International Journal of Biological Macromolecules xxx (2015) xxx–xxx

Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac

Ag^0 nanoparticles containing cotton fabric: Synthesis, characterization, color data and antibacterial action

Q1 Hossam E. Emam [a,*], M.K. Zahran [b]

[a] Textile Research Division, National Research Centre, Dokki, Cairo 12622, Egypt
[b] Chemistry Department, Faculty of Science, Helwan University, Ain-Helwan, Cairo 11795, Egypt

ARTICLE INFO

Article history:
Received 5 September 2014
Received in revised form
15 December 2014
Accepted 19 December 2014
Available online xxx

Keywords:
AgNPs
Xanthan
Reducing sugars
Silver content
Color data
Antibacterial activities

ABSTRACT

The main objective of the current research was to successfully employ the reducing and stabilizing features of xanthan gum to synthesize nanosilver, then coating cotton fabrics with the net produced nanosilver in order to obtain finished fabrics valuable in medical applications. Pre-hydrolyzed xanthan gum was used to reduce Ag^+ to Ag^0 in nano size using a simple one-step rapid synthetic route. The reduction step was followed up by measuring the concentration of reducing sugars eliminated in the reaction medium. The optimum concentration of xanthan gum was 3 g/L to reduce 1 mmol/L Ag^+, as 2.66 ± 0.4 g/L was the maximum concentration of reducing sugars obtained in the reaction. Transmission microscope images show that the AgNPs are spherical in shape with mean size 9.1 ± 4.8 nm. Cotton fabrics were then coated with the produced AgNPs using pad-dry-cure method. Well dispersed layer from Ag^0 on cotton surface was showed under electron microscope. The biocidal activities of the coated fabrics were tested against *Staphylococcus aureus* and showed excellent results for antibacterial even after 20 washing cycles. This method has the advantage of not necessitating aggressive conditions such as the presence of organic solvents to produce durable antibacterial cotton fabrics.

1. Introduction

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Studies and preparation of silver nanoparticles is one of the most progressively growing areas in the nanotechnology research. Silver is a historically well known metal for its broad antimicrobial activity while nanocrystalline silver was reported to inhibit microbial growth more rapidly and completely than its cationic form [1]. The main problem of silver nanoparticles application is an aggregation process, causing the loss of their unique properties. Therefore, the research focuses on finding suitable compounds that increase the aggregate stability of nanoparticles. Suitable substances can be found between the polymers (polyvinyl alcohol, poly-acrylamides) and the surfactants (Tweens). Using natural substances instead of toxic synthetic polymers can positively influence the toxicological properties.

Many researchers tried to use various modifiers or dispersants based on natural substances to coat the nanoparticles to increase steric and electrostatic hindrance among the particles which results in the stability of the nanoparticles suspension [2–4]. Different chemicals were used to modify the particle surface such as cellulose [1,5,6] and carboxymethyl cellulose [7].

Xanthan gum (Fig. 1a) is a polysaccharide secreted by the bacterium *Xanthomonas campestris* [8], used as a food additive and rheology modifier [9], commonly used as a food thickening agent (in salad dressings, for example) and a stabilizer (in cosmetic products, for example, to prevent ingredients from separating). It is composed of penta-saccharide repeat units, comprising glucose, mannose, and glucuronic acid at the molecular ratio of 2.0:2.0:1.0 [10,11].

Xanthan is produced by the fermentation of glucose, sucrose, or lactose. After a fermentation period, the polysaccharide is precipitated from a growth medium with isopropyl alcohol, dried, and ground into a fine powder. Later, it is added to a liquid medium to form the gum. The molecular weight of xanthan gum can reach up to 6 million Daltons, which makes it possible to create extremely viscous solutions at very low concentrations [12].

It has been reported that, concentrations above 0.5% (w/v) of xanthan gum attain relatively high viscosity indicating the pseudoplastic behavior of the gum. Such behavior is a characteristics feature shown by polymeric solutions of microbial polysaccharides with large molecular weight. Xanthan solutions are specific in their ability to retain their viscosity until it reaches its melting temperature. At such temperature, the viscosity sharply decreases

* Corresponding author. Tel.: +20 201008002487.
E-mail address: hossamelemam@yahoo.com (H.E. Emam).

http://dx.doi.org/10.1016/j.ijbiomac.2014.12.050
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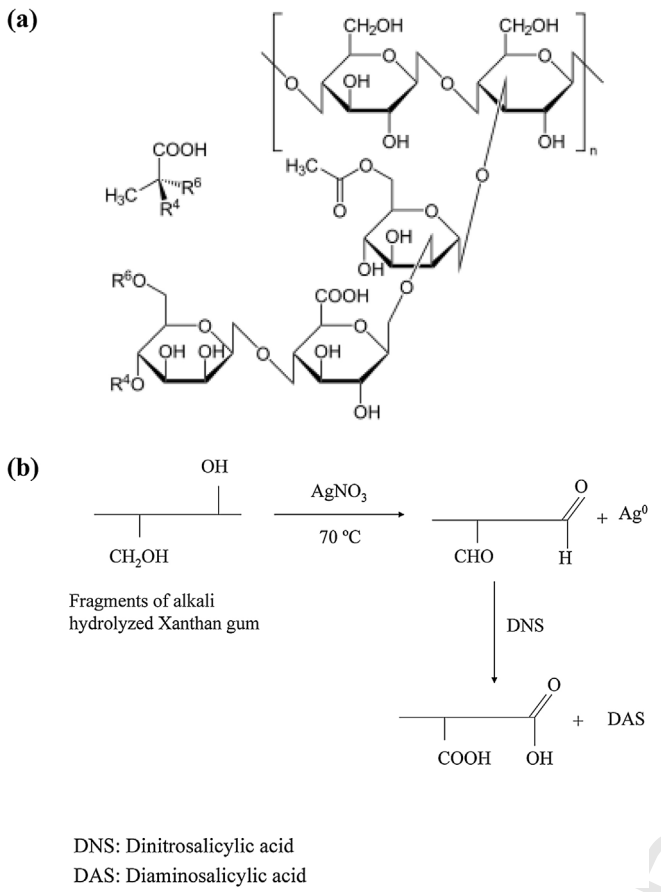

