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Influence of surfactants in synthesizing of AgCl-doped silica spheres

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Abstract

Silver chloride-doped silica (AgCl-SiO_2) particles with the crystallite size of AgCl ranging from 130 – 240 nm have been prepared via the precipitation of Ag^+ and Cl^-. The Ag^+ were adsorbed onto the silica surfaces during the Stöber process, followed by the addition of Cl^- and surfactant for the formation of AgCl-SiO_2 particles. Polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP) with different molarities were used as surfactants. Samples also were prepared without surfactant. The synthesized samples were characterized by using X-ray diffraction (XRD), scanning electron microscope (SEM) and UV-visible spectroscopy (UV-Vis). XRD patterns confirmed the formation of AgCl for all samples. For low molarity of surfactants (50 mM), smaller size of 133 nm AgCl was formed by using the PEG while at higher molarity (75 mM), the PVP gives smaller size of 134 nm AgCl particles. From SEM, there is no significant difference in morphology of all samples. The strong absorption in ultraviolet region indicates the presence of AgCl particles.

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*Keywords:*polyvinylpyyrolidone; polyethylene glycol; silver chloride

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1. Introduction

The massive issues on environment pollutant and energy crisis urge the researchers to hunt for efficient semiconductor photocatalyst to minimize the problem. Silver chloride (AgCl) is well identified as a good candidate for photocatalytic activity especially for splitting of water into hydrogen using solar energy and its application in pollutant degradation[1]. Moreover, recent interest in AgCl particles as vital component materials for visible-light-driven photocatalyst has aroused[2-4]. It also has wide applications in sensitized paper and electroplate due to its indirect gap semiconductor property[5-6]. Interestingly, it also exhibits a good antimicrobial characteristic that can be used for the fabrication, bone cement and antiseptic catherers[7]. As for photocatalytic application, larger surface area provides more adsorption capacity that can increase the efficiency of the material. Thus, the size of the synthesized AgCl particles plays important roles. In this work, the demonstration via facile route to synthesize AgCl nanoparticles were done by mixing Ag^+ ion from $AgNO_3$ and Cl^- ion from NaCl in ethanolic medium. Apparently, AgCl nanoparticles have distinctive properties such as catalytic and optical properties in which depend on the shape and the size of produced nanoparticles. However, the evolution of AgCl occurred rapidly and it is challenging to produce small particle especially in nano size.

In order to solve this matter, many research testified on using amorphous silica (SiO_2) particles, which were synthesized via Stöber method as a substrate for the deposition of various type of nanoparticles[8]. The properties of silica that is chemically inert and able to conduct high stability against aggregation make it suitable to use as the substrate. Apparently, Stöber method is the most modest and elegant method for preparing monodisperse spherical silica particles. The process involved hydrolysis and condensation of a silica precursor, tetraethyl orthosilicate (TEOS) in an ethanol as the solvent with the presence of ammonia at room temperature[9].

Polymers such as polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP) are known to have long hydrocarbon chain structures with hydrophobic ends. It is believed that this structure is critical in manipulating particle sizes. The rate of particle aggregation is a major factor that controls the morphology and crystallinity of the final product. Besides that, the discrepancy in the capping behavior between PEG and PVP could be attributed to the difference in adhesion strength. However, PVP has been widely known as a capping reagent for microparticles as the adhesion of PVP was stronger than that of PEG[11]. Thus, the usages of PVP or PEG are expected to results in difference rate of formation, deposition, size and shape of synthesized AgCl particles.

2. Methodology

2.1 Materials

The reagents applied were ammonium hydroxide 25%(NH_4OH, J.T Baker), tetraethyl orthosilicate 98% (TEOS, Acros), ethanol 96 % (Altia), silver nitrate ($AgNO_3$, Sigma-Aldrich), hydrochloric acid 37% (HCl, Sigma-Aldrich), polyvinylpyrrolidone m.w.10,000 (PVP, Sigma-Aldrich), poly(ethylene glycol) (PEG) m.w. 380 and deionized water. All the materials were used without prior purification.

2.2. Synthesis of AgCl-doped SiO_2 spheres

The synthesis of the silica spheres was based on the modified Stöber method[10]. An amount 200 ml ethanol and 2 ml of tetraethylorsolicate (TEOS) was mechanically mixed in 500 ml beaker for 5 minutes, followed by the addition of 14 ml of ammonium hydroxide (NH_4OH) dropwise. Then, the solution was stirred for 15 minutes followed by the addition of 10 ml of 20 wt% $AgNO_3$ solutions. The solution was stirred under mechanical mixing for 1 hour to ensure the adsorption of Ag^+ species onto the silica surfaces. After that, 10 ml of 50 mMpolyvinylpyrrolidone (PVP) and a certain amount of 300 mMCl^- was added simultaneously into the Ag^+–rich silica solution. The solution was continued to stir for 2 hours. The sample was washed with deionized water for the removal of excess PVP by centrifuge, dried in vacuum oven at 323 K and ground. This step is repeated for other samples with different parameters as shown in Table 1.