Introduction

Double emulsions are complex liquid dispersion systems in which the droplets of one dispersed liquid are further dispersed in another liquid. These systems are recently emerging as oil-in-water-in-oil (o/w/o) double emulsions. Previous studies revealed that the inner w/o emulsion of w/o double emulsions can be stabilized by using ethyl cellulose as a polymeric emulsifier [3]. However, the long-term stability of such emulsions was not satisfying. The aim of the present study was to investigate if the overall performance of such w/o emulsions can be improved by adding a hydrophilic polymer to the aqueous phase.

Experimental methods

Materials

Eutanol G (octyldecaneol) was purchased from Cognis, Düsseldorf, Germany. Hypromellose (Metolose 90SH-100) was supplied by Shin-Etsu, Tokyo, Japan. Ethocel 10 (ethyl cellulose) was supplied by Dow, Schwalbach, Germany.

Methods

a) Preparation of emulsions

Emulsions were prepared by homogenising using an Ultra Turrax (Janke & Kunkel, Staufen, Germany) for 4 minutes at 10000 rpm. Alternatively, emulsions were in addition passed through a high pressure homogeniser (Niro Soavi, Parma, Italy) for 15 cycles at 750 bar. The temperature in both homogenisation steps raised from 30 °C to 55 °C. Emulsions consisted of 20 % water phase and 80 % oil phase. The oil phase was Eutanol G containing 2 % ethyl cellulose. The water phase was either distilled water (A) or a 2.5 % aqueous hypromellose solution (B).

b) Determination of water content

The water content of the samples was determined via thermo gravimetry using a TG/DTA 320 (Seiko Instruments, Tokyo, Japan). The temperature raised from 25 °C to 120 °C with a rate of 5 °C per minute. 120 °C were kept constant for 45 minutes.

c) Centrifugation

For centrifugation an Allegro 64R Centrifuge (Beckman Coulter, Krefeld, Germany) was used. 10 ml of the emulsion were centrifuged for 2 hours at 800xg at 20 °C. The “brighter” supernatant was separated. The “brighter” supernatant was determined in ml.

d) Particle size measurements

The size distribution of the emulsion droplets was investigated using an image analysis software. Microscopic images of the emulsions were taken with a digital camera. The images were converted using the filter “Trenner” of the program analySIS 3.1 (Soft Imaging System GmbH, Münster, Germany). This filter creates a one pixel wide black line at the borderline of the droplets on a white background. In these transformed images all areas and their corresponding perimeters were determined by a self written software program. The diameter of each area was then calculated based on the area as well as on the perimeter. For circular particles the ratio of these two diameters should be 1. The particle size distribution was analysed using a 200x microscope. The number distribution all diameters of all droplets with a diameter > 0.82 were considered (Fig. 1).

Results and discussion

TG measurements revealed that the phase volume fraction of all emulsions was practically identical (all numerical data shown in Tab. 1). Centrifugation showed substantial differences between the two formulations. Formulation A without a hydrophilic polymer in the inner phase displayed more voluminous supernatant after centrifugation. The effect of the processing method was less pronounced. However, sedimentation was reduced after high pressure homogenisation. As expected, formulation B yielded higher Dv0 values due to the viscosity enhancing effect of the added hypromellose in dispersed aqueous phase. The additional use of a high pressure homogeniser did not further decrease the size of the water droplets.

Conclusions

Based on these results the addition of hypromellose to the inner phase and the use of the high pressure homogeniser is recommended to prepare primary emulsions for a surfactant-free double emulsion system.

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References