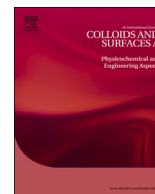




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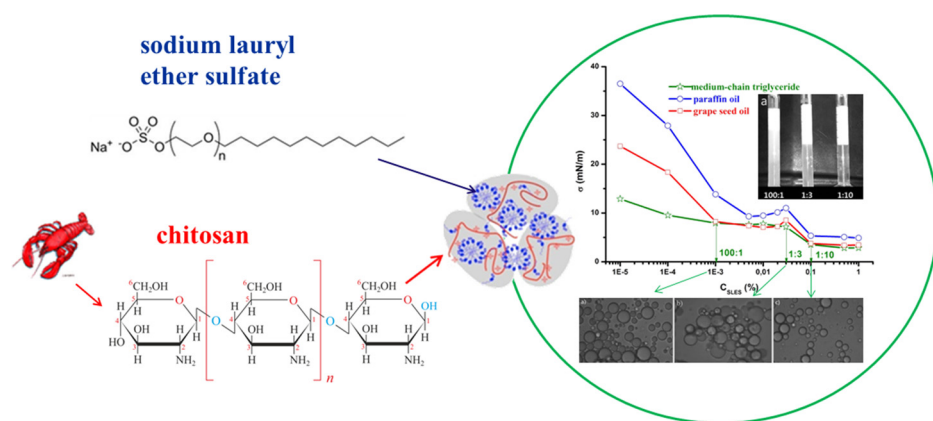
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Interfacial and emulsifying properties of chitosan/sodium lauryl ether sulfate system

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GRAPHICAL ABSTRACT



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ABSTRACT

Many pharmaceutical, cosmetic and chemical products exist in form of emulsions. A common problem with emulsions is their instability. Interaction between polymer and surfactant could change the adsorption layer around the oil droplets in emulsion which affects their stability. In order to understand the stabilization mechanism, the interface of oil/water systems that contained mixtures of chitosan (Ch), cationic polyelectrolyte and oppositely charged anionic surfactant, sodium lauryl ether sulfate (SLES) was studied by measuring the interfacial tension. Considering the fact that the properties of the oil phase influence the adsorption process, three different types of oil were investigated: medium-chain triglycerides (semi-synthetic oil), paraffin oil (mineral oil) and natural oil obtained from the grape seed. Based on the results of the measurements of interfacial tension, system medium-chain triglycerides/water was selected, for obtaining the 20% oil-in-water (O/W) emulsions. Emulsions were stabilized by Ch/SLES mixture, in mass ratios that correspond to different regions of their interaction. Also investigation on the emulsions characteristics (stability, droplet size and size distribution, zeta potential) was carried out. Results of this study are important for understanding the influence of polymer-surfactant interactions on the properties of solution and stability of dispersed systems that could be useful in microencapsulation processes.

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1. Introduction

Emulsions are thermodynamically unstable systems and tend to separate in order to minimize the interfacial area between the aqueous and oil phase, what is a common problem. In commercial products, besides hydrocolloids, some other components like low molecular mass surfactants are often present as emulsifiers. However, for environmental and safety reasons, the use of a large quantity of low molecular mass surfactants is not recommended. On the other hand, interaction between polymer and surfactant could change the adsorption layer around the oil droplets influencing the emulsion stability [1–4].

In recent years the number of studies dealing with mixtures containing surfactant and polymer has been increased due to their importance in a variety of industrial applications like cosmetics, personal-care products, pharmaceuticals and detergents [5]. The emulsion stabilized by the polymer/surfactant complexes is generally more stable than the one stabilized by a low molecular mass surfactant solely. This is because it is stabilized not only by the electrostatic repulsion but also by the steric hindrance of the bulky oppositely charged polymer/surfactant complexes [6]. Also, adsorbed layer of polymer/surfactant complexes exhibit better mechanical properties that contribute to the increased stability of system. The interactions between polymers and surfactants in aqueous media give rise to the formation of association structures, thereby modifying functional and interfacial properties of the solution. When surfactants are introduced into a solution containing oppositely charged polymer, strong electrostatic interactions are referred as the attractive electrostatic forces that are extremely important for the oppositely charged polymer and surfactant. There has been significant research work focusing on the interaction of oppositely charged polyelectrolytes and surfactants using different techniques (tensiometry, turbidity, viscosity, isothermal titration calorimetric, conductivity, electrophoretic mobility) in recent years [7–14].

