585d CO2-Soluble Gras Solvents for Extracting Nutraceuticals by a Gas Anti-Solvent (Gas) Process

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Nutraceuticals are polar organic molecules with associated health benefits that can be extracted from plants. This paper describes a novel extraction process for obtaining high value nutraceuticals from plants. It utilizes CO2-soluble GRAS (generally recognized as safe) ester solvents to extract the nutraceuticals from plant tissues. CO2 is then used to recover the product via a gas anti-solvent (GAS) process(1-3).

The GAS process requires the nutraceutical of interest to be appreciably soluble in the extraction solvent and insoluble in the CO2-expanded solvent. Alkyl aryl esters are promising solvents for this purpose since their substituent groups can be chosen to achieve the desired polarity and hydrophobicity to target a specific product. In addition, many of these esters are completely miscible with dense CO2, making them suitable for GAS precipitation and for the extraction of residual solvent. GAS precipitation is followed by an increase in pressure to dissolve and recycle the liquid solvent. Since the liquid extraction step, the GAS process, and the dissolution of the liquid solvent can all operate effectively at room temperature, this process can reduce nutraceutical degradation and thus increase yields. This process also offers possibility to fractionate a complex mixture of components into its various constituents by applying CO2 over a broad pressure range(4,5). In this way, materials with lower solubility will precipitate at lower pressures. As model systems, the recovery of sclareol from clary sage, anthocyanins from purple sweet potato, and lutein and zeaxanthin from marigold flowers is described.

Screening of GRAS solvents is done by measuring the solubility of each solute of interest in various ester solvents. Solubility measurements are done using UV visible spectroscopy for anthocyanins and marigold derivatives. With sclareol and solanesol, gas chromatography is used to measure solubility.

The apparatus for performing the GAS process consists of a high pressure cell, CO2 syringe pump, line filter (0.5 ml pore diameter), pressure transducer, and temperature controller. The solutions containing the target molecule are introduced into the cell. CO2 is then introduced into the cell until the desired pressure is reached. While maintaining the temperature and pressure of the system constant, the mixture is drained through the line filter to a solvent trap. The amount of product in the solvent trap is measured using UV visible spectroscopy or gas chromatography allowing a calculation of the yield of the GAS process as a function of initial solute concentration, gas pressure and temperature. The results from solubility measurements as well as from GAS precipitation process as a function of system conditions, such as initial solute concentration, temperature and pressure, will be presented and discussed.

It is important to explore the role of temperature, pressure, and composition in determining the efficacy of the process. Fundamental thermodynamic modeling is required to understand the solid-vapor-liquid equilibrium phase behavior of the ternary system consisting of antisolvent + solvent + nutraceutical solute, most importantly the solubility phenomena. Due to the scarcity of the parameter data for the nutraceutical systems of interest, a group contribution approach through a UNIFAC activity coefficient model coupled with the Peng-Robinson equation-of-state is being used in order to capture the phase behavior of these complex systems(6,7). Thus, the phase equilibria of the solute in the gas-expanded ester solvent is predicted as a function of CO2 pressure and temperature. The model is compared to existing literature phase equilibrium data and applied to the solubility of nutraceuticals in CO2-expanded ester solvents. The relationship between the solubility and system conditions such as pressure and temperature and other factors that govern process optimization will be presented and discussed.

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