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Sonochemical Synthesis and Characterization of Silver Nanoparticles using Lactose as the Reducing Agent

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ABSTRACT

The present study focuses on the exploration of a synthetic procedure for the preparation of green silver nanoparticles. Using lactose as the reducing agent, the reaction mixture containing the silver precursor was exposed to ultrasonic irradiation at ambient temperature and conditions. To optimize the synthesis, the pH levels and concentration of lactose were varied. Using lactose at acidic level, no silver nanoparticles were produced since there was no color change observed after the sonication. On the other hand, silver nanoparticles were produced using alkaline lactose solutions as evidenced by the formation of yellow-colored products. Infrared spectrometry revealed the functional groups responsible for the reduction and capping of the nanoparticles. The sonochemical route provided a synthetic strategy for the production of quality nanoparticles with potential chemical and biological activities.

Keywords: Lactose, Reducing agent, Silver nanoparticles, Sonochemical technique, Ultrasound.

INTRODUCTION

Continuous development of methods for the preparation of functional nanomaterials such as silver nanoparticles (AgNPs) that will yield reproducible physical and chemical properties is an important area of research in the field[1]. Various modern techniques for the preparation of AgNPs such as photochemical, chemical reduction, thermal methods, and irradiation methods require costly experimental set-up and harsh reaction conditions[2]. Further, these methods use toxic reagents and produce toxic by-products that impose risks and hazards. Alongside the methods used, chemical reagents include sodium citrate and sodium borohydride as the reducing agents, and alkanethiols and alkylamines as the capping agents affect the quality of AgNPs synthesized[3]. A typical synthesis of AgNPs can either be a top-down approach or a bottom-up approach[4]. In brief, the top-down approach is based on the production of nanoparticles from bulk material while the bottom-up approach is about the formation of nucleation sites that grow into nanosized particles.

Besides the thermal method, a sonochemical technique that utilizes sound waves from a vibrating object that produces

mechanical energy and pressure is gaining interest due to its advantages of cost-effectiveness, rapid, and simplicity[5,6]. It is reported that the sonochemical approach produced well-dispersed nanoparticles with minimal agglomeration at room temperature conditions[7]. For a greener strategy, the use of biological substances as the reducing agents instead of toxic reagents such as sodium borohydride has been demonstrated to be superior due to its low cost and eliminates toxic synthesis by-products[8]. Such biomolecules utilized in the synthesis of silver nanoparticles are carbohydrates[9,10] and plant extracts[7,11,12].

In the present study, a simple and green procedure for the preparation of AgNPs was explored. The objective is to prepare AgNPs via an ultrasound-assisted method and utilize mild reaction conditions such as ambient reaction temperature and green reducing agents. Parameters such as pH and concentration of reducing agent were varied but the reaction temperature and reaction time were held constant. The reaction temperature was maintained at 30°C and the reaction time was fixed at 10 minutes. Typically for green reducing agents, either plant extracts or carbohydrates were utilized. The common structural features of phytochemicals such as polyphenols and carbohydrates are the reducing capability of the hydroxyl (-OH) moieties[13]. In this exploration, lactose is used as the green reducing agent as it has a number of hydroxyl groups as its structural feature that is capable of the reduction of metal ions[10]. The synthesized AgNPs from the proposed method can be further utilized for chemosensor and biological applications and activities.

MATERIALS AND METHODS

Chemicals and Equipment

Chemical reagents such as silver nitrate ($AgNO_3$), lactose, and sodium hydroxide (NaOH) were used without further purification. All the solutions were prepared in distilled water. A Lambda 365 UV-Vis spectrophotometer (PerkinElmer, USA) was used for the characterization and estimation of the size and concentration of the synthesized AgNPs. The wavelength range is set to 300-600 nm and a scanning rate of 120nm/minute. Fourier transform infrared spectrometer (FTIR) IRSpirit using the attenuated total reflectance (ATR) accessory (Shimadzu, Japan) was used for the IR spectral characterization. The following parameters are used for the FTIR analysis: (i) wavelength range: 700 to 4000 cm^{-1}; (ii) resolution: 4 cm^{-1}; (iii) number of scans: 20, and; (iv) apodization: Happ-Genzel. The ultrasonic bath used in the synthesis is a PowerSonic 410 with a 40 kHz frequency (Hwashin Technology, Korea).

