Exploring Reduction Time and Precursor Effects on Cu Nanoparticle Size in A Nontoxic Chemical Reduction Method

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Abstract

In order to develop the properties of nanomaterials, controlling the crystallite size, shape and synthesis conditions comes into picture. Here, copper (Cu) nanoparticles, having been synthesized by chemical reduction, employed copper nitrate and copper sulfate as precursors, prepared with L-ascorbic acid as reducing agent, without using any capping agent. The various times of reduction include 4, 8, 12, 16, 20, and 24 hours. Characterization is carried out by using XRD, TEM, FESEM, and EDS. Results showed that increased crystallinity was obtained from longer reduction times. It was also noted that the utilization of copper nitrate-generated nanoparticles resulted to crystallinity being higher than that of copper sulfate. Although the size of crystallites remained constant for 4-20 hours, it increased to the maximum size of 45 ± 11 nm during the 24-hour reduction. Copper nitrate-derived nanoparticles, however, were always bigger than copper sulfate-derived nanoparticles. Fine crystalline pure Cu was obtained after 20 hours of reduction of copper nitrate. This study thus emphasizes on the way the duration and type of precursor have a great role in determining the actual quality of Cu nanoparticles, thus finding a use in guiding researchers in modifying nanomaterials for electronics, catalysis, and material science applications.

Keywords: Cu nanoparticles; Chemical reduction method; Reduction time; Reduction agent; crystallite size

1. Introduction

Nanotechnology is a rapidly emerging field with a wide range of applications, including sensing, diagnostics, biomedical imaging, and medicines. Differentiable physicochemical properties of nanoparticles include their size, shape, and surface-to-volume ratio. Their typical size ranges from 1 to 100 nm [1-3]. These characteristics make nanoparticles essential in a wide range of applications, including biomedicine, color degradation, wastewater treatment, catalysis, medicinal delivery, and diagnostics. They also enhance surface activity, catalytic function, and interactions with other particles [4-6]. Copper (Cu) nanoparticles are one of the most well-known synthetic nanomaterials due to their unique mechanical, electrical, magnetic, and thermal characteristics. These characteristics are applied to surgical instrument antimicrobial coatings, heat transfer systems, and water treatment. Since copper is widely available and reasonably priced, it is economically viable to produce Cu nanoparticles; yet, a major barrier still remains as a result of their oxidation susceptibility in aquatic environments [7-9]. Inorganic nanoparticles are made via a variety of physical and chemical processes, such as chemical co-precipitation, sol-gel processes, hydrothermal methods, chemical vapor deposition, electrochemical processes, and microwave synthesis. However, because these techniques typically include dangerous chemicals and potent reducing agents that can produce harmful byproducts, there is a risk to the environment and public health [10, 11]. Because it is so versatile and easy to utilize, the chemical reduction method is frequently used to produce nanoparticles. It is capable of working with a wide range of energy sources and reaction devices, and it can take as substrates common compounds that reduce metal ions or natural molecules. This approach is scalable and economical, allowing for precise control

over nanoparticle properties including size and shape without requiring high temperatures or pressures. It has proven essential for tailoring metal nanoparticles for particular uses in both organic and aqueous solvents, frequently leading to reduced toxicity when natural chemicals are utilized [12-14]. In this work, an excellent non-toxic chemical reduction method was developed for producing high-purity Cu nanoparticles without the use of a capping agent. The size and clarity of the crystallites may be precisely controlled thanks to L-ascorbic acid, an environmentally friendly reducing agent. The current study comprehensively evaluated copper nitrate and copper sulfate, evaluating their effects on nanoparticle production in order to estimate the relevance of precursor selection. Furthermore, in order to identify the ideal circumstances for reaching the smallest crystallite size with good crystallinity, the current study examined the effects of reduction times (4, 8, 12, 16, 20, and 24 hours). Since this work not only solves the need for harmful chemicals but also enhances the tunability of nanoparticle production, hence its technology is scalable and sustainable. The findings provide significant insights into the controlled synthesis of Cu nanoparticles and offer potential for applications needing high-purity and well-crystallized nanostructures.

