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IMPACT OF PLANT SPECIES IN GREEN SYNTHESIS OF COPPER NANOPARTICLES ON THEIR MORPHOLOGY AND SIZE

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Abstract:
This article describes the preparation of copper nanoparticles (Cu-NPs). Natural sources as a green method were used in procedure. Then the properties of the products were compared to each other by XRD spectroscopy. The Copper nanoparticles were prepared using environmentally friendly manner using $CuSO_4$ salt as a source of copper metal with the help two different aqueous extracts of *grape and fig* leaves as a reducing factor and NaOH solution as a precipitating agent. The copper nanoparticles (Cu-NPs) were obtained by slow addition of plants leaves extracts and sodium hydroxide solution as precipitating agent at room temperature. Copper nanoparticles were characterized by XRD, FE-SEM and TEM techniques. X-ray diffraction (XRD) examination showed that the size of copper nanoparticles was 36.67 and 29.57 nm for *grape and fig* leaves, respectively. FE-SEM images gave different sizes and shapes of copper nanoparticles at 327.39 and 123.74 nm to sample prepared by *grape and fig* leaves, respectively. The average size range from approximately 53 to 74 nm for Cu-NPs synthesized using *grape* leaf extract, and from 33 to 61 for those synthesized using *fig* leaf extract this confirmed by TEM techniques examination.

Keywords: copper nanoparticles; leaves plant extract; XRD, FE-SEM, TEM.

1. INTRODUCTION

Nanomaterials and nanotechnology have gained great importance in modern research. Michael Faraday's research on gold colloids in the nanometer range in the middle of the 19th century is one of the first accounts of nanoparticles in the scientific literature.[1] The word "nanoparticles" refers to particles that are at least one of the three potential sizes and fall between 1 and 100 nm. The physical, chemical, and biological characteristics of nanoparticles vary significantly in this size range compared to those of individual atoms or molecules and the comparable bulk materials.19. Although materials of many different chemical types may be used to create nanoparticles, metals, metal oxides, silicates, non-oxide ceramics, polymers, organics, carbon, and biomolecules are the most often used materials.[1] Nanoparticles come in a variety of shapes, including tubes, spheres, cylinders, and platelets. etc.[2,3] Nano copper, or copper nanoparticles, are utilized in a variety of applications, including food packaging, textiles, and even medical contexts, their antimicrobial properties make them useful for extending shelf life in food packaging, creating odor-resistant textiles, and potentially reducing infections in medical devices.[4] Copper has also been used in drinking water and swimming pool water filters because of its antibacterial qualities.[5] Several techniques, such as spark discharge, electrochemical reduction, solution irradiation, and chemical synthesis, have been used to design metallic copper into ultrafine particles in order to create nano copper.[6] Nanocopper particles are composed of around 30–15,000 copper atoms and are typically smaller than 100 nm.[7] As, copper is a soft blue element, an important use of copper nanoparticles is give a products a copper finish.[8] Still, the remarkably strong antimicrobial activity is the major direction for development of nano copper products.[9] Copper nanoparticles, a promising area in nanotechnology, are increasingly synthesized using eco-friendly, "green" methods, particularly through the bio-reduction of copper ions using plant extracts. This approach utilizes various plant parts like leaves, flowers, and roots to create copper nanoparticles, offering a more sustainable alternative to traditional chemical and physical methods.[10,11]

In this study described the preparation of copper nanoparticles (Cu-NPs) by two natural sources as a green method were used in procedure, then the properties of the products were compared to each other by XRD, FE-SEM and TEM techniques.

Experimental part

1.1. Preparation of plant leaves extract: Plant Leaf Extract Preparation: *grape* and *fig* leaves obtained from trees in Iraq were thoroughly washed with tap water, followed by deionized water, and air- dried. The leaves were cut and ground well, and stored in dry conditions. After adding 10 g of powdered grape or fig leaves to 100 mL of deionized water, the mixture was heated for three hours at 80 oC until the solution became green. It was then allowed to cool to room temperature before being filtered through Whatman paper No. 1 to produce a clear filtrate. To eliminate any fine plant particles and biomaterials, centrifuge the filtrate for 20 minutes at 1200 rpm. The extract should then be stored at -4 °C to be employed in the production of copper nanoparticles, as seen in figure 1.

