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Synthesis of copper oxide nanoparticles at various pH values and temperatures by applying green chemistry using Bardi plant (Typha domingensis)

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***Abstract*—In the field of materials science, "green" synthesis has received significant attention as a reliable, sustainable, and environmentally friendly method for producing diverse materials and nanomaterials, such as hybrid materials, bioinspired materials, and metal/metal oxide nanomaterials. Green synthesis is a crucial tool for mitigating the harmful effects of conventional nanoparticle synthesis techniques widely used in laboratories and industry. In this study, we provide an overview of the basic procedures and mechanisms of action of "green" synthesis methods, specifically for metal nanoparticles and metal oxides such as copper oxide (CuO), made using natural extracts. Key phytochemicals, such as flavonoids, alkaloids, and terpenoids, serve as solvent systems and reducing agents. The synthesized nanoparticles were examined using several techniques, such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and ultraviolet–visible spectroscopy. UV–vis: The best results were obtained at 70 °C and pH 9 and pH10,45 ℃. Spectroscopic analysis of the synthesized copper solution showed an absorption peak at 242 nm. XRD spectra of the biosynthesized copper oxide nanoparticles yielded a series of diffraction peaks at 2θ angles, which correspond to the h, k, and l values of the reflections. SEM revealed that the resulting copper oxide particles had a spherical shape, with an average particle size ranging from 32 to 65 nm. This study aimed to produce nanoparticles using green methods using the aqueous extract of Typha domingensis at low cost and low toxicity.**

***Keywords*—: nanoparticles: copper nanoparticles: SEM: XRD: UV-visible: Typha domingensis.**

I. INTRODUCTION

Nanotechnology and nanoscience are multidisciplinary fields that use innovative approaches to manufacture exceptional and distinctive qualities of a nanomaterial at the nanoscale and to utilize the wide range of the developing field from fundamental disciplines including physics, chemistry, electronics, and material science [1-2]. The size range of nanoparticles is 1–100 nm [3]. Nanoparticles can be categorized as 0D, 1D, 2D, or 3D according to their morphology [4]. And there are two types of nanoparticle synthesis methods: top-down and bottom-up [5]. Utilize microorganisms and plant extracts to fabricate nanoparticles; this constitutes the foundation of green synthesis [6]. The active ingredients found in plants exhibit a great deal of biological activity, including powerful antibacterial and anticancer effects, strong antioxidant and anti-inflammatory qualities, and other substantial therapeutic benefits [7].

In recent years, microorganisms have emerged as evidence against several antimicrobial treatments. In particular, antibiotic resistance has developed in the two most significant bacteria, streptococcus and pseudomonas aeruginosa. Metallic nanoparticles (MNPs) were suggested as an alternative to antibacterial medications due to their efficacy against fungus and bacteria. Because of their length distribution, shape, and too high floor-to-amount ratio—which calls for smaller dosages—MNPs have a greater biological energy than their equivalents in bulk metals. There is an excessive amount of interplay between the surface and the microbial membrane of MNPs [8]. The composition, size, shape, surface area, and material type of MNPs are important factors for both commercial and medicinal uses. The cleanliness and homogeneity of the nanomaterials are very important in various packaging because impurities in cosmetics could negatively affect customers [9]. Nonetheless, nanoparticles produced using environmentally friendly methods that employ plant extracts continue to have excellent potential for increasing nanoparticle production without the need for costly and hazardous chemicals. This study examines the copper oxide nanoparticle (CuO NP) production using plant extract and its structural, morphological[10]. Copper nanoparticles were created in this investigation using the plant Typha domingensis.

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II. MATERIALS AND METHODS

A. Typha domingensis plant collection

Typha domingensis (Bardi plant) was collected from the marshes in Al-Chibayish, dated to the city of Nasiriyah in Iraq in the month of July 2024. The collected leaves were cleaned with purified water and allowed to air dry for 14 days at room temperature in the shade, crushed by a high-speed grinder to obtain fine powder, and saved in the powder in a refrigerator 4 ℃ until use.

B. Preparation of plant extract

Five hundred milliliters of deionized water were used to extract fifty grams of dried leaves, which were then incubated for forty-eight hours in a water bath. Thereafter, the extract was filtered three times using a Tetron cloth and once using a piece of gauze. A 45°C rotary evaporator was used to remove the solvent after the extract was collected, and the crude extract was then stored for additional testing at 4°C.

