

ELECTROSTATIC COALESCENCE

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BACKGROUND OF THE INVENTION

The invention relates to a method for segregating a dispersion having a continuous organic phase and a disperse aqueous phase from a caprolactam production process.

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DISCUSSION OF PRIOR ART

In a caprolactam production process caprolactam is prepared from cyclohexanoxime after a Beckmann-rearrangement in the presence of sulphuric acid or oleum. The rearrangement-mixture is then neutralised with ammonia whereby ammonium sulphate is formed. Subsequently caprolactam is extracted from water with an organic solvent, for example benzene. In the resulting organic phase also a disperse aqueous water phase is present. This aqueous phase comprises besides caprolactam also ammonium sulphate. The presence of ammonium sulphate is disadvantageous in the subsequent processing and purifying of caprolactam. Removing the disperse aqueous phase as much as possible is therefore highly desired.

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There are many processes in which it is necessary to separate dispersions.

One can use settling tanks for the separation of dispersions where the densities of the two phases differ sufficiently. The denser phase sinks below the less dense phase and given a sufficient time the two phases can be separated sufficiently to be drawn off. However, such a process requires the use of very large tanks taking up a correspondingly large amount of space. Furthermore, this separation procedure would be the slowest stage of the more extensive caprolactam production process and would therefore determine the throughput of the complete process.

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It is also possible to use coalescours or washing treatments to remove a disperse phase as for example described in German patent application 1,031,308. The addition of caustic washing material in the caprolactam production process is disadvantageous to the quality of caprolactam.

US patent 3,528,907 describes the possibility of separation of water dispersed in a hydrocarbon, for example crude oil, from said oil by the use of an electric field. However, this patent also teaches that the voltage of the applied electric field must definitely not exceed 1000 volts per inch, or 400 volts per cm, because this would cause a short-circuit between the electrodes. A disadvantage of this method is that, in particular, a dispersion containing at most only about 5 wt.% aqueous phase of the combined total liquid phases cannot be readily separated from the organic phase.

European patent application No. 51463 describes settling of liquid emulsions by means of electrostatic coalescence. This process however requires the presence of high volumes of conducting liquid (the aqueous phase), for example 50%. Especially insulated electrodes are needed because otherwise short-circuiting would occur.

German patent application No. 3,709,456 and European patent application 438790 both describe separation of liquid membrane systems where the aqueous phase is the continuous phase.

In general, dispersions exhibit a continuous particle size distribution. Dispersions can be subdivided into primary and secondary dispersions. In the case of primary dispersions the particles forming the disperse phase have an average particle size > 0.1 mm. In many cases these dispersions can easily be segregated gravimetrically with the aid of settling tanks. In the case of secondary dispersions, however,

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the particles forming the disperse phase have an average particle size of only $\ll 0.1$ mm. Segregating these latter dispersions by means of settling tanks takes an unacceptably long time, however, for industrial applications. To shorten this segregation time it is therefore possible and advantageous to employ electrostatic coalescence according to this invention.

10 GENERAL DESCRIPTION OF THE INVENTION

 This invention has as its primary object the provision of a method whereby a dispersion from a caprolactam production process, in particular a dispersion containing at most only about 5 wt. of aqueous phase, can be segregated very efficiently.

 This object is achieved by applying electrostatic coalescence at an electric field of at least 500 volts per centimetre and a frequency of at least 5 Hz. Electric fields with a (peak) strength of at least 1000 volts per cm, preferably at least 3000 volts per cm, more preferably at least 5000 volts per cm and especially at least 10,000 volts per cm can also be advantageously used, and by so doing the segregation results achieved become increasingly better. In spite of use of these high electric fields, no short-circuits occur, yet the segregation of the aqueous phase from the organic phase is very efficient. It was found, for example, that in continuous organic liquid phases, at a frequency of 50, Hz 50% of the aqueous disperse phase present can be separated after 10 minutes at 733 volts per cm. At 6,666 volts per cm, as much as 90% of the aqueous phase present can be segregated after 10 minutes. With a settling tank it would take several days to a week to achieve the same effect.