2. Materials and methods

2.1.Cu Nanoparticle Synthesis: Experimental Overview

A 0.2 M solution of copper nitrate ($Cu(NO_3)_2.3H_2O$, Sigma-Aldrich) was made and heated to 80°C for 1 hour in a reflux system until it turned light blue. This was done in order to make Cu nanoparticles utilizing the aqueous solution reduction technique reported in Westhauser *et al.* [15] study. Following this, the copper nitrate solution turned transparent green when a 0.4 M solution of L-ascorbic acid (Sigma-Aldrich) was added dropwise. Two hours later, the solution took on a light brown hue. The color alterations are shown in Figure 1. To investigate the effect of reduction

time, the resultant Cu nanoparticles were evaluated 4, 8, 12, 16, 20, and 24 hours following the addition of the L-ascorbic acid solution. At each time point, the morphological and structural properties of the produced Cu nanoparticles were assessed.

To further explore the influence of the Cu precursor on the structural and morphological properties of the resulting Cu nanoparticles, copper sulfate ($CuSO_4$, Sigma-Aldrich) was substituted for copper nitrate. This substitution occurred in the aforementioned procedure after 24 hours of reduction.

2.2.Assessment of Phase Structure and Crystallite Size

The synthesized Cu nanoparticles' phase structure was assessed using X-ray diffraction (XRD) analysis, which was done with an XRD PMD Philips X-Pert device (Philips, The Netherlands). CuKα radiation with a wavelength of 1.54 Å, a step size of 0.05 Å, an applied voltage of 40 kV, and a current of 25 mA were employed to obtain the diffraction patterns in the 2θ range from 10° to 90°. The crystallite size of the synthesized Cu nanoparticles was determined using the Debye-Scherrer method and the X'pert highscore Plus software, accounting for precursor and reduction time changes.

2.3.Exploring Morphology Through FESEM and TEM Analysis

After gold coating in a sputter coater, the shape of the produced Cu nanoparticles was evaluated using Field Emission Scanning Electron Microscopy (FESEM, TESCAN MIRA3, Czech Republic). Images from a Zeiss EM900 transmission electron microscope (TEM, Germany) were also used to look into the shape and size of the Cu nanoparticles. The hydrogels were subjected to elemental analysis using Energy-Dispersive Spectroscopy (EDS, TESCAN, Czech Republic).

3. Results and discussion

Figure 2(a) displays the X-ray diffraction patterns of the Cu nanoparticles formed after the reduction of copper nitrate for 4, 8, 12, 16, 20, and 24 hours. Aligning with the diffraction angles with 2θ values of 19.6º, 22.7º, 32.2º, 34.4º, 38º, 39.8º, and 46.2º, respectively, are the (111), (200), (220), (311), (222), and (400) planes. These patterns match the JCPDS data No. 96-410-5041 for an FCC lattice, suggesting that contaminants like copper oxide are not present. Figure 2(c-i) displays the results of EDS studies of all Cu nanoparticles made using different precursors and reduction durations in order to verify the absence of contaminants. According to the analyses, Cu is the only element found. Furthermore, the unlabeled remaining peaks are indicative of the gold coating that was used on the Cu nanoparticles to improve their conductivity in the FESEM. Longer reduction times result in taller diffraction peaks, as seen in Figure 2(a), which suggests that the Cu nanoparticles have greater crystallinity. This is because extended reduction times allow more atoms to occupy the same lattice planes, resulting in a more repeated pattern. Consequently, the electron density on the crystallographic planes also increases with the reduction time, then the crystallinity is enhanced [16, 17]. The XRD patterns of Cu nanoparticles produced from precursors of copper nitrate and copper sulfate after a 24-hour reduction are shown in Figure 2(b). The Cu nanoparticles' FCC lattice structure is matched by the peaks that correspond to JCPDS data No. 96-410-5041. It is noteworthy that Cu nanoparticles generated with copper nitrate had greater peak intensities than those synthesized with copper sulfate. This suggests that Cu nanoparticles synthesized from copper nitrate have a higher electron density on the crystallographic planes and, consequently, a higher crystallinity than those derived from copper sulfate. The reason for this is because copper nitrate is more reactive than copper sulfate [18], which leads to a larger atomic density on the planes of the Cu nanoparticles that are made from copper nitrate.