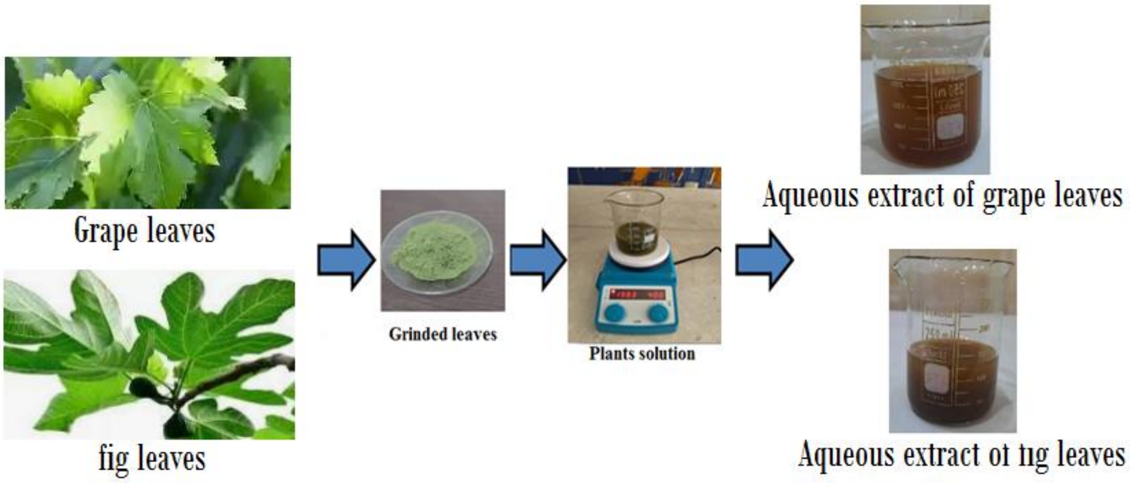


Figure 1. The steps of the preparation plant leaves extract

1.2. Green synthesis of copper nanoparticles: CuSO4 (3.0 g, 0.018 mole) was dissolved in 100 mL of deionized water to create the salt solution. To get the pH at about 12, the mixture was stirred continuously for three hours while the extract solution was added gradually at 40 °C. The temperature of the mixture was then raised to 80 °C while being stirred, and sodium hydroxide solution (0.1 M) was added dropwise to the mixture (leaves and brine extract). After filtering the mixture, the precipitate was collected and cleaned with ethanol and deionized water to bring the pH down to around 7. The copper nanoparticles were then made (figure 2).

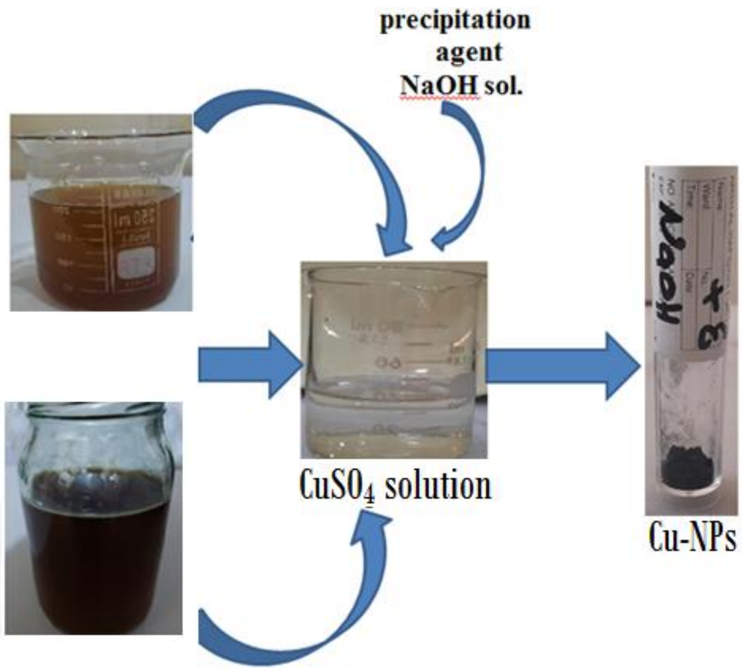


Figure 2. The biosynthesis of copper nanoparticles (Cu- NPS) using two plants

2. RESULT AND DISCUSSION

The first indication of biosynthesis synthesis of copper nanoparticles (Cu-NPs) is often a noticeable color change in a copper sulfate solution, typically observed within 10 to 15 minutes of adding a reducing agent. This color change, shifting from the initial blue or brown to a darker hue, signifies the formation of Cu-NPs. Specifically, the dark color is attributed to the surface plasmon resonance of the newly formed Cu-NPs, figure 3.[12]

