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(54) Title: ACCELERATION OF THE PHASE SEPARATION OF LIQUID PHASES WHICH CONTAIN POLYMERS

(57) Abrégé/Abstract:
The invention relates to the acceleration of the separation of liquid phases which contain polymers by using branched instead of linear solvents.

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Mit internationalem Recherchenbericht.
Vor Ablauf der für Änderungen der Ansprüche zugelassenen Frist; Veröffentlichung wird wiederholt falls Änderungen eintreffen.

(54) Title: ACCELERATION OF THE PHASE SEPARATION OF LIQUID PHASES WHICH CONTAIN POLYMERS

(54) Bezeichnung: BESCHLEUNIGUNG DER PHASENTRENNUNG VON POLYMER–ENTHALTENDEN FLÜSSIGEN PHASEN

(57) Abstract

The invention relates to the acceleration of the separation of liquid phases which contain polymers by using branched instead of linear solvents.

(57) Zusammenfassung

Die Patentanmeldung beansprucht die Beschleunigung der Abscheidung flüssiger, Polymer–enthaltender Phasen durch Verwendung verzweigter statt linearer Lösungsmittel.

ACCELERATION OF THE PHASE SEPARATION OF LIQUID PHASES WHICH CONTAIN POLYMERS

Mixed polymers may be separated by a liquid-liquid phase separation [1]. Thereby, polymers are present in all phases, but selectively separated. Liquid phases with no insignificant polymer contents have high viscosities. Polymers are understood to be technically or biologically produced macromolecules having a molar weight of 1000 Daltons or more. Polymers of different kind are understood to be chemically or structurally different polymers, such as HDPE, LDPE, PP or PVC.

For the economic of such a thermal separation process, the times of sedimentation in the separation steps are decisive, since space-time-yields are determined thereby. Further, long residence times contribute that decomposition reactions take place.

Liquid systems having two or more phases include a continuous phase (KP) and one or more discontinuous phases (DP) in a dispersed state. The literature [2] subdivides coalescence and sedimentation of the discontinuous phase into several stages:

- coalescence of single drops to form larger drops
- ascending or descending of larger drops controlled by density
- unification of larger drops with already formed phase.

In all these processes, in particular the rising or settling velocities, respectively, of single drops are important. A comparatively simple mathematical modellation is obtained when the drops are considered as rigid spheres in the KP.

The modellation of the motion of sperically shaped solid particles in a continuous phase is made according to Clift R. et al [3] and Brauer [2] by using the equation of motion. The equation of motion (eq.1) represents a term for the stationary velocity of falling spherically shaped particles.

$$w_P = \sqrt{\frac{4}{3}\frac{\rho_P - \rho_F}{\rho_F} \cdot g \cdot dp \cdot \frac{1}{\xi}} \qquad \text{eq. (1)}$$

Therein, w_P represents settling or rising velocity of particles, ρ_P and ρ_F respectively, the density of the discontinuous (index P) and the continuous phase (index F), respectively, g the acceleration due to gravity, d_P the particle diameter and ξ the drag coefficient.

Equation 1 is generally valid for solid or fluid high viscous particles. The special characteristics of the phase boundary surface are described by the drag coefficient for which the following interrelation is valid:

$$\xi \propto \frac{1}{\mathrm{Re}} \qquad \text{eq. (2)}$$

wherein

$$\mathrm{Re} = \frac{w_P d_P}{\nu_F} = 18\left(\sqrt{1+\frac{\sqrt{Ar}}{9}} - 1\right)^2 \qquad \text{eq. (3)}$$

In equation 3, ν_F represents the kinematic viscosity of the continuous phase and Ar the Archimedian number. The introduction of the dimensionless Archimedian number is useful, since hereby the settling velocity w_P of particles in equation 1 can be represented in explicit form. It is:

$$Ar = \frac{\rho_P - \rho_F}{\rho_F} \cdot \frac{g \cdot d_P^3}{\nu_F^2} \qquad \text{eq. (4)}$$

Equation (4) teaches that only the viscosity of the continuous phase, the particle diameter of the discontinuous phase and the density difference between continuous and discontinuous phase are important for the separation. Short residence times are advantageous to save operation and investment costs and to suppress chemical reactions.

According to the teaching of the literature, the phase separation time can be improved in particular by reducing the viscosity of KP. Possibilities known in the literature are increasing the temperature or changing the nature of the solvent which forms KP together with the polymer, both measures with the aim to reduce the viscosity of KP. The said measures do not lead to the goal in the separation of mixed polymer by liquid-liquid-phase separation, since

- the temperature does not only change the viscosity, but due to the thermodynamics in particular the purity/yield of the polymers to be separated. Therefore, an improvement of the separation time must be paid for with a deterioration of selectivity;

- changing the nature of the solvent changes the thermodynamics to an extent that the separation temperatures are either shifted into ranges which are uneconomic or the phase separation does no longer exist in the range of temperatures which can be technically reached.