The Debye-Scherrer method is used to assess the crystallite size of Cu nanoparticles made with various precursors and reduction periods. The results are shown in Table 1 and Figure 3. Figure 3(a) illustrates that the crystallite size of Cu nanoparticles formed with copper nitrate increases somewhat from 34 ± 14 nm to 32 ± 11 nm when the reduction duration is increased from 4 hours to 20 hours. This has no variance that is statistically significant. However, after a 24-hour reduction period, the crystallite size increases considerably to 45 ± 11 nm. This shows that while the quantity of Cu nanoparticles does not increase after 24 hours, the crystallites themselves do. This is probably because the extended reduction time promotes the formation of crystallites.

The crystallite sizes of Cu nanoparticles made from copper nitrate and copper sulfate are shown in Figure 3(b). When compared to nanoparticles made from copper sulfate, those made from copper nitrate seem to have larger crystallite sizes at first appearance. This is probably because nitrate is more reactive than sulfate [18]. Nevertheless, a statistical comparison of the mean ± SD values for the crystallite sizes in Figure 3(b) reveals that these differences are not significant.

Figure 3(c) presents the TEM image of Cu nanoparticles synthesized using copper nitrate after a 24-hour reduction time. The Debye-Scherrer method yielded results that were in agreement with the observed particle sizes, which are less than 50 nm. Furthermore, the FESEM images of Cu nanoparticles synthesized with copper nitrate and copper sulfate at different reduction periods are displayed in Figure 4 (a-g). The synthesized nanoparticles' morphologies show aggregation, which is explained by their high surface energy and van der Waals forces [19, 20].

The influence of reduction duration and precursor type on Cu nanoparticle size is described by nucleation and growth dynamics. Initially, a high concentration of Cu^{2+} ions stimulates fast nucleation, resulting in tiny nanoparticles [21-23]. Atomic diffusion promotes nanoparticle development and increases crystallinity as the reduction time increases [24, 25]. Ostwald ripening

induces extra particle growth after a certain threshold duration. Copper nitrate has better solubility and reactivity than copper sulfate, which allows for a more uniform and efficient reduction process. This produces larger, more defined crystallites, whereas copper sulfate's slower reduction kinetics limit atomic movement and crystal growth [26-28]. XRD and TEM investigations confirm these patterns, with stable crystallite sizes up to 20 hours and a considerable rise at 24 hours, especially for Cu nanoparticles produced by copper nitrate.

In summary, the increased crystallinity of Cu nanoparticles with increasing reduction time is responsible for the enhanced atomic diffusion and rearrangement of Cu atoms during the production process. More Cu^{2+} ions are reduced to Cu^0 as the reduction process progresses, enabling newly formed atoms to form a more stable crystalline structure. Longer reduction durations give these atoms enough energy and time to migrate and align into well-ordered lattice structures, lowering structural flaws and boosting crystallite size. Furthermore, the prolonged reduction time increases electron density on the crystallographic planes, resulting in sharper and more intense diffraction peaks in XRD patterns, indicating increased crystallinity. This phenomenon is consistent with previously reported studies on metal nanoparticle synthesis, where controlled reaction times significantly impact crystallite growth and structural refinement [29-32].

Additionally, Cu nanoparticles generated from copper nitrate are more crystallin than those made from copper sulfate, which can be attributed to variations in reactivity and solubility between the two precursors. Copper nitrate's increased solubility in aqueous solutions and faster dissociation into Cu^{2+} ions result in a more consistent and efficient reduction process. With the help of a more regulated nucleation and growth process, Cu atoms can arrange themselves into crystalline structures that are more organized and have fewer defects. In contrast, the less soluble and reactive nature of copper sulfate can lead to slower atomic organization and less efficient reduction, both

of which can compromise crystallinity [33-36]. Cu nanoparticles made from copper nitrate showed sharper and more intense diffraction peaks than those made from copper sulfate, suggesting a higher degree of crystallinity, which is supported by the XRD results. The higher crystallinity of copper nitrate was further confirmed by the Debye-Scherrer analysis, which showed that after 24 hours of reduction, the crystallite size of Cu nanoparticles made from copper nitrate was 45 ± 11 nm, whereas those made from copper sulfate were smaller at 35 ± 14 nm.