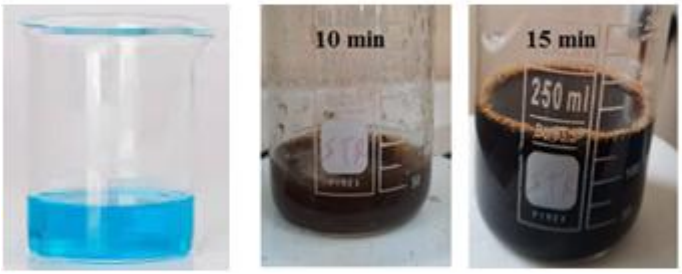


Figure 3. Visual detection after 10 min and 15 min

2.1. Characterization of copper nanoparticles synthesized:

Synthesis rout with NaOH solution as precipitation agent was taken to study the synthesis copper nanoparticles, and were analyzed using XRD, FE-SEM, and TEM techniques.

2.1.1. XRD analysis

The size of Cu-NPs nanoparticles have calculated from Scherrer formula as:

$$D=K\lambda/\beta \cos\theta$$

where θ is the Bragg's angle, β is the full-width at half-maximum (FWHM) in radians, λ is the wavelength of the x-ray source, and D is the average crystalline size. Narrow, symmetrical delta function peaks arranged in accordance with a certain unit cell plane make up an excellent diffraction pattern. Peak broadening is the term used to describe the deviations from the ideal pattern. This broadening of diffraction peaks, which happens when atoms in crystal unit cells are moved from their ideal position by small crystallites (size broadening) below one micrometer and an abundance of lattice defects (strain broadening) like dislocations, is what the XRD method uses to quantify the dislocation density.[12]

The copper nanoparticles Cu-NPs was characterized using powder XRD. In which to confirm the particles of copper and to know the structural information for product. Figures 4 and 5 shows the XRD pattern of copper nanoparticles that synthesized using *grape* and *fig* leaves extract. The pattern of Cu-NPs for *grape* in Figure 4 clearly shows the main peaks at (2θ) 28.26°, 33.47°, 40.11°, 53.7°,66.5° and 78.23° corresponding to the (110), (111), (020), (202), (-222), (220), (-311) and (311) planes, respectively, the typical pattern of green-synthesized Cu-NPs was found to possess an cubic structure with average crystalline size 36.67 nm.[13]

