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Measurement Of Mordant Brown - Supercritical Carbon Dioxide Phase Equilibrium

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In this work, the experimental solubility of Mordant Brown in supercritical carbon dioxide (SC-CO_2) are determined by a dynamic method in the pressure range 195-350 bar and at temperature of 333 K and the obtained values are in the range 10^{-5} mole fraction. The measurements were performed using an apparatus that was especially designed in our laboratories. On the basis of the obtained experimental results, the solubilities increased by increasing the pressure of the experiments. The molar fraction and solubility of the Mordant Brown dye ranged from [(2,72-10,36) $\cdot 10^{-5}$ and (1,91 – 7,27) $\cdot$ 10^{-4}] $g \cdot L^{-1}$, respectively.

1. Introduction

The textile industry uses large amounts of water in its dyeing processes. Due to environmental problems such as the emission of organic materials in wastewater, a new dyeing process has been developed in which supercritical carbon dioxide (SC-CO_2) is used as the solvent for dyes (Özcan et al, 1998; Beltrame et al, 1998). The advantages of SC-CO_2 are that it can be recycled, it is inexpensive, essentially non-toxic, and non-flammable, and it has easily accessible critical conditions. It is an alternative dyeing process, which is able to replace the conventional wet process. In this process, water, surfactants, dispersing agents and the drying process are eliminated (Saus, 1995).
The application of the Supercritical Fluid Technology in each one of these processes takes implicit the study of the phase equilibrium formed by each one of the solutes and the supercritical solvent, that is to say, the fluid-solid phase equilibrium. Solubility measurements of some dyes in SC-CO_2 have been reported (Lee et al, 2001;Tamura and Shinoda, 2004).These results are commonly correlated using theoretical or semiempirical models (Huang et al, 2002; Gordillo et al, 2005; Joung et al, 1998; Mishima et al, 2004).

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2. Experimental section

The apparatus used in this study was especially designed in our laboratories in order to measure solubility. The flow diagram of the installation is shown in Fig. 1, and the equipment and its main operating steps are described below.

The gaseous solvent, carbon dioxide, was pumped by a high pressure pump capable of providing a maximum pressure of 700 bar. The CO_2 was delivered by the pump through a heating coil so that the CO_2 could attain the desired operating temperature prior to entering the equilibrium cell. The equilibrium cell was constructed from stainless steel tubing (42 cm long and an ID of 0.88 cm). The cell was packed with alternate layers of the dye and glass beads and was surrounded by a heating jacket through which hot water was passed in order to achieve the appropriate process temperature. Flow control was maintained with a flow-metering valve. The dye that was dissolved in supercritical CO_2 was separated from the carbon dioxide and collected in two traps containing ethanol. The CO_2 then passed through a flowmeter of Hastings Instruments. A UV_ visible spectrophotometer Shimadzu (Multipurpose Recording Spectrophotometer MPS-2000) was used to analyze the dye per ethanol samples. It showed a sharp absorption maximum at 425 nm.

Mordant brown (2-nitroso-1-naphtol) was supplied by Aldrich (minimum purity of 95%) and the carbon dioxide was supplied by Carburos Metálicos, S.A. with a minimum purity of 99%.

The validity of the experimental technique was first determined by measuring the solubility of naphthalene in supercritical carbon dioxide at 120 and 300 bar and 308, 318 and 328 K (Gordillo et al, 2002). The data are comparable to those reported by Tsekhanskaya et al. (1964). The flow rate was controlled within the range of 0.1-1 ml/min.

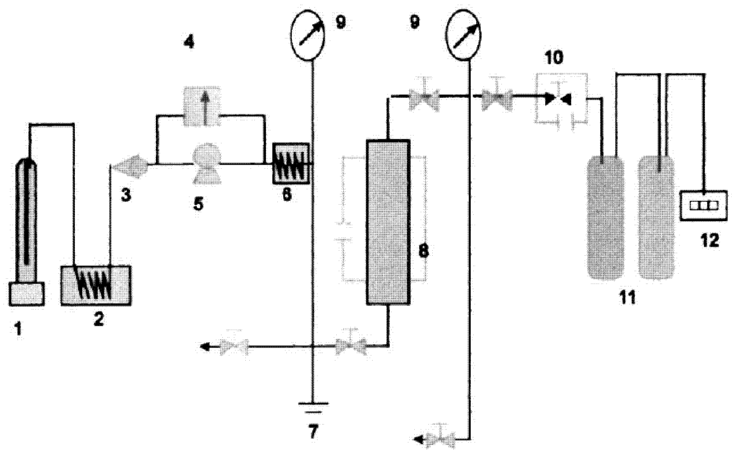

Fig. 1. Schematic diagram of the experimental apparatus: (1) gas tank; (2) constant temperature bath; (3) check valve; (4) pressure regulating valve; (5) liquid pump; (6) preheater; (7) rupture disc; (8) equilibrium cell; (9) pressure gauge; (10) metering valve; (11) cold traps; (12) volumetric flow meter.

3. Results and Discussion

Experimental solubility data as mole fraction for Mordant Brown in supercritical carbon dioxide at 333 K in the pressure range of 195-350 bar are shown in Fig. 2 and Table 1. From the obtained results it can be seen that Mordant brown solubility always increases as the pressure does.This is because the CO_2 density increases,so the solvent power does also.This efect occurs because of the decrease in intermolecular distance, so the solute-solvent interactions increase (King and List, 1996). Guzel and Akgerman (1999) measured the solubility of Mordant Brown at lower pressures and reported higher values than ours. In the future, we are going to study the solubility of Mordant Brown at 323 and 353 K in the pressure range of 195-350 bar, and we are going to analyze how is the behaviour of the solubility of this dye with the temperature.

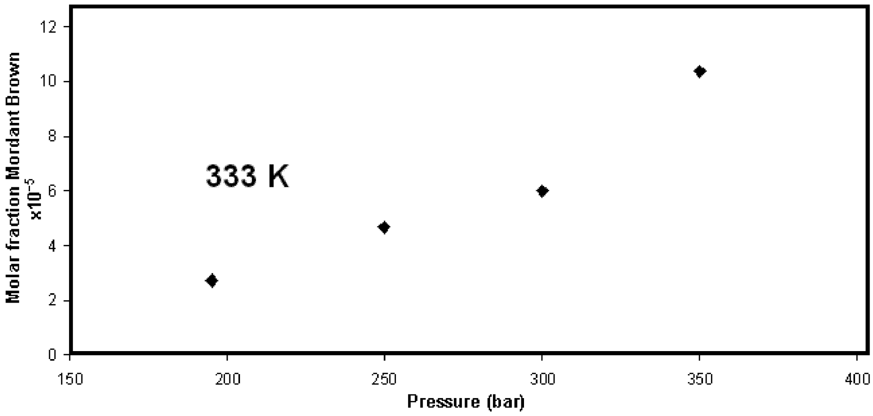

Fig. 2. Molar fraction of Mordant Brown in Supercritical Carbon Dioxide

Table 1.Solubility of Mordant Brown in Supercritical Carbon Dioxide

Mordant Brown	
333K	
P(bar)	$X \cdot 10^{-5}$
195	2,72
250	4,66
300	5,98
350	10,36

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