

ORIGINAL RESEARCH PAPER

Preparation of Chitosan-Copper Nanoparticles Coated Kraft Paper, Characterization and its Antimicrobial Activity

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ABSTRACT

In this paper, Chitosan-copper nanoparticles were successfully coated onto the Kraft paper using a dip coating process. First, chitosan-copper nanoparticles solution was prepared by reduction method and the use of ultrasonic radiation. It was employed copper sulphate pentahydrate as precursor salt, NaOH as a reducing agent and Chitosan as capping agent to prepare Chitosan-copper nanoparticles solution. The solution was analyzed by UV-Vis spectroscopy and dynamic light scattering (DLS). After the coating process, the coated Kraft paper was characterized by X-Ray diffraction and scanning electron microscopy (SEM). Then, the antimicrobial activity of the Kraft paper, in contrast to *Bacillus subtilis* and *Escherichia coli* was measured by disc diffusion method.

Keywords: Chitosan, Copper, Nanoparticles, Kraft paper, Coating.

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INTRODUCTION

Nanotechnology plays a crucial key role in various technologies of the new millennium. Metal nanoparticles exhibit unique electronic, magnetic, catalytic and optical properties that are different from those of bulk metals. Among different metal particles, copper nanoparticles, due to their unique physical and chemical properties and low-cost preparation, have been of great interest recently [1]. Copper nanoparticles have great applications as heat transfer systems, antibacterial materials, super strong materials, sensors, and catalysts [2, 3, 4]. Copper nanoparticles acts as an anti-biotic, anti-microbial, and anti-fungal agent when added to plastics, coatings, and textiles [5].

Generally, there are various methods to synthesize copper nanoparticles. Many methods

are on the basis of the reduction of Cu^{2+} ions in a solution. In these methods, it is used from a reducing agent for the reduction of Cu^{2+} ions in solution (especially). It could state the difference of these methods on base this same reducing agent [6]. In chemical reduction methods, reducing agent is a chemical solution such as polyols, NaBH_4 , and N_2H_4 , whereas in biological methods, collection of enzymes especially reductases play such role. From among mentioned methods, chemical method is most application to synthesize copper nanoparticles. However, nowadays, it is used for such methods to synthesize copper nanoparticles in big scales. In addition, Physical methods are usually fast and do not involve toxic chemicals. The process of preparing nanoparticles using physical methods is faster than chemical and biological methods. The general disadvantage of physical

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methods is the high cost of equipment to prepare the nanoparticles [7]. Pinto et al. (2012) also studied the nucleation, growth and chemical stability of copper nanoparticles in vegetable and bacterial cellulose. They suggested delaying surface oxidation of bacterial cellulose fibers as the most efficient substrate [8]. Vainio et al. (2007) prepared Cu, CuO, and Cu₂O particles in microcrystalline cellulose by adding a copper salt solution to an insoluble cellulose matrix and reducing the copper ions with several reducing agents [9]. Li et al. (2012) reported a simple hydrothermal process to produce Cu from CuO using cellulose as a reducing agent under mild hydrothermal conditions of 250 °C for 1.5 h in NaOH [10]. Recently, Komeily-Nia et al. (2013) reported synthesis of copper nanoparticles on nylon fabric with antibacterial properties [11].

In the current study, we employed copper sulphate pentahydrate as precursor salt, NaOH as a reducing agent and chitosan as capping agent to prepare chitosan-copper nanoparticles solution. In other hand, it includes in situ generation of nanoparticles suspension in the reaction container and the coating on the Kraft paper. The coated Kraft paper showed great antibacterial properties against *B. Subtilis* and *E. Coli*.

MATERIAL AND METHODS

Materials

Copper sulphate pentahydrate (CuSO₄·5H₂O), Ascorbic acid (C₆H₈O₆), Sodium hydroxide (NaOH) and chitosan were purchased from Sigma Aldrich. Distilled water was used for all the experiments. Kraft paper was provided by Pars Golden Trade, Company, Tehran, Iran.