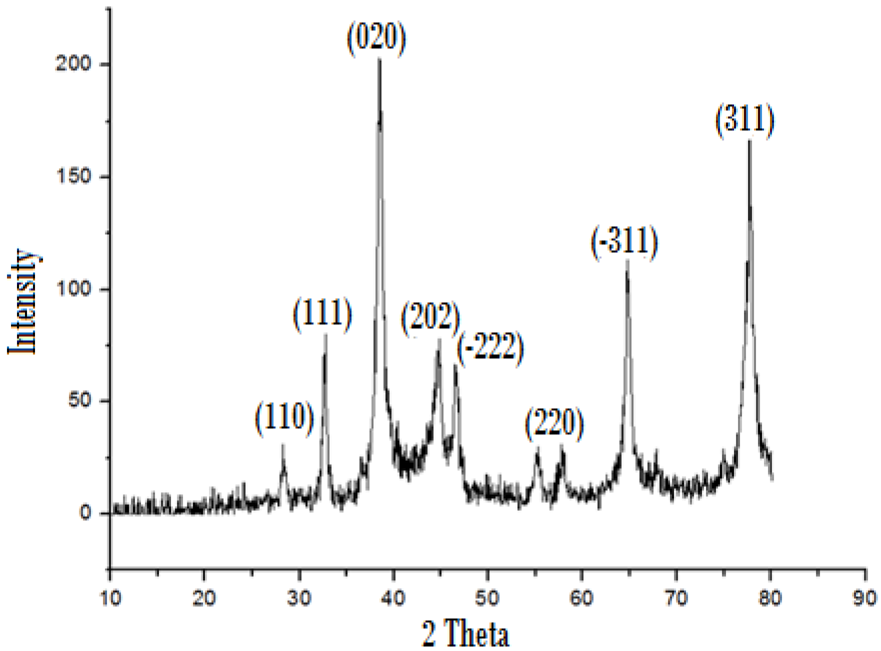


Figure 4 XRD pattern of synthesized Cu-NPs using *grape* leaves extract

While, the data of synthesized Cu-NPs using *fig* leaves extract is shown in figure 5, the main peaks at (2θ) 28.06°, 33.17°, 38.28°, 45.52°, 47.39°, 67.10°, and 78.61° corresponding to the (110), (111), (020), (202), (-222), (220), (-311) and (311)planes, respectively. Additionally, the Cu-NPs have found to possess a cubic structure with crystalline average size 29.57. Addition, many unassigned peaks appeared at (28.06 °, 33.17°, 38.28°) and (45.52°, 47.39°, 67.10°) for Cu-NPs , these peaks were weaker than those of copper nanoparticles, figure 5.

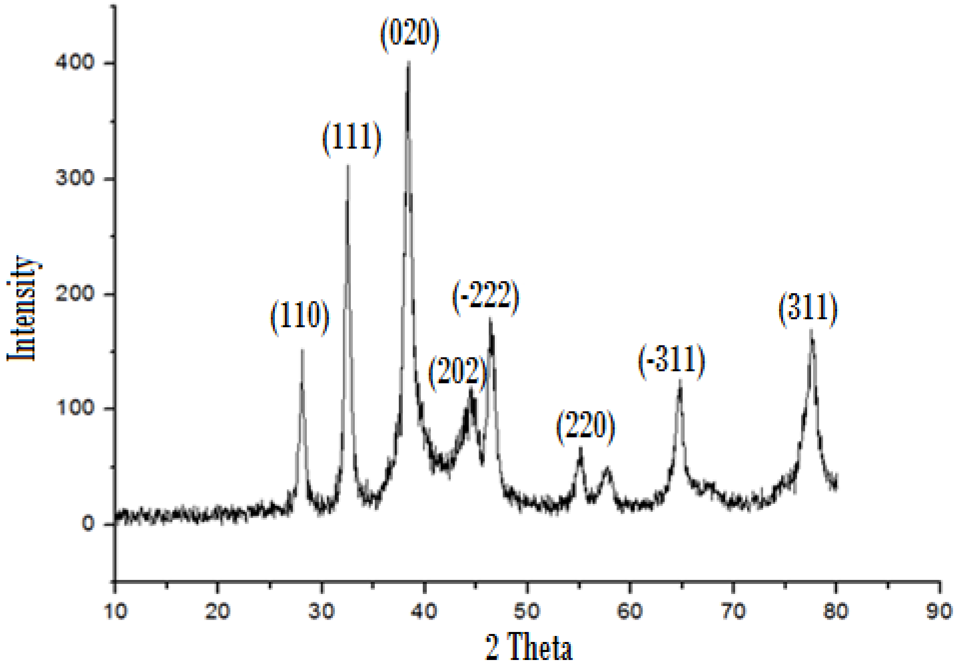


Figure 5 XRD pattern of synthesized Cu-NPs using *fig* leaves extract

The illustrated figures showed that there was a clear effect of the type of plant extract that used as a reducing agent, which had a role in the affecting the average size of the copper nanoparticles. Accordingly, the comparing of X-ray diffraction pattern of prepared copper nanoparticles using different plants extracts showed clearly effect on the average diameter size of copper nanoparticles.

2.1.2. FE-SEM images

FE-SEM technique has used to visualize the size and shape of the nanoparticles. FE-SEM micrographs of copper nanoparticles have given in figures 6, and 7 with 50 kx magnifications. However, the NaOH solution and the secondary metabolites present in the plant also played a vital role in the morphological changes. The particles have aggregated with one another; this similar result has discussed by reference. [14] Figure 6 illustrate the FE-SEM images of synthesized Cu-NPs using *grape* leaves extract plant confirmed the presence of cubic shapes and agglomeration nanoparticles with average diameters about 327.39 nm, while in the FE-SEM image of synthesized Cu-NPs using *fig* leaves extract was confirmed the presence of regular spherical shapes nanoparticles with average diameters about 123.74 nn, figures 6 and 7.

