.30 |
| 4 | 6400 | 1.54 |
| 5 | 6400 | 0.62 |
| Solvent | 5400 | 0 |

The following explains, in Table 2, the amount in each receiver and the amount of solvent:

It was found that about 400 g of solvent were lost in each run by evaporation. Consequently, the solvent used per batch was increased to 5400 g from the 5200 g, corresponding to a 4:1 solvent ratio; the final miscella withdrawn was decreased to 4400 g from the calculated 4600 g. 7800 g were stored in receiver 2 so there would be enough to make final miscella and provide additionally for a flooded bed. 6400 g were stored in the other receivers so the interstage flow was more than the solvent by roughly the difference between flooded and pressed hold-up in the flakes. All these criteria are consistent with the operation of a percolation extractor.

Before pressing, the drained flakes contained 67% volatiles, based on wet flakes, and the protein content on a dry basis was 70.5%. After pressing, the pressed flakes contained 48% volatiles based on wet flakes, and the protein content on a dry basis was 71.0%.

Note that the final miscella concentration of 6.85% (corrected from the 7.14% listed in Table 2 by multiplying by 4400/4600) is much higher than the 4.63% reported by Circle (2). Approximately 7% is the optimum miscella concentration, much higher than could have been anticipated from a batch extraction.

A commercial extractor can be designed with confidence from such an experiment, since all steps faithfully simulate plant practice. The technique clearly is not limited to oilseeds.

## REFERENCES

1. Karnofsky, G., and R.J. Hansotte, U.S. Patent 4,359,417, Nov. 16, 1982.
2. Karnofsky, G., U.S. Patent 4,144,229, March 13, 1979, U.S. Patent 4,219,470, August 26, 1980.
3. Circle, S.T., and R.W. Whitney, U.S. Patent 3,365,440, January 23, 1968.

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