

# Use of polysaccharide based surfactants to stabilize organically modified clay particles aqueous dispersion



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## ARTICLE INFO

### Article history:

Received 1 August 2012

Received in revised form 17 October 2012

Accepted 15 January 2013

Available online 4 February 2013

### Keywords:

Organically modified clay particles

Inutec SP1 surfactant

Electrophoretic mobility

DLS

Contact angle

Dispersion stability

## ABSTRACT

Pure as well as organically modified clay minerals are widely applied particles in different research areas. For the incorporation of hydrophobic organically modified clay particles into the hydrogel matrix, a stable aqueous dispersion must be prepared. In this article we report on the stabilization of aqueous dispersions of hydrophobic organically modified clay particles by using a non-ionic polysaccharide-based surfactant system-Inutec SP1 (based on chicory inulin). Different concentrations of surfactants were tested. Properties of the particulate surfactant-stabilized aqueous colloidal system were determined by electrophoretic mobility and dynamic light scattering measurements. Determination of contact angles gave us insight into the particles' surface interaction ability with water and also some information regarding the conformation of adsorbed surfactant molecules on the particle surface. By using Inutec SP1, the wettability of clay particles was improved, particle size was reduced and consequently, enhancement of their dispersion ability in water-based systems was observed.

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## 1. Introduction

Various types of montmorillonite (MMT) and closely related minerals are the most important and widely used phyllosilicate fillers in polymer composites (e.g. Fedulo et al., 2007; Le Baron, Wang, & Pinnavaia, 1999; Sfligoj Smole et al., 2009). Their specific characteristics like large active surface area, ion exchange capacity and ability to swell remarkably in water improves functional properties and mechanical properties of nano-composites (Ray & Okamoto, 2003). By organic modification clay minerals obtain an organophilic character and the capacity to adsorb heavy metals (Bhattacharyya & Gupta, 2008), organic dyes (Baskaralingam, Pulikes, Elango, Ramamurthi, & Sivanesan, 2006; Koswojo et al., 2010; Kurečič & Sfligoj, 2012; Liu & Zhang, 2007; Özcan, Ömeroğlu, Erdoğan, & Özcan, 2007; Yang, Han, Fan, & Uqbolue, 2005), pesticides (Groisman, Rav-Acha, Gerstl, & Mingelgrin, 2004) and herbicides (Hermosin, Celis, Facenda, Carrizosa, Ortega-Calvo, & Cornejo, 2006). Since organically modified clay particles are good adsorbents for pollutants, these particles can be incorporated as active fillers also in composite hydrogels through hydrogel in situ polymerization. Kurečič & Sfligoj (2012) has reported on the

synthesis of PNIPAM/O-MMT nanocomposite hydrogels for adsorption of acid dyes.

Polymer formation takes place from monomer aqueous solution containing dispersed particles. Polymerization occurs between the silicate layers after their swelling by the liquid monomer or monomer solution (Kiliaris & Papaspyrides, 2010). Therefore, the most decisive step in formation of hydrogel composites with the desired properties is the preparation of stable particle dispersions prior to polymerization (Landry, Riedl, & Blanchet, 2008).

Organically modified clay particles are hydrophobic and are therefore difficult or even impossible to disperse in aqueous solution. The most common strategy for tackling this issue is to introduce stabilizers into the system. Stabilizer quality is based on their ability to provide wetting of particles' surface and to offer a barrier to prevent particles from agglomeration. (Gabrielli, Cantale, & Guarini, 1996).

Polymeric surfactants are essential materials for preparation of many disperse systems (Tadros, 2009). As an answer to the growing demand for products made from renewable resources, there has been a considerable interest in development of polysaccharide based surfactants (Kjellin & Johansson, 2010; Tadros & Tharwat, 2005).

The present work describes the use of polysaccharide based surfactants for preparation of stable aqueous dispersions of organically modified clay particles. The polymeric surfactant, based on the polysaccharide-inulin, which is hydrophobized by grafting several alkyl groups (B) onto the linear polyfructose chain (A), is

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commercially available as Inutec SP1 from Orafti, Belgium. It is mainly used for stabilization of oil-in-water emulsions (Tadros, 2008). Due to the large carbohydrate part in the molecule, the surfactant is biodegradable and it is not toxic (Kjellin & Johansson, 2010). In our research the Inutec SP1 was used for enhancement of clay particles wettability and consequently, to improve particles dispersion ability in water-based systems. Different concentrations of surfactants were tested. Properties of stabilized aqueous colloidal systems were followed by zeta potential, dynamic light scattering measurements, contact angle determination and turbidity observation. Knowledge about electrokinetic and scattering data and established hydrophobic/hydrophilic character with analysis of particles sedimentation behavior, are important for future formation of hydrogels containing organically modified clay particles and subsequent properties and functionality of these composites.

## 2. Experimental

### 2.1. Materials

Inutec SP1 was supplied by Orafti Non-food (Tienen, Belgium). As mentioned above Inutec SP1 is a copolymer based on polyfructose backbone with grafted alkyl groups. Its average molecular mass is about  $4500 \text{ g mol}^{-1}$  (Esquena et al., 2003) and degree of polymerization for linear polyfructose backbone is above 23 monomer units (Nedyalkov, Alexandrova, Platikanov, Levecké, & Tadros, 2010).