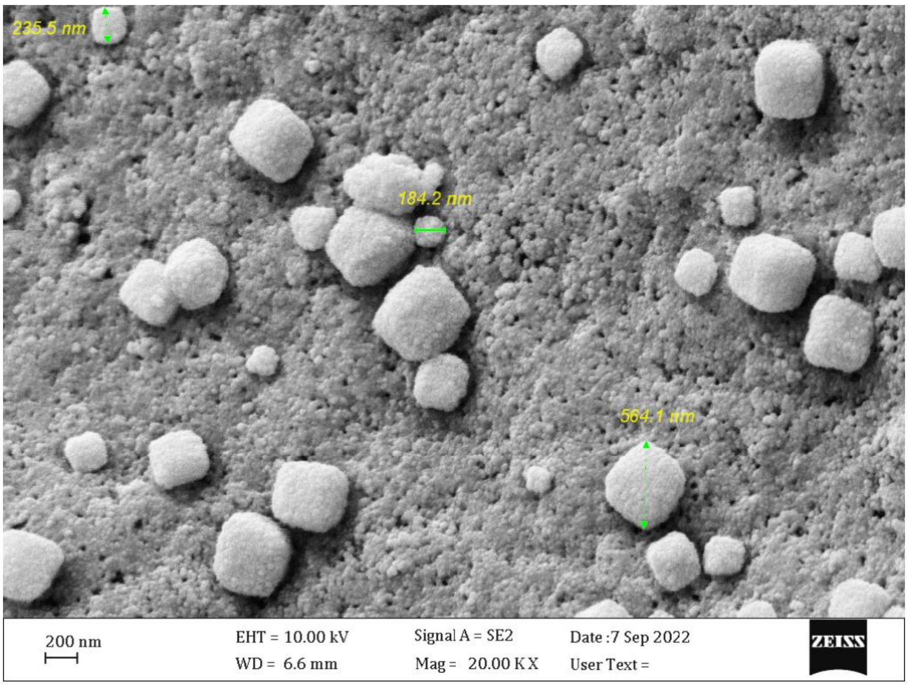


Figure 6 FE-SEM images of copper nanoparticles synthesized using *grape* leaves extract

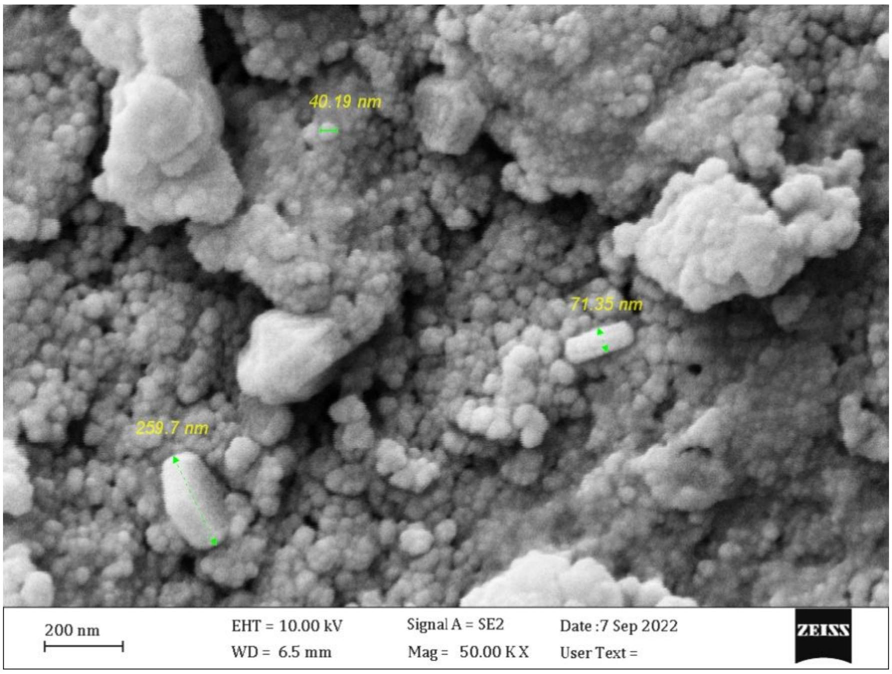


Figure 7 FE-SEM images of copper nanoparticles synthesized using *fig* leaves extract

2.1.3. TEM images

Figures 8 and 9 show transmission electron microscope (TEM) images of copper nanoparticles (Cu-NPs) at a 200 nm. The nanoparticles appear highly agglomerated with heterogeneous shapes. The average size range from approximately 53 to 74 nm for Cu-NPs synthesized using *grape* leaf extract, and from 33 to 61 for those synthesized using *fig* leaf extract.