Synthesis and Coating of Kraft Paper

The chitosan-copper nanoparticles were synthesized by chemical reduction process using CuSO₄·5H₂O as precursor salt and chitosan as capping agent. First, chitosan solution was added to CuSO₄·5H₂O solution. Then, ascorbic acid solution was added to the synthesis solution under continuous fast mixing. Subsequently, NaOH (0.1 M) solution was slowly added to the prepared solution with mixing process and heating at 90 °C for 1 h. After the completion of reaction (overnight), the precipitates were separated from the solution by centrifuging at 5000 rpm for 20 min. The precipitate was

suspended in water, centrifuged again, and then freeze-dried. The freeze-dried chitosan-copper nanoparticles were then resuspended for coating purpose. Uv-vis spectroscopy was applied to confirm the existence of nanoparticles. The size was determined by dynamic light scattering (DLS). The chitosan-copper nanoparticles suspension was sonicated for 20 min. A piece of Kraft paper was submerged into the bath containing the prepared nanoparticles solution, and after 30 min, it was withdrawn from the bath. The coated Kraft paper was dried in a hot-air oven to remove the water solvent and further stabilized under room environmental conditions for at least one day to characterization.

Characterization of Coatings

To confirm the coating of chitosan-copper nanoparticles on the Kraft paper, XRD spectra were used. Also, the structure of the chitosan-copper nanoparticles was studied using X-ray diffraction. The morphology and the size of the nanoparticles were studied with a scanning electron microscopy (SEM).

Antibacterial activity

The disc diffusion method was used to specify the antibacterial activity of coated Kraft paper in contrast to *B. subtilis* and *E. coli*. This method was performed in nutrient agar medium solid agar Petri dish. Samples of coated Kraft paper (1 cm²) and Kraft paper (control sample) were placed in the nutrient agar Petri dish and kept for incubation at 37°C for 1 day. Zones of inhibition for two antibiotics, coated Kraft paper and Kraft paper were measured. The experiments were accomplished three times, and the average values of zone diameter were determined.

RESULT AND DISCUSSION

UV-Vis spectrum of solution at different wavelengths ranging from 400 to 800 nm showed strong absorption peak with centring at 588 nm indicated the formation of copper nanoparticles (Fig. 1). As illustrated the SPR band centered 588 nm confirms the formation of chitosan-copper nanoparticles in the solution. On the other, the size of nanoparticles synthesized are measured by dynamic light scattering (DLS). This technique is used widely for the determination of nanoparticles size in a colloidal solution. The average size of nanoparticles is about 60

nm which shows perfect distribution of the nanoparticles (Fig. 2).

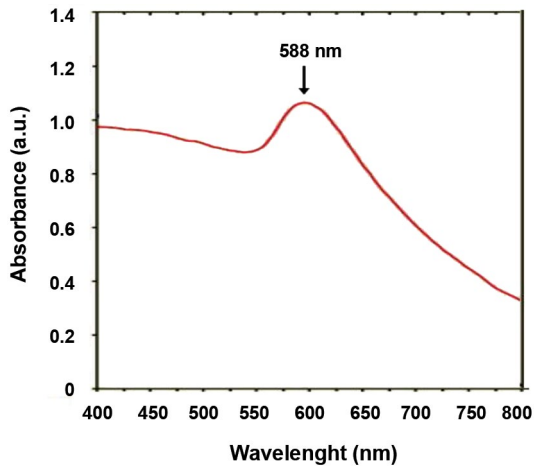


Fig. 1. UV-vis spectra of chitosan-copper nanoparticles solution.

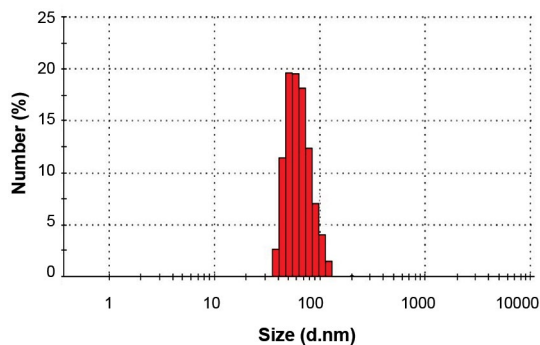


Fig. 2. The size distribution of chitosan-copper nanoparticles by number.