4. Conclusion

Copper nitrate and copper sulfate were utilized as precursors in this study's aqueous solution reduction method to synthesize Cu nanoparticles over reduction periods of 4, 8, 12, 16, 20, and 24 hours. The results revealed that increasing the reduction time enhanced the crystallinity of the nanoparticles, with those synthesized from copper nitrate exhibiting superior crystallinity compared to those derived from copper sulfate. For the first 20 hours, the crystallite size was comparatively constant, but after 24 hours, it had grown considerably to 45 ± 11 nm. Furthermore, the crystallite diameters of Cu nanoparticles made using copper nitrate were consistently greater than those made with copper sulfate. This study develops an ecologically safe, scalable, and optimal chemical reduction process for the production of Cu nanoparticles that employs L-ascorbic acid as a non-toxic reducing agent. The findings indicate that a 20-hour reduction period provides the best balance of crystallite size and purity, and that copper nitrate is a more effective precursor for producing high crystallinity. This method produces high-quality Cu nanoparticles for a wide range of commercial and biological applications without the need of stabilizing chemicals, making it a feasible and sustainable option. Future study will focus on improving the synthesis process by investigating other parameters such as temperature and concentration in order to precisely control the size and form of the nanoparticles. The long-term stability and biocompatibility of these

extraordinary Cu nanoparticles will be explored, as well as their prospective uses in electronics, biomedical devices, and catalysis. Furthermore, studies will be done to assess the method's scalability for industrial production and to compare its environmental impact to other nanoparticle synthesis methods.

Conflict of interest

The authors have no conflicts of interest to declare.

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Figure captions

Figure 1: The color changes during the reduction of Cu nanoparticles.

Figure 2: (a) X-ray diffraction patterns of Cu nanoparticles synthesized at various reduction times. (b) X-ray diffraction patterns of Cu nanoparticles synthesized with different precursors. EDS analyses of Cu nanoparticles synthesized using copper nitrate for (c) 4 hours, (d) 8 hours, (e) 12 hours, (f) 16 hours, (g) 20 hours, and (h) 24 hours. (i) EDS analysis of Cu nanoparticles synthesized using copper sulfate after 24 hours.

Figure 3: (a) Crystallite size of Cu nanoparticles synthesized at various reduction times and (b) with different precursors, calculated using the Debye-Scherrer method with X'Pert HighScore Plus software. (c) TEM image of Cu nanoparticles synthesized using copper nitrate after a 24-hour reduction time.

Figure 4: FESEM images of Cu nanoparticles synthesized with copper nitrate after reduction times of (a) 4 hours, (b) 8 hours, (c) 12 hours, (d) 16 hours, (e) 20 hours, and (f) 24 hours, and with copper sulfate after a reduction time of (g) 24 hours.

Table captions

Table 1: The crystallite size of the Cu nanoparticles synthesized in various reduction time and with different precursors calculated with Debye-Scherrer method using X'pert highscore Plus software

Figure 1

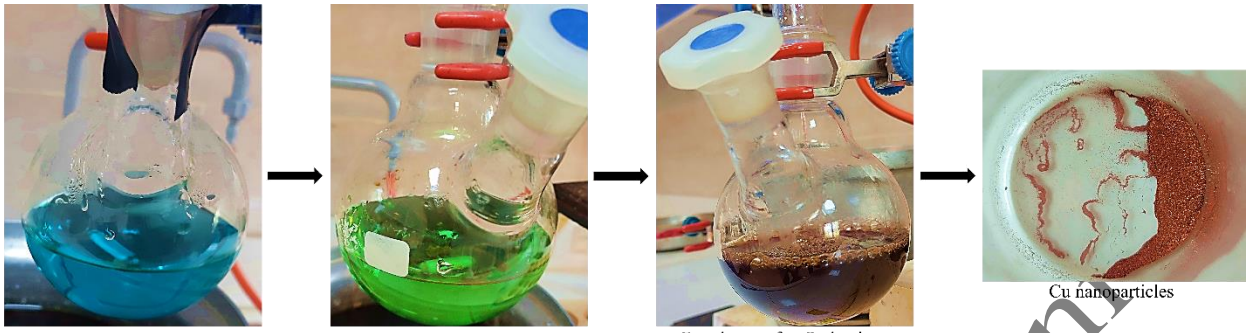
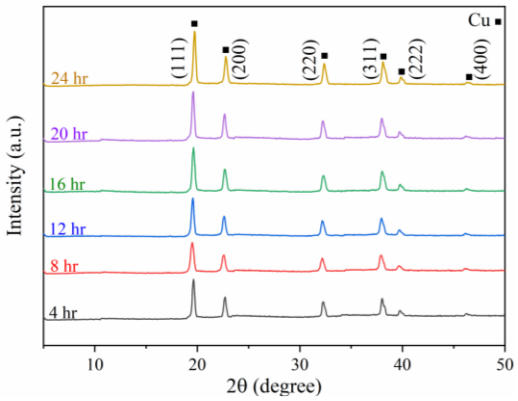


