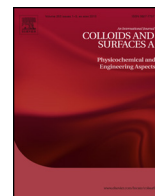




Contents lists available at ScienceDirect

Colloids and Surfaces A: Physicochemical and Engineering Aspects

journal homepage: www.elsevier.com/locate/colsurfa

Synthesis and characterization of surfactant stabilized nanocolloidal dispersion of silver chloride in aqueous medium



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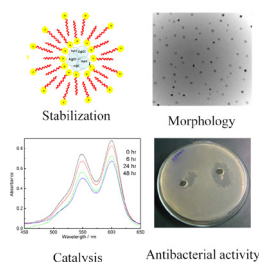
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HIGHLIGHTS

- Surfactant stabilized AgCl nanoparticles in aqueous medium were synthesized.
- AgCl nanoparticles remained stable for at least six months.
- Nanoparticles were stabilized by a bilayer like envelope of surfactants.
- AgCl nanoparticles catalyzed degradation of pinacyanol chloride.
- AgCl nanoparticles evidenced antibacterial activity.

GRAPHICAL ABSTRACT

Surfactant stabilized AgCl dispersions: proposed model, morphology, catalytic and antibacterial activity.



ARTICLE INFO

Article history:

Received 9 September 2013

Received in revised form 26 October 2013

Accepted 31 October 2013

Available online 7 November 2013

Keywords:

Silver chloride

Surfactant

DLS

TEM

Catalysis

Antibacterial activity

ABSTRACT

Nanocolloidal dispersion of silver chloride was successfully synthesized in aqueous medium using three different cationic surfactants. In the one pot synthesis, surfactant molecules served dual purposes: they acted as the source for the chloride ion as well as the capping agents. Formation of the surfactant stabilized nanoparticle was established from Raman spectra. Surfactant molecules stabilized the nanoparticles through the formation of a double layer like structure. Surfactant stabilized growth process for the nanoparticles followed first order kinetics as revealed from the quasi-elastic light scattering measurement. Size and morphology of the particles were found to be dependent on the concentration of silver ion as well as the surfactant head group. A catalytic amount of silver chloride nanoparticle degraded the carcinogenic dye pinacyanol chloride. Antibacterial activity of AgCl nanoparticle was assessed using a gram positive bacteria, *Bacillus subtilis*.

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1. Introduction

Clusters of 10–1000 atoms having size dependent physical, chemical, optical and electronic properties are categorized as nanoparticles [1]. Their unique properties make them immensely

useful in electrical and optical fields. Due to manifold increase in the surface to volume ratio, catalytic activity of nanoparticle increases significantly compared to their bulk materials [2]. Hence various methods of preparation of nanoparticles involving innovative chemical, colloid-chemical and electrochemical techniques are being developed by researchers [3]. Inorganic nanoparticles with attractive physico-chemical properties are being extensively studied, and among them silver halides have much interest [4]. The silver chloride nanoparticle is reported to have many

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photochemical and biomedical applications [5]. Use of silver chloride in photographic films [6], photocatalysis in splitting of water and removal of organic wastes are well documented in the literature [7,8]. In another report of Schruch et al. [9], it has been stated that nanostructured silver chloride, in the presence of excess silver ion, can act as a good catalyst in the generation of active oxygen species. AgCl nanoparticles also finds many biomedical applications, viz., preparation of antiseptic catheters, bone cements and fabrics owing to its antibacterial properties [10–12]. In spite of its immense applications, few successful methods are known for the preparation of silver halides with controlled size. The available reports include microemulsion technique [13], use of gelatin matrix [14], double decomposition reaction of AgNO₃ and NaCl in the presence of polyethylene glycol [15], mixing of AgNO₃ and hydrochloric acid (HCl) in the presence of poly(vinyl pyrrolidone) (PVP) as a capping agent in ethylene glycol media [5], etc. However, preparation of AgCl nanoparticles using a simple technique of reacting AgNO₃ with a chloride ion containing surfactant molecule, where surfactant molecules itself can act as capping agents, are not much reported. Recently such a technique for the successful preparation of AgBr nanoparticles has been reported by the present research group [16]. Keeping in mind the manifold applications of AgCl nanoparticles and the difficulty in preparation due to its high sensitivity to photo reduction [17], we have extended the previous work for the preparation of nanocolloidal dispersions of silver chloride.

In order to further investigate the stability dependence of the synthesized silver chloride colloidal nanoparticles on the surfactant head group, three different surfactants hexadecyltrimethylammonium chloride (CTAC), hexadecylpyridinium chloride (CPC) and benzyldimethylhexadecylammonium chloride (BDHAC) were used as the source for the chloride ion as well as the stabilizing agents. The surfactants have similar hydrocarbon chain length, however, their head groups are different. The judicious choice of the specific surfactants lies with the previous work where hexadecyltrimethylammonium bromide (CTAB) served as the stabilizing agent. Here along with CTAC, two other surfactants CPC and BDHAC (containing aromatic rings), were chosen. Moieties comprising aromatic rings with π -electrons should act as better stabilizing agents for their stacking abilities which can favor the formation of surfactant bilayers around the nanoparticles [16].

