



## On-line light backscattering tracking of the transitional phase inversion of emulsions<sup>☆</sup>

Aldo Pizzino<sup>a,b</sup>, Marianne Catté<sup>a</sup>, Elisabeth Van Hecke<sup>a</sup>, Jean-Louis Salager<sup>b</sup>, Jean-Marie Aubry<sup>a,\*</sup>

<sup>a</sup> LCOM UMR CNRS 8009 Equipe "Oxydation et Physico-Chimie de la Formulation" – USTL, Cité Scientifique, 59655 Villeneuve d'Ascq, France

<sup>b</sup> Laboratorio FIRP, Ingeniería Química, Universidad de Los Andes, Mérida, Venezuela

### ARTICLE INFO

#### Article history:

Received 30 January 2008

Accepted 26 May 2008

Available online 3 June 2008

#### Keywords:

Emulsions

Phase inversion

Light backscattering

### ABSTRACT

A surfactant/oil/water system (Brij 30/decane/brine) is stirred while continuously changing the temperature in order to induce the swap of the emulsion morphology from O/W to W/O or vice versa. The transitional phase inversion is detected by monitoring both the electrical conductivity and the light backscattering with a Turbiscan (on line) equipment. The optical technique pinpoints the inversion and exhibits several advantages over the conductivity measurement: (i) it does not require any addition of electrolyte in the aqueous phase, (ii) it provides some information on the drop size, and (iii) it works with both O/W and W/O morphologies whereas conductimetry is blind for the latter case.

© 2008 Elsevier B.V. All rights reserved.

## 1. Introduction

### 1.1. General background

Emulsions are thermodynamically instable dispersions of two immiscible liquids, most often stabilized by surfactants. The emulsion type, so-called oil-in-water (O/W) or water-in-oil (W/O) morphology essentially depends on the physicochemical formulation.

For a water-to-oil ratio (WOR) not far from unity, Bancroft's rule and similar more sophisticated prediction yardsticks [1–6] indicate that systems in which the surfactant hydrophilic (respectively lipophilic) affinity dominates, results in an O/W (respectively W/O) emulsion upon stirring.

For polyethoxylated nonionic surfactant systems, an increase in temperature tends to trim down the interactions between the polyether chain and the water molecules. Consequently it turns the surfactant less hydrophilic, and triggers the emulsion inversion from O/W to W/O morphology at the so-called phase inversion temperature PIT [3]. The opposite inversion from W/O to O/W takes place upon cooling at or close to the PIT.

Emulsion inversion is a way to produce emulsions with fine droplets at a low energy expense even with viscous phases, and the

so-called PIT emulsification-method [7] has been used for decades for such purpose.

In practice, it is important to monitor morphology changes taking place close to or at the inversion, in order to control the manufacture of nutrient emulsions for fermentation processes, epoxy or polyurethane waterborne paints, silicone putties. This monitoring is particularly critical when the evolution of the system exhibits a complex behaviour such as three phase or multiple emulsion occurrence, which are often impossible to follow by visual observation because of the fast evolution taking place near inversion.

The classical way to detect the emulsion inversion consists in measuring the conductivity of the emulsion [8], which is typically high for the O/W morphology and low for the W/O one, particularly if the aqueous phase contains some electrolytes as often the case in practice. However, the conductimetry method has some drawbacks. First, to get a clear-cut detection of the inversion, it requires the aqueous phase to be conductive, i.e. to contain some electrolytes. Secondly, the variation of conductivity likely to indicate some morphology change prior to inversion, only supplies information in the O/W → W/O direction of change, i.e. on the eventuality of multiple w/O/W morphology. It is essentially ineffective to detect multiple emulsions of the o/W/O type. Finally, the conductivity does not give any valuable information about the drop size, which is often an important issue in practical cases.

As the temperature approaches the PIT from both sides, the interfacial tension decreases, thus inducing the formation of smaller droplets. However, the coalescence rate increases, thus tending to produce larger drops. Because of these opposite effects as the PIT is approached, a continuously stirred emulsion exhibits a

<sup>☆</sup> Paper from the "Frontiers in Formulation Science (Formula V-2007)" Special Issue.

\* Corresponding authors.

E-mail addresses: [pizzino@ula.ve](mailto:pizzino@ula.ve) (A. Pizzino), [salager@ula.ve](mailto:salager@ula.ve) (J.-L. Salager), [jean-marie.aubry@univ-lille1.fr](mailto:jean-marie.aubry@univ-lille1.fr) (J.-M. Aubry).