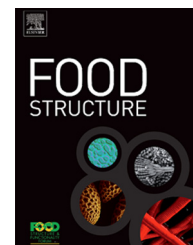


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# Self assembly and rheology of emulsions-mimicking food emulsion rheology

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## ABSTRACT

An investigation has been carried out to understand the microstructure of amino based surfactant (cocoyl glutamic acid, CGT) and viscoelastic properties of emulsions mimicking the food emulsion rheology. The structural modulation of the surfactant aggregates depends on the degree of neutralization, temperature and oil concentration. Phase behaviour of the CGT surfactant system behaves similar to a short chain ( $C_{12}EO_8$ ) (Aramaki & Khalid, 2004) or a long chain ( $C_{12}EO_{25}$ ) (Rodriguez, Shigeta, & Kunieda, 2000) non-ionic surfactant systems, depending on the degree of neutralization. Krafft point of the CGT surfactant has been decreased with the addition of triethanolamine (TEA) and temperature. In presence of oil liquid crystal structure has been modified, which is due to the changing of the surfactant layer curvature (Kunieda, Umizu, & Aramaki, 2000). It is interesting to note that the  $I_1$  phase solubilizes large amount of oil. High viscosity with large amount of oil emulsions were obtained when the degree of neutralization,  $n = 1.2$ . The decrease in viscosity with the oil content in the  $O/I_1$  emulsions is ascribed to the lower volume fraction of the continuous phase ( $I_1$  phase).

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## 1. Introduction

In food industry, most of the products are somehow emulsion type, e.g. milk, cheese, butter, mayonnaise, gel (Dave, Sharma, & Muthukumarappan, 2003; Mezzenga, 2007; Tipettes & Martini, 2012). Generally emulsions consist of two immiscible liquids where one phase is dispersed phase and another phase is continuous phase. The emulsion stability could be tuned by using different types of continuous phase (Rodriguez & Lazzari, 2008; Rousseau & Hodge, 2005; Rousseau, 2013) or dispersed phase (Pons, Erra, Solans, Ravey, & Stebe, 1993). Among the continuous phases, liquid crystal promisingly increases the emulsion stability due to its high viscosity (Alam & Aramaki, 2008; Friberg, Mandell, & Larsson, 1969; Kunieda, Tanimoto, Shigeta, & Rodriguez, 2001; Mele, Murgia, &

Monduzzi, 2003; Mosca, Murgia, Ceglie, Monduzzi, & Ambrosone, 2006; Rodriguez, Roman, & Kunieda, 2004), as a result, a matter of interest to the researchers. Recently, lyotropic liquid crystals (LC) are emerging as an interesting subject due to their microstructure and viscoelastic properties, which make them useful in the field of food, cosmetics, fertilizer, encapsulation and drug delivery (Farkas, Kiss, & Zelko, 2007; Krog, 1997; Larrson, 2000; Mariani, Rustichelli, Saturni, & Cardone, 1999).

The most common liquid crystals reported in the literature are cubic (I), hexagonal (H), bicontinuous (V) and lamella ( $L_\alpha$ ) (Hyde, Andersson, Ericsson, & Larsson, 1984; Mitchell, Tiddy, Waring, Bostock, & McDonald, 1983). In contrary to conventional gel emulsions or highly concentrated emulsions (HCE), LC based emulsions are formed in the region of ternary phase diagram in which excess oil or water phase coexists with the LC (Alam, 2009; Alam & Aramaki, 2009; Alam, Shrestha, &

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Aramaki, 2009; Rodriguez et al., 2000, 2007; Takahashi, Sugiyama, Watanabe, & Aramaki, 2011). LC based emulsions show high viscosity even at low volume fraction of dispersed phase (<50%) (Kunieda et al., 2001; Rodriguez et al., 2004), where HCE shows high viscosity only when the dispersed phase exceeds the volume fraction 74% (Pons et al., 1993; Rajinder, 2006). Among the liquid crystals, the cubic phase ( $I_1$ ) has the highest viscosity (Rodriguez, Acharya, Aramaki, & Kunieda, 2005) and thus gives opportunity to stabilize O/ $I_1$  emulsions (Alam et al., 2009; Alam, Sugiyama, Watanabe, & Aramaki, 2010; Rodriguez et al., 2000). It should be noted that the LC based emulsions were mainly reported in the literature based on non-ionic (ethoxylated) fatty alcohol (Alam et al., 2009, 2010; Rodriguez et al., 2000) tri-block copolymers (May, Aramaki, & Gutiérrez, 2011; Rodriguez et al., 2007) and silicon surfactant (Uddin et al., 2001). Recently, we have reported the LC based emulsion in a sugar based surfactant system (Alam, Hoshida, Arima, & Aramaki, 2012). We described a simple way to manipulate the thermal behaviour of the liquid crystals (Garti, Aserin, & Fanun, 2000; Nilsson, Soderman, & Hansson, 1998; Sadtler, Guely, Marchal, & Choplin, 2004). Rheological behaviour of the LC and related emulsions were also discussed.

