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Mixing polymer mixtures in the dissergation process by SEDS method

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Abstract

The analysis of phase equilibria was carried out in order to establish the optimal paracetamol dispersion parameters by the supercritical fluid anti-solvent (SEDS) method. The solubilities of the SEVA-113 and SEVA-115 polymers in supercritical carbon dioxide at a pressure range of 10-20 MPa are investigated. The results of an experimental study of the characteristics of phase equilibria for the "toluene-SEBA- $\rm CO_2$ " system at temperatures of 313 and 323 K at pressures of 8 MPa and 10 MPa are presented.

Taking into account all the thermodynamic characteristics studied, optimal regime parameters for the joint dispersion of the polymers SEVA-113 and SEVA-115 were established. The results obtained in the SEDS dispersion process were the Scanning Electron Microscopy (SEM) method using the *AURIGA Cross Beam* with an INCA X-MAX energy dispersive spectrometer. The results of the investigation of the process of joint dispersion of polymer mixtures SEVA and PEVD according to the SEDS method performed in the pressure range 8.0÷25 MPa at temperatures T = 313, 323 and 333 K.

Studies on the kinetics of crystallization and phase transformation in mixtures of copolymers prepared by melt mixing and using the SEDS method were carried out using the DSC-200 TA differential scanning calorimeter (DSC) with the Pyris software. Mixtures of SEAB with different content of vinyl acetate units and HDPE are obtained by melt mixing at a temperature of 110-115 °C on Gerhard Koch rolls after 4-5 minutes after loading the original components.

When studying melting diagrams it was found that for all polymer pairs, the heat of melting of mixtures obtained by "mixing" in the SEDS method is greater than the heat of melting of mixtures obtained by melt mixing. As a result, it can be concluded that mixing by the SEDS method leads to an increase in the degree of crystallinity and, accordingly, to the improvement of the structure of the polymer matrix.

References

- [1] U.N. Kahramanly. Incompatible polymer blends and composites based on them. *Baku. «ELM»*. **2013**. 152p.
- [2] S.K. Ivanovsky, A.R. Ishkuvatova, K.V. Trifonova. Technological and thermodynamic aspects of obtaining molecular polymer compositions. *The Successes of Modern Natural Science*. **2014.** No.12. P.92. (russian)
- [3] F.M. Gumerov, A.N. Sabirzianov, G.I. Gumerova. *Sub- and Supercritical Fluids in Polymers Processing. Kazan.* **2000**. 328p. (russian)
- [4] M.F. Kemmere, T. Meyer (Eds.) Supercritical Carbon Dioxide in Polymer Reaction Engineering. **2005**. *WILEY-VCH Verlag GmbH*. 1131p.
- [5] P.S. Timashev, L.I. Krotova, D.A. Lemanovskiy, V.K. Popov. Dispersion copolymerization of methyl methacrylate and styrene in supercritical carbon dioxide. *Supercritical Fluids. Theory and Practice.* **2010**. Vol.5. No.2. P.70. (russian)
- [6] V.F. Khayrutdinov, F.R. Gabitov, F.M. Gumerov, B.Le. Neindre, E.S. Vorob'ev. Thermodynamic Bases of Polycarbonate Dispersing Process Using SAS Method. *Supercritical Fluids. Theory and Practice*. **2011**. No.3. P.62-78. (russian)
- [7] I.M. Gilmutdinov, V.F. Khayrutdinov, I.V. Kuznetsova, A.A. Mukhamadiev, F.R. Gabitov, F.M. Gumerov, A.N. Sabirzyanov. Dispersion of polymeric materials using supercritical fluid media. *Supercritical Fluids. Theory and Practice.* **2009**. No.3. P.25-38. (russian)
- [8] I.M. Gilmutdinov, A.N. Sabirzyanov, F.M. Gumerov. The effect of solvent density and channel geometry

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- MIXING POLYMER MIXTURES IN THE DISSERGATION PROCESS BY SEDS METHOD on the morphology and size of the resulting microparticles during the rapid expansion of the supercritical solution Supercritical Fluids. Theory and Practice. 2008. Vol.3. No.1. P.43-49. (russian)
- [9] D.U. Zalepugin, A.I. Gamzadze, N.A. Tilkunova. Preparation of micro- and nanoparticles of chitosan and its derivatives by spraying from solutions using supercritical solvents. Supercritical Fluids. Theory and Practice. 2008. Vol.3. No.1. P.25-32. (russian)
- [10] J. Jung, M. Perrut. Particle Design Using Supercritical Fluids: Leterature and Patent Survey. J. Supercritical Fluids. 2001. No.20. P.179-219.
- [11] S.D. Yeo, E. Kiran. Formation of polymer particles with supercritical fluids: A review. J. Supercritical Fluids. 2005. No.34. P.287-308.
- [12] K. Mishima. Biodegradable particle formation for drug and gene delivery using supercritical fluid and dense gas. Advanced Drug Delivery Reviews. 2008. No.60. P.411-432.
- [13] M. Hanna, P. York. "Method and apparatus for the formation of particles" Patent WO 95/01221, 1995.
- [14] O.V. Stoyanov, R.M. Khuzakhanov, L.F. Stoyanova, V.K. Gerasimov, A.E. Chalykh, A.D. Aliyev, M.V. Vocal. The structure of binary mixtures of ethylene-vinyl acetate copolymers. *Adhesives*. Sealants. Technologies. 2010. No.11. P.15-17. (russian)
- [15] L.F. Utrachki. Clay-Containing Polymeric Nanocomposites. *Monograph to be published by Rapra*. 2004. 600p.
- [16] Heng-Joo Ng, Donald B. Robinson Equilibrium Phase Properties of the Toluene-Carbon Dioxide System. Journal of Chemical and Engineering Data. 1978. Vol.23. No.4. P.325
- [17] William 0. Morris and Marc D. Donohue Vapor-Liquid Equilibria in Mixtures Containing Carbon Dioxide, Toluene, and I-Methylnaphthalene. J. Chem. Eng. Data. 1985. Vol.30. P.259-263.
- [18] F.E. Wubbolts, O.S.L. Bruinsma, G.M. Rosmalen. Dry-spraying of ascorbic acid or acetaminophen solutions with supercritical carbon dioxide. J. Cryst. Growth. 1999. No.198. P.767-772.
- [19] A. Shariati, C.J. Peters. Measurements and modeling of the phase behavior of ternary systems of interest for the GAS process: I. The ternary system carbon dioxide+1 propanol +salicylic acid. J. Supercrit. Fluids. 2002. Vol.23. P.195.
- [20] V.G. Bortnikov. Basics of plastics processing technology. *Leningrad: Chemistry.* **1983**. 304p. (russian)