



Temperature effect on the inter-micellar collision and maximum packaging volume fraction in water/AOT/isooctane micro-emulsions



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ABSTRACT

We have studied the viscosity behaviour of water/AOT/isooctane micro-emulsions as a function of the volume fraction of the dispersed phase over a temperature range from the (298.15 to 328.15) K. For all the studied temperature range, a sharp increase of the viscosities is observed when the droplets concentration was varied. Several equations based on hard sphere model were examined to explain the behaviours of micro-emulsions under temperature and concentration effects. According to these equations, the shape factor and the inter-particle interaction parameters were found to be dependent on temperature which is in contradiction with experimental results reported in the literature. A modified Vand equation, taking into account the inter-particle collision time, is used to interpret the results obtained. This deviation is attributed to the aggregation of the droplets which becomes important by increasing temperature. The maximum packaging volume fraction of particles Φ_{dm} and the intrinsic viscosity $[\eta]$ were determined according to the Krieger and Dougherty equation through the temperature range studied. These two parameters were shown to be dependent on temperature but their product was found to be constant and close to 2 as reported in theory.

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1. Introduction

Micro-emulsions made of water; surfactant and oil are widely studied, because of their importance in various industrial applications such as food digestion [1], pharmaceutical industry [2], biochemical field [3], drug delivery [4,5]. The reverse micelles [6] are composed of surfactant that around a nano-sized aqueous core in oil. The structure and the transport properties of these systems have been studied experimentally by using NMR self-diffusion [7–10], dynamic light scattering (DLS) [11], small-angle neutron scattering (SANS) [12], small-angle X-ray scattering (SAXS) [13], electrical conductivity [14–16] and viscosity measurements [17–19]. By increasing temperature and/or the volume fraction of droplets, the viscosity of micro-emulsion exhibits a sharp increase, which is the signature of a percolation phenomenon [20,21]. In micro-emulsion, percolation manifests itself also by the increase of the electrical conductivity [22–24] and/or the dielectric constant [25,26]. Percolation in micro-emulsions [27–29] is associated with droplet clustering and internal structure changes. This

phenomenon has been quantitatively described in terms of a percolation transition theory [30,31] with scaling type equations.

When the viscosities of dispersed and continuous phases are close to each other, percolation of the viscosity cannot be verified quantitatively [32–34]. However, many equations [35–45] were proposed to describe the dependence of the viscosity on the volume fraction of the dispersed phase. These equations were deduced from the hard sphere model which is the most used by scientists to interpret the viscosity behaviour of micro-emulsion versus the volume fraction of the dispersed phase.

In this work we examine some of these equations to study the viscosity behaviours of the AOT (sodium bis(2-ethylhexyl) sulfosuccinate)/water/isooctane (2,2,4-trimethylpentane).

First, the viscosity of the micro-emulsions was measured by a rotational co-cylindrical viscometer as a function of the volume fraction of the dispersed phase over the $T=(298.15$ to $328.15)$ K temperature range. The results obtained were analysed and discussed according to the Mooney and modified Vand equations [35]. The last equation takes into account the collision time constant between droplets. In the second part of this work, the Krieger and Dougherty equation was used to determine the effect of temperature on the maximum packaging of the volume fraction droplets and on their geometrical properties.

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2. Experimental

2.1. Sample preparation

AOT (sodium bis(2-ethylhexyl) sulfocinate) (\geq mass fraction purity 0.96) and isooctane (\geq 0.99) were respectively purchased from Fluka (Switzerland) and Sigma–Aldrich (U.S.A.) respectively and used without further purification as indicated in [table 1](#). Deionised water was used to prepare the various solutions.

First, a suitable mass of AOT was dissolved in isooctane in order to obtain a volume fraction ratio, $\Phi_m = \frac{V_{AOT}}{V_{iso}}$, where V_{AOT} and V_{iso} are respectively the volumes of AOT and isooctane. Then a known amount of water was added to the solution in order to obtain a calculated molar ratio of water to surfactant concentration W_0 . The mass is determined by using a digital electronic balance (OHUAS AS 200) with a resolution of 10^{-4} g. The relative uncertainty in mole fractions is 1%. The ternary system is completely described by the calculus of the mole fraction of water and AOT denoted respectively by x_w and x_{AOT} .