This problem is solved by a method according to claim 1 in an elegant and novel manner. Preferred embodiments are subject-matter of the subclaims.

As an example, the thermal separation of polyolefine mixtures as polymers of different type is considered, with n-alkanes having five to seven carbon atoms in the molecule as solvent. The teachings of the patent are applicable to all other separation methods which include the separation of polymer mixtures by forming liquid phases.

The method described in [1] separates polyolefines in that two liquid phases are formed in n-hexane at 180°C, wherein the upper light phase contains under certain conditions polyethylene from high pressure synthesis (LDPE), and the lower one polyethylene from low pressure synthesis (HDPE). Due to the amount, the low viscous upper phase is KP, and the high viscous lower phase is DP in the dispersed system. The low viscosity of the upper phase, however, does not lead to technically acceptable separation times, so that the above mentioned disadvantages (costs, reactions) become effective.

Experimental runs, however, have surprisingly shown that the use of branched solvents with same carbon number significantly and advantageously changes the separation times. Therein,

the viscosity of KP is about the same for the non-branched and the branched solvent, so that the effect cannot be explained at the moment.

Branched solvents are such aliphatic materials in which at least one carbon atom has more than two carbon atoms as neighbors and which may also carry functional groups along with further C and H atoms.

To illustrate the proceedings according to the patent, the branched solvent 2,3-dimethylbutane shall be compared with the unbranched solvent n-hexane.

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Example 1

Two experimental runs were performed having the separation of a polyethylene-polypropylene mixture as a goal. As polyolefines, HDPE (high density polyethylene), LDPE (low density polyethylene) and polypropylene PP were supplied in a proportion of 15/43/42, wherein the total content of polyolefine in solution was 20 weight %.

In the first experimental run V_1 n-hexane was used as a solvent, for the second experimental run V_2 2,3-dimethylbutane. The phase separation times as well as the viscosities of the so-
20 lution were observed. They are represented in table 1. It is noticeable that periods of 300 min cannot be realized with an unbranched solvent or only in a difficult way.

The purities of the different polymers were practically identical in both phases in both runs, since the nature of the solvent had not been changed (in both cases, a hexane is concerned).

However, the considerably shorter separation times in the experimental run V_2 using the branched solvent are surprising. Even though the viscosity of KP and the density difference ρ_P-ρ_F did not change significantly in both runs, it has been detected surprisingly that the system with the branched solvent had been separated faster by a factor of 150 than the system with n-alkane as a solvent.

Experimental run	Solvent	$w_{Feed.Pol}$ [weight %]	System temperature	Separation time of phases [min]	Viscosity of upper phase KP	Viscosity of lower phase DP
V_1	n-hexane	20	180	300	Low	High
V_2	2,3-dimethylbutane	20	180	2	Low	Low

Table 1: Experimental runs to separate polyolefine with a branched (experimental run V_2 and an unbranched solvent (experimental run V_1)

According to the teaching of the literature, these reduced separation times lead to smaller separation containers and therefore to a reduction of investment costs and of residence times at the necessary high temperatures.

The teaching of this patent can be applied to all separations of liquid phases which contain polymers and solvents of which branched and unbranched representatives exists.

Citation

[1] DE 199 05 029 A, published after the priority date of the present application

[2] Brauer, H.: Grundlagen der Einphasen- und Mehrphasenströmungen; Verlag Sauerländer, Aarau, 1971

[3] Clift, R.; Grace, J.R.; Weber, M.E.: Bubbles, Drops and Particles Academic Press, New York, 1978

Patent claims:

1) A method for acceleration of the separation of phases containing different polymers, characterized in that

a) said polymers are solved in a branched solvent or solvent mixture and

b) the resulting solvent phases which include solved polymers of different type in different concentrations are separated after sedimentation.

2) The method of claim 1, characterized in that said solvent is selected such that the sedimentation times of the phases in the branched solvent or solvent mixture are shorter than those in a non-branched solvent or solvent mixture.

3) The method of claim 1 or 2, characterized in that said polymers of different type are mixtures of polyolefins and said solvent is a branched alkane having a carbon number between 4 and 16.

4) The method of claim 3, characterized in that said solvent is a branched alkane having a carbon number between 4 and 10.

5) The method of claim 3, characterized in that said solvent is a branched alkane having a carbon number between 6 and 8.

6) The method of any of claims 1 and 5, characterized in that said solvent is dimethylbutane or methylpentane.

7) The method of any of claims 1 to 6, characterized in that the supplied polyolefins originate from waste to be recycled.