To confirm the coating of chitosan-copper nanoparticles on the Kraft paper, XRD spectra were used. XRD spectra recorded from coated Kraft paper after 30 min of sonication are shown in figure 3. The XRD pattern recorded from Kraft paper showed peaks at 2θ value of 42.89° (111), 49.99° (200) and 73.61° (220) characteristic of copper and were in good agreement with literature report and confirmed copper nanoparticles [12]. In addition, SEM results also support this observation. The chitosan-copper nanoparticles coated Kraft paper was studied by scanning electron microscopy (SEM) and images show and confirm chitosan-copper nanoparticles

on Kraft paper. SEM images of nanoparticles on Kraft paper are shown in figure 4.

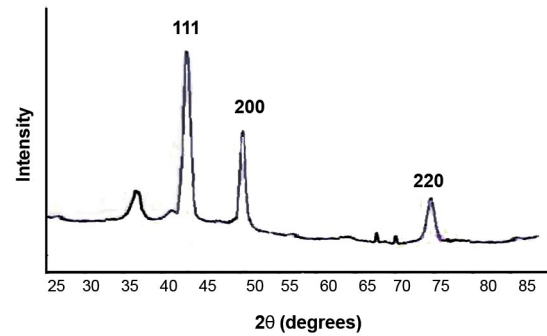


Fig.3. X-ray diffraction pattern of coated Kraft paper.

The chitosan-copper nanoparticles show an effective antibacterial activity against gram negative and gram-positive bacteria. The result suggests that chitosan-copper nanoparticles undergo a contact with bacterial wall and showed the strong action against *B. subtilis* and *E. coli*. In this work, the chitosan-copper nanoparticles coated Kraft paper was tested on bacterial strains *B. subtilis* and *E. coli*. The formation of clear zone around the hole is a sign of antibacterial activity. As can be realized, the antibacterial activity of nanoparticles coated Kraft paper on *E. coli* is more than *B. subtilis* (Figure not shown). The zone of inhibition of diameters was determined. However, it is obvious that the chitosan-copper nanoparticles coated Kraft paper shows perfect antibacterial activity against both *B. subtilis* and *E. coli* whereas Kraft paper (control sample) show no zone of inhibition. The results have been summarized in Table 1.

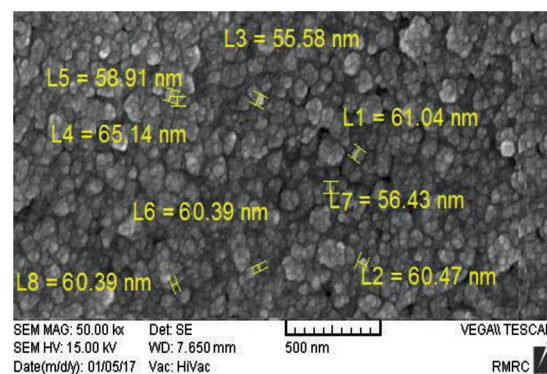


Fig. 4. SEM image of coated Kraft paper.

Table 1. The antibacterial activity of the chitosan-copper nanoparticles coated Kraft paper, zone of inhibition (mm)

Organism	Coated Kraft paper	Kraft paper
B. subtilis	7.5	0
E. coli	9.8	0

CONCLUSIONS

In current work, the chitosan-copper nanoparticles were successfully synthesized by reduction technique using NaOH as reducing agent, and copper sulphate pentahydrate as precursor salt and chitosan as capping agent. The size range of the produced chitosan-copper nanoparticles was about 60 nm. These chitosan-copper nanoparticles were successfully coated onto the Kraft paper using a dip coating process. The coated Kraft paper established very perfect antibacterial activity in contrast to gram negative and gram positive bacteria.

CONFLICT OF INTEREST

The authors declare that there is no conflict

of interests regarding the publication of this manuscript.

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