FRACTIONATION OF AQUEOUS FRUCTOSE–GLUCOSE MIXTURES BY ADSORPTION AND SUPERCRITICAL DESORPTION

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Fractionation of aqueous fructose glucose mixtures with the aid of supercritical fluids was investigated. The monosaccharides were first adsorbed onto activated carbon from an aqueous solution and then desorbed into a stream of supercritical carbon dioxide containing a small amount of ethanol. Use of an entrainer such as ethanol is essential for solubilizing sugars in supercritical carbon dioxide as well as enhancing the relative selectivity. Experiments were conducted at pressures 82.7 and 151.6 bar and temperatures 35, 50, and 65 degrees centigrade.

The ultimate capacity of activated carbon for both fructose and glucose is low. However, the two components are not adsorbed to the same extent. The relative selectivity of fructose over glucose ranges from 2 to 75 when adsorbing from aqueous solution, and from 2 to 300 when desorbing into supercritical carbon dioxide.

INTRODUCTION AND BACKGROUND

The rising cost of sucrose was responsible for earlier work in search of alternative sweeteners. One possible replacement is glucose that could be obtained from starch. Another is fructose which happens to be sweeter than either sucrose or glucose. The food and beverage industry prefers sweeteners that contain a higher percentage of fructose due to the resulting dietetic and low calorie products. Several attempts have been made to concentrate fructose from mixtures of fructose and glucose. Complexing of either component preferentially in sodium or calcium salts and solvent extraction were studied in the early seventies. Barker and Thawait (1983) used columns packed with ion exchange resin in the calcium charged form. When a mixture of fructose and glucose is passed through such a column, a complex of calcium ions and fructose molecules forms. Thus, the fructose is retarded and separation occurs. Neuzil and Priegnitz (1984) conducted similar experiments using an X zeolite charged with potassium as the adsorbent. In this case, glucose is preferentially adsorbed and a fructose rich product is obtained directly. Barker (1984) achieved the same result with an anion resin in the bisulphite form. Kim (1985) attempted the desired separation using membranes of cellulose acetate and polyvinyl alcohol. When salts such as NaCl, Na_2CO_3 and $NaHCO_3$ were added to the feed, complexes with glucose formed and a fructose-enriched product was obtained. D'Souza (1986) applied the more recently developed separation process: supercritical-fluid extraction. When equimolar mixtures of fructose and glucose were introduced in the liquid phase, good separation and selectivity were achieved with supercritical carbon dioxide when ethanol was used as the entrainer. Commercial processes utilize cation exchange chromatography to separate fructose and glucose.

More recently, SuperCritical Extraction (SCE) started receiving considerable attention as a means of accomplishing separations that are difficult, particularly in high value added product applications. SCE uses solvents that are at temperatures and pressures above their critical points. At these conditions, solubilities in supercritical fluids are comparable to those in liquid solvents; mass transfer resistance is lower than would be obtained with a liquid solvent, diffusion coefficients are intermediate between those of gases and liquids, density is roughly half that of a liquid, and viscosity is slightly greater than that of a gas (Randall, 1982). Superior transport and solubility characteristics, coupled with their sensitivity to changes in temperature and pressure, provide SCE with the following advantages: less energy is consumed when compared with some conventional methods; the extracted material can be separated from the solvent very efficiently and in ultra-pure form; high selectivity is obtainable with a prudent choice of operating conditions; and rather inexpensive, environmentally safe and non-inflammable substances such as carbon dioxide become effective solvents.

The addition of a small amount of a cosolvent to the supercritical solvent can result in considerable improvement of both selectivity and solubility. The presence of the cosolvent, also known as an entrainer, also allows the separation to be conducted at a different operating temperature and/or pressure. However, the additional component increases the complexity of the solvent recovery process. Peter and Brunner (1978) studied the solubility of glycerides in supercritical CO_2 and discovered that separation was enhanced by the addition of a small amount of entrainer, acetone. The issues involved in entrainer selection have been discussed by Sunol *et al.* (1984).

Currently, many investigators are looking into the use of supercritical fluids as regenerants for spent adsorption beds. The approach provides an alternative to the currently used capital and energy intensive thermal regeneration processes or liquid solvent regeneration schemes that suffer from high solvent losses and environmental concerns stemming from the use of special solvents. An earlier process developed by Arthur D. Little, Inc. (deFilippi *et al.*, 1980) uses carbon dioxide as a desorbent for regenerating activated carbon. Other adsorbents were studied by Picht *et al.* (1982). Thermody-

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