



Research paper

Synthesis, characterization and antibacterial properties of novel nano-silver loaded acid activated montmorillonite



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ABSTRACT

This paper reports synthesis, characterization and antibacterial properties of a novel silver-exchanged montmorillonite (Ag-Mt) synthesized from acid activated sodium montmorillonite (Na-Mt). The silver-loaded nanostructures (Ag-Mt) were synthesized in a two-step approach in which Na-Mt was first converted into acid activated Na-Mt and further treated with two concentrations of silver nitrate solution to obtain two types of acid activated Ag-Mt for comparison, Ag-Mt without the acid activation step and isotropic silver nanoparticles were synthesized as control samples. EDX confirmed successful loading of elemental silver in all types of Ag-Mt and WXR analysis showed undisturbed crystal structure with increased interlayer space compared to Na-Mt. TEM micrographs revealed formation of spherical silver nanocrystals on the supporting silicate layers although no external reducing agent was employed due to high reduction potential of silver ions. The results were further confirmed in UV spectroscopy where strong absorption bands related to the surface plasmon resonance (SPR) was observed. The bactericidal efficacy of the clay minerals were evaluated against Gram-negative bacteria *Escherichia coli* and Gram-positive bacteria *Staphylococcus aureus* using disk diffusion method which showed excellent inhibitory properties and large inhibition zones of all synthesized Ag-Mt. The minimum inhibitory concentration (MIC) of the clay minerals was also quantified and compared with synthesized isotropic silver nanoparticles. Acid activated Ag-Mt showed antimicrobial activity marginally lower than silver nanoparticles alone, although the silver content was ~10 times lower than Ag-NP. This antibacterial agent is thus very effective and more attractive in terms of cost and environment friendly nature.

1. Introduction

In the past few years, bacterial resistance to antibiotics has been rising at an alarming rate due to rampant overuse of antibacterial medicines and gene mutation undergone by pathogenic bacteria. Numerous cases of outbreak of infectious diseases have been reported as a direct effect of antibiotic resistance (Rai et al., 2009). In such times, inorganic metal based nanoscale materials has emerged as a novel class of antibacterial agent due to advantages to high surface area to volume ratio, chemical stability and thermal resistance (Morones et al., 2005). Also bacteria are not known not to develop resistance to inorganic antimicrobial agents. Though nanoparticles of gold, copper, zinc and titanium have showed high bioactivity, silver nanoparticles is the most promising one and has shown broad spectrum activity against as many as twelve species of Gram positive and Gram-negative bacteria (Kim et al., 2007; Tran et al., 2013) as well as yeast (Kim et al., 2007).

Though some researchers debate whether silver nanoparticles possess inhibitory effect, there is a general consensus which agrees that the

silver ions released from unstable silver nanoparticles are responsible for its excellent biocidal action (Chopra, 2007). Bacterial cell wall has a net negative charge under physiological conditions due to the presence of functional groups such as carboxyl, phosphate and hydroxyl on the lipoprotein containing outer cell wall. An electrostatic attraction is created between silver ions and the cell wall in which the functional groups strongly bind to silver ions via ion-dipole interaction causing shrinkage of the cytoplasm membrane or detachment of the cell wall. Silver ions also interact with the thiol groups present in protein of cytoplasm, causing denaturation of proteins which impairs its replicating ability. (Bragg and Rainnie, 1974; Tran et al., 2013).

High performance antibacterial activity is only obtained if silver nanoparticles are of uniform shape and have narrow particle size distribution (Girase et al., 2011). However, silver nanoparticles have a strong tendency to form aggregates due to instability stemming from high surface area to volume ratio. In order to overcome such problem, inorganic carriers for silver nanoparticles e.g. zeolites (Top and Ülkü, 2004), titanium dioxide (Viana et al., 2013) and clay minerals (Dizman

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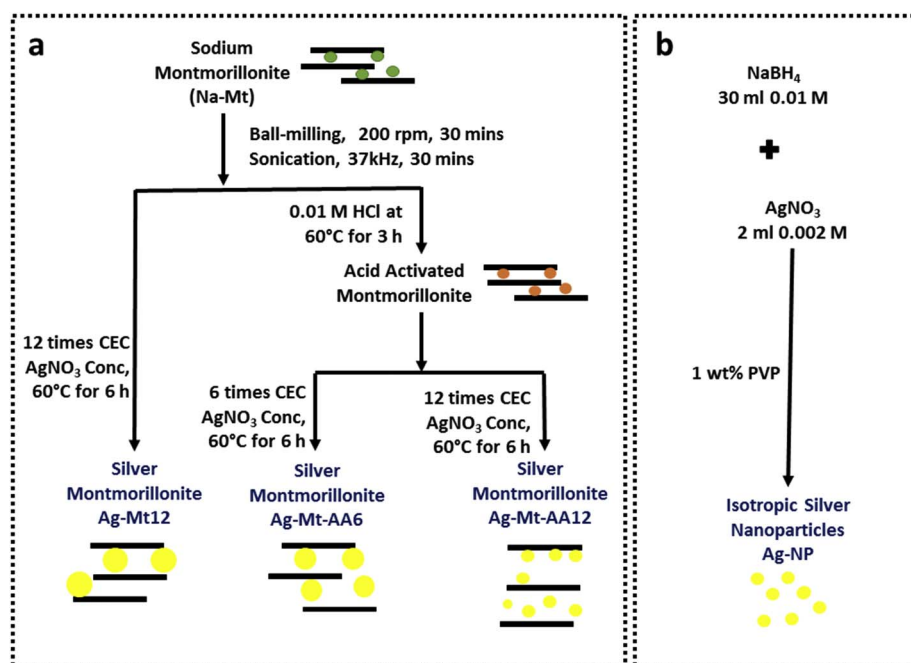