Sonochemical synthesis and characterization of AgNPs

For the sonochemical synthesis, the pH and the concentration of the lactose solutions were varied. Initially, the pH of the lactose is 6, which is an acidic pH. Therefore, 0.1 M NaOH was used to vary the pH of the lactose solutions. Lactose solutions with pH levels of 8 and 10 were prepared after adding specific volumes of 0.1 M NaOH to the lactose solutions. For the effect of lactose concentration, lactose solutions with varying concentrations were prepared. A 0.5 g, 1.0 g, and 2.0 g of lactose were dissolved in water to prepare 0.5% w/v, 1.0% w/v, and 2.0% w/v solutions, respectively. In brief, 0.5 mL of 1 mM $AgNO_3$ solution was added to 10 mL of the lactose solution with different pH. The resulting mixture was exposed to ultrasound irradiation under ambient temperature for 10 minutes. The visible change in color of the solution from colorless to yellow signified the synthesis of AgNPs. The synthesized AgNPs were characterized using UV-Vis spectroscopy and FTIR spectrometry. To estimate the size and concentration of the AgNPs, the absorbance values at the maximum absorption wavelength (λ_{max}) and the molar extinction coefficient from a tabulated reference data set[14] were used.

RESULTS AND DISCUSSIONS

To test the effect of pH on the synthesis of AgNPs, the pH levels of the lactose solutions were varied. Initially, 1.0% w/v of lactose was used and the pH level was adjusted using 0.1 M NaOH solution. Using the pH 6 lactose solution as the reducing agent, no color change was observed after the ultrasonic irradiation which signified that there was no formation of AgNPs in the reaction mixture, as compared to the reaction mixtures with pH-adjusted lactose solutions (Fig. 1). The pH values of the lactose solution were then adjusted to pH 8 and pH 10 and a formation of yellow color was observed after the 10-min ultrasonic irradiation. As observed from Fig. 2, no peak was observed for the AgNPs prepared from the acidic lactose solution after the ultrasonic irradiation. The

AgNPs synthesized using lactose solution with pH levels of 8 and 10 exhibited a yellow color solution and a λ_{max} range of 400-406nm. The peak observed within the wavelength range 320-580nm corresponds to the surface plasmon resonance (SPR) of AgNPs in aqueous solutions and is dependent on the morphological conditions[15,16]. This suggests that changing the pH of the lactose solution affected the production of AgNPs from its precursors. This observation can be attributed to the fact that at alkaline pH, disaccharides such as lactose that contains -OH groups were deprotonated in the basic environment[9]. Reducing sugars such as lactose contain a free carbonyl group that can donate electrons to initiate the reduction of Ag^{+} to Ag^{0}. Moreover, the -OH moieties and the oxygen molecules can cap the silver nanoparticles to prevent aggregation, therefore the lactose can also act as a capping agent (Figure 3).

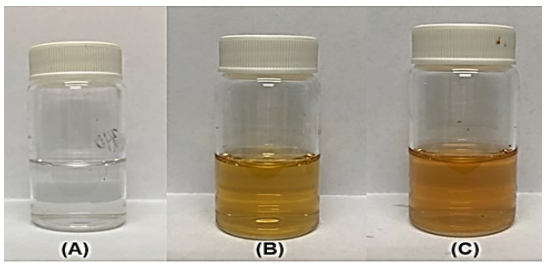

Fig. 1. Synthesized AgNPs using lactose as the reducing agent with different pH values: (A) pH=6; (B) pH=8; (C) pH=10

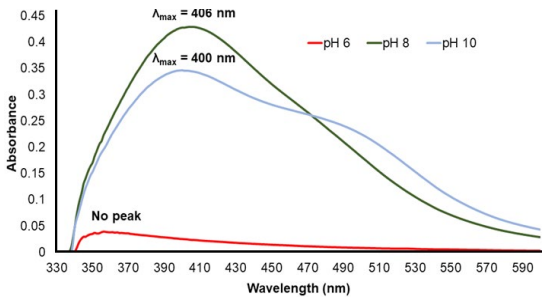

Fig. 2. UV-Vis spectra of the synthesized AgNPs using lactose with different pH levels

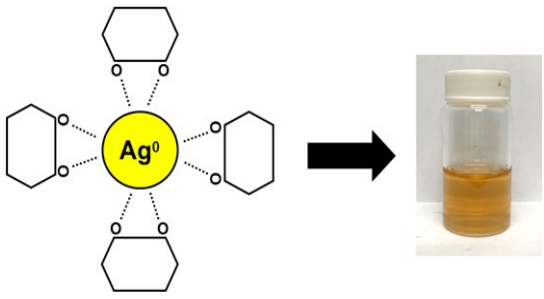

Fig. 3. Schematic representation of the synthesized AgNPs using lactose as the reducing agent