The volume fraction of the dispersed phase Φ_d was deduced from the following equation:

$$\Phi_d = \frac{V_w + V_{AOT}}{V_w + V_{AOT} + V_{iso}}, \quad (1)$$

where V_w is the volume of water. equation (1) can be expressed as a function of α , β , x_{AOT} , x_w and x_{iso} :

$$\Phi_d = \frac{1}{1 + \frac{1}{\alpha x_{iso} + \beta x_w}}, \quad \alpha = \frac{V_{m,AOT}}{V_{m,iso}} \text{ and } \beta = \frac{V_{m,w}}{V_{m,iso}} \quad (2)$$

where $V_{m,w}$, $V_{m,iso}$ and $V_{m,AOT}$ are respectively the molar volume of water, isooctane and AOT. The mole fraction of isooctane x_{iso} is deduced from the mole fraction of water and AOT. In this study, the volume fraction of the dispersed phase was ranged from 0.06 to 0.36.

2.2. Rotational concentric cylinder viscometer

The viscosity measurements were performed with a Brookfield (U.S.A.) LVDV-II + Pro viscometer. The apparatus measures the torque required to rotate a cylindrical spindle (rotor) in the sample contained in a cylindrical geometry (stator). The used cylindrical spindle is adapted to characterize samples of low viscosities and is driven by a motor through a calibrated spring. Temperature controlled water was pumped through the fixed cylinder by a Hakke K. (U.S.A.) circulating bath. For this apparatus, the shear rate $\dot{\gamma}$ at the

surface of cylindrical spindle and shear stress σ are given by the following equations [46,47]:

$$\dot{\gamma} = \frac{2\omega R_c^2}{(R_c^2 - R_b^2)} (s^{-1}) \quad (3)$$

$$\sigma = \frac{M}{2\pi R_b^2 L} (\text{dPa}), \quad (4)$$

where R_c and R_b are respectively, the container and spindle radius, ω is the angular velocity of the spindle ($\text{rad} \cdot \text{s}^{-1}$), M is the torque ($\text{dyn} \cdot \text{cm}$) and L is the spindle's length. The viscometer was calibrated by using standard fluids as it reported in our earlier paper [48]. The dynamic viscosities were measured with a relative uncertainty less than 3%. Viscosity measurements were performed between (150 and 250) s^{-1} . In this range of shear rate, the water/AOT/isooctane system behaves as Newtonian fluid whatever the temperature [48,49].

3. Results and discussion

The experimental values of viscosity η for the AOT/water/isooctane micro-emulsion at temperatures $T = (298.15, 303.15, 308.15 \text{ and } 313.15) \text{ K}$ as a function of the mole fraction of water x_w and AOT x_{AOT} have been reported in [table 2](#). The calculated values of the viscosity η_c versus Φ_d at each studied temperature were obtained from the following equation:

$$\eta_c = \sum a_k \Phi_d^k, \quad (5)$$

where k is the number fitted parameters and a_k represents the coefficients. The value of Φ_d was calculated according equation (2). Adjustable parameters of a_k were evaluated by last square method, and the values of the standard deviation s were obtained according to equation (6).

$$s = \left(\frac{\sum_{i=1}^N (\eta_i - \eta_{c,i})^2}{N - 1} \right)^{1/2}, \quad (6)$$

where η_i , $\eta_{c,i}$ and N are, respectively, the experimental, the calculated data and the number of experimental points. Adjustable parameters of equation (5) and standard deviation s values are presented in [table 3](#).

We report in [figures 1a and 1b](#) the variation of the Newtonian viscosity versus the volume fraction of the dispersed phase Φ_d over the (298.15 to 328.15) K temperature range. All the curves, [figures 1a and 1b](#), are almost superposed until $\Phi_d = 0.25$ from which an

TABLE 1
Detailed description of chemical compounds used.

Chemical name	Formula	Structural Formula	Supplier	Molar mass (g mol ⁻¹)	Purity	Purification method
Sodium bis(2-ethylhexyl) sulfosuccinate	C ₂₀ H ₃₇ NaO ₇ S		Fluka	444.56	\geq 96%	-
2,2,4-triméthylpentane	C ₈ H ₁₈		Sigma–Aldrich	114.23	99%	-

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