Synthesis and characterization of copper oxide nanoparticles using *Moringa Oleifera* leaves Extract

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Abstract

Moringa oleifera leaves extract Yemeni was used for green synthesis of copper oxide nanoparticles (CuO). The obtained copper oxide nanoparticles were characterized using ultraviolet–visible spectroscopy (UV–VIS), Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), energy-dispersive X-ray analysis (EDX), and X-ray diffraction (XRD). The aqueous suspension of (CuO NPs) showed a UV-Vis. absorption maxima peaks at 236 and 240 nanometers respectivel primarily in its formation. The FTIR analysis to be confirmed functional groups of biosynthesized CuO NPs the absorption band at 608cm^{-1}. The XRD analysis revealed that the biosynthesized NPs appear to be more than crystalline of the copper oxide nanoparticles. The characteristic peaks obtained by EDX analysis icon firmed the presence of the element copper (89.8), and the element oxygen (10.2). SEM images show that the particles are round, granular, and nanosized and the nanoparticle has more aggregation ,and conglomeration. Extraction of *moringa* leaves serves as a promising agent for the balance of particle size. The result of medical value shows significant antibacterial activity.

Keywords: Green synthesis, Copper oxide Nanoparticles, *Moringa oleifera*, Nanotechnology.

1. Introduction

Recently, nanoparticles synthesized by the green method have received a great deal of attention because of their unique physical and chemical properties Khan, & Lee, (22). Nanoscience has modified and developed the physical and chemical properties of metal by transforming them at the nanoscale. Extensive efforts are in progress to characterize and utilize metal oxide nanomaterials in numerous technological of fields medicine, including food, energy, and environment Ahamed, *et al.,* (2). The nanoparticles can be prepared by several methods like chemical, physical, and biological, but chemical and physical methods are associated with high energy demand, sometimes generating poisoned and parlous chemicals, which may lead to consanguineous menaces Kharissova, *et al.,* Khatamifar, & Fatemi, There are a lot of applications of copper oxide in medicinal, biological, environmental, and engineering fields. Copper oxide NPs have several medicinal and biological applications, such as in anticancer Gnanavel, *et al.,* (16) antimicrobial activity Usman, *et al.,* (52), antioxidant activity Muthuvel, *et al.,* 2020 (30), antifungal efficiency (Khatami, *et al.,*; Shammout, & Awwad, (24,44), and antibacterial materials Gowri*, et al.,* 2019; Rabiee, *et al.,* 2020; Ramzan, *et al.,* 2021;Ebrahimi, *et al.,* 2017 & Begum, *et al.,* 2019 (17,20,37,13,9), whereas, in environmental and engineering applications such as in environmental remediation Kumar, *et al.,*; Rather, & Sundarapandian, 2022 (28,38), including sensors Haghparas, *et al.,* 2020 & Ambardekar, *et al.,* (18,6), catalysts Muthuvel*, et al.,* & Begildayeva, *et al.,* (30,8), optical and electronics devices Sarwar, *et al.,* 2021; Sahai, & Varshney, (42,40), and solar cells Siddiqui, *et al.,* (46), etc. An eco-friendly green-intermediated preparation of nanoparticles, there has been a huge demand for a green synthesis strategy due to its eco-friendly, sustainability, reliability and the presence of photochemical in plants especially in leaves like flavonoids, phenols, terpenoids, amides that reduce the toxicity exist in chemicals and ascorbic acids Doble, *et al.,* (12). Green synthesis of nanostructures using *Verbascum Thapsus* leaf extract Weldegebrieal, (54), *Aloe vera* leaf extract Kumar, *et al.,* (27), *Cynodon dactylon* and *Cyprus rotundus grass* extracts Suresh, *et al.,* 2020 (48), *Brassica oleracea* var. italic extract Renuga, *et al.,* 2020 (39), *Ocimum Basilicum* leaf extract Malik, *et al.,* (29) flower extracts of *Cordia Sebestena* Prakash, *et al.,* (33) and leaf extract of mangosteen Veerasamy, *et al.,* (53). Copper oxide nanostructures revealed antimicrobial activities even at minor appropriate and concentrations