



Microwave assisted antibacterial chitosan–silver nanocomposite films



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ABSTRACT

In the current approach, antibacterial chitosan–silver nanocomposite films were fabricated through microwave irradiation. During the process, by utilizing chitosan as reducing agent, silver nanoparticles were synthesized within 11 min by microwave irradiation. Further, films were fabricated within 90 min. It involved an energy consumption of just 0.146 kWh to synthesize silver nanoparticles. This is many times less than the energy consumed during conventional methods. The silver nanoparticles were examined through UV–vis spectrum and transmission electron microscopy (TEM). The fabricated films were characterized by using scanning electron microscopy coupled with an energy dispersive spectrometer (SEM–EDS), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), and contact angle (CA) measurements. The films exhibited antibacterial properties against both Gram-negative micro-organisms (*Escherichia coli*; *E. coli*) and Gram-positive micro-organisms (*Staphylococcus aureus*; *S. aureus*). In overall, the procedure adopted for fabricating these antibacterial films is environmental friendly, time-saving and energy-saving.

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1. Introduction

Antibacterial metal nanocomposite films have gained much significance in antimicrobial field. These films were fabricated by adopting conventional routes for synthesizing the metal nanoparticles (inorganic nanopartilces) during their fabrication protocol. The conventional routes include sonication, sol gel method, microemulsion, evaporation–condensation, chemical methods, electro-chemical method, etc. [1–5]. These conventional routes utilize either more time or more energy or both. This associated problem can be minimized if microwave radiation is employed as an alternative energy system. Since, microwave heating enables efficient formation of nanostructures with small sizes in shorter reaction times with reduced energy consumption compared with conventional routes [6].

Chitosan, a polysaccharide composed of β (1 → 4) linked 2-amino-2-deoxy- β -D-glucopyranose (*N*-acetylglucosamine) is generally used as a potential material for antibacterial film production due to its attractive properties like renewable, biodegradable, antimicrobial and excellent film-forming properties [7,8]. Chitosan has also been reported to be a mild reducing agent for the reduction of silver ions and is frequently employed as an ion capping agent

to control the growth of nanoparticles and avoid their aggregation [9].

Silver nanoparticles (AgNPs) have gained notoriety to combat a broad spectrum of micro-organisms covering not only the common food prone bacteria but also various Gram-positive and Gram-negative bacteria, fungi and multidrug resistant bacteria [10,11]. The advantage of employing AgNPs in nanocomposite films is that these metal nanoparticles do not act via cell receptors to kill microorganisms [12]. Hence, the problem of disease transmission/contamination through various micro-organisms could be greatly eradicated without bringing any resistance in micro-organisms, after the film disposal [13,14].

To date, no work has been reported on microwave irradiation based fabrication of chitosan silver nanocomposite films. In the view of this, based on microwave radiation, a series of chitosan silver nanocomposite films (CSNFs) were fabricated, characterized and evaluated for antibacterial applications.