According to a recent report, about 8.0×10^5 tons of textile dyes is used worldwide per [18]. The huge dye waste in water are not only aesthetically unwarranted but also the existence of dye in wastewater severely can affect the ecosystems. Therefore, removal of dyes from the wastewater is considered to be an important issue. There are several methods, viz., adsorption, flocculation, photocatalysis, etc. [18], for the abatement. Among them, the catalytic approach is most economical and environmentally benign method [19,20]. Hence, the application of the prepared AgCl nanoparticles in the degradation of the carcinogenic dye pinacyanol chloride (PIN) is considered. Besides, as AgCl is known to have antimicrobial activities, hence the antimicrobial activity of AgCl nanoparticles has also been investigated.

2. Experimental

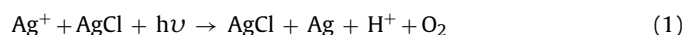
2.1. Materials

The cationic surfactants hexadecylpyridinium chloride (CPC) and benzyldimethylhexadecylammonium chloride (BDHAC) were purchased from Sisco Research Laboratories Pvt. Ltd., Mumbai, India. Hexadecyltrimethylammonium chloride (CTAC) was a product of E. Merck, Germany. All the three cationic surfactants were stated to be >99% pure and were used as received. A.R. grade

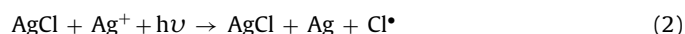
AgNO₃ was purchased from S.D. Fine Chemicals Ltd., India. The pseudoisocyanine dye pinacyanol chloride, PIN [1,1'-diethyl-2,2'-carbocyanine chloride, 2,2'-trimethinequinocyanine chloride] was a product from Sigma–Aldrich Chemicals, USA. The dye was used as received (stated to be >99% pure). Nutrient broth and nutrient agar used were from Himedia Chemicals, Mumbai, India. The composition of the nutrient broth consisted of peptic digest of animal tissue (5 g/L), beef extract (1.5 g/L), yeast extract (1.5 g/L), sodium chloride (5 g/L). Nutrient agar was comprised of peptic digest animal tissue (5 g/L), beef extract (1 g/L), yeast extract (2 g/L) and agar (15 g/L). All the chemicals were used as received. The solutions were prepared with double distilled water with a specific conductance of $2\text{--}4 \mu\text{S cm}^{-1}$ at 25 °C. Further details are available elsewhere [21,22].

2.2. Methods

Stable colloidal dispersion of AgCl was synthesized by adding an aqueous solution of AgNO₃ into the aqueous surfactant solution. Solution with five different concentrations of silver chloride (10, 20, 30, 40, 50 μM) were prepared. Details are available in the previous publications [16,23]. Briefly, a stock solution of 1 mM AgNO₃ was prepared and quantitative amount of that stock solution was added drop-wise with a Hamilton (USA) microsyringe to a 10 mL aqueous surfactant solution with constant stirring. The optimum concentrations of CPC, CTAC and BDHAC for the experiments, were determined by the method of trial and error. While for CPC and CTAC the optimum concentration was fixed at 0.4 mM, for BDHAC the appropriate working concentration was found to be 0.15 mM for the final synthesis. Herein all the solutions would refer to the systems comprising the above mentioned surfactant concentrations. Below the aforementioned concentration, not enough surfactant molecules were available in stabilizing the AgCl nanoparticles. From the conductance studies (discussed later) it was evidenced that a higher concentration of surfactants (up to 1 mM) would also give stable dispersions, however, at the optimum concentrations, with minimum usage of the surfactants, yielded the same results. Hence all experiments were carried out with 0.4 mM CPC and CTAC and 0.15 mM BDHAC. As the surfactant concentration was more than thousand fold higher than the silver ion concentration, hence it could be assumed that all the silver ions have been completely transformed into its chloride. In a previous work where AgBr nanoparticles were prepared by a similar technique, surfactant stabilized AgBr nanoparticles could be isolated from the aqueous suspension or by a phase transfer method [16]. However, as AgCl in the present case undergoes rapid photodecomposition whereby Ag⁺ ions were reduced to black metallic silver within minutes [17]. The detailed mechanism for such degradation has explicitly been explained by Schruch et al. [9]. Isolated silver chloride nanoparticle, in the presence of water and Ag⁺ ion, undergoes the following chemical changes upon UV–vis irradiation:



Also the other possible photochemical change leads to the formation of chlorine free radicals and subsequently chlorine molecule.



This particular property of AgCl nanoparticles, were, in fact beneficial for its catalytic activity in the degradation of the dye PIN, which will be discussed in subsequent section. However, the aforementioned difficulty has refrained us from isolating surfactant stabilized AgCl nanoparticles. The colloidal dispersions of the

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