Figure 2



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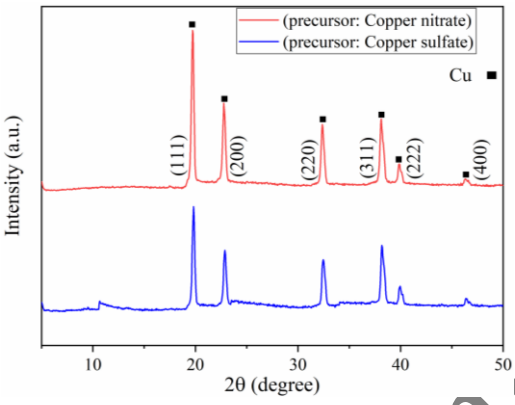

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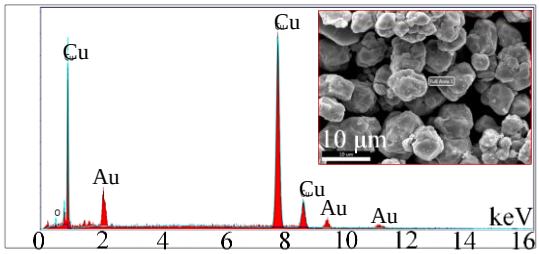

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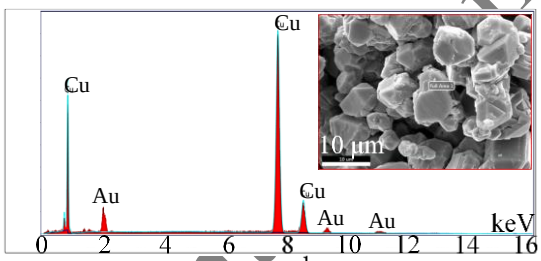

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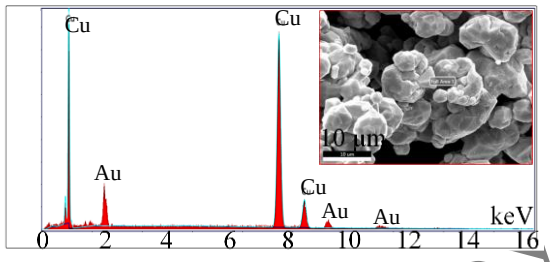

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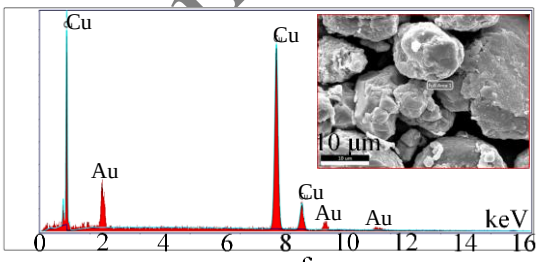

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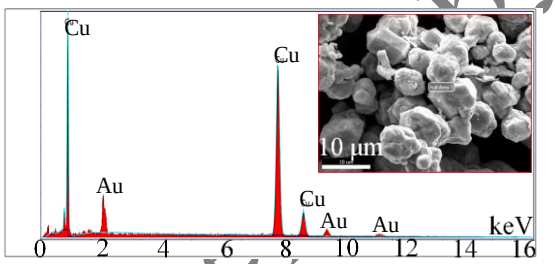

