INDUSTRIAL SONOMECHANICS™

NANOEMULSIONS FOR FOOD AND BEVERAGES

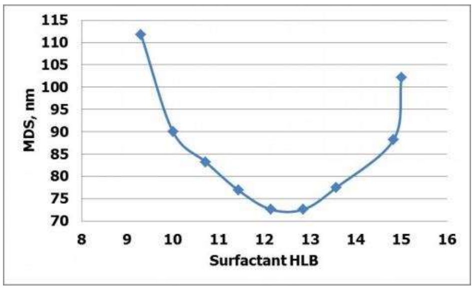
This project consisted of two main steps: 1. lab study aimed at optimizing the formulation and ultrasonic processing conditions for the production of a non-toxic, translucent nanoemulsion of soybean oil in water and 2. transferring the process from lab to pilot scale using BHUT, determining the production rate scale-up factor, and estimating the potential industrial-scale production rate.

The nanoemulsions were produced using ISM's 1200 W bench-scale ultrasonic liquid processor, BSP-1200, operating in the flow-through and batch modes. High-purity soybean oil, Tween 80 and Span 80 were purchased from Sigma-Aldrich, St. Louis, MO. The ingredients were initially pre-mixed using a magnetic stirrer at 500 rpm for 2 min. Mean oil droplet sizes (MDS) were measured using Beckman Coulter N4 Plus Particle Size Analyzer.

LABORATORY-SCALE EXPERIMENTS

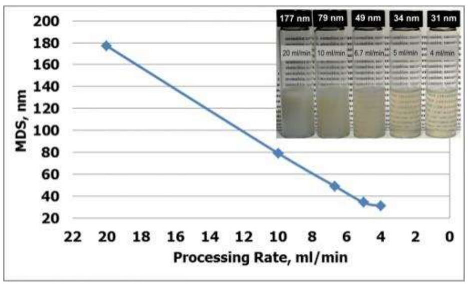
A set of lab experiments was carried out in order to optimize the nanoemulsion's formulation and ultrasonic processing parameters: processing rate, vibration amplitude, power requirement. The requested concentration of oil was 10%. Tween 80 and Span 80 surfactants were chosen due to their Generally Recognized As Safe (GRAS) FDA status. Lab experiments were conducted using the processor fitted with a conventional converging ultrasonic horn (CH), having an output tip diameter of 15 mm. For the nanoemulsion to be translucent, its MDS had to be below 100 nm.

Dependence of MDS on Surfactant HLB



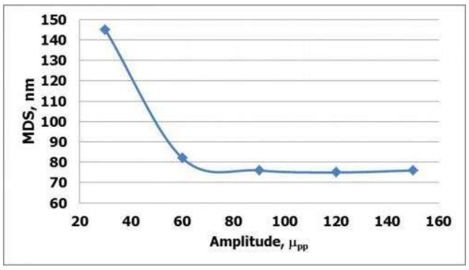
These experiments were conducted in a batch mode using an 80 ml glass beaker inserted into an ice bath. The ultrasonic amplitude was 90 microns. Concentrations of Tween 80 and Span 80 were varied from 10% of Tween 80 and 0% of Span 80 (HLB=15) to 4.67% of Tween 80 and 5.33% of Span 80 (HLB=9.3). Batch sizes were 50 ml and processing times were 5 min (processing rate of 10 ml/min). Minimum MDS was achieved at HLB of about 12 – 13. At these values, the resulting nanoemulsions were sufficiently translucent, with MDS falling to about 73 nm. Based on these results, all further experiments were carried using the following formulation: 10 % soybean oil, 8 % Tween 80, 2 % Span 80 (HLB = 12.86), and 80 % water.

Dependence of MDS on Processing Rate



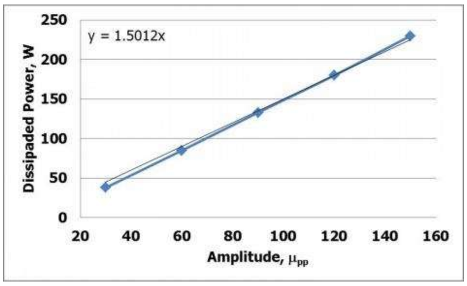
The effect of the processing rate on MDS was investigated next. These experiments were conducted in a flow-through mode using the same CH, operating at the same amplitude, placed in a small reactor chamber. A series of 50 ml samples of the working liquid were re-circulated through the reactor chamber for 2.5 min, 5 min, 7.5 min, 10 min and 12.5 min, resulting in processing rates of 20 ml/min, 10 ml/min, 6.7 ml/min, 5 ml/min and 4 ml/min. MDS decreased as the processing rate was lowered, falling to 31 nm when the rate was 4 ml/min. The nanoemulsion became increasingly translucent as the droplet size decreased. Since the product nanoemulsion was intended by our client to be used in a diluted formulation, the degree of clarity achieved at the processing rate of 10 ml/min was determined to be sufficient.