Nanofil 8, an organically modified montmorillonite (Fig. 1), was purchased from Süd-Chemie (Germany). O-MMT particles were organically modified by distearyldimethylammonium chloride (DSDMAC).

### 2.2. Methods

#### 2.2.1. Preparation of Inutec SP1 solutions

A water solution of 1 g/L Inutec SP1 surfactant in deionized water was prepared. This stock solution was used further on for preparation of diluted surfactant solutions. The Inutec SP1 surfactant concentrations used were in the range from 0.01 to 1 g/L.

#### 2.2.2. Preparation of aqueous based O-MMT particle dispersions in surfactant solutions

O-MMT particles with the concentration 1 g/L were dispersed in Inutec SP1 solutions of different concentrations using homogenizer

Ultraturax (IKA) for 10 min at 10,000/min and subsequently exposed to ultrasound for 20 min at an amplitude 50% using an ultrasound probe (Vibra-cell, Sonics). Dispersed samples were centrifuged and sedimented, in order to remove all surplus surfactant, using a Centric 322A centrifuge (Tehtnica) for 20 min (4500/min). The supernatant was removed and replaced with fresh water. The particles were redispersed in water using the ultrasound bath (Transsonic 820/H, Elma) for 30 min. This procedure was repeated three times and finally O-MMT particles modified with Inutec SP1 dispersed in water were obtained.

### 2.3. Analytical procedures

#### 2.3.1. Determination of critical micelle concentration (CMC)

Surfactant's critical micelle concentration CMC was determined by measuring the surface tension ( $\gamma$ ) of water upon addition of Inutec SP1. Krüss tensiometer K12 was used, employing a Wilhelmy plate method with a platinum plate which has been cleaned through heating to red hot. The method is described in detail (Peršič et al., 2011). Inutec SP1 solution with a concentration of 2 g/L was dosed into 30 ml of water. Surface tension was constantly determined over a range of addition steps 0.01 g/L of Inutec SP1 solution. Surfactant's critical micelle concentration was graphically determined from the plot of surface tension versus concentration of Inutec SP1 by modeling two straight lines of solution properties below and above the CMC which intersect each other. The intersection represents the CMC of Inutec SP1 surfactant.

#### 2.3.2. Surface morphology observations by SEM

Surface morphology of particles prior and after adsorption of Inutec SP1 was studied by a scanning electron microscope (SEM) FE-SEM-ZEISS Gemini Supra 35 VP. Samples were prepared by deposition of a drop of O-MMT dispersion on an adhesive carbon tape, which was placed on a sample holder. Samples were dried in a vacuum oven at  $40^\circ\text{C}$ .

#### 2.3.3. Particle size determination

To determine the particle size distribution, DLS measurements were carried out. The analysis was performed using a ALV 5000 digital correlation spectrometer (Brookhaven Zeta Plus). A laser beam, with the wavelength of 532 nm and maximum output power 5 W, was used to cover the wide size range involved. Measurements were carried out at a scattering angle of  $90^\circ$  and at the constant temperature of  $25^\circ\text{C}$ . The particle size is measured from the autocorrelation function of the intensity fluctuation of scattered lights. From this, one obtains the diffusion coefficient,  $D$ , which is used to calculate the particle hydrodynamic radius,  $R_H$  using the Stokes–Einstein equation (Brar & Verma, 2011):

$$D = \frac{kT}{6\pi\eta R_H} \quad (1)$$

where  $k$  is the Boltzmann constant,  $T$  is the absolute temperature, and  $\eta$  is the viscosity of the continuous phase. The value  $\eta$  for water at  $25^\circ\text{C}$  was  $8.9 \times 10^{-4} \text{ Pa}$ .

For the purpose of measurements samples were diluted using 0.001 M KCl.

#### 2.3.4. Zeta potential determination

The zeta potential of the dispersed particles was determined with a Zeta meter (Brookhaven Zeta Plus), where the electrophoretic mobility of particles in dilute suspensions was measured using Zeta PALS (Phase Analysis Light Scattering) method at  $25^\circ\text{C}$  and then converted to zeta potential ( $\zeta$ ) by utilizing the

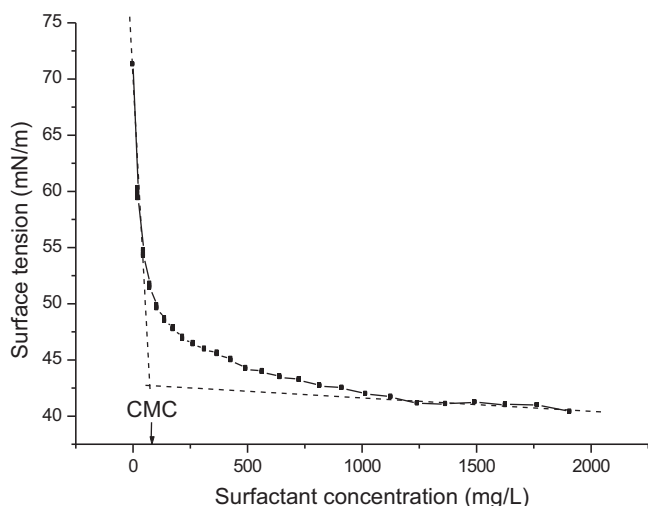


Fig. 1. Plot of water surface tension vs. Inutec SP1 concentration.

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