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Usage of Carbohydrate in situ Green Synthesis of Nano Copper On Morphology Properties Cotton Fabric Focuses on Absorbency

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Abstract

In this study, the effect of copper nanoparticles synthesized via a green method on the waterproofing properties of cotton fabric was investigated. Copper nanoparticles were prepared using aqueous solutions with varying concentrations of copper(II) nitrate (1–2%), sucrose (1–3%), and a 2% dispersing agent at pH 5 and 50 °C for one hour. Subsequently, pre-scoured cotton fabrics were treated with the synthesized nanoparticles at 50 °C for 60 minutes. The structure and morphology of both scoured and treated fabrics were characterized using X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). The particle size of the copper nanoparticles was analyzed by dynamic light scattering (DLS). The waterproofing performance of the samples was evaluated using the standard wicking test (AATCC 197-2013). The results revealed that the most effective waterproofing was achieved when copper nanoparticles were synthesized in a bath containing 1% copper nitrate, 1% sucrose, and 2% dispersing agent, resulting in nanoparticles smaller than 100 nm. This environmentally friendly approach provides a cost-effective and sustainable method for enhancing the functional properties of cotton fabrics, paving the way for advanced and sustainable practices in the textile industry.

Keywords: Green synthesis, copper nanoparticles, water repellency, cotton fabric, morphology.

1. Introduction

Nanotechnology has rapidly emerged as a critical field in modern research, enabling the design and manipulation of nanoparticles with significant impacts across a wide range of applications [1–3]. Reducing materials to the nanoscale dramatically increases their surface area-to-volume ratio (A/V), which in turn leads to pronounced changes in both their physical and chemical properties [4]. Research on nanostructures focuses on their unique ability to modify and enhance the properties of materials at the macroscopic scale.

Among metallic nanoparticles, copper nanoparticles have garnered particular attention due to their low cost and favorable properties compared to other metals [5]. Numerous synthesis methods for copper nanoparticles have been investigated, including chemical reduction in aqueous solutions [6], colloidal synthesis [7], and the evaporation-condensation of metal vapor onto a cold substrate [8,9]. However, some of these chemical reduction methods employ reducing agents that may pose potential risks to human health and the environment, and are typically costly [10]. Therefore, there is an increasing need for the development of safer and more environmentally benign synthesis approaches to improve the biocompatibility of nanoparticles.

Green synthesis, or biosynthesis, has emerged as an important alternative due to its clean, non-toxic, and environmentally friendly procedures [11]. It has been reported that metal nanoparticles such as Au, Ag, Pt, and Pd can be synthesized using commonly available sugars—including glucose, fructose, and sucrose—as reducing agents, without the need for additional stabilizers or capping agents. Furthermore, polysaccharides, which are polymers of monosaccharides containing various reactive groups (such as hydroxyl, carboxyl, and amino groups), have been widely used in the development of nanoparticle-based drug delivery systems [12]. However, relatively few studies have examined the use of polysaccharides as reducing agents for metal salt precursors to produce metallic nanoparticles [13–16].

A key criterion for textile materials is their ability to absorb and repel water. This complex mechanism involves structural modifications of fibrous materials during water absorption or repulsion. Sorption isotherms are frequently used to describe the relationship between the equilibrium moisture content of a substance and the surrounding relative humidity (RH) at a constant temperature. Water absorption in fibers is of particular significance due to its impact on their mechanical properties; as a result, various researchers have proposed and investigated different isotherms to characterize the moisture absorption behavior of fibers. The chemical nature of the fiber plays a dominant role in these properties. The presence of diverse adsorption and desorption sites, both on the surface and within the core of hydrophilic fibers, enhances the formation of hydrogen bonds with water vapor molecules compared to hydrophobic fibers [17].

In this study, we investigated the waterproofing properties of cotton samples—both scoured and treated with green-synthesized copper nanoparticles. Copper nanoparticles were synthesized under various conditions at 50 °C and pH 5 for 60 minutes, followed by treatment of pre-scoured cotton fabrics. The samples were characterized using scanning electron microscopy (SEM) to assess surface morphology, Fourier-transform infrared spectroscopy (FTIR) to identify functional groups, energy-dispersive X-ray spectroscopy (EDX) for elemental analysis, and X-ray diffraction

(XRD) to evaluate crystallinity and morphology. The particle size of synthesized copper nanoparticles was determined using dynamic light scattering (DLS). The waterproofing properties of the treated cotton fabrics were examined using the standard wicking test. The results presented in this study provide valuable insights into the application of copper nanoparticles for enhancing the functional performance of cotton textiles in the industry.