C. F. synthesis of copper oxide nanoparticle

For the synthesis of plant Typha domingensis copper oxide nanoparticles,50 mL (5 m M) copper sulfate ($Cu\ SO_4.\ 5H_2O$) solution was mixed with 5 mL of aqueous plant Extract the pH value (9,10) adjusted for the mixture by the addition of NaOH (1 N) solution and temperature 70,45 ℃ Further, a green-colored solution was formed.". The two mixtures were constantly stirred on a magnetic stirrer for 2 h. followed by centrifugation at 5000 rpm for 25 min. Residues were washed many times with de-ionized water. The above-mentioned processes were repeated several times to remove impurities (if any) from the copper oxide nanoparticles. The precipitates so obtained were dried overnight in an oven at 60 ℃ [11]. As Figure 1 show.

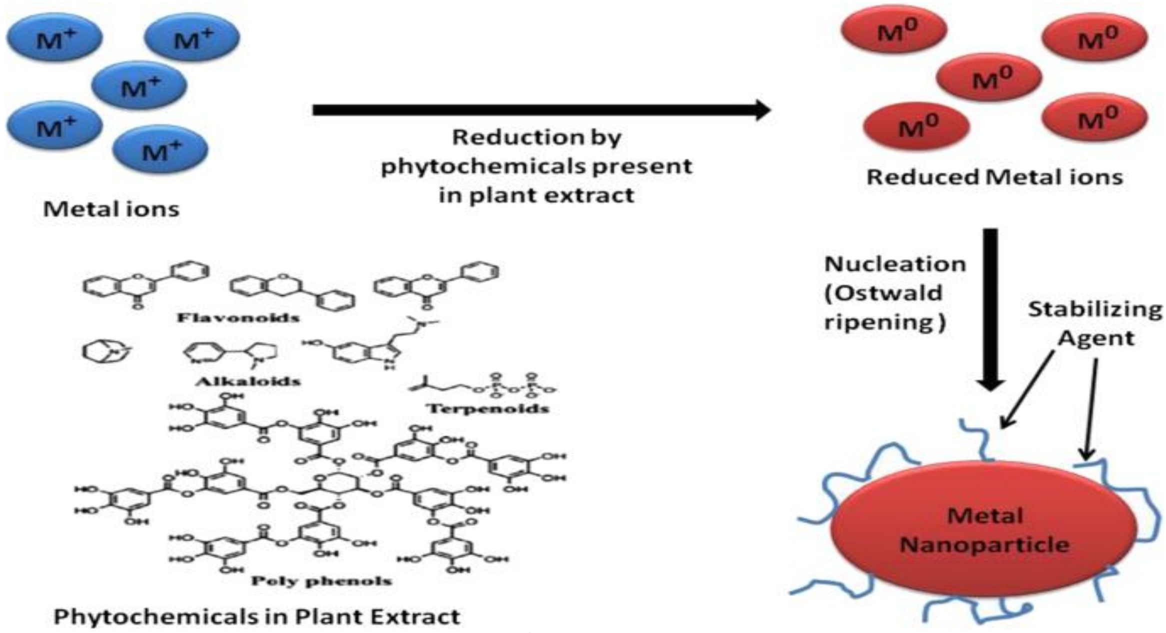

Fig. 1: Synthesis of copper oxide Nanoparticle [12]

D. Plants' role in the production of green nanoparticles

Alkaloids, citric acid, phenols, polyphenols, ascorbic acid, terpenes, flavonoids, and other active ingredients are important reducing agents found in plant extracts. Given the reducing and capping properties of plant extracts, the production of nanoparticles from plant biomass is crucial for nanotechnology [13].

E. Factors influencing the synthesis of nanoparticles

Nanoparticle production is impacted by a number of physical and chemical factors. Plant extracts, temperature, pH, reaction duration, and substrate concentration are some of these variables. Copper nanoparticles have been produced using a range of factors, such as temperature, pH [14].

