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Simple one step synthesis of nonionic dithiol surfactants and their self-assembling with silver nanoparticles: Characterization, surface properties, biological activity

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ABSTRACT

Simple esterification of 2-mercaptoacetic acid and polyethylene glycol with different molecular weights was done to form the desired nonionic dithiol surfactants. The chemical structures of synthesized thiol surfactants were confirmed using FT-IR and ^{1}H NMR spectra. The surface activity of the synthesized surfactants was determined by measurement of the surface tension at different temperatures. The surface activity measurements showed their high tendency towards adsorption and micellization. The thermodynamic parameters of micellization (ΔG_{mic}, ΔH_{mic} and ΔS_{mic}) and adsorption (ΔG_{ads}, ΔG_{ads} and ΔS_{ads}) showed their tendency toward adsorption at the interfaces and also micellization in the bulk of their solutions. The nanostructure of the synthesized nonionic dithiol surfactants with silver nanoparticles was prepared and investigated using UV and TEM techniques. Screening tests of the synthesized dithiol surfactants and their nanostructure with silver nanoparticles, against gram positive bacteria (*Bacillus subtilis* and *Micrococus luteus*), gram negative bacteria (*Escherichia coli* and *Bordatella pertussis*) and fungi (*Aspergillus niger* and *Candida albicans*) showed that they are highly active biocides. The presence of silver nanoparticles enhancement the biological activities of the individual synthesized nonionic dithiol surfactants.

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1. Introduction

The chemical reactivity of thiol surfactants make it different from other surfactants. Thiols are a class of organic compounds that contain a sulfhydryl group (SH), also known as a thiol group, that is composed of a sulfur atom and a hydrogen atom attached to a carbon atom. This molecular structure is what distinguishes thiols from other organic chemical compounds with an oxygen-to-carbon bond configuration, such as phenols and alcohols. It's also what gives many high velocity thiols a persistent and highly unpleasant odor that is reminiscent of rotten eggs [1]. They are also used in the formation of nanoparticles of many other materials. The thiol compounds undergo self-assembling with metal nanoparticles such as gold nanoparticles [2]. In the case of silver nanoparticle (NP) usage as an antibacterial agent, the bacterial resistance has not been observed up to the present moment. This fact is supposedly caused by a difference in the mechanism of the antibacterial actions of the diverse forms of silver [3]. The fact of the non-existing bacteria resistance against the destructive effects of the silver NPs, that are observed already at low concentrations (units of milligrams per liter), lead to a recent rapid development in the field of synthesis of the silver NPs conveying the antibacterial activity [4–9]. The performed research had proven that antibacterial activity of the silver NPs is dependent not only on their size [4,5] but also on their shape [8]. The antibacterial activity of silver NPs have been currently applied as disinfecting agents in general practice, for example, for the antibacterial modification of textile materials [10,11]. The objective of the present study is the synthesis of dithiol surfactants with their silver nanostructures and investigates the influence of the structure on surface tension, interfacial tension, emulsion stability and thermodynamic parameters of dithiol surfactants. The biological activity of the synthesized compounds and their silver nanostructures were investigated.

2. Materials and measurements

2.1. Chemicals

Polyethylene glycol (PEG) of different molecular weights (Mwt = 600, 1000 and 1500) were purchased from El Goumhoria

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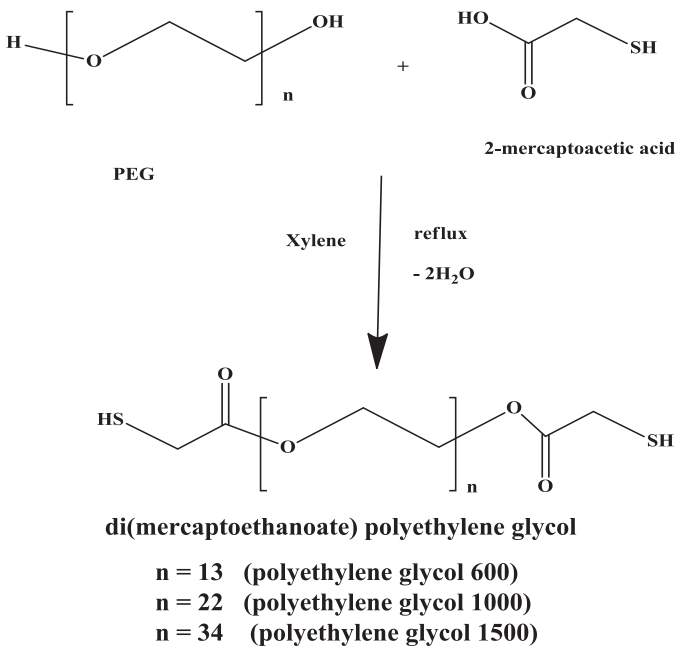

Scheme 1. Synthesis of nonionic dithiol surfactants.