2. Experimental

2.1. Materials

A woven 100% cotton fabric with a density of 25 ends/picks per centimeter was used in this study. Copper nitrate, sodium carbonate, sucrose, ascorbic acid, and the dispersing agent (Setamol XP-D) were all purchased from Merck. Diadavin UN served as a nonionic detergent.

2.2. Instrumentation

Scanning electron microscopy (SEM; Kyky-EM3200 Model) was utilized to examine the surface morphology of the cotton samples. Fourier-transform infrared spectroscopy (FTIR; Bruker Tensor Model 27) was employed to analyze the functional groups present in the samples. X-ray diffraction (XRD; Equinox 200 Model) was used to study crystallinity and morphology. Elemental analysis was carried out using an EDX microprobe (Horiba XGT 7200). The particle size distribution of the synthesized copper nanoparticles was determined using dynamic light scattering (Zetasizer Nano-ZS, Malvern Instruments, Ltd.).

2.3. Methods

2.3.1. Scouring and Preparation of Cotton Fabric

The cotton fabric was scoured in a bath containing 2 g/L nonionic detergent and 1 g/L soda ash at 70 °C, with a liquor ratio of 30:1, for 30 minutes. After scouring, the samples were washed thoroughly with cold water and air-dried.

2.3.2. Synthesis of Copper Nanoparticles

Copper nanoparticles were synthesized in aqueous solutions containing different concentrations of copper nitrate (1–2%), sucrose (1–3%), and 2% dispersing agent at pH 5 and 50 °C for 1 hour.

2.3.3. Treatment of Cotton Samples

The pre-scoured cotton samples were immersed in the copper nanoparticle solution and treated at 50 °C for 60 minutes. Following the treatment, all samples were air-dried at room temperature.

2.3.4. Wicking Test

The wicking test was conducted according to AATCC 197-2013 to evaluate moisture absorption and regain in both scoured and treated samples.

3. Results and Discussion

3.1. Wicking Test

The moisture absorption of the cotton samples—both scoured and treated with copper nanoparticles—was evaluated using the wicking test according to AATCC 197-2013. The results are presented in Figures 1 and 2. It was observed that the best waterproofing effect was achieved in the sample treated with 1% copper nitrate, 1% sucrose, and 2% dispersing agent.

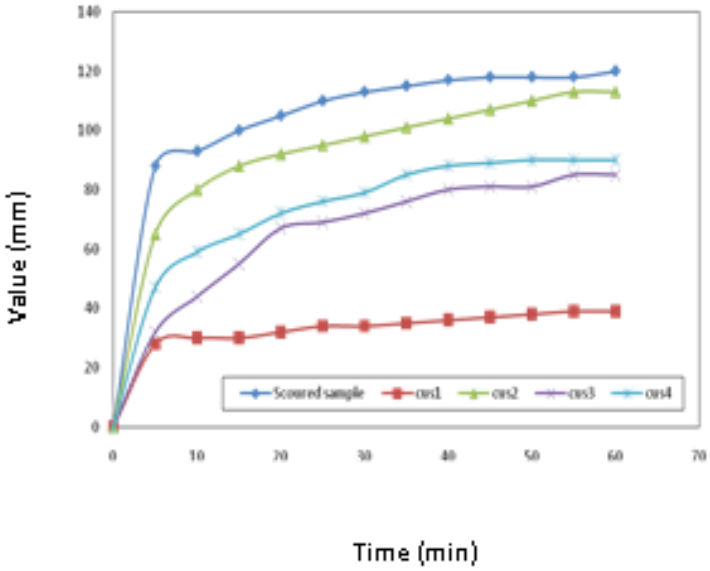


Figure 1. Wicking test results of cotton samples at different times. (Cus1: copper nitrate 1%, sucrose 1%, and dispersing agent 2%. Cus2: copper nitrate 2%, sucrose 1%, and dispersing agent 2%. Cus3: copper nitrate 1%, sucrose 3%, and dispersing agent 2%. Cus4: copper nitrate 2%, sucrose 3%, and dispersing agent 2 %.

