THE VISUAL INVESTIGATION OF SOLUBILITY OF BIOLOGICAL ACTIVE SUBSTANCES

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In the work there are presented the results for visual investigations of a solubility of biological active substances (BAS) in example of substances complex extracted from the *sage* by the supercritical CO_2 extraction.

INTRODUCTION

Today the supercritical solvents are widely used in many branches of industry. The most useable supercritical solvent is carbon dioxide (CO_2). CO_2 is applied both in pure condition and in correlation with co-solvents. It is known that co-solvents strongly influence on critical parameters if critical solvents. In that connection it becomes urgent to study an influence of extract and dissolved substances in the supercritical fluids upon the critical parameters. It is important for extraction of constituents from complex molticomponent systems, utilization of complex compounds, and extraction of biological active substances from materials of plant and animal origin.

In works [1, 2, 3, 4, 5] are given effective supercritical parameters of the extraction much more exceeding real critical parameters of solvents at which ought to be a maximum solubility. To research these phenomena, and also to study a solubility, separation and extraction we offer to use a very convenient and visual method. The offered methodic allows to test all phenomena and assumptions concerning the processes near the critical and in the supercritical region of the substance state parameter.

EXPERIMENTAL DEVICE

An experimental device is assembled on the basis of the experimental apparatus described earlier [6], but a device, which is described below, allows to carry out more accurate researches. A principle scheme of the experimental device is presented in Figure 1. A main part of the experimental device is the high pressure sapphire tube (1), which will do for us both by its solidity and by its chemical stability. The tube is squeezed between two flanges (2) and (8) out of stainless steel. On the reverse the lower flange (8) is a piston with rubber rings (9-lower couple), which is put into the base (11), where it may move up and down owing to feeding of oil in a base of the flange (10). The oil does into the press from a vessel with oil (15). For the hermeticness between sapphire tube and flanges are placed fluorine-plastic washers (5), which can, if it is necessary, be substituted for more thermostable washer, for example carbon-graphite. The sapphire tube is heated by a special heater (3). It is done out of copper for uniform distribution of the temperature. There are six heating elements are put inside along the whole length of the copper casing. In the heater (3) are done windows to

observe and light the sapphire tube. The windows are hermetically sealed up by a transparent glass (4). For a rapid thermostating a gap between the sapphire tube and heater is filled up by distilled water (7). The hermeticness of the gap is provided by upper couple of rubber rings (9) on the lower flange (8).

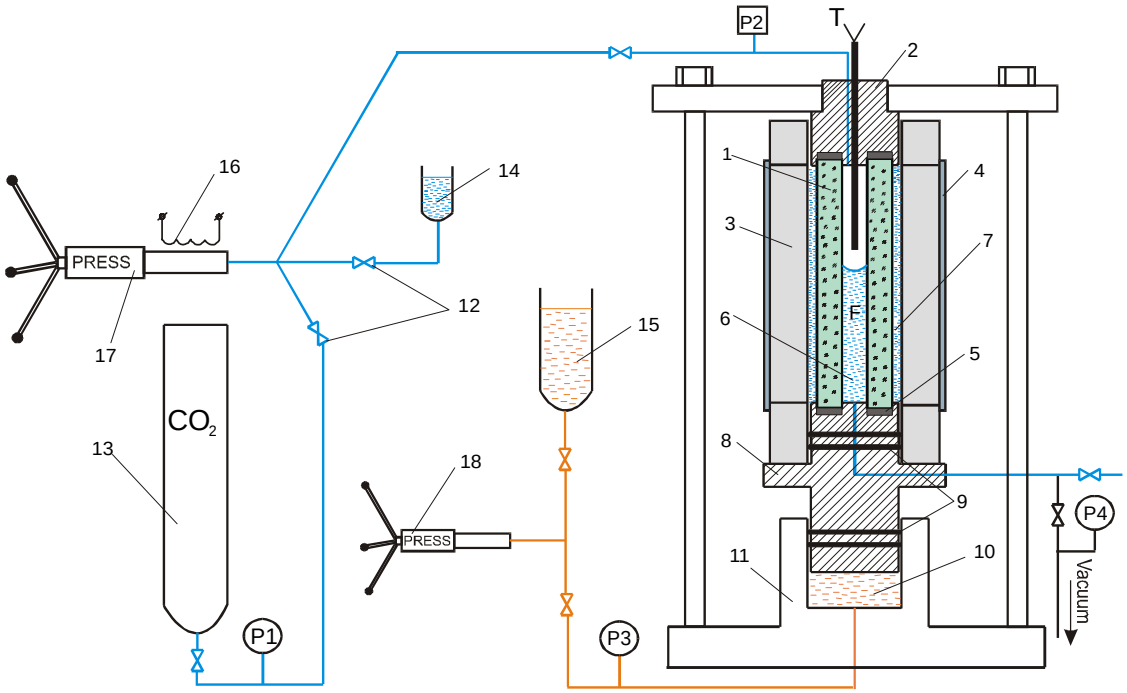

Figure 1. Principle scheme of experimental device

A thermostating precision of the heater to within 0,02°C. Number 6 marks a studied fluid or extract inside the sapphire tube. A temperature inside the sapphire tube is measured by copper-constantan thermopair T. A precision of temperature measuring is to within 0.005°C. The studied substance is fed in the measuring cell by a press (17) from a vessel with studied substance (14). Also CO_2 is fed in the measuring cell with the help of hand press (17). A precision of press feeding is 0,01 cm^3. Substances may be heated up to necessary temperature in the press by a. heater 16. A pressure is measured in the cell by a special electronic pressure transducer P2. To exclude a "parasitic" volume of measuring cell is used the electronic pressure transducer P1, P3 are standard manometer. They show a pressure in CO_2 tank and in a system of the hydraulic squeezing of sapphire tube correspondingly. Before the beginning of the investigations the system is utterly vacuumed. P4 is the vacuum manometer. Number 12 marks the valves. The experimental device allows to observe visually and fix any phase transitions near critical and supercritical regions of substance state parameters, to see an appearance and disappearance of menisci.

