83c Influence of Process and Formulation Variables on the Rheological Properties of Highly Concentrated Water-in-Oil Emulsions

Oscar A. Alvarez, Veronique Sadtler, Lionel Choplin, Philippe Marchal, and Marie José Stébé Concentrated emulsions are mixtures of two immiscible liquids (water and oil, generally), in which the volume fraction (Φ v) of the dispersed phase is higher than 0.74. The two main emulsion types are direct oil-and-water emulsion (O/W) and reverse water-in-oil emulsion (W/O). If Φ v is higher than 0.90, these emulsions are defined as highly concentrated emulsions.

In the preparation of these emulsions, the process and formulation variables have important coupled effects that determine the final rheological characteristics. There are two main processes for the preparation of these products: -1) Semi-batch process: it consist in two steps, the first one is the incorporation of aqueous phase into the surfactant-oil mixture under given mixing conditions, and the second one is the homogenization step in the same equipment (with possible modifications in the mixing conditions); -2) Batch process: all the components are mixed together and the final concentrated emulsions is obtained through a phase inversion process.

In this paper, we will consider the semi-batch process, and study the influence of process and formulation variables on rheological characteristics of highly concentrated water-in-oil emulsions.

The experimental set-up consists in a mixing vessel equipped with a 45° pitched blade turbine, whose diameter is 71% that of the mixing vessel. The turbine is positioned at the oil phase free surface (before the aqueous phase incorporation).

The studied mixing variables are: The agitation speed (N) during the incorporation (between 150 and 2000rpm), the aqueous phase mass flow rate (Qw) (between 2 and 20g/min), the homogenization speed (N') (between 150 and 2000rpm) and the homogenization time (th) (between 1 and 10min).

The ingredients are: n-dodecane, deionized water and sorbitan monooleate (Span 80). The studied formulation variables are: The electrolyte concentration (NaCl) in aqueous phase (0-5wt%), the surfactant/oil ratio (0.20-0.45) and the volume fraction of the dispersed phase (0.88-90.5).

The Rheology of the highly concentrated water-in-oil emulsions is evaluated, one hour after their preparation at room temperature, through mechanical spectrometry in the linear viscoelastic domain. These emulsions generally have a strong gel-like behavior with a storage modulus (G') much higher than the loss modulus (G'), and essentially independent of the frequency (ω).

All results confirmed that the rheological characteristics of highly concentrated water-in-oil emulsions are determined by strong coupling effect between process (mixing) conditions and formulation variables. Among all variables, the agitation speed as well as the homogenization speed play a critical role.