Raja, *et al.,* (36). Chemical techniques of synthesis of NPs are also excessive in the atmosphere Akintunde, *et al.,* (3). So, for many causes, mainly for eco-friendly benefits, the green synthesis techniques or green preparation of nanostructures Thakur, *et al.,* (49). *Moringa oleifera* tree also known as the drumstick tree is native to Asia particularly India ,and also is grown in the Philippine, Sudan, Egypt, and South Africa. It is a member of the *Moringa* species and the Moringaceae family, and is renowned for its safe to eat soft beans, flowers and leaves. The leaves, notably, are well-known for their prophylactic and therapeutic powers and are often devoured in meals. Extracts obtained from the leaves have powerful antioxidant capabilities and high antibacterial action against Gram-positive and Gram-negative bacteria. Additionally, phytochemicals in *Moringa oleifera* leaves show incredible properties such as anticancer, anti-inflammatory, anti-diabetics and are suitable for applications in different industries such as foodstuff, cosmetics, and drug delivery Bhalla, *et al.,* (10). The leaves of the *Moringa oleifera* serve as a medical plant, due to the presence of essential phytochemical and the phytochemical profile of leaves *Moringa oleifera* exhibited the presence of essential minerals, anthraquinones, terpenoids, saponins, flavonoids, alkaloids, tannins, sterols, and vitamins. These phytoconstituents lead to properties against different bodies diseases like rheumatism, hyperthyroidism, anemia, arthritis, blindness, herpes simplex virus, Crohn's disease, epilepsy, gout, Espenti, *et al.,* 2020; Dejen, *et al.,* Bandeira, *et al.,* 2020; Srivastava, & Choubey, (14,11,7,47). antiulcer, antidiabetic, anti-inflammatory Timah, et al., (50), antioxidant, antifungal, and antimicrobial properties Sannasi, & Subbian, (41). anticancer and sexually diseases transmitted Raj, *et al.,* (35). In recent times there are huge challenges in evolving and innovating strong antibacterial agents. As per the last 5 years, the Food and Drug Administration (FDA) have banned the most common antimicrobial agent like triclosan, triclocarban, and 18 others from the personal care industry Halden, (19). In recent research, GCMS analysis revealed an extraction of a range of phytochemicals of the *Moringa Oleifera* leave's aqueous and alcoholic extracts Bhalla, *et al.,* (10). *Moringa oleifera* leaf extract acts as a biodegradable, natural and non-toxic reducing agent for the manufacture of copper oxide at the nanometer scale. The evaluation and characterizations of the green synthesis of CuO-Nps are performed by using advanced technical tools such as UV Vis, FTIR, XRD, SEM-EDX, and antimicrobial studies. The synthesis of nanoparticles from the extraction of leaves using plants is simpler to use. Environmentally friendly, reliability It is non-toxic and no poison is released into the environment. Due to the primordial facts mentioned about the medical ideals of this plant, therefore, the green synthesis of copper oxide selected for the present work has a wide application. This research will further help increase the application of green synthesized versatile nanomaterials in medicine and healthcare.

2. Material and Methods

2.1. Materials and Reagent

For the preparation of nanoparticles extracted from *Moringa* leaves, copper acetate monohydrate 99.5% [$(CH_3COO)_2$-H_2O], sodium hydroxide [NaOH] and deionized water were bought from shops of chemicals. All tools are purified then dried up by putting them in the oven before the next procedure. The leaves of *Moringa oleifera* were collected from Faculty of Science Hadhramout, University, Yemen.

2.2. Synthesis of Aqueous Extract from *Moringa oleifera* Leaves

About twenty grams of powdered *Moringa Oleifera* leaves were mixed with 100 ml of deionized water (WD). and boiled for 2 h at 80 ˚C to 90 °C. During the process, a light brown coloured solution was formed. Then the prepared extract was allowed to cool at room temperature and finally, it was filtered using Whatman's no. 1 filter paper. and stored in a refrigerator for further use.