Chitosan (Ch) is the deacetylated form of chitin, which is the second most abundant biopolymer in nature. Due to the combination of its properties, such as biocompatibility, biodegradability, nontoxicity, bioactivity and antifungal activity, chitosan has many applications in medicine, cosmetics and drug controlled release systems [15–18]. Since chitosan is positively charged at low pH values ($\text{pH} < 6.5$), it spontaneously associates with oppositely charged polymers in solution to form polyelectrolyte complexes [17]. Also, chitosan can associate with anionic surfactant giving chitosan/surfactant complexes of different properties. A great number of studies investigate interaction between chitosan and sodium dodecyl sulfate (SDS) [11–13,19,20], as well as emulsions stabilized by this complexes [21–24]. Hence, in the present study we used sodium lauryl ether sulfate (SLES), which unlike SDS possesses ethylene oxide groups between the hydrophilic moiety and dodecyl hydrophobic part of the surfactant. In our previous study interaction between Ch and SLES has been investigated and explained in detail [14].

For these reasons, the main goal of this study was to investigate interfacial and adsorption properties of Ch/SLES complexes in order to be used in the emulsification process. The structure of the emulsions was analyzed microscopically and droplet size and size distribution were determined. Stability and zeta potential of this emulsions were investigated, as well.

2. Materials and methods

2.1. Materials

Low molecular weight (50,000–190,000 Da) Ch (product number: 448869) was obtained from Sigma-Aldrich (China), Ch degree of deacetylation, determined by potentiometric titration according to the procedure described by Yuan et al. [25], is found to be 81.8%. SLES with 2–3 ethylene oxide groups, active matter 70%, was purchased from Clariant (Germany). Different oils were used: medium-chain

triglycerides or caprylic/capric triglyceride (CAS number 73398-61-5, Saboderm TCC, Comcen, Zemun), extra pure paraffin oil (> 98%, Centrohem, Stara Pazova) and grape seed oil containing 71% linoleic acid and 20% oleic acid (Olitalia, Forlì (FC), Italy). In all experiments buffered water was used as a solvent, and pH was adjusted using 0.2 M water solution of acetic acid (Zorka-Pharma, Serbia) and 0.2 M water solution of sodium acetate (Centrohem, Serbia).

2.2. Preparation of solutions

The experiments were carried out at pH 4. The pH was measured by means of 827 lab pH-meter (Metrohm, Switzerland). Stock solutions of 1% (w/w) Ch were prepared by dissolving a given mass of polymer in the buffered water, at pH 4, while stirring and after relaxation at room temperature during 24 h, pH value of the solutions was checked. SLES stock solution of 2% (w/w) was prepared by the same procedure. Ch/SLES mixtures were prepared by slow addition of required volumes of SLES stock solution into Ch stock solution under continuous stirring. For interfacial tension measurements concentrations of SLES were varied from 0.00001% (w/w) up to 1% (w/w), while Ch concentrations were kept constant at 0.01% (w/w). Mixtures were left for 24 h at room temperature in order to equilibrate. Before measurements, pH value of the mixtures was checked.

2.3. Interfacial tension

Interfacial tension measurements between oil and water phase containing SLES or mixtures of chitosan and various concentrations of SLES, were carried out on a Sigma 703D tensiometer (KSV Instruments, Finland) using the Du Noüy ring method. Prior to the measurements the ring was immersed in water phase (below the surface), then oil phase was slowly added to the top and the system was left for 15 min to equilibrate the interface. The interfacial tension of each oil/water system was measured by pulling the ring out of heavier, aqueous phase into lighter, oil phase. In all measurements the temperature was kept constant at 30 °C. The reported values of the interfacial tension were average of three measurements, at least.

2.4. Preparation of O/W emulsions

Emulsions were prepared with 20% (w/w) of oil and 80% (w/w) of aqueous phase using the Ultra Turrax T25 (IKA, Germany) homogenizer at 5000 rpm and temperature of 30 °C. Mixtures of Ch (0.1% (w/w)) and SLES in mass ratios of 100:1, 1:3 and 1:10 were used as the aqueous phase of emulsions. Medium-chain triglycerides were used as the oil phase. The aqueous phase was homogenized 5 min before the emulsion preparation started. After this the oil phase was gradually added to the aqueous phase during the first minute of homogenization and continued with emulsifying for the next 9 min.

2.5. Droplet size analysis

Droplet size analysis of freshly prepared emulsions was carried out by microphotography analysis using BELView software. Microphotographs were taken on an optical microscope, Biooptica BEL-3000, Germany at 40x magnification. Particle mean diameter, expressed as volume-surface mean value, d_{vs} (μm) and standard deviation σ (μm) were calculated from the experimental data, Eqs. (1) and (2), respectively:

$$d_{vs} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \quad (1)$$

$$\sigma = \left(\frac{\sum n_i (d_i - d_{vs})^2}{\sum n_i} \right)^{1/2} \quad (2)$$

where d_i is droplet diameter and n_i is number of droplets. Droplet size distribution curves were obtained by fitting the experimental data with the gamma-equation (3):

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