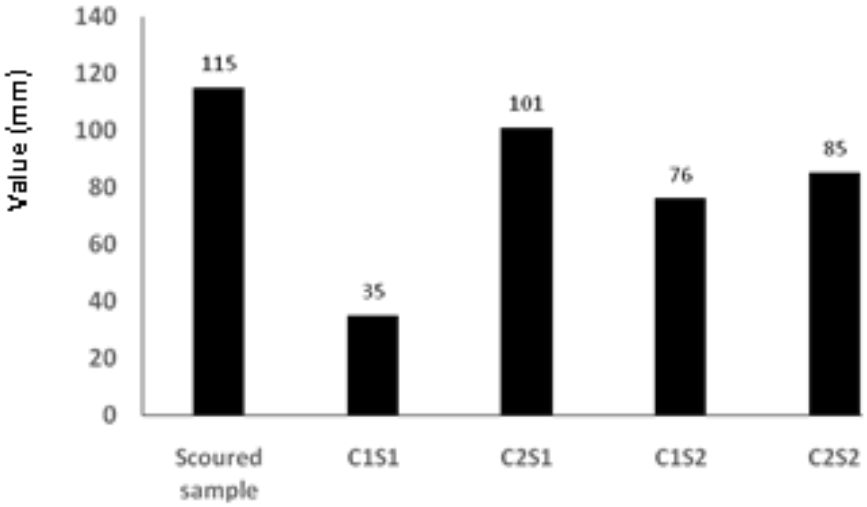


Figure 2. Wicking Test results of cotton samples after 60 mins. (Cus1: copper nitrate 1%, sucrose 1%, and dispersing agent 2%. Cus2: copper nitrate 2%, sucrose 1%, and dispersing agent 2%. Cus3: copper nitrate 1%, sucrose 3%, and dispersing agent 2%. Cus4: copper nitrate 2%, sucrose 3%, and dispersing agent 2 %.)

3.2. FTIR/ATR Analysis

The functional groups present on the surface of the cotton samples were investigated using FTIR spectroscopy (Bruker Tensor Model 27) in the range of 500–3500 cm^{-1}. The FTIR spectra are shown in Figure 3. As illustrated in Figure 3(a), the characteristic absorption of hydroxyl groups (–OH) appears at approximately 3440.02 cm^{-1}. In the treated sample (Figure 3(b)), this peak is shifted to 3507.96 cm^{-1}. Similar bands are observed between 3000–3600 cm^{-1} in both spectra [19], corresponding to –OH groups. The shift in the OH band for the treated cotton sample indicates a modification of the cellulose structure, which can be attributed to the presence and interaction of the synthesized copper nanoparticles.

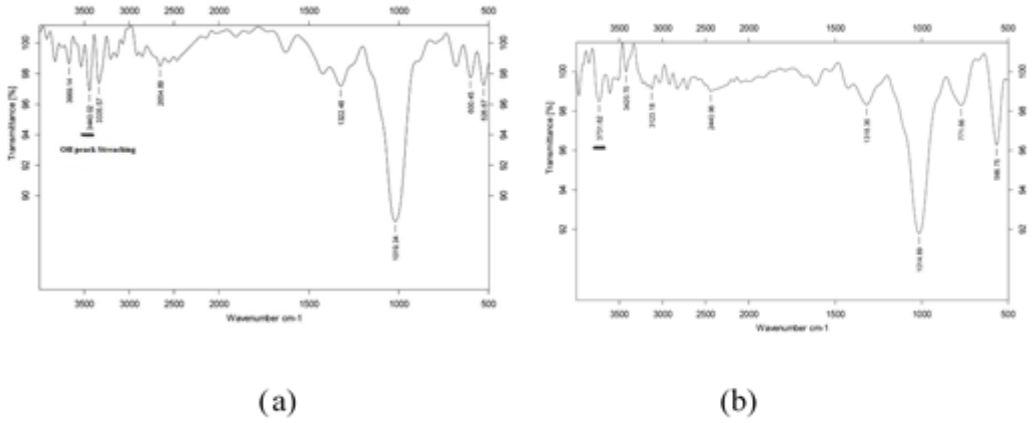
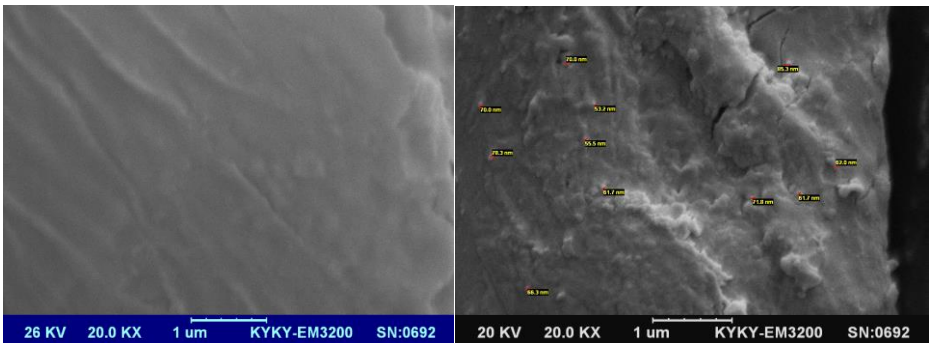