2.3. Synthesis of Copper Oxide Nanoparticles from Leaf Extraction

In the present study, the copper ion was obtained mainly from used copper acetate monohydrate, which was bought from the chemical shop, Mukalla City, Yemen. The solution of copper acetate monohydrate), was prepared in deionized water. In phytochemical-mediated synthesis of copper oxide nanoparticles fabrication, 20 ml form the aqueous leave extract of *Moringa oleifera* (capping and stabilizing agent) was added to the aqueous solution of 0.05M copper acetate monohydrate [Cu $(CH_3COO)_2.H_2O$], and stirred using a magnetic stirrer heated at 80°C is nonstop until the homogenous solution pH was adjusted to 7.5 by using of sodium hydroxide (NaOH) (0.2 M) solution. The aqueous solution of copper acetate and leave extract of *Moringa oleifera* was continuously stirred at the same speed and temperature for another ninety minutes. Color changes were observed during the pH adjustment of extract and solution spontaneously from dark greenish into brownish black which is indicative of the formation of get green-synthesized copper oxide nanoparticles. Finally, the product was dried twenty four hours at 50°C Kumar, *et al.,* (6).

2.4. Characterization Techniques

The UV-visible absorbance and reflectance spectra of the copper oxide nanoparticles were recorded in the range of 200–800nm using Shimadzu's UV-730, UV-visible spectrophotometer. Fourier transform-infrared spectroscopy (FT-IR) spectrum was recorded using KBr pellets in the range of 500–4000cm^{-1}. X-ray diffraction (XRD-Shimadzu X-ray diffractometer) analytical technique was used to reveal the crystalline nature of copper oxide nanoparticles. The scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) model were used for understanding morphological and structural features of copper oxide nanoparticles Tsade, *et al.,* 2019 (51).

3. Results and Discussion

3.1. UV-Vis Analysis of CuO NPs from *Moringa oleifera* Leaf Extract

As illustrated in Figure 1, the synthesized of copper oxide nanoparticles was further identified by using UV–Vis spectrophotometer analysis the range between 200 to 800 nanometers, UV-Visible spectra revealed the absorbance peak in absorbance spectra at 236 nanometers, which is attributed to the formation of cuprous oxide nanoparticles Renuga, *et al.,* (39), Figure 1. Which is in good agreement with the results reported by the different researchers Gebremedhn, *et al.,* 2019; Abel, *et al.,*; & Hazaa, *et al.,* (1,15,20).