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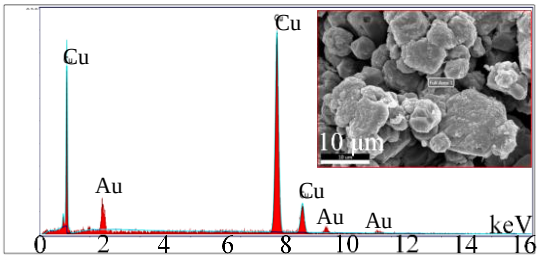

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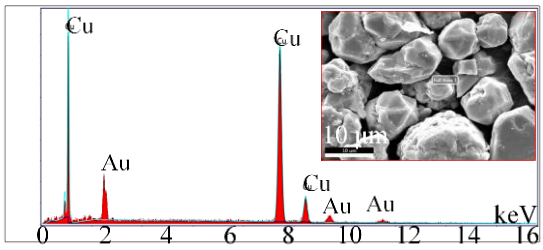

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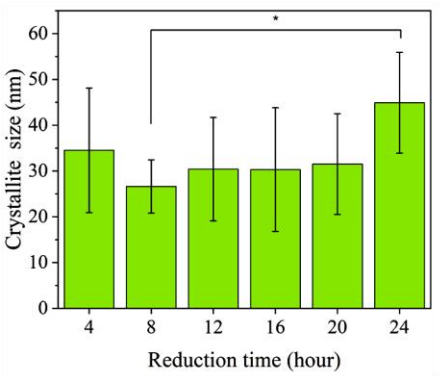
Figure 3



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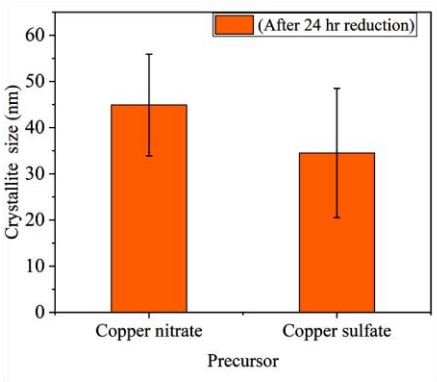
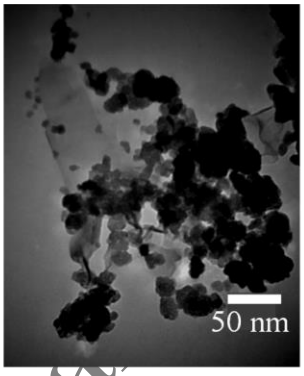