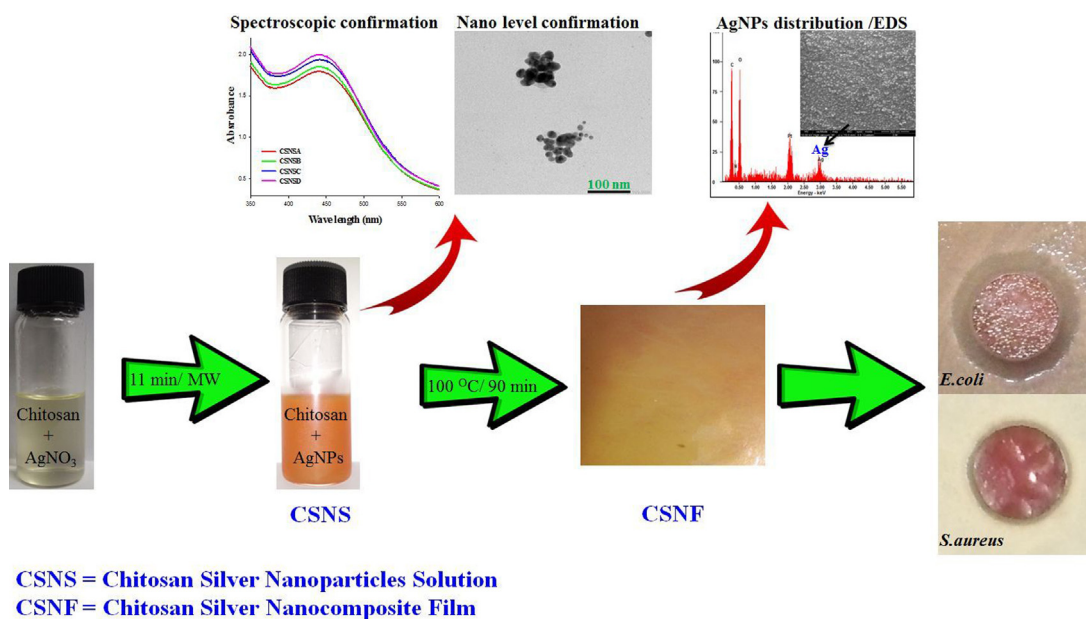
2. Experimental

2.1. Materials

Chitosan (degree of deacetylation 75–85%) and silver nitrate (AgNO_3) (ACS reagent, $\geq 99.0\%$) were purchased from Sigma Aldrich Co. (St. Louis, MO, USA). Acetic acid (10%) was purchased from DUKSAN Pure Chemical Co., Ltd., Korea. Glycerin, used as plasticizer, was purchased from Daejung Reagents Chemicals Co., Ltd., Korea.

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Scheme 1. Pictorial illustration of green fabrication of chitosan silver nanocomposite films (CSNFs) and its antibacterial efficiency.

All chemicals were used as-received, without further purification. Deionized water was used throughout experimentation.

2.2. Synthesis of silver nanoparticles (AgNPs)

Silver nanoparticles (AgNPs) were synthesized using the reducing ability of chitosanas follows. A solution was prepared by dissolving 1.5 wt% Chitosan in a 2.0 v/v% acetic acid solution by vigorous stirring over a heating magnetic stirrer at 60 °C for 3 h. The obtained chitosan solution was cooled to room temperature and sonicated until transparent. Subsequently, silver nitrate, previously diluted with 3 mL of distilled water, was added to the solution and stirred until complete dissolution was achieved. Various chitosan solutions corresponding to 17.64, 23.53, 29.41 and 35.29 mM concentrations of AgNO₃ were prepared. These solutions were then subjected to microwave irradiation in a modified microwave oven (MWO-2015, 800 W) for 11 min with an intermediate pause time of 45 s every 1 min of irradiation. The modified microwave oven for the green synthesis of silver nanoparticles contained a condenser and a Teflon-coated overhead stirrer, accessed through a hole in the microwave's top, which stirred at 75 rpm during course of reaction. The contents of the reaction vessel changed from colorless to a brownish-yellow over the course of the reaction, indicating formation of AgNPs. The chitosan AgNPs solutions were allowed to come to room temperature and then used for fabricating chitosan silver nanocomposite films. Depending on the concentration of the AgNO₃ precursor used, the chitosan silver nanoparticle solutions (CSNSs) were designated as CSNSA, CSNSB, CSNSC or CSNSD, corresponding to 17.64, 23.53, 29.41 and 35.29 mM, respectively.

2.3. Fabrication of chitosan silver nanocomposite films (CSNFs)

Glycerin (30% wt of chitosan) was added to the various chitosan silver nanoparticles solutions (CSNSs) and stirred magnetically (300 rpm) for 10 min at room temperature. The chitosan silver nanocomposite films (CSNFs) were obtained by the solvent-cast method wherein the solutions were poured into polystyrene Petri dishes and the solvent was allowed to evaporate for 90 min at 100 °C. The obtained films were peeled off and stored in air-tight polyethylene containers to protect them from light. Based on the solution chosen, the chitosan silver nanocomposite films (CSNFs)

were named CSNFA, CSNFB, CSNFC or CSNFD corresponding to the CSNSA, CSNSB, CSNSC and CSNSD solutions, respectively.

The green fabrication protocol adopted for fabricating the CSNFs is schematically presented in [Scheme 1](#).

3. Characterization

3.1. UV-vis analysis

Formation of AgNPs was spectroscopically confirmed by using a UV-Vis spectrophotometer (JASCO V-650, Japan) operated from 350 to 600 nm.

3.2. Transmission electron microscopic (TEM) analysis

To confirm the dimensions of the synthesized AgNPs, transmission electron microscopic (TEM) images were taken (Tecnai G2 Spirit, FEI Company, USA) at an accelerating voltage of 120 kV. The TEM samples were prepared by diluting the solutions and placing them onto carbon-coated copper grids (400 mesh). All specimens were plasma treated before TEM examination.

3.3. Fourier transform infrared (FTIR) spectroscopy

The interaction of Chitosan's functional groups with the synthesized AgNPs was investigated using FTIR. FTIR spectra were created for all fabricated films using a Spectrum 65 FTIR spectrometer (PerkinElmer Co., Ltd., MA, USA). These scans were performed with a wave number between 500 and 4000 cm⁻¹ using sixteen accumulated scans in attenuated total reflection (ATR) mode.

3.4. Scanning electron microscopy-energy dispersive spectrum (SEM-EDS)

A Quanta FEG250 scanning electron microscope equipped with an energy dispersive X-ray spectrometer (SEM-EDS) (FEI Co., Ltd.,) was used to observe the morphology and elemental makeup of the fabricated films. All films were scanned on both their top and fracture surfaces. Prior to examination, films were coated in platinum for 60 s a vacuum chamber.

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