

Fundamental data for design of processes using supercritical fluids

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1. Introduction

High pressure technologies involving sub and supercritical fluids offer the possibility to obtain new products with customer designed properties or to design new processes, which are environmentally friendly and sustainable.

For the design of all high pressure process, data are required on the operating parameters, the type and quantity of the solvent, the recirculation rate and energy consumption. This information can be obtained from phase equilibrium and mass transfer measurements. Therefore, several parameters influencing solubility, mass transfer of target compounds in the SCF, and consequently extraction yield have to be considered prior to choosing the suitable processing solvent and process parameters¹.

2. Methods

Determination of thermodynamical and transport data: For evaluation of basic thermodynamical and transport data describing the behavior of the system at certain conditions phase equilibria, density, viscosity, dielectric constant, diffusion coefficient and interfacial tension have to be considered. However, scientific literature offers a variety of these data, measured at a variety of pressures and temperatures, for several pure compounds. Data on behavior of multicomponent systems at elevated pressures and temperatures are still relatively scarce and comprise methods that are expensive and time consuming.

The thermodynamic properties of multicomponent mixtures and their analysis in terms of interpretative models constitute a very interesting subject, crucial for design and set up of industrial processes which continue to drive research in the study of multicomponent systems.

Specific interest should be dedicated to the fact that thermodynamic and transport properties are associated with heat and fluid flow characteristics. Viscosity and interfacial tension of systems containing supercritical fluid are among the most influential parameters on fluid behavior.

In order to determine the hold-up times, transfer studies are performed and for determining mass transport coefficients different mathematical models were tested.

Extraction: Extract quality depends on processing pressure and temperature which can seriously influence the composition of the final extracts. In addition, pressure drop effect has to be evaluated and taken into account when optimizing parameters to obtain the best ratio between yield and solvent amount and extraction time. There is an additional requirement, namely, highest possible loading of SC solvent should be achieved in extraction step of the processes, while in separation step of the process the solubility of solute in solvent should be the lowest.

Particle formation/impregnation: The traditional methods for processing of solids and solid mixture for applications in food, feed and pharma industries involve either high temperatures, necessary for melting or viscosity reduction or hazardous organic solvents and chlorofluorocarbons. Due to the undesirable environmental and biological impact of these solvents, intensive research is focused on seeking new and cleaner methods for the processing of those compounds.

Applying supercritical fluids for particle formation may overcome the drawbacks of conventional particle size reduction processes. Powders and composites with special characteristics can be produced. Several processes for formation and design of solid particles using dense gases are studied intensively. The unique thermodynamic and fluid-dynamic properties of SCFs can be used also for impregnation of solid particles, for formation of solid powderous emulsions, particle coating e.g., for formation of solids with unique properties for the use in different applications.

3. Discussion

The lecture will give special attention to determination of thermodynamic and mass transfer fundamentals. Behavior of carriers and bioactive binary systems with different gasses (CO₂, SF₆, propane, argon, xenon) have been investigated in details. The “external balance method” was developed for determination of the solubility of gas into substrates which are soluble in gas. Densities and viscosities of gas saturated solutions of polymers were measured by new methods developed by the authors. Capillary rise method was applied to investigate the interfacial tension of substances with various gasses²⁻⁴.

Obtained fundamental data were used to determine process parameters for micronization of polymers with PGSSTM (Particles from Gas Saturated Solutions) process co invented by author of this manuscript (USP 6056791)⁵ for the formation and formulation fine particles.

4. Conclusions

This is a primary step in design, modification and optimization of processes using sub or supercritical fluids with different gasses like propane, argon, chlorinated hydrocarbons, sulfur hexafluoride and carbon dioxide⁶. This is related to the fact that high solubility of compound of interest in the supercritical solvent is essential for the economy of extraction process the practical analyses shall verify if extraction using supercritical fluid is the suitable technique for the isolation of the target compound. For production of particles with micron and submicron size, several methods using supercritical fluids are described in the literature. The bases of practically all processes are fundamental thermodynamic data and transport data for the system polymer/dense gas. An overview of methods for investigation of the thermodynamic properties of the binary systems containing polymer and gas will be presented.

References

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