F. Factors affecting synthesis sate, Size and shape of nanoparticles

1) Temperature Effect

Various levels of research are being conducted worldwide to understand the temperature control of nanoparticles. Temperature is the most important factor influencing the size and shape of nanoparticles, as well as their synthesis level. The various shapes (triangle, octahedral platelets, spherical, and rod-shaped) and the size of nanoparticle synthesis can be adapted depending on the temperature. As the temperature increases, the reaction rate also enhances the formation of nucleation centers [15]. In the green synthesis process of nanoparticles, the reaction time is a key factor that critically affects the shape, size and yield of the synthesized nanoparticles [16].

2) PH

The response pH plays an important role in the structure of nanoparticles. Indeed, pH and temperature also control the formation of nucleation centers. Increasing pH automatically leads to an increase in the number of nucleation centers, which is essential to stimulate the formation of metal nanoparticles. It is recognized that pH plays an important role in formulating the structural morphology and size of nanoparticles [17].

III. CHARACTERISATION OF COPPER OXIDE NANOPARTICLES

The morphology and structure of the generated nanoparticles were examined using a UV-visible spectrophotometer, X-ray diffraction, and scanning electron microscopy (SEM), respectively.

IV. RESULTS AND DISCUSSION

A. Percent yield of Typha domingensis CuO NPS

For aqueous extract of Typha domingensis:

The weight of Typha domingensis used :50 g

The weight of aqueous extract: 11g

Percentage of extract = (weight of Extract /weight of plant) *100%

Percentage of extract = (11 /50g) *100%

Percentage of extract =22%

B. Aqueous extract qualitative tests of a few active compounds

Table 1. Initial screening of phytochemicals for aqueous extract [18]

No.	Constituents	Test	Results
1	***Saponin***	***HgCl2 (5%)***	-
2	Carbohydrates	Molisch reagent	+
3	Tannins	(a) FeCl3 (b) Lead Acetate	+
4	Phenols	FeCl3 1%	+
5	Alkaloids	Wagner's test	+
6	Glycosides	Fehling's reagent (a)Before glycoside dissociation (b)After glycoside dissociation	+
7	Setrols	H2SO4	-
8	Triterpenoids	H2SO4	+
9	Flavonoids	Lead acetate test	+

C. UV-vis. Spectroscopy

The UV-vis absorption spectra of the synthesized green copper nanoparticles were recorded at different wavelengths within the range of 200-600 nm, as shown in Figure 2. The copper nanoparticles were synthesized from copper sulfate (CuSO4.5H2O) and plant extracts. The absorption peak appeared when the extract was used as a reducing agent, where Cu^{2+} was converted to CuO. This peak is attributed to the synthesis of copper nanoparticles based on plant extracts [19]. The wavelength in this study was 242 nm [15]. As Figure 2-5 shows.

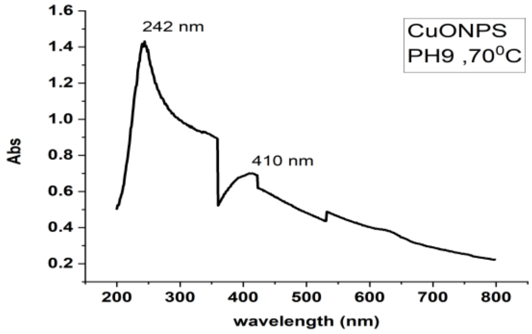

Fig.2: The plasmon spectrum of copper oxide nanoparticles prepared at pH9 and temperature 70°C is shown

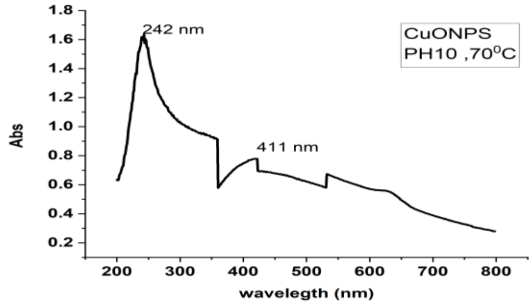

Fig .3: The plasmon spectrum of copper oxide nanoparticles prepared at pH10 and temperature 70°C is shown