Figure 3. FTIR spectrum of cotton samples: (a) scoured (b) treated cotton fabric with copper nanoparticles. Synthesis condition: copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

3.3. SEM Analysis

The surface morphology of the cotton samples was examined using a KYKY-EM3200 scanning electron microscope. Imaging was conducted at an accelerating voltage of 26 kV and a magnification of 20,000×. Prior to imaging, the samples were sputter-coated with a thin layer of gold to prevent surface charging and enhance image clarity under high-vacuum conditions. As shown in Figure 4, the untreated cotton sample (Figure 4(a)) exhibits a relatively smooth surface, whereas the treated sample (Figure 4(b)) is covered with copper nanoparticles uniformly distributed along the fiber surface. Image analysis revealed that the average size of the copper nanoparticles adsorbed onto the cotton surface was approximately 79.21 nm.



(a) (b)

Figure 4. SEM micrographs of cotton sample: (a) scoured and (b) treated cotton fabric with copper nanoparticles—synthesis conditions: copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

3.4. XRD Analysis

The morphology and crystallinity of the synthesized copper nanoparticles were examined using X-ray diffraction (XRD, Equinox 200 model). Prior to analysis, both scoured and treated cotton samples were ashed in a Nabertherm furnace (30–3000 °C, B170 controller) at 600 °C for 3 hours to obtain appropriate specimens for the test. The XRD measurements were conducted using Cu Kα radiation (λ = 1.5406 Å), over a 2θ range of 10°–120°, with a step size of 0.02°. Figure 5 shows the diffraction patterns of the scoured and treated cotton samples. Several peaks are in good agreement with the standard reference patterns of metallic copper and copper oxide, confirming the successful synthesis and deposition of copper-based nanoparticles on the surface of the treated cotton fabric [20].

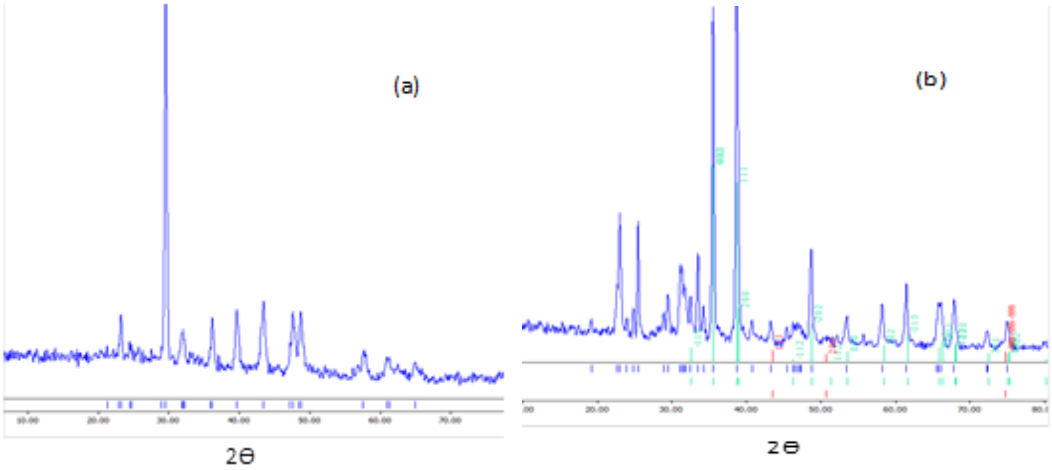


Figure 5. XRD graphs of cotton sample: (a) scoured and (b) treated cotton fabric with copper nanoparticles. Copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

In X-ray diffraction and crystallography, the Scherrer equation (Equation 1) is used to relate the size of sub-micrometer particles or crystallites in a solid to the broadening of the diffraction peaks in the XRD pattern [18,19].

$$\tau = K\lambda/(\beta\cos\theta) \qquad \text{equation 1}$$

where:

- **τ** is the mean size of the ordered (crystalline) domains, which may be smaller than or equal to the grain size;
- **K** is a dimensionless shape factor, typically close to unity (commonly taken as 0.9), but its exact value depends on the actual shape of the crystallites;
- **λ** is the X-ray wavelength;
- **β** is the full width at half maximum (FWHM) of the diffraction peak (in radians) after correction for instrumental broadening, sometimes denoted as Δ(2θ);
- **θ** is the Bragg diffraction angle.