b



c

Figure 4

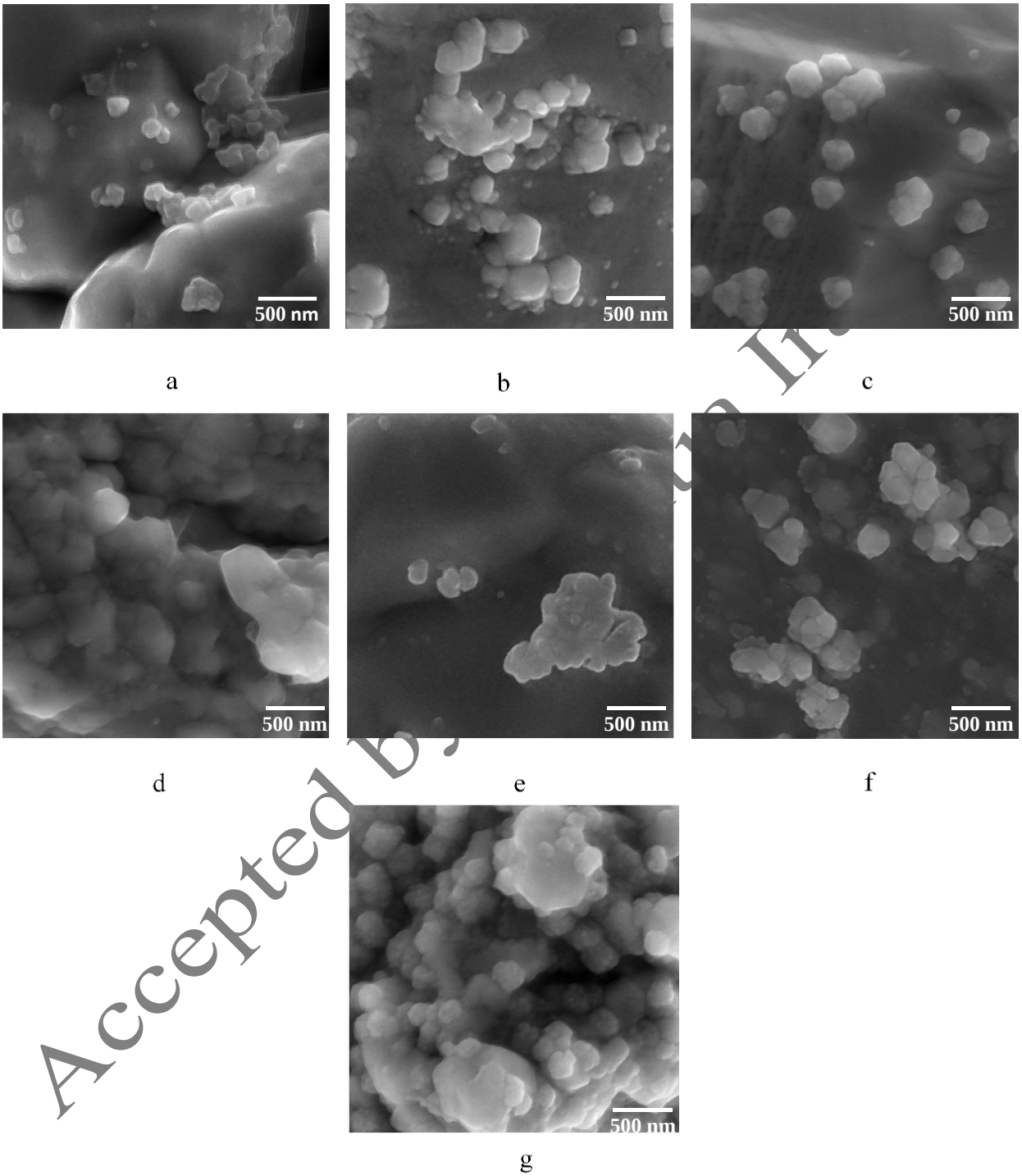
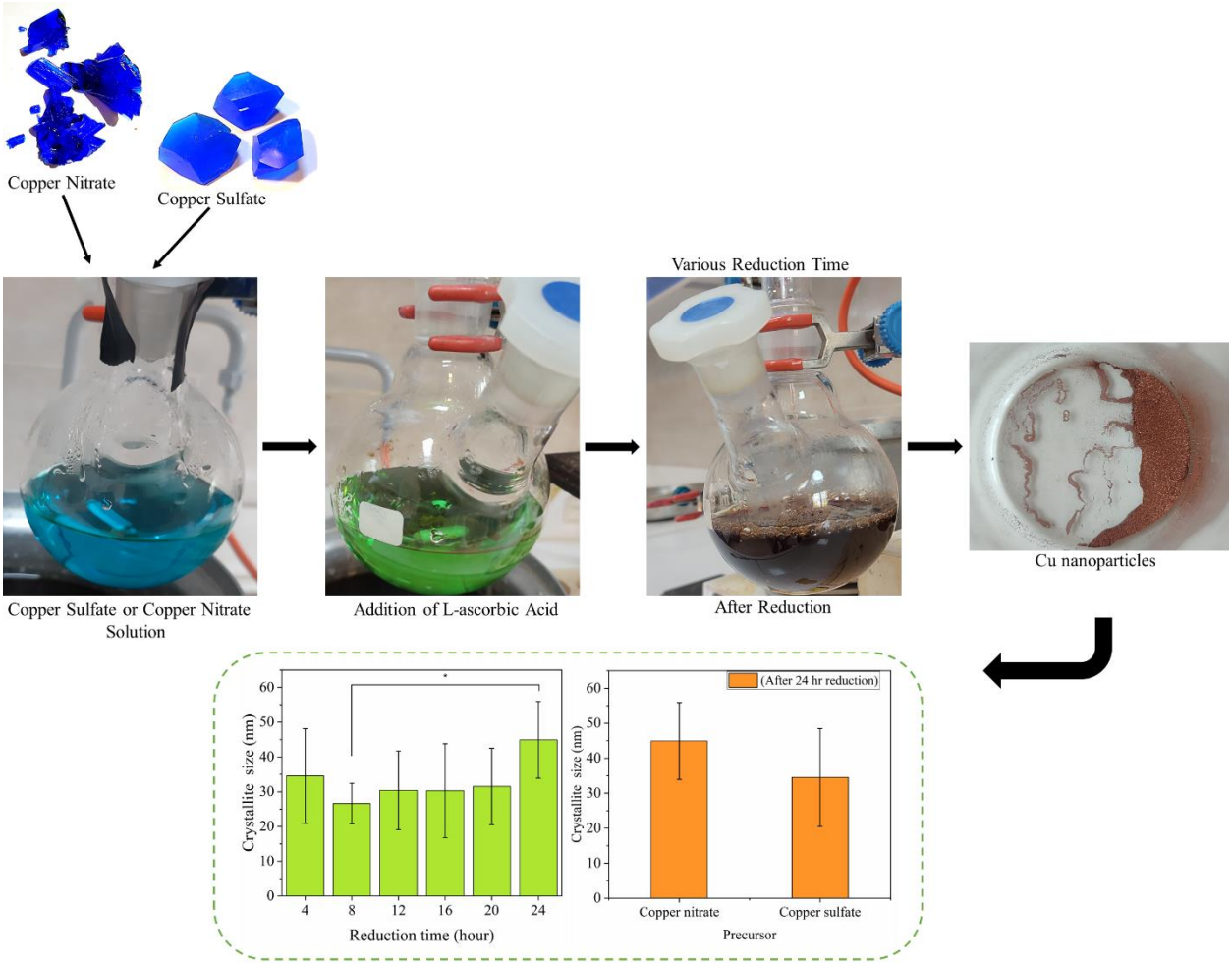