Trade Pharmaceuticals & Chemicals Company, Cairo (Egypt). 2-mercaptoacetic acid, tri-sodium citrate and silver nitrate were analytical grade chemical was obtained from Merck chemical company. All chemicals used as received without further purification.

2.2. Synthesis of nonionic dithiol surfactants

2-mercaptoacetic acid (0.2 mol.) and PEG-600 (14 U of ethylene glycol per molecule), PEG-1000 (23 U of ethylene glycol per molecule), PEG-1500 (34 U of ethylene glycol per molecule) were esterified individually in xylene (250 mL) as the solvent under reflux conditions at 138 °C and 0.01% *p*-toluene sulphonic acid was used as dehydrating agent. The reaction was stopped after complete removal of the water of the reaction (0.2 mol., 3.6 mL). Then the solvent was removed using vacuum rotary evaporator. The catalyst was extracted from the reaction medium using petroleum ether. Subsequent purification was done by means of vacuum distillation to remove the excess and residual materials [12]. The nonionic dithiol surfactants obtained was designated as SH600, SH1000 and SH1500. Scheme 1 shows the chemical structures of the synthesized compounds.

2.3. Synthesis of silver nanoparticles

The colloidal silver nanoparticles solution was prepared by using chemical reduction method. All solutions of reacting materials were prepared in distilled water. Fifty milliliter of 1×10^{-3} M $AgNO_3$ solution was heated to boiling. To this solution 5 mL of 1% trisodium citrate was added drop by drop. During the process solution was mixed vigorously. Solution was heated until color's change is evident (pale yellow). Then it was removed from the heating element and stirred until cooled to room temperature [13].

2.4. Synthesis of the nanostructure of nonionic dithiol surfactants with silver nanoparticles

The silver nanoparticles solution (20 mL) was mixed with 5 mL saturated solution of the synthesized dithiol surfactant (SH600, SH1000 and SH1500) in deionized water and stirred effectively. Stirring was continued for 24 h until the yellow faded [14]. The resulting solution was used for ultraviolet experiments and TEM image.

2.5. Dithiol surfactants and their nanostructure characterization

The chemical structures of the synthesized nonionic dithiol surfactants were confirmed using FTIR and ^{1}H NMR spectroscopy. The FTIR analysis was performed by means of a Nicolet IS-10. All spectra were recorded with 2 cm^{-1} resolution at an angle of incidence 80° relative to the surface normal. ^{1}H NMR measurements were carried out using a Varian-Germini-200 instrument.

The UV measurements for the solution of AgNPs and solution of the nanostructure of the synthesized dithiol surfactants with AgNPs were carried out by UV–vis photometer.

Transmission electron microscope used to investigate the nanostructure of the prepared samples. A convenient way to produce good TEM samples is to use copper grids. A copper grid pre-covered with a very thin amorphous carbon film. To investigate the prepared AgNPs, the nanostructure of synthesized dithiol surfactants with AgNPs using TEM, small droplets of the liquid was placed on the carbon-coated grid. A photographic plate of the high resolution transmission electron microscopy (Type JEOL JEM-2100 operating at 200 kV attached to a CCD camera).

2.6. Surface activity measurements

Surface tension measurements were performed using a Kruss K6 tensiometer by the platinum ring detachment method (±0.5 mN/m). Freshly prepared aqueous solutions of the synthesized nonionic dithiol surfactants were used with a concentration range of 0.01–0.000005 M/L at 20, 40 and 60 °C. The solutions were poured into a clean Teflon cup with a mean diameter of 28 mm (Teflon cup was used to prevent the adhesion of the surfactant to the glass cup walls). The solutions were left for 2 h to allow the stabilization and complete adsorption at the solution surface, then the apparent surface tension values were measured a minimum of three times and the recorded values were taken as the average of these values [15,16]. The interfacial tension measurements were obtained between aqueous solutions of the synthesized nonionic dithiol surfactants at a concentration of 0.1% by weight and light paraffin oil at 25 °C using the same procedures of the surface tension measurements [16,17].

Emulsion stability was measured by vigorously stirring a mixture of 10 mL (0.5%) of the synthesized surfactant solutions and 10 mL of paraffin oil at 25 °C [18]. Emulsifying power (emulsion stability) of the surfactants was expressed as the time required for separation of 9 mL of pure surfactant solution.

2.7. Biological activity

The antimicrobial activity of synthesized nonionic dithiol surfactants was measured against a wide range of test-organisms comprising: (bacteria and fungi).

The different species of tested organisms were obtained from the unit of operation development Center, Egyptian Petroleum Research Institute.

Three media were used in the antimicrobial activity of synthesized products, the bacterial species grow on nutrient agar, while fungi mold grow on Czapek's dox agar. (i) Nutrient agar consists of Beef extract (3.0 g/L); Peptone (5.0 g/L), Sodium chloride (3.0 g/L) and Agar (20.0 g/L), then, completes the volume to 1 L, heated the mixture until the boiling, and sterilizes the media by autoclave. (ii) Czapek's Dox agar consists of sucrose

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