
REFERENCES


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Acetone as a Selective Solvent for Vegetable Oils

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The modification of drying and semi-drying oils by extraction with a selective solvent has received considerable attention in recent years. A wide range of solvents has been investigated (9), but only furfural (2, 4) and liquid propane (1, 6) have warranted development to the point of commercial application. A third solvent which may find application in this field is acetone containing small proportions of water. Such a system was suggested by Jenkins (3) in a patent dealing with the use of "wet" furfural. It is a solvent which is readily available, is easily stripped from the oil, and requires no special equipment for handling. It has the added advantage that by varying the water content it may be "tailored" to the particular oil and separation involved.

Solubility and Selectivity

From the viewpoint of extraction efficiency the important properties of a solvent are a) solubility of the oil in it and b) selectivity, that is, the difference in iodine value between extract and raffinate on batch extraction. The effect of water content of acetone on these two properties is shown in Figures 1 and 2 for the batch extraction of a sample of soybean oil, I. V. 129.3, and a sample of linseed oil, I. V. 188.0. Solubility has been shown as the ratio of solvent to oil as this form is to be used later. Since a low solvent to oil ratio and high selectivity are desired, the variations of these properties with water content are conflicting. With reference to existing processes it appears however that a useful compromise can be obtained in the range of 3 to 7 parts water per 100 parts acetone by volume.

Equilibrium Diagrams

The evaluation of the solvent for use in countercurrent column operation rather than in batch extraction is the important consideration. As in distillation the prediction and evaluation of extraction column operation is based on equilibrium data between phases over the concentration range involved. The particular type of equilibrium diagram used here is one adapted to extraction calculations by Maloney and Schubert (5) in which the ratio of solvent to solute is plotted against the concentration of components in the solute. As pointed out by Pratt (7) and illustrated by Rushton (8), in dealing with multi-component systems it is necessary to replace concentration by the value of some additive property of the system. The obvious choice of property in this system is iodine value. The required data are the solvent to oil ratios and iodine values of the oils in extract and raffinate phases over the range of iodine values likely to be encountered in fractionation. Ruthruff and Wilcock (9) in their
work on soybean oil and furfural covered a limited range of iodine values by varying the fraction of oil extracted. In the present work a wider range was covered by a stepwise batch extraction process in which the extract and raffinate oils from an initial extraction process were used as feed for second extractions and so on. It was found that nonglyceride materials present in the original oil rapidly built up in successive extract fractions and markedly affected solubilities and selectivities. Such material was therefore removed as completely as possible from the original oils by passing the oils in a hydrocarbon solvent through an alumina column. The linear relation between refractive index and iodine value for glycerides was used as a measure of the effectiveness of this treatment. This relation was checked after each successive extraction, and where necessary the alumina treatment was repeated. Samples obtained in this way were equilibrated with the solvent at a temperature of 25°C.

Figure 3 shows the equilibrium diagram obtained for soybean oil, using 6 parts water per 100 parts acetone as solvent. Over the range covered the iodine values of the extract and raffinate phases varied linearly with solvent to oil ratio, and the selectivity remained constant. The conjugate line, located as shown by the broken lines from the ends of the tie lines, was plotted to facilitate interpolation between tie lines. A similar diagram, Figure 4, was obtained for soybean oil using 3.5 parts water per 100 parts acetone. Again the iodine values varied linearly with solvent to oil ratio, but the selectivity decreased at the higher iodine values. Since the extract lines were straight and nearly parallel for these two cases, the whole family of equilibrium curves over the useful range of water contents can be filled in from solubility and selectivity data. A similar diagram, Figure 5, was also obtained for linseed oil, using 5 parts water per 100 parts acetone as the solvent.

Pilot Plant Operations

To evaluate the reliability of these diagrams for prediction of column operation and to estimate column efficiencies a number of runs were made in a packed column, 2 in. in diameter and 20 ft. high, and H.E.T.S. values were computed. Figure 6 is a schematic diagram of the column used. Oil was fed 11 ft. from the top of the column, and solvent was sprayed in at the bottom. The interface was maintained at the top of the column so that the oil-rich phase formed the continuous phase throughout the extraction zone. Extract overflowing from the top of the column passed to an evaporator where the solvent was stripped off and a portion of the extract oil was returned to the top of the column as reflux.