Dependence of MDS on Ultrasonic Amplitude



The next part of the study was to investigate the effect of the ultrasonic amplitude on MDS. These experiments were conducted in the flow-through mode at the processing rate of 10 ml/min. Based on the results of these experiments, the ultrasonic amplitude of 90 microns was selected for the production of this nanoemulsion. It is important to point out that all presented data is self-consistent: MDS of about 75 nm is consistently obtained at the processing rate of 10 ml/min and the ultrasonic amplitude of 90 microns, for both batch and flow-through setups.

Power Requirement Versus Amplitude



In preparation for the pilot-scale studies, the dependence of power requirement on the ultrasonic amplitude delivered to the liquid was also investigated. These tests were carried out in the flow-through mode. The results show a nearly linear dependence on the amplitude, with a power draw of 130 W at the amplitude of 90 microns.

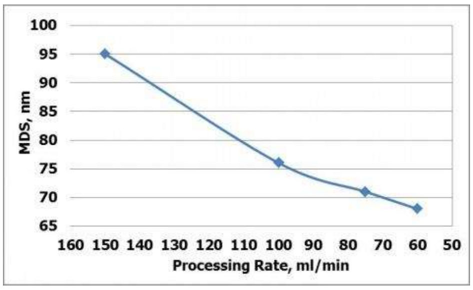
Summary of Lab Experiments' Results

The results of the laboratory experiments were, therefore, as follows: 1. Surfactant HLB = 12.86, 2. Processing rate = 10 ml/min, 3. Ultrasonic amplitude = 90 microns, 4. Power requirement (15 mm horn output diameter) = 130 W.

PILOT-SCALE EXPERIMENTS

An estimation of the scale-up factor was first made based on the following considerations. The common converging horn (CH) with the output tip of 15 mm in diameter creates one active cavitation zone under the tip. The radiating area of this horn is, therefore, 1.8 cm^2. A Half-wave Barbell horn (HBH) with the output tip diameter of 35 mm (used in pilot-scale studies) creates two active cavitation zones, one under and one above its output section. The total radiating area of this horn is, therefore, approximately 19.2 cm^2. Therefore, if the ultrasonic amplitude produced by both horns is similar, the size of the cavitation zone is about 10 times greater for the HBH than for the CH. Therefore, a factor of approximately 10 increase in the processing rate and power requirement was expected.

Dependence of MDS on Processing Rate After Scaleup



Pilot-scale experiments were conducted in the flow-through mode and utilized an HBH, having an output section diameter of 35 mm. The ultrasonic amplitude was maintained at 90 microns. A series of 300 ml samples of the working liquid were re-circulated through the reactor chamber for 2 min, 3 min, 4 min and 5 min, resulting in processing rates of 150 ml/min, 100 ml/min, 75 ml/min, and 60 ml/min. The closest MDS match to the final nanoemulsion obtained during lab experiments was achieved at the processing rate of 100 ml/min. This corresponded to the scale-up factor of about 10, as anticipated. The power draw was approximately 1,150 W, which is consistent with the fact that the same ultrasonic amplitude was now radiated via an area about 10 times greater than in the lab setup.

CONCLUSIONS

Formulation and ultrasonic processing conditions optimization for the production of a non-toxic, translucent nanoemulsion of soybean oil in water for the use in the food industry was carried out. Laboratory-scale results were obtained using ISM's 1200 W bench-scale ultrasonic liquid processor, BSP-1200, equipped with a CH, having the output diameter of 15 mm. The optimized processing rate was 10 ml/min, the ultrasonic amplitude was 90 microns, and the power requirement was 130 W.

The process was then successfully transferred to a pilot-scale flow-through setup using the same processor equipped with an HBH, having the output section diameter of 35 mm. The scale-up factor was approximately 10, resulting in the pilot-scale processing rate of 100 ml/min at the amplitude of 90 microns. The power requirement at this scale was 1,150 W.

By extension, it was concluded that a further scale-up factor of approximately 2.5 can be obtained by using ISM's 3000 W industrial-scale ultrasonic liquid processor, ISP-3000, equipped with an HBH with the output section diameter of 55 mm. This scale-up would result in the production rate of 250 ml/min (15 L/hour) per processor.

It is important to point out that the process described in this document is among the most challenging, as it involves the generation of extremely small droplet sizes. Nanoemulsions requiring MDS on the order of 200 - 300 nm can commonly be produced about 5 - 10 times faster.

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