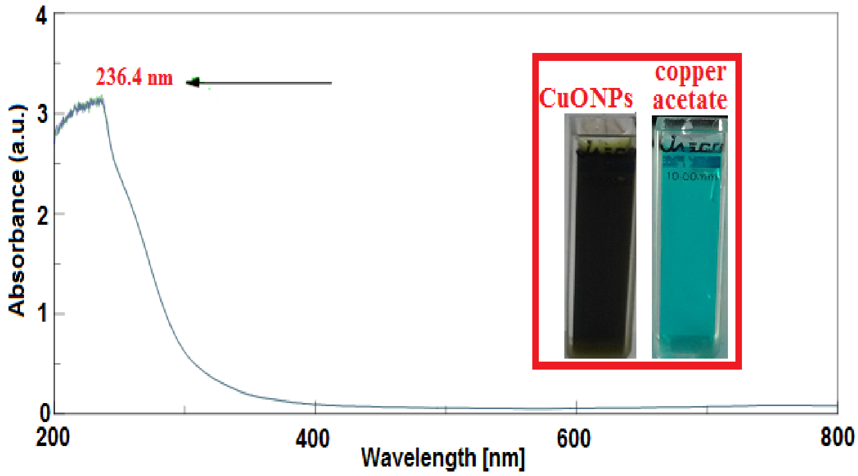

Figure 1: UV-visible analysis of CuO nanoparticle

3.2. FT-IR Spectral Analysis of CuO NPs from *Moringa oleifera* Leaf Extract

The infrared spectrums of the copper oxide nanoparticles CuO NPs and plant extract were recorded using Jasco V-730 -type device operating between 500-3750cm^{-1}. FT-IR spectra of *Moringa oleifera* leaf, copper oxide nanoparticles are as shown in Figure. 2. The peak of copper oxide nanoparticles at 3450 cm^{-1} broad band shows N-H stretching (amine group), bands at around 2927 cm^{-1}and 2856 cm^{-1} can be assigned to C-H asymmetric and symmetric stretching respectively. The peak at 1749 cm^{-1} reveals the presence of stretching vibrations of hydroxyl groups (O-H). The peaks at 1631cm^{-1} show C=C stretching (alkenyl), and 1463 cm^{-1} shows C-H asymmetric bending. The peak at 1030 cm^{-1} shows C-N stretching. The peaks found at 708 cm^{-1} and 550 cm^{-1} are assigned to the presence of biosynthesis of copper oxide nanoparticles Aliya, *et al.,* & Sathiyavimal, *et al.,* (5,43).

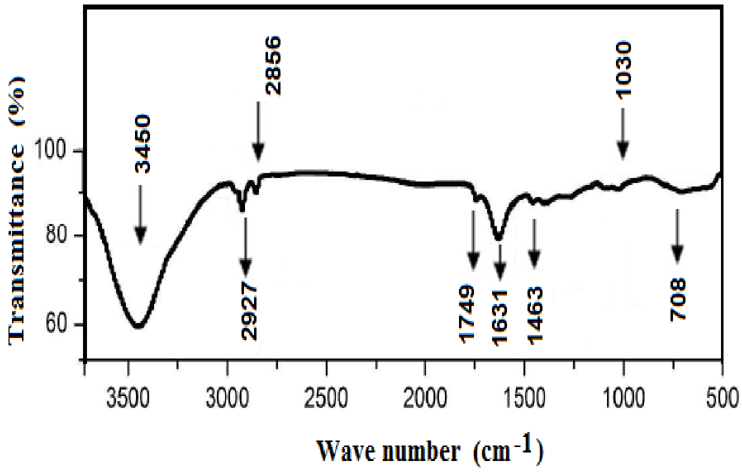

Figure 2: FT-IR spectra analysis of CuO nanoparticle

3.3. X-Ray Diffraction Analysis of CuO NPs from *Moringa oleifera* Leaf Extract

The XRD pattern has used to exactitude identification of synthesized copper oxide nanoparticles is shown in Figure. 3. The spectrum of copper oxide characteristic intensity sharp peaks at **2θ = 33.49, 35.49, 39.63, 48.72, 53.45, 58.33, 61.53, 66.35, 67.90, 72.42, 75.72** that can be ascertained to **(1 1 0), (0 0 2), (2 0 0), (–2 0 2), (0 2 0), (2 0 2), (–1 1 3), (0 2 2), (1 1 3), (3 1 1)** and **(0 0 4)** diffraction planes, respectively. The position of intensity peaks is perfectly assigned to the monoclinic structure of synthesized copper oxide nanoparticles (JCPDS file no.45-0937) Sharma, & Kumar, (45). Dry husk mediated bio-synthesis of copper oxide nanoparticles was revealed a monoclinic cubic structure Nwanya, *et al* (31). The XRD pattern of copper oxide is in accord with recent studies.

