Towards supercritical extraction of valuable compounds from waste: Phase equilibrium measurements of CO_2 with diethyl succinate

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Abstract

Diethyl succinate occurs naturally in plants. This ester is used as a flavoring agent and in fragrances. A potential method of recovery from residual plants is its supercritical carbon dioxide extraction. However, for the design of a larger scale process phase equilibrium data are imperative. Phase equilibrium data at high pressures for supercritical fluid systems are in general becoming more important because of their increasing. Carbon dioxide as it is nontoxic, and has low critical temperature and pressure is often used for these measurements.

Experimental measurements for carbon dioxide with esters are useful for thermodynamic modeling and process design. The main goal of this work is to report experimental phase equilibrium data for the binary system (CO_2 + diethyl succinate). The experiments were carried out in a high-pressure variable volume view cell at temperatures of 303, 313, 323, 333, 343 and 353 K; CO_2 mole fraction ranged from 0.4246 to 0.993. Briefly, the experimental procedure includes a syringe pump for injecting CO_2 into the cell and for adjusting the pressure into the equilibrium unit, and an electrical heating jacket for the temperature control. Two sapphire windows, one on the side and another at the front of the cell were serving for the visual observations. For each composition, the procedure was the same: flushing the cell with CO_2 to remove any residual air; loading the cell with the respective amount of liquid solute (diethyl succinate) and then with the CO_2; and setting the pressure to reach the homogeneous phase condition (one phase). The phase transition pressure was measured by the slow depressurization of the system at each tested temperature.

Keywords: *vapor-liquid equilibria; ethyl acetate; added value extracts; plant-pharmaceuticals*

1. INTRODUCTION

Diethyl succinate occurs naturally in plants. This ester is used as a flavoring agent and in fragrances. A potential method of recovery from residual plants is its supercritical carbon dioxide extraction. However, for the design of a larger scale process phase equilibrium data are imperative. Phase equilibrium data at high pressures for supercritical fluid systems are in general becoming more important and their application

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is increasing. Carbon dioxide is often used for these applications. Supercritical carbon dioxide has been studied widely as a solvent for chemical reaction and separation processes because it is nontoxic has low critical temperature and pressure (T_c=304.2 K, P_c=7.38 MPa, respectively). The available in the literature vapor-liquid equilibrium (VLE) data at high pressures for carbon dioxide with esters are not sufficient. Thus, the main goal of this work is to report experimental phase equilibrium data for the binary system [CO_2(1)+DES(2)]. The experiments were carried out at temperatures from 308.15 to 358.15 K; CO_2 mole fraction ranged from 0.4246 to 0.9925.

2. MATERIALS AND METHODS

2.1 Chemicals

Diethyl succinate [DES(2)], ethyl acetate (EA) and carbon dioxide with a purity greater than 99% were employed in conducted in a high-pressure variable-volume view cell containing a movable piston, which allowed pressure control inside the cell.
Vapor liquid equilibrium measurements were first conducted for [CO_2(1)+EA] mixture in order to ensure the reliability of the employed equipment and procedures. The results were in excellent agreement with those reported in the literature.

Typical experimental results for the binary system [CO_2(1)+DES(2)], in the temperature range 308.15 to 358.15 K are shown in Figure 1.

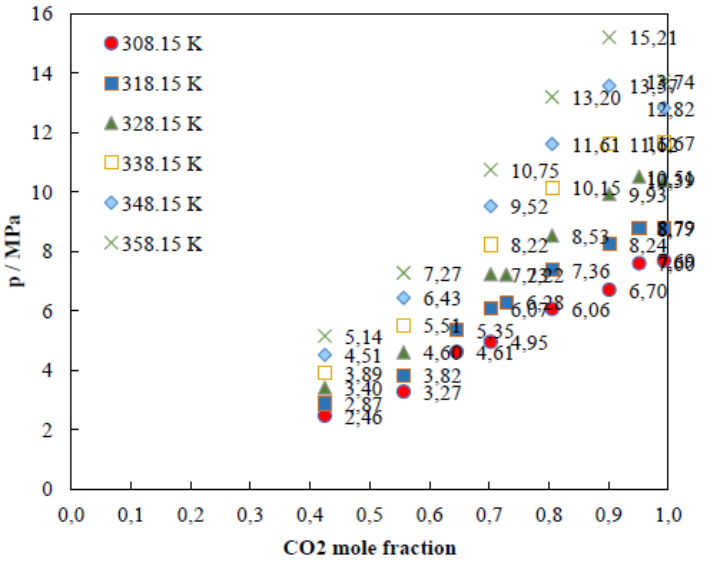

Figure 1. Pressure-composition diagram for the system [CO2(1)+DES(2)] at different temperatures.

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