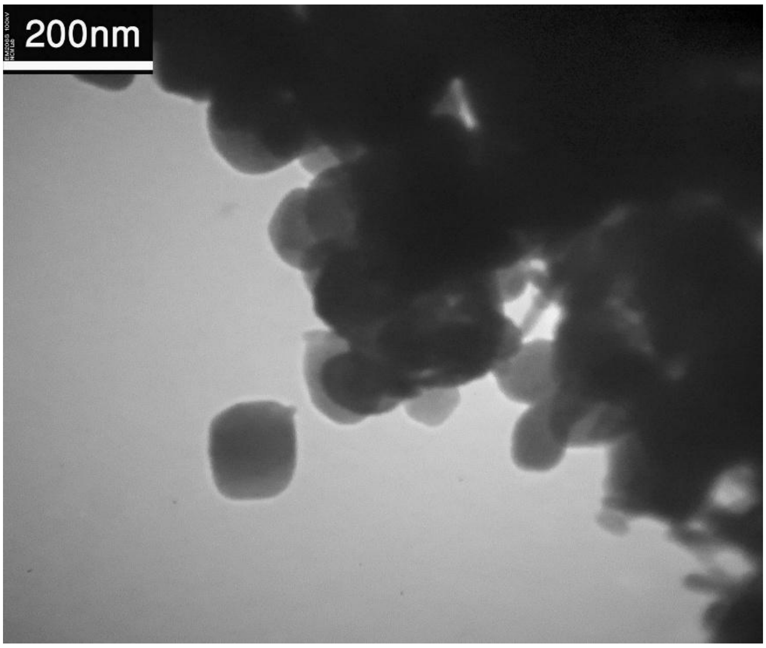
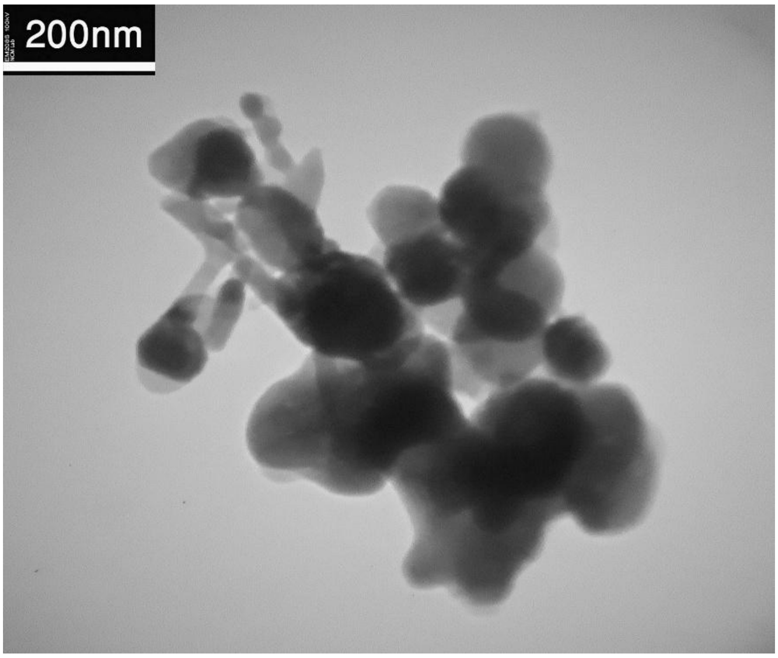


Figure 8. TEM image shows Cu nanoparticles made by extract via Grape leaves extract.



.Figure 9. TEM image shows Cu nanoparticles made by extract from fig leaves

CONCLUSION

In this work, stable copper nanoparticles (Cu-NPs) were synthesized via a green approach using aqueous extracts of *grape* and *fig* leaves as both reducing and stabilizing agents with NaOH solution as a precipitation factor Pure Cu-NPs were successfully produced through this eco-friendly method, which is less toxic and more cost-effective than conventional techniques. The production of cubic-phase Cu-NPs was verified by X-ray diffraction (XRD) analysis; the average crystallite diameters of the nanoparticles made using fig and grape leaf extracts were 29.57 nm and 36.67 nm, respectively. The choice of plant extract as a reducing agent had a pronounced influence on the morphology and particle size of the Cu-NPs, as evidenced by FE-SEM and TEM analyses.

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