Fig. 1. (a) Xanthan macromecule and (b) schematic diagram for detection of reducing sugars content using DNS reagent.

due to a reversible molecular change. Decrease in the viscosity of xanthan solution was observed by increasing temperature up to 80 °C, though it was only a transient phenomenon but the solution returned to their original viscosity upon cooling. Since xanthan is a neutral and nonionic polymer [13], its viscosity was independent of pH and remained stable under pH 2–10. Such stability of xanthan at various pH ranges makes it preferably applicable in food products [11,14–17].

Comba and Sethi [18] used the biopolymer xanthan gum to disperse nano Fe^0, and found that highly concentrated nano Fe^0 slurries (15 g/L) could be stabilized for more than 10 d after adding 6 g/L of xanthan gum. The sand-packed column experiment also proved that xanthan gum is an excellent stabilizing agent and delivery vehicle of nano Fe^0 particles with high potential for use in real scale remediation interventions [18,19].

The current work aimed to study the preparation of silver nanoparticles (AgNPs) using xanthan gum, which was used as both a reducing and capping agent for the net produced nanoparticles, as synthesis of Silver nanoparticles (AgNPs) in combination with biodegradable, biocompatible polymers can be an interesting field of research for preparation of antimicrobial materials [1,3,20,21]. Polymers like xanthan in solutions and as hydrogels were shown to be effective capping agents for AgNPs providing in the same time biocompatibility and diversity of forms and structures, and thus possibilities for variety of biomedical applications such as antimicrobial coatings, wound dressings and, potentially, tissue implants.

The reduction process followed up by measuring the concentration of reducing sugars. The characterization of AgNPs was performed by UV–vis spectroscopy and transmission electron microscope (TEM). The particle size was determined by image analysis of TEM images. The produced AgNPs was then coated on the surface of cotton fabrics for medical applications. Coated fabrics were examined for surface characteristics by using scanning electron microscopy. Color coordinates, silver content and silver release percent were calculated to evaluate the laundering durability of nanosilver coated cotton fabrics. The biocidal effects of the coated fabrics were detected against *Staphylococcus aureus* (Gram +ve) bacterial strains.

2. Experimental

2.1. Materials and chemicals

Silver nitrate (99.5%), xanthan gum $(C_{35}H_{49}O_{29})_n$, supplied from JK Bio-Chem Co., Limited—China), sodium hydroxide, 3, 5-dinitro salicylic acid (DNS), sodium sulphite, potassium sodium tartarate, glucose, phenol, and sodium carbonate monohydrate, sodium hydroxide and nitric acid (55%) were all used without further purification. Desized, scoured, and bleached 100% cotton fabrics were kindly supplied from El-Mahalla Company for Spinning and Weaving, El-Mahalla El-Kubra—Egypt and used as received without any treatments.

2.2. Methodology

2.2.1. Preparation of silver nanoparticles

Different weights of xanthan (0.1–6 g/L) were treated with sodium hydroxide solution using magnetic stirrer to prepare different solutions of hydrolyzed xanthan with pH ≈12 in order to serve the dual role as a reductant of silver nitrate and stabilizer for the produced nanosilver. After complete dissolution, the temperature of the reaction medium was raised to the desired degree (30–80 °C). In this moment, certain amount from silver nitrate solution (0.1 M) was added dropwise (keeping in mind that the total volume of the reactants is 100 mL). The reaction was kept under continuous stirring for different durations (1–60 min). After addition of silver nitrate, the reaction medium acquires a brownish color indicating that silver nanoparticles may be produced. The progression of the reaction was controlled by UV–visible absorption and reducing sugars content; aliquots from the reaction bulk were withdrawn at given intervals time and both spectra and reducing sugars were evaluated.

2.2.2. Coating process

Incorporation of AgNPs in cotton fabrics was performed by pad–dry–cure method. A 20 cm × 20 cm piece of cotton fabrics were immersed in AgNPs solution bath containing 100 ppm of AgNPs for one minute and squeezed to 100% wet pick up using laboratory pad at constant pressure. The samples were dried at 75 ± 5 °C for 20 min and cured at 120 ± 5 °C for 3 min as thermal fixation of nanoparticles on fabrics surface.

3. Analyses and measurements

3.1. Determination of reducing sugars contents

The dinitrosalicylic acid reagent (DNS) was used for the determination of concentrations of reducing sugars. This method was carried out to detect the free carbonyl groups of the remaining reducing sugars at the end of redox reaction which is supposed to take a place between silver nitrate and hydrolyzed xanthan gum (Fig. 1b). The reagent is composed of dinitrosalicylic acid, Rochelle salt (sodium potassium tartarate), phenol, sodium bisulfite, and sodium hydroxide. According to Sumner [22], Rochelle salt is added to prevent the reagent from dissolving oxygen, phenol is used to