35 With electrostatic coalescence it is obviously also possible to vary the frequency. Good results according to this invention are achieved if the

frequency is at least 20 Hz. Frequencies up to 500 Hz can very well be used. Good results can also be obtained at frequencies outside the preferred ranges.

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BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates in schematic form one type of apparatus which may be used in the practice of this invention.

10 Fig. 2 illustrates in schematic form another type of apparatus which may be used in the practice of this invention.

DETAILED DESCRIPTION OF THE INVENTION

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The shape of the vessel or container in which the electrostatic coalescence can take place is less important. The vessel may be cylindrical, square, rectangular, etc.

20 The electrostatic coalescence technique of this invention can be conducted either continuously or batchwise. When continuous application of electrostatic coalescence is carried out, however, care should be taken regarding the internal surface configuration of the vessel or the container, to ensure that laminar
25 flow is established; the length/diameter ratio may be significant in this context. In particular, turbulent flow should be avoided, since this turbulence adversely affects segregation or, as the case may be, segregation time requirements.

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Electrodes which can be used may be made from various materials, e.g. conductive glass, metal etc.

The shape of the electrodes employed is not essential to the invention. Examples include rod-shaped or plate-shaped electrodes. The electrodes may be either
35 insulated or noninsulated. The use of noninsulated electrodes may make suggest an advantageous cost difference for the procurement. On the grounds of

safety and/or energy consumption, however, one may alternatively opt for insulated electrodes.

The temperature at which segregation with the aid of electrostatic coalescence can take place is not really essential to the invention and may preferably be at room temperature. While, in general higher temperatures favor a more rapid achievement of the phase segregation, it is generally necessary to guard against the temperature rising too high, such that the dispersion to be segregated starts to boil or lead to generation of a gas, thereby inducing turbulence and resulting reformation of the dispersion.

It is of course always possible to apply the electrostatic coalescence under elevated pressure in order thus to prevent the dispersion from boiling.

Both alternating current and direct current can be used. When alternating current is applied the pulse can be either sinusoidal or rectangular, sawtooth-shaped, or a combination of these, there is only little apparent effect on the segregation results.

The term organic phase as used in this patent, refers to one or more organic compounds composed of carbon and hydrogen, for example linear and cyclic hydrocarbons, and which may also contain other atoms such as oxygen, sulphur, nitrogen, halogens etc. Examples of such compounds include benzene, toluene, cyclohexane, heptane, dimethyl sulphoxide, chloroform, trichlorethane, etc.

In addition to water, the aqueous phase may also contain other compounds, for example salts or even organic compounds dissolved in the aqueous phase. The aqueous phase may even contain up to 60 wt.% of organic compound(s), for example caprolactam. Nonetheless, in the context of this invention, the "aqueous phase" is considered to be aqueous provided that its water content is at least 20 wt.%.

The aqueous phase may also contain water

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soluble salts, for example between 0.1 and 5 wt.% ammonium sulphate.

The now segregated aqueous disperse phase can be separated from the organic continuous phase by means which will be apparent to those skilled in the art, for example by drawing off the aqueous layer, by decantation, and the like. It is possible to recycle the separated aqueous phase into the caprolactam production process.

The preparation of caprolactam involves various preparation steps in which such dispersions are formed. These then generally require an effective liquid/liquid separation technique. Examples at various process steps include the cyclohexane oxidation, the preparation of hydroxylammonium salts, the top and bottom streams from extraction columns.

EXAMPLES OF THE INVENTION

DETAILED DESCRIPTION OF THE DRAWINGS

The invention will now be explained in more detail with reference to the following examples, without being limited thereto.