Fig. 1. Schematic of (a) Types of Ag-Mt samples prepared and (b) Synthesis of isotropic Ag-NP.

et al., 2007; Miyoshi et al., 2010) have been under extensive research focus. Smectite clay minerals like montmorillonite (Mt) have been widely studied as a carrier because it possess numerous advantages like natural abundance and cost effectiveness, atoxic nature, chemical inertness, high intercalation and sorptive capabilities and large specific surface area (Carretero, 2002; Saha et al., 2014). In most cases, silver ions are first exchanged into Mt sheets and during after-treatment in the next step exchanged cations are transformed into nanoparticles. Silver ions loaded Mt has been after-treated with reducing agents like formaldehyde (Praus et al., 2010), sodium borohydride (Praus et al., 2010), ethanol (Wei et al., 2013), ethylene glycol (Sohrabnezhad et al., 2015) or calcined or UV-irradiated (Magaña et al., 2008; Girase et al., 2011) to obtain silver nanoparticles decorated Mt. Slow diffusion of silver ions from nanoparticles immobilized on Mt helps to provide excellent bioactivity with prolonged duration. The synthesized silver-clay structures has been shown to be effective against wide range of bacteria e.g. *Staphylococcus aureus*, *Pseudomonas aeruginosa* (Gram-positive) and *Escherichia coli* (Gram-negative) (Xu et al., 2011; Costa et al., 2011; Tian et al., 2014; Joshi et al., 2015).

High adsorptive capabilities for silver ions and nanoparticles in Mt are largely responsible for the superior antimicrobial properties of the silver-clay nanohybrids. The adsorptive properties in Mt are governed by large specific surface area and with increase in net negative charge on the sheets. Concentrated inorganic acids at elevated temperatures causes the exchangeable cations in the interlayer space to be replaced with protons and dissolution or leaching out of some structural cations like Mg²⁺, Al³⁺ and Fe^{2+/3+} in the octahedral sheet without altering the SiO₄ groups. The acid treated clay gradually transforms into a partially dissolved, mesoporous, amorphous silica rich crosslinked structure. These structural alterations during acid treatment result in higher negative overall charge, increasing the surface acidity and therefore the specific surface area and pore volume (Bhattacharyya and Sen Gupta, 2008; Komadel, 2016). All these attributes assist in increased adsorptive capabilities in acid-activated Na-Mt. Characterization techniques like FT-IR (Tyagi et al., 2006) and SEM (España et al., 2016) have been employed to understand morphological changes during acid treatment. Bhattacharyya and Sen Gupta (2008) have showed an increase of 123% in CEC with acid activation of Mt with enhanced mesoporosity. Acid activated Mt has found widespread applications as catalysts and catalyst support, in paper industry, in

chemicals and foodstuff (Zhao et al., 2016).

However, the increase in adsorptive capabilities as a consequence of acid activation has not been exploited yet to obtain higher loadings of silver cations on the silicate layers. Higher loadings of silver directly translate to increased antimicrobial efficiency and therefore Ag-Mt prepared from acid activated Mt is expected to exhibit higher microbial resistance. This paper reports the preparation, characterization and antibacterial properties of silver loaded Mt based on acid-activated Na-Mt. The synthesized Ag-Mt shows deposition of fine silver nanoparticles (~10 nm and ~30 nm) on clay surface and thus leads to excellent antibacterial activity only marginally lower than silver nanoparticles alone.

2. Experimental

2.1. Materials

Sodium montmorillonite (Na-Mt) having a cation exchange capacity of 120 mequiv/100 g was procured from Southern Clay Products (Japan) and was used as received. Silver nitrate (AgNO₃, 99.9% purity, molar mass 169.87 g/mol), hydrochloric acid (HCl, 37%) and sodium borohydride (NaBH₄, molar mass 37.83 g/mol) was obtained from Merck Chemicals, Mumbai. Polyvinyl pyrrolidone (PVP, K-40) was purchased from Sigma Aldrich. All chemicals were of reagent grade and have been used without further purification.

2.2. Synthesis of silver loaded montmorillonite (Ag-Mt)

Three kinds of silver modified montmorillonite (Ag-Mt) have been prepared through cation exchange reaction of Na-Mt according to the following procedures as outlined in Fig. 1.

Prior to the exchange operation, Na-Mt was ball-milled (Retsch ball milling machine, Germany) at 200 rpm for 30 min and sonicated at 37 kHz for 30 min to obtain peeled apart individual particles. Acid activation treatment was carried out by refluxing 5 g of Na-Mt dispersed in 150 ml deionized water with 100 ml 0.01 M HCl at 60 °C for 3 h. The resulting dispersion of activated clay mineral was centrifuged (Sigma Centrifuge) at 7000 rpm for 30 min, washed with distilled water several times till it became free of residual chloride ions (silver nitrate test). The precipitate was gently dried in vacuum oven at 60 °C until

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