In reference to the data set[14], the size and concentration of the synthesized AgNPs were estimated via interpolation from the reference values, the estimated size and concentration for AgNPs using lactose with pH 8 are 30.67nm and 0.02737±0.0000 1nM, respectively. On the other hand, the values for the size and concentration of the AgNPs using lactose with pH 10 are 18.55nm and 0.1014±0.000 1nM, respectively. In this regard, the lactose solution with a pH value of 10 was used in the subsequent experiments since it yielded the AgNPs with the smaller size and higher concentration.

To optimize the concentration of the lactose solution that was used as the reducing agent, two other concentrations of lactose with a pH 10 value, 0.5% w/v and 2.0% w/v were utilized. Following the same synthesis setup, AgNPs were synthesized using the varying lactose concentrations. The synthesized AgNPs exhibited SPR peaks from 398-408nm (Fig. 4). The estimated size and concentration of AgNPs synthesized using 0.5% w/v lactose is 16.46nm and 0.07348±0.0000 4nM, respectively. While the AgNP size and concentration when using 2.0% w/v of lactose was 33.85nm and 0.00760±0.0000 5nM, respectively.

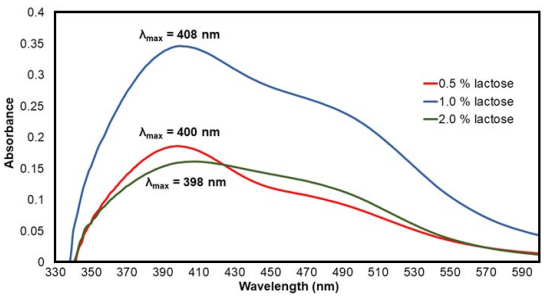

Fig. 4. UV-Vis spectra of AgNPs synthesized using various concentrations of lactose

An FTIR spectral analysis of the lactose reagent and synthesized AgNPs showed characteristic peaks that correspond to functional groups and moieties that were involved in the synthesis of AgNPs. The peak at 3314 cm^{-1} for the synthesized AgNPs Fig. 5 and the 3246 cm^{-1} for the lactose can be attributed to the O-H groups and the intramolecular hydrogen bonds from the water molecules[17]. The peak at 1633 cm^{-1} for the synthesized AgNPs was due to the C=O stretching[18] Peaks at 1033 cm^{-1} to 1070 cm^{-1} for the lactose are due to the intermolecular stretching of carbohydrates[19]. The observed peaks can be

assigned to the presence of molecules that are responsible for the reduction and stabilization of the nanoparticles[8].

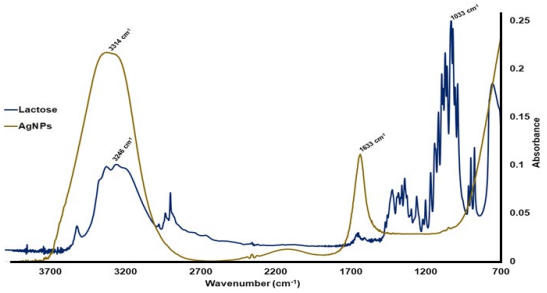

Fig. 5. FTIR spectra of lactose and the synthesized AgNPs

Table 1: Summary of the estimated size and concentration of AgNPs from the varying synthesis parameters

pH	Concentration of lactose (% w/v)	Size (nm)	Concentration (nM)
8	1.0	30.67	0.02737 ± 0.00001
10	1.0	18.55	0.1014 ± 0.0001
10	0.5	16.46	0.07348 ± 0.00004
10	2.0	33.85	0.00760 ± 0.00005

CONCLUSION

The study presented a simple and green protocol for the preparation of AgNPs. Without using extreme reaction conditions such as high temperature and strong reducing agents, ultrasound irradiation and lactose were utilized for the synthesis of AgNPs. The synthesis of AgNPs using lactose depends on the pH of the reducing agent since there were no synthesized AgNPs using lactose at acidic pH. Sonochemical synthesis of AgNPs offered another route in preparing nanomaterials with potential applications as bioactive agents and colorimetric probes in various sensing assays.

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Conflict of interest

The author declares that there is no conflict of interest related to this article.

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