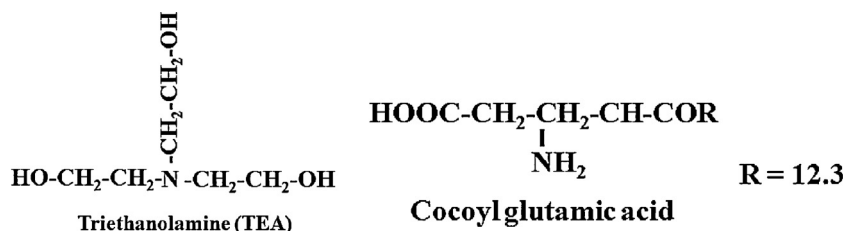
Physical properties of the amino based surfactant show low stimulus (temperature, pH) responsiveness and thus promising for the field of cosmetic, foods, pharmaceuticals applications. Although amino based surfactant has high potential for industrial application, few reports are found in the literature (Aramaki, Iemoto, Ikeda, & Saito, 2010; Shrestha, Shrestha, & Aramaki, 2007; Yamashita, Kunieda, Oshimura, & Sakamoto, 2007). One drawback of this surfactant is high Krafft point, which makes it difficult to formulate. However, an easy way to control the Krafft point of the surfactant is to change the degree of neutralization without changing the chain length of surfactant (Aramaki et al., 2010; Shrestha et al., 2007). To our knowledge, this could be the first article using amino acid based surfactant to study liquid crystal based emulsions to understand the physical properties of food emulsions under shear as well as an interesting matter of colloid science.

In the present contribution, we described the self assembly of an amino based surfactant in water and the rheological properties of the emulsion mimicking the rheology of the food emulsion.

## 2. Experimental

### 2.1. Materials

An amino acid based anionic surfactant, cocoyl glutamic acid (termed as CGT) was received from Ajinomoto Co., Japan.



Scheme 1 – Structure of triethanolamine (TEA) and cocoyl glutamic acid (CGT).

Average carbon number in the cocoyl chain is about 12.3. Triethanolamine (TEA) (purity > 99%) was purchased from Tokyo Kasei Kogyo Co., Japan. Decane was purchased from Tokyo Chemical Industry. Millipore filtered water was used as a solvent. Structure of the TEA and CGT is shown in Scheme 1.

### 2.2. Methods

#### 2.2.1. Determination of the phase diagram

The CGT surfactant was first neutralized by TEA at different molar ratio from 0 to 2.0 and is denoted as CGT- $n$ ;  $n$  indicates the degree of neutralization defined as (mol of TEA)/(mol of CGT). The samples were prepared individually by weighing the appropriate amounts of components in glass ampoules. The ampoules were closed with a plastic cap and sealed with para film and tap. The sample mixtures were homogenized by heating and vortex mixing. The samples were kept in a temperature-controlled bath at 25 °C for several days to bring the system in equilibrium. The phase equilibrium was determined by visual inspection and a cross polarizer. In general, transparent, highly viscous, isotropic and birefringent phases are cubic ( $I_1$ ) and hexagonal ( $H_1$ ) phase, respectively; clear, low viscous, isotropic and birefringent phases are micellar ( $W_m$ ) and lamellar ( $L_\alpha$ ) phase, respectively; all turbid phases are two-phase. Liquid crystal structure was determined by small angle X-ray scattering technique.

#### 2.2.2. Characterization of the liquid crystals

Small angle X-ray scattering (SAXS) measurements were performed to characterize the liquid crystal structure. For the SAXS measurements, a SAXSess camera (Anton Paar) attached to a PW3830 laboratory X-ray generator (PANalytical) with a long fine focus sealed glass X-ray tube (Cu  $K\alpha$ , wavelength of 0.1542 nm) (PANalytical) was used. The sample temperature was controlled with a thermostatted sample holder unit (TCS 120, Anton Paar). The apparatus was operated at 40 kV and 50 mA. The structures of the liquid crystal were identified from the SAXS peak ratios. For example, the SAXS peak ratios for the  $L_\alpha$  and  $H_1$  phases are 1:2:3 and 1:1.73:2 respectively (Kunieda, Ozawa, & Huang, 1998).

#### 2.2.3. Preparation (formation) of liquid crystals based emulsions

All components were added together at a specific composition, melted at 80 °C and instantly mixed with a vortex mixer in open air-cooling. Highly viscous emulsion was observed that did not flow when the sample tube was turned upside down.

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