



# Studying the effect of newly synthesized cationic surfactant on silver nanoparticles formation and their biological activity

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## ABSTRACT

The chain length of the synthesized cationic surfactant effect on the amount and stability of the formed silver nanoparticles. The stability of prepared silver nanoparticles increases by increasing the chain length of the synthesized cationic surfactants. The amount of the formed silver nanoparticles increases by increasing the chain length of synthesized cationic surfactants. The chemical structure of synthesized surfactants has been confirmed using FTIR and  $^1\text{H}$ NMR spectroscopy. The used method for synthesizing silver nanoparticles produces uniform with very narrow size range. Transmission electron microscope, dynamic light scattering, UV–Vis spectroscopy and energy dispersive X-ray have been used to confirm the formation of silver nanoparticles and the effective role of the used cationic surfactants. The synthesized cationic surfactants and their silver nanoform showed a good antibiotic effect against fungi and bacteria. The silver nanoparticles enhanced the antibiotic effect of synthesized cationic surfactants.

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## 1. Introduction

The silver nanoparticle colloids have an impressive electrical, optical and thermal properties so they are used in many applications like conductors, chemical sensors and catalyst [1–3]. Due to these potential important a several methods were developed to prepare silver in the nano-form. Some authors used different reducing agents in the reduction process like ascorbic acid, sodium borohydride, hydrazine, and formaldehyde [4–7]. Many of the authors studied the effect of reducing agent (its strength and concentration) on the size, shape and size distribution of the formed nanoparticles. The synthesis of nanoparticles requires protective agents, which prevent the aggregation of the formed nanoparticles and hence control the size and size distribution [8–9]. The protecting agent used two techniques; self-assembly or capping process [10–11]. The protective agent may be polymer, surfactants or any suitable organic material which carries some functional groups that can chelate with the formed nanoparticles. The mainly used stabilizing agents are surfactants and many of the researchers studied the effect of surfactant concentration, time and solution temperature on the nanoparticle formation [12–15]. The present study aimed to prepare a new series of cationic surfactants with different alkyl chain and confirming their chemical structures using FTIR and  $^1\text{H}$ NMR spectroscopy. The work aimed to study the effect of chain length of the synthesized cationic surfactants on the stability, size distribution, shape and amount of silver nanoparticles formed. The formed silver nanoparticles were

confirmed using transmission electron microscope, dynamic light scattering, energy dispersive X-ray and UV–Vis spectroscopy.

## 2. Materials and methods

### 2.1. Chemicals

The chemicals, which are used in the synthesis of the cationic surfactants and the silver nanoparticles were of analytical grade and purchased from different companies. The fatty acids used, which were lauric, myristic and palmitic acid were purchased from Merck chemical company. The silver nitrate ( $\text{AgNO}_3$ , 99%) and dimethylaminopropyl amine were provided from Sigma-Aldrich/Germany.

### 2.2. Synthesis

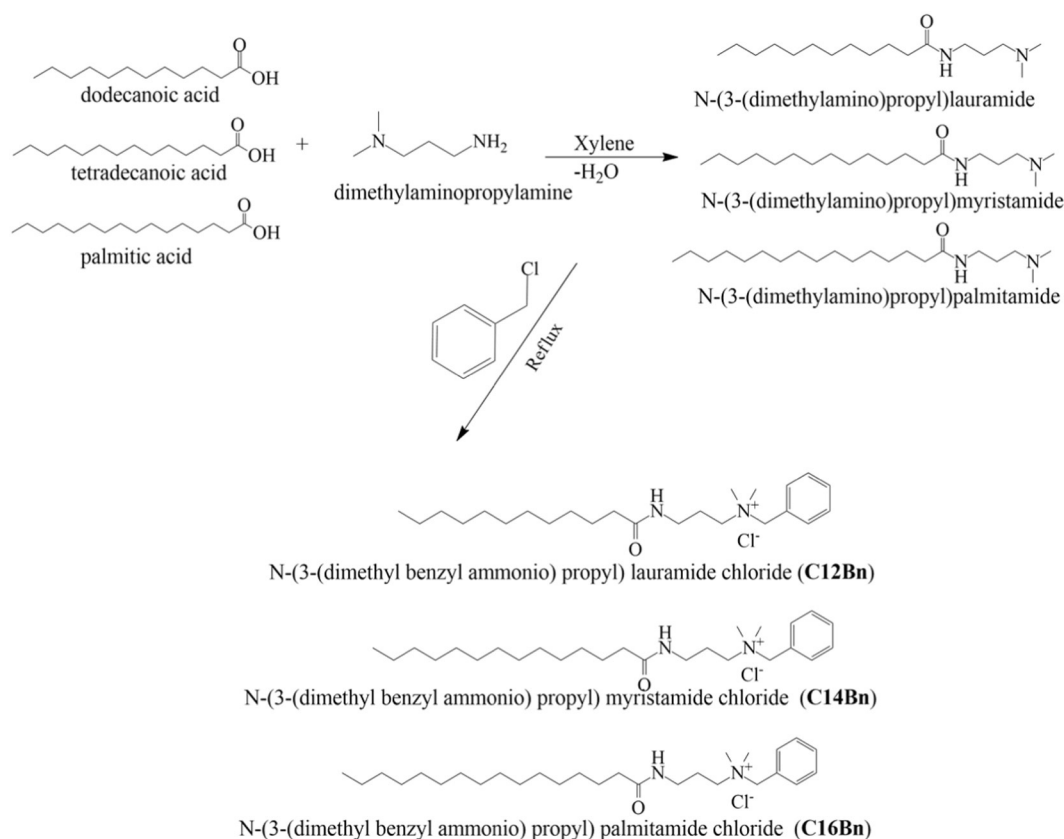
#### I. Preparation of cationic capping agents:

