

Light Backscattering as an Indirect Method for Detecting Emulsion Inversion

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Many phenomena take place during different types of emulsion inversions, particularly a change in interface curvature and drop size, which could be detected by backward light scattering. Monitoring the backscattering signal allows us to detect the emulsion inversion in three main cases, one transitional and two catastrophic types. The backscattering data could give some clue as to emulsion morphology, which is not available from conductivity measurements.

Introduction

Emulsion inversion is an important phenomenon because it is a way to produce emulsions with characteristics such as fine drops at a low energy level¹ or with an extremely viscous internal phase² when a very energetic method is not suitable.³ There are several inversion mechanisms,⁴ and most of them depend on many variables.⁵ In practice, it is extremely important to monitor morphology changes, particularly those related to the intermediate stage (e.g., three phase or multiple emulsion occurrence), which are often impossible to follow by visual (microscopic) means, particularly because of the fast evolution taking place near inversion.

The classical method of monitoring inversion is to measure the emulsion conductivity, which is a way to estimate the external or continuous phase proportion when it is an aqueous phase. In most applications, the aqueous phase contains some electrolyte at a concentration large enough to provide a high enough conductivity. The monitoring of viscosity has recently been proposed as a substitute or complementary measurement, and it offers promising results.^{6–8} However, the emulsion viscosity depends on many variables other than the emulsion type, and the interpretation is not always clear cut, particularly with viscous fluids and high internal phase content or with multimodal emulsions.^{9,10} Drop size variations also correlate with inversion in some cases, such as transitional inversion, though not in a

simple way.^{6,11} But in any case, the drop size and distribution depend on many other variables, particularly formulation and composition.¹²

Because the structure of the emulsion and the interfacial curvature drastically change during inversion, it is believed that an optical signal related to curvature (e.g., some reflectivity measurement) should allow us to detect the inversion.¹³ This is sustained by the fact that the curvature swaps at inversion, at least in some specific cases, and therefore goes through a near-zero value. This letter reports the detection of emulsion-phase inversion by means of light backscattering measurements.

Experimental Section

The two main kinds of emulsion dynamic inversion (i.e., transitional and catastrophic protocols¹⁴) are carried out in a conventional way,^{15,16} with continuous monitoring of the backscattering through an available commercial apparatus (i.e., Turbiscan on-line model (TOL) from Formulation, France). This equipment produces a backscattering signal that is a measurement of the near-infrared ($\lambda = 850$ nm) light diffused at an angle close of to 135° . The backscattering percentage is reported with respect to a reference suspension of $0.3 \mu\text{m}$ latex particles in silicone oil. The equipment has been routinely used to evaluate the variation of particle size in suspensions and emulsions.¹⁷

The emulsion sample to be evaluated is circulated with a peristaltic pump through the cylindrical cell of the apparatus in a closed circuit connected to the emulsion vessel.¹⁸ The conductivity is measured with a Radiometer Analytical conductimeter with a platinized platinum cell located inside the emulsion vessel. The emulsion vessel is a jacketed glass cylindrical tank continuously stirred with a Rushton turbine rotating at approximately 1000 rpm. The changes in water

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