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One-pot synthesis of nanoscale silver supported biomass-derived silica

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Abstract

In this study, silver nanoparticles $(AgNP/SiO_2)$ with an average diameter of 25 nm were successfully fabricated using silica rice husk as support via a simple yet cheap sol-gel route at room temperature. The synthesized AgNP/SiO₂ was characterized by several spectroscopic methods such as FTIR, SEM/EDX, XRD, N₂ adsorption-desorption and TEM analyses. FTIR and EDX analyses showed the successful inclusion of silver into the silica framework. Preservation of the amorphous nature of silica in AgNP/SiO₂ was confirmed from the high angle powder XRD analysis. High mesoporosity of AgNP/SiO₂ (BET surface area = $514 \text{ m}^2\text{g}^{-1}$ and pore volume = 0.50 ccg⁻¹) was clearly evidenced from the N₂-sorption studies. Well-dispersion of spherical Ag nanoparticles was observed through the TEM investigation. This research proves the successful conversion of uneconomical biomass into discreet AgNP/SiO₂ via a facile and novel sol-gel method.

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1. Introduction

The advent of nanotechnology secures a great prospect in the field of science, technology, catalysis, electronics and magnetism [1]. Without a doubt, the manipulation of discreet size materials (1-100 nm) offers unique and different physical properties in comparison to its bulk size. Currently, the fabrication of metal nanoparticles such as

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Ag had indisputably received wide attention among global researchers due to its inherent potential for biological applications and pose non-toxicity [2]. Owing to its broad applications, various protocols have been employed to produce unique Ag metal nanoparticles which includes laser ablation [3], γ -ray irradiation [4], hydrothermal [5], electrochemistry [6] and thermal decomposition at higher temperature [7]. Most of these existing procedures is complicated, needs expensive instrument and consume high energy [8]. Besides, Huang et al. [9] had fabricated Ag nanoparticles using CO₂-expanded liquid at pressure of 5.52 MPa, which undoubtedly requires elevated energy. Despites this, physical methods also requires toxic and polluting chemicals and solvents [10]. Extensive uses of these noxious/harsh chemicals have a strong impact on the eco-system and human as well. 'Green Chemistry' is currently the key concern in chemical industry. Thus, designing a secure route for the production of Ag nanoparticles has been a battle among researchers towards safeguarding our mother earth.

Rice husk (RH), the by-product from rice milling process is well-known for its high-grade silica content [11]. Due to its high surface area, it is widely used as catalyst support for the production of various fine chemicals [12-14]. However, there is very few published literature on silica from RH being used as support for nanoparticles production. On the other hand, sol-gel process is a simple, environmentally friendly route for the immobilization of metals at low temperature. It offers homogeneity, better control of grain size and porosity of the material [15]. Recently, Rahman and Padavettan, [16], obtained monodispersed nanoparticles with narrow size distribution at mild condition using simple sol-gel technique.

Herein, we report a facile sol-gel route to fabricate Ag nanoparticles supported on RH silica. This work will focus on the synthesis and the nanoparticles characterization.

2. Experimental

2.1. Materials

Rice husk (RH) was obtained from a local rice mill in Penang, Malaysia. AgNO₃ (R&M Chemicals, 99.8%) was used as the precursor of silver. Other chemicals used were nitric acid (Systerm, 65%) and sodium hydroxide (QReC, 99.0%).

2.2. Preparation of AgNP/SiO2

Sodium silicate was prepared according to the method by Adam et al. [12]. In brief, RH was washed thoroughly with copious amount of distilled water to remove soil and contaminants prior drying at room temperature. Later, about 30 g of cleaned RH was stirred with 500 mL of 1 M HNO3 for overnight and subsequently oven dried at383K for overnight. The acid treated RH was transferred into a flask containing 500 mL of 1 M NaOH and mechanically stirred for 1 day. Then, it was suction filtered yielding a dark brown solution of sodium silicate. Briefly, to prepare AgNP/SiO2, the prepared sodium silicate solution was titrated slowly with 120 mL of 3M HNO3 containing 10 wt.% AgNO3 until pH 3. The resulting gel was aged at room temperature for 48 h, centrifuged, washed with distilled water and oven dried at 383 K for 18 h. The obtained solid material was ground into fine powder and designated as AgNP/SiO2.

2.3. Material Characterizations

The prepared material was characterized by X-ray diffraction (XRD, Kristallofex Siemens, model D5000), scanning electron microscope (SEM, model 50VP) equipped with energy dispersive spectrometry (EDX, Falcon System), transmission electron microscope (TEM, model Phillips CM12), Fourier Transform Infrared (FT-IR, Perkin Elmer System 2000), N₂-sorption (NOVA 2200e porosimeter) and Atomic Absorption Spectroscopy (AAS, Perkin Elmer, model 3100).

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