The used cationic surfactants were prepared in two steps

#### a. Synthesis of N-(3-(dimethylamino) propyl) alkanamide derivatives:

Equimolar amount from 1,3-dimethylamino-1-propyl amine (DMAPA) (0.15 mol) and fatty acid (dodecanoic acid, tetradecanoic acid and hexadecanoic acid) (0.15 mol) were dissolved in 120 ml xylene with 0.01% from p-toluene sulphonic acid as catalyst. The reaction mixture was refluxed using Dean–Stark apparatus and the reaction was completed by receiving the released water (0.15 mol, 2.7 ml). Petroleum ether was used to get rid of catalyst [16].

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**Scheme 1.** the chemical structure of prepared cationic surfactants.

#### b. Synthesis of N-(3-(dimethyl benzyl ammonio) propyl) alkanamide chloride derivatives:

The prepared amide (0.1 mol) from the previous step was refluxed with benzyl chloride (0.1 mol) in 120 ml ethanol as solved from 25 to 30 h depending on the fatty alkyl chain length of the synthesized amide. The solvent was evaporated under vacuum and the residue subject to crystallization using diethyl ether. The obtained amido-cationic surfactants named C12Bn, C14Bn and C16Bn and the general procedures for the synthesis were depicted in *Scheme 1*.

#### II. Preparation of silver nanoparticles (AgNPs):

The silver nanoparticles were prepared using simple one-step method. Green synthesis of silver metal nanoparticles was developed using sun light as reducing agent in the presence of the laboratory synthesized amido-amine cationic surfactants as capping agent. In a typical experiment, 20 ml of 2 mM aqueous solution of  $\text{AgNO}_3$  were mixed with 20 ml of 2 mM aqueous solution of each prepared amido-cationic surfactants individually. After good mixing, the solution was exposed to sun-light. Fast change in the color was observed in 5 min as maximum [17].

#### 2.3. Instrumentation

##### I. Fourier transform infrared spectroscopy (FTIR):

The chemical structure of the synthesized cationic structure was confirmed using Fourier transform infrared spectroscopy (Bench top 961, ATI Mattsonm Infinity series™, controlled by Win First™ V2.01 software Egyptian Petroleum Research Institute).

##### II. Proton nuclear magnetic resonance ( $^1\text{H}$ NMR):

Proton nuclear magnetic resonance in  $\text{DMSO-d}_6$  (GEMINI 200 ( $^1\text{H}$  500 MHz), National research center was used to determine the proton distribution of synthesized cationic surfactants.

#### III. Transmission electron microscope (TEM)

Small droplets of silver nanoparticles colloid were placed on carbon-coated grid pre-covered with a very thin amorphous carbon film. A photographic plate of the transmission electron microscope employed in the present work to investigate the microstructure of the prepared silver nanoparticles using TEM model "Joel JeM – 2100 (Japan)". (Egyptian Petroleum Research Institute EPRI).

#### IV. UV-Visible spectroscopy

The effect of chain length on silver nanoparticles formation was monitored using UV-visible spectrophotometer (Shimadzu, UV-2550, Japan) equipped with a quartz cell with optical path of 10 mm, and spectral resolution of 1 nm at a  $25 \pm 1$  °C in the wavelength range (190–700 nm). Before the measurement step, blank (the same water used in synthesis step) was placed inside of the sample cell to adjust the 100% transmittance signal.

#### V. Dynamic light scattering (DLS)

The stability of the formed silver nanoparticles and their size distribution were determined using dynamic light scattering (DLS) (a Malvern Zetasizer Nano Instruments Ltd., Worcestershire, UK). Each DLS measurement was operated in triplicate using automated, optimal measurement time and laser attenuation settings. The recorded correlation functions and measured particles mobility's were converted into size distributions and zeta potentials, respectively, using the Malvern Dispersion Software (V5.10, <http://www.zetasizer.com/>).

#### VI. Energy dispersive X-ray (EDX) spectroscopy

The energy-dispersive X-ray (EDX) spectroscopy was recorded with an EDX detector (Oxford LINKISIS 300) equipped on a Transmission electron microscope (TEM, Hitachi S-520) operated at 10 kV accelerating voltage.

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