RESULTS

We carry out the researches of the *sage* extract, preliminary which was extracted on a semi-industrial experimental device at 10 MPa, 20 MPa, 30 MPa and temperature 31.5°C. The extract is viscous, single-phase, not transparent substance at the atmospheric pressure and room temperature. The extraction is loaded into the experimental device for visual researches. A pressure in the measuring cell is gradually increased, a temperature was kept constant and

equal to 31.5°C. In Figure 2. A is shown the beginning of a phase transition at the pressure 5.5 MPa . Extract is phase I , CO_2 is phase II . A reciprocal solution of CO_2 and extraction in the cell begins with increasing of pressure. An extraction level rises . A new phase with the lowest number of carbon atoms forms out of CO_2 and extraction molecules. Observing the meniscus level one can see a considerable decrease of extraction viscosity. A number of liquid phases rises with increase of pressure.

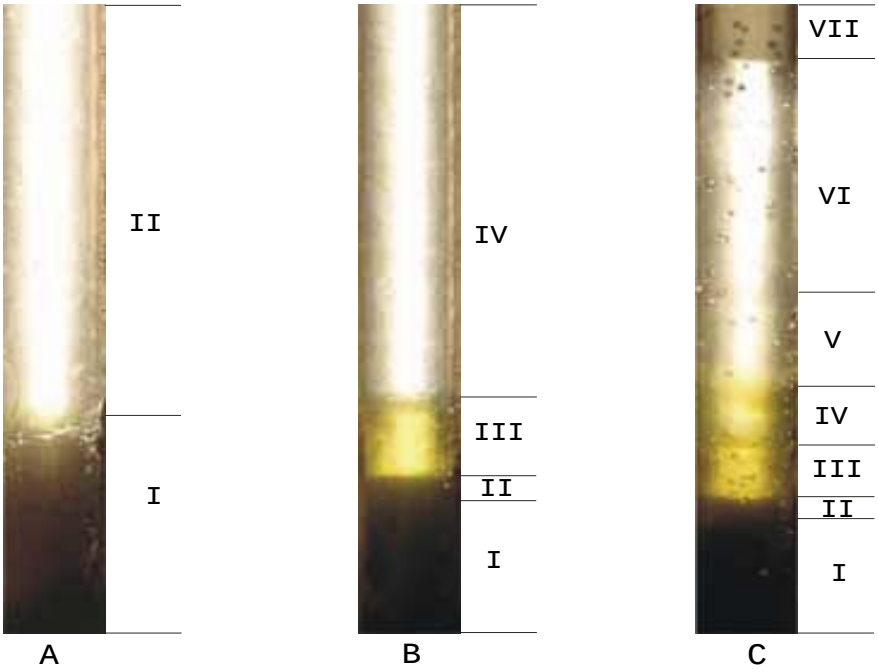

Figure 2. Phase foliation of *sage* extract

When pressure increases, CO_2 is bound up with large molecules of a great number of carbon atoms and forms new liquid phases .At pressure 6.5 MPa the extraction is foliated in to three liquid phases (Figure 2B). All these stages of separation are related to the liquid CO_2 extraction. Further increasing of the pressure and approximation to critical parameters of CO_2 continue a phase separation from bottom to top. But simultaneously phases mix from top to bottom. During the separation any considerable changes not occur in a region of CO_2 critical state. Intensive density fluctuations are observed in upper layers at pressure 13 MPa. The most upper phase reduces into dimmed “fog” (Figure 2 C, phase VII). All properties of phase VII are point to that it is a supercritical region CO_2. At pressure increase region of intensive fluctuations decreases dissolving lower phases and reducing in to homogeneous one- colour phase of intensive fluctuations. The process is going on up to reaching of more dense phases III, II, I. Reaching the dense layers one can judge of solubility only by disappearance of meniscus, the dense layers retains their colours, intensive fluctuations are stopped. Figure 2C shows a disappearance of meniscus between phase VI and V. Parameters, at which the meniscus disappears and lower phases mix with upper phase, are effective extraction parameters for extraction components dissolved in CO_2.

CONCLUSION

The researches show that extraction influence upon the critical parameters of CO_2. The extraction dissolved in CO_2 changes its critical parameters and consequently effective critical parameters of extraction.

Changing the thermodynamic parameters of CO_2 a dissolved extract may be separated into dozen phases consisting of molecules with similar number of carbon atoms in one phase. Each phase forms from molecules with certain number of carbon atoms. This methodic may easily solve a problem of BAS separation in to the components. The selectivity increases with decrease of temperature.

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