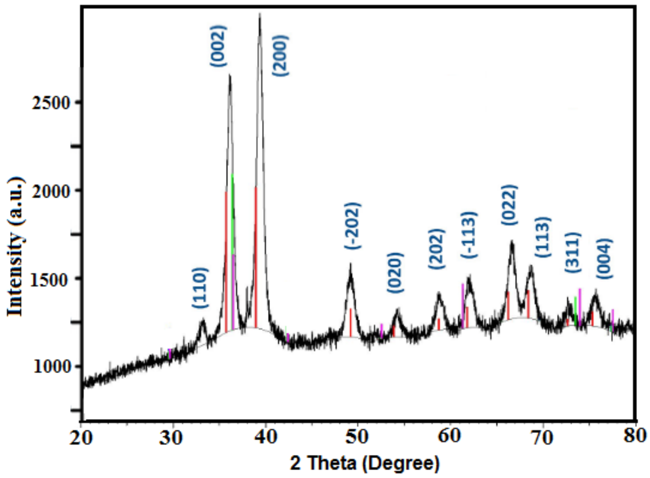

Figure 3: XRD pattern of CuO nanoparticles

3.4. SEM and EDX analyses of CuO NPs from *Moringa oleifera* Leaf Extract

The morphology, structure, and shape of the crystal, of the copper oxide nanoparticle were revealed by SEM image. The size of the crystal, the shape of the crystal, and the morphology of the copper oxide nanoparticle are clearly shown by the scanning electron microscope image as displayed in Figure 4. Detailed structural characterizations demonstrate that the synthesized products are (flower-like shapes), SEM images show that the particles are round, granular, and nanosized and the nanoparticle is more aggregation and conglomeration. These results also agree with those previously reported by Pakzad, *et al.* Iranbakhsh, *et al.,* & Al Baloushi, (32,21,4). EDX analysis was used to verify the chemical elements found in the biosynthesized CuO NPs Figure 5. The EDX spectrum confirms that CuO NPs contain both oxygen (O) and copper (Cu) with a weight percentage of approximately 89.8 % and 10.2 % respectively as shown in Table 1.This finding agrees well with previous works by Pakzad, *et al.,* & Iranbakhsh, *et al.,* (32,21).