Table 1 presents the XRD results for the cotton samples. The data show that copper nanoparticles were synthesized in various forms and sizes, and the observed peaks correspond to the standard reference peaks for copper nanoparticles. These findings confirm the successful synthesis of copper nanoparticles.

Table 1. XRD analysis of treated cotton sample with copper nanoparticles. Synthesis condition: copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

[°2Th.]	FWHM of Intense peak (β) radians	Size of the particle (τ) nm	Plane
43.23	0.2509	5.952	(111)
53.48	0.2509	6.192	(200)
74.93	0.2509	11.558	(220)

3.5. EDX Analysis

The elemental composition of the treated cotton samples was examined using energy-dispersive X-ray spectroscopy (EDX). Figure 6 presents the EDX spectrum of the treated fabric. The results confirm the presence of synthesized copper nanoparticles on the cotton surface, with a measured copper content of 0.66%. Additionally, other elements were detected in the EDX analysis, which are likely attributable to the inherent mineral impurities present in the cotton samples.

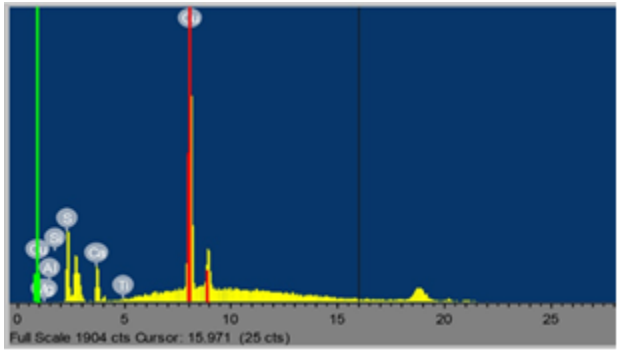


Figure 6. The EDX graph of the treated cotton sample. Synthesis condition: copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

3.6. Particle Size Analysis

The particle size distribution of the synthesized copper nanoparticles is illustrated in Figure 7. The results indicate that, under optimal synthesis conditions—specifically, a bath containing 1% copper nitrate, 1% sucrose, and 2% dispersing agent—the average size of the copper nanoparticles was less than 100 nm.

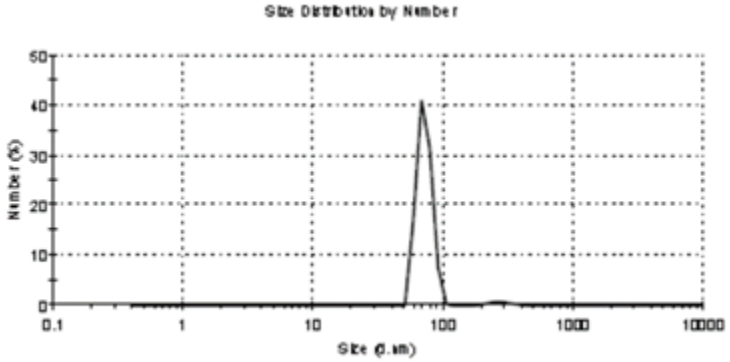


Figure 7. The DLS graph of synthesized copper nanoparticles. Synthesis condition: copper nitrate 1%, sucrose 1%, and dispersing agent 2%.

4. Conclusion

In this study, the effect of green-synthesized copper nanoparticles on the waterproofing properties of cotton fabric was investigated. Copper nanoparticles were synthesized in solutions containing various concentrations of copper nitrate, sucrose, and dispersing agents at pH 5 and 50 °C for one hour. The pre-scoured cotton fabrics were then treated with the synthesized nanoparticles at 50 °C for 60 minutes. Comprehensive characterization of the untreated and treated samples was performed using XRD, EDX, SEM, and FTIR, while the particle size was assessed by DLS. The waterproofing performance was evaluated using the wicking test according to AATCC 197-2013. The findings demonstrated that the optimal waterproofing effect was achieved when copper nanoparticles were synthesized in a bath containing 1% copper nitrate, 1% sucrose, and 2% dispersing agent, yielding nanoparticles with an average size below 100 nm. Treatment of cotton fabric with these copper nanoparticles significantly enhanced its water-repellent properties. The outcomes of this research highlight the potential of green-synthesized copper nanoparticles for the development of advanced, sustainable textile finishes in the industry.

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