Table 1

Precursor	Reduction time (hours)	Crystallite size (nm)
Copper nitrate	4	34 ± 14
	8	27 ± 6
	12	30 ± 11
	16	30 ± 14
	20	32 ± 11
	24	45 ± 11
Copper sulfate	24	35 ± 14

1. Ms. Fatemezahra Zeraati attained a BSc in Materials Engineering from the College of Engineering at Golpayegan, Isfahan University of Technology, Iran. She studied biomaterials during her BSc, more specifically their applications in polymeric scaffolds for tissue engineering and wound healing. She was able to actively participate in research alongside multidisciplinary teams. She completed research in biomedical materials at the undergraduate level, with a keen interest in chemistry and strong laboratory skills.
2. Dr. Narges Johari is an Assistant Professor at the Materials Engineering Group, at Golpaygan College of Engineering, Isfahan University of Technology. She has a Ph.D. in Materials Science and Engineering, specializing in biomaterials and tissue engineering. Her research is concerned with the development of polymeric scaffolds and nanocomposites for regenerative medicine, as well as in novel methods of 3D bioprinting. Dr. Johari has further distinguished herself by publishing peer-reviewed articles on these topics. She has also been actively working with researchers worldwide in several interdisciplinary biomedical engineering projects, all with a view toward contributing in the improvement of healthcare technologies all around the world.
3. Ms. Fatemeh Rafati has obtained her Master's degree from the Materials Engineering Group at Golpayegan College of Engineering, Isfahan University of Technology, Iran. Her research has widely focused on biomaterials for tissue engineering, mainly the development of polymeric hydrogels and electrospun scaffolds for wound healing purposes. She has published scientific works in this domain and has actively collaborated with various teams to advance regenerative medicine technologies.

Graphical Abstract


Copper Nitrate
Copper Sulfate
Various Reduction Time
Copper Sulfate or Copper Nitrate Solution
Addition of L-ascorbic Acid
After Reduction
Cu nanoparticles
Crystallite size (nm)
Reduction time (hour)
4
8
12
16
20
24
(After 24 hr reduction)
Copper nitrate
Copper sulfate
Precursor