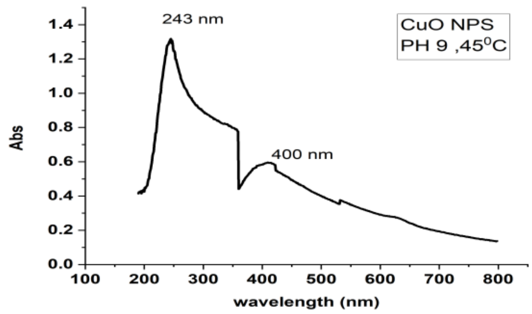

Fig .4: The plasmon spectrum of copper oxide nanoparticles prepared at pH9 and temperature 45°C is shown

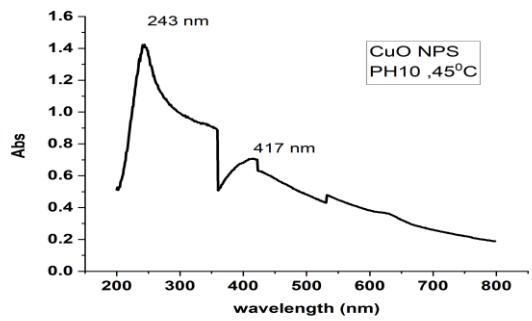

Fig .5: The plasmon spectrum of copper oxide nanoparticles prepared at pH10 and temperature 45°C is shown

D. X-ray diffraction XRD

The XRD image of copper nanoparticles (Cu NPs) green synthesis from Typha domingensis extract is shown in Figures 3 to 6. The Scherer equation was used to determine these nanoparticles' dimensions:

D=(Kλ)/(b cos θ)

where k = 1.5406 Å is the frequency of Cu-Ka radiation, b is the peak's half-width, D is the diffraction size in nanometers, and k is a constant equal to 0.9. Several diffraction peaks are visible in X-ray diffraction analysis [20]. This study observed X-ray diffraction peaks with 2θ angles of 31.75°,35.6° ,43.37°,46.25° ,51° ,58.36° ,61.52° ,48.8° ,53.49° ,75.08° , 73.58° ,38.73° ,55.02° and77.49° corresponding to the h, k, l values of the reflections from (327.32), (002), (111), (202), (200), (202), (113), (202), (200), (303), (044), (111) ,(200), and (222) The results obtained were close to the study conducted by [21]. The synthesized copper oxide's crystal structure was discovered via X-ray diffraction data. The produced nanoparticles' crystalline characteristics were ascertained by X-ray diffraction (XRD). Diffraction peaks were visible at 2-theta angles, i.e., 20.1, 21.7, 24, 30.9, and 34.12 degrees, in the agglomerated biomaterial's diffractogram. These peaks could be brought on by pollutants [22]. As shown in Figures 6 to 9.

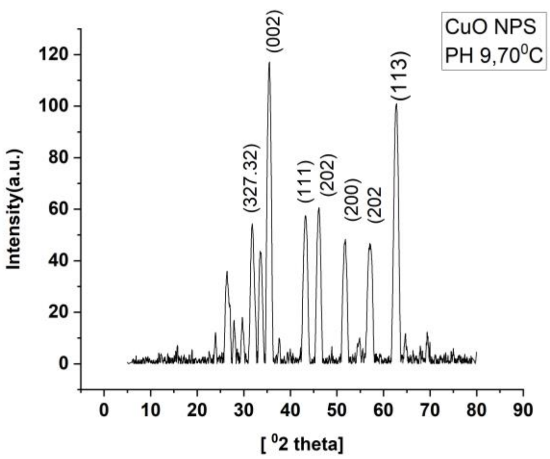

Fig. 6 XRD spectrum of copper oxide nanoparticles at pH9, 70 ℃

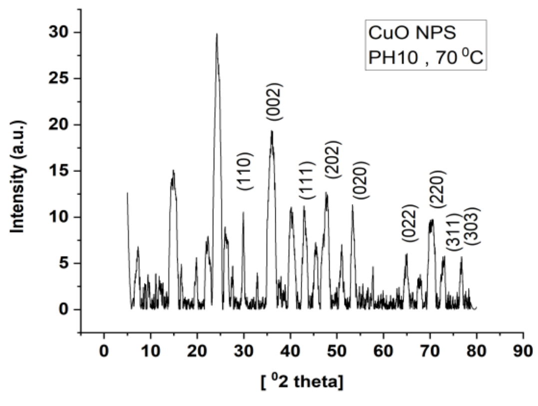

Fig.7: XRD spectrum of copper oxide nanoparticles at pH10, 70 ℃