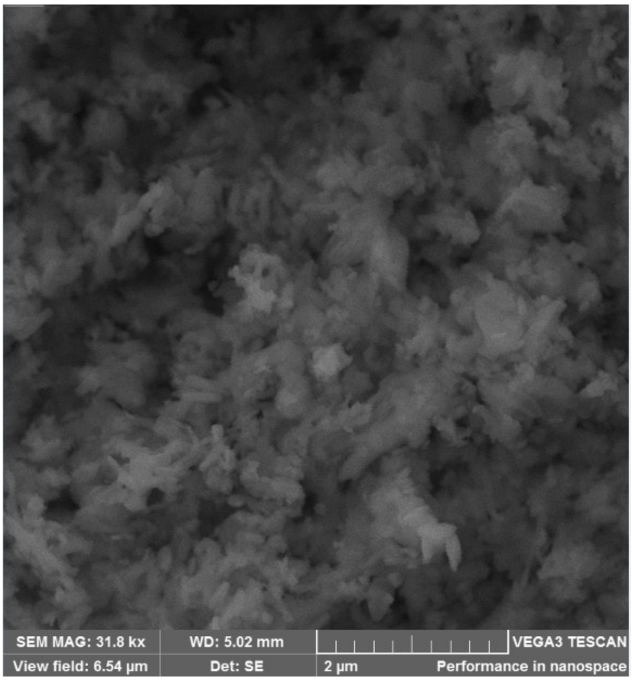

Figure 4: Scanning electron microscope image of copper oxide nanoparticles

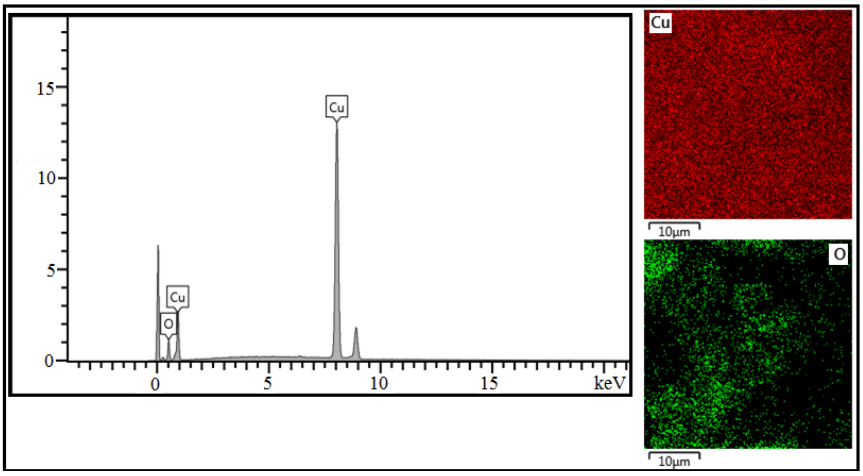

Figure 5. Energy dispersive X-ray analysis of CuO nanoparticles

Table 1. Weight percentage of Cu and O atoms present in CuO-NPs synthesized using *Moringa oleifera* Leaf Extract

Spectrum Number	Element	Series	Weight (%)	Atomic (%)
Cu	29	k	89.8	68.91
O	8	k	10.2	31.09
Total			100	100

4. Conclusion

The green synthesis of copper oxide nanoparticles was efficaciously performed using copper acetate as a precursor and *Moringa oleifera* plant leaf extract as a powerful reducing/oxidizing chelating agent. The applied biosynthetic technique is undemanding and easily biodegradable due to the presence of phytochemicals and is carried out in a short time. The visible green color indicates the production of copper oxide nanoparticles, further demonstrating the reduction of Cu ions in copper oxide nanoparticles using ultraviolet-visible spectroscopy. The ultraviolet spectroscopic absorption peak is at 236 nanometers. The XRD, SEM and EDX analyzes show that the detailed structural characterizations show that the synthesized products are (flower-like shapes), that the particles are round, and granular, and that the nanoparticles show more aggregation and conglomeration.

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اصطناع وتشخيص جزيئات أكسيد النحاس النانوية باستخدام مستخلص أوراق *Moringa oleifera*

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الملخص

تم استخدام مستخلص أوراق *Moringa oleifera* اليمني للتخليق الأخضر لجسيمات أكسيد النحاس النانوية. تم تمييز الجسيمات النانوية لأكسيد النحاس التي تم الحصول عليها باستخدام التحليل الطيفي المرئي فوق البنفسجي (UV-VIS)، والتحليل الطيفي للأشعة تحت الحمراء (FT-IR)، والفحص المجهري الإلكتروني (SEM)، والتحليل الطيفي للأشعة السينية المشتتة للطاقة (EDX) وقياسات حيود الأشعة تحت السينية (XRD). أظهر المعلق المائي لـ (CuO NPs)الأشعة فوق البنفسجية. قمم الامتصاص القصوى عند 236 و 240 نانومتر فيما يتعلق بشكل أساسي بتكوينها. يجب تأكيد تحليل FTIR لمجموعات وظيفية من CuO NPs التي تم تصنيعها حيوياً في نطاق الامتصاص عند 608 سم $^{-1}$. كشف تحليل XRD أن الجسيمات النانوية من اكسيد النحاس المُصنَّع حيوياً يبدو أنه أكثر بلورية. أكدت القمم المميزة التي تم الحصول عليها بواسطة تحليل (EDX) وجود عنصر النحاس بنسبة (89.8)، وعنصر الأكسجين بنسبة (10.2). تُظهر صور SEM أن الجسيمات مستديرة وحبيبية ومتناهية الصغر وأن الجسيمات النانوية أكثر تجمعًا وتكتلًا. يعمل مستخلص أوراق*Moringa oleifera* كعامل واعد لتوازن حجم الجسيمات. تظهر نتيجة القيمة الطبية نشاطًا كبيرًا مضادًا للبكتيريا.

الكلمات المفتاحية: التخليق الأخضر، جزيئات أكسيد النحاس النانوية، *Moringa oleifera*، تقنية النانو.