Examples

An Ultra-Torrax7 T50 mixer was used to disperse water having caprolactam dissolved therein (50 wt.%) in benzene as the organic phase with 2 wt. of water phase dispersant based on the total mixture, at 5000 revolutions per minute (rpm) over a period of 5 minutes. Within 1 minute, this dispersion was introduced into an electrostatic coalescer, the frequency and the voltage being subsequently set. The examples employed alternating current in each case and were carried out at room temperature.

The coalescer used in Examples I-III is schematically illustrated in Figure 1, where 1 is an insulated glass electrode, 2 is a glass jacket

containing 3 sulphuric acid as the outer electrode and 4 is the voltage source with frequency controller. The gap between electrodes 1 and 3 was 1.5 cm.

5 A coalescer as employed in Examples IV and V is depicted in Figure 2, where 10 is a thermostatted vessel within which a glass vessel 20, (100:30:100 mm) has been placed which is sealed by means of a cover 30 through which two noninsulated stainless steel electrodes 40 have been positioned. The gap between the
10 electrodes was 1.5 cm. After various intervals, samples of the dispersion were taken and analysed.

The experiments were carried out at room temperature. To determine how much water had separated, or the concentration of aqueous phase in the
15 dispersion, the turbidity was measured in accordance with the 'Deutsche Einheitsverfahren C2, DIN 38404, Teil 2', this is also a measure for the percentage of the water phase which had separated. Via a calibration curve the degree of turbidity was related to the
20 percentage of water which had separated.

Examples I-III

Examples I-III were carried out as described above, the frequency was 50 Hz, the electric field was
25 sinusoidal. The voltage was varied and is shown in Table I, together with the corresponding separation results.

Table I

Example	Voltage (Volts/cm)	wt.% sepa- rated after 1 min	wt.% sepa- rated after 5 min	wt.% sepa- rated after 10 min
I	733	15	40	51
II	3333	42	68	80
III	6666	60	88	90

Table I clearly shows that the higher the voltage, the better and more complete the separation result.

Examples IV and V

These examples were carried out as described above, the frequency was 50 Hz and the voltage was varied. The voltage field was sinusoidal. The results are shown in Table II.

Table II

Example	Voltage (Volts/cm)	wt.% sepa- rated after 1 min	wt.% sepa- rated after 5 min	wt.% sepa- rated after 10 min
IV	733	48	70	73
V	2067	62	91	92

Comparative Example A

A sample of the dispersion of Example ??? was introduced into a cylindrical vessel allowed to settle with no application of electrical coalescence. After 1 minute, only 6% of aqueous layer had separated, after 5 minutes this amounted to 9% and after 10 minutes only to 12%.

This invention has been described with reference to specific embodiments thereof but the scope and extent of rights under the applicable patent laws is limited only by the terminology of the following claims.

CLAIMS

1. Method for segregating a dispersion having a continuous organic phase and a disperse aqueous phase from a caprolactam production process, characterized in that electrostatic coalescence is applied to the dispersion at an electric field of at least 500 volts per centimetre and a frequency of at least 5 Hz, whereby the formation of a separate aqueous layer is induced.
2. Method according to claim 1, characterized in that an electric field of at least 1000 volts per centimetre is used.
3. Method according to claim 2, characterized in that an electric field of at least 5000 volts per cm is used.
4. Method according to claim 3, characterized in that an electric field of at least 10,000 volts per cm is used.
5. Method according to any one of claims 1-4, characterized in that the frequency is at least 20 Hz.
6. Method according to claim 5, characterized in that the frequency is at most 500 Hz.
7. Method according to any one of claims 1-6, characterized in that the amount of aqueous phase is at most 5 wt.% of the combined total liquid phases.
8. Method according to any one of claims 1-7, characterized in that the organic phase is benzene.
9. Method according to any one of claims 1-8, characterized in that the disperse phase is water.
10. Method according to claim 9, characterized in that the disperse phase comprises up to 60 wt.% caprolactam.



11. Method according to claim 9, characterized in that the disperse phase comprises between 0.1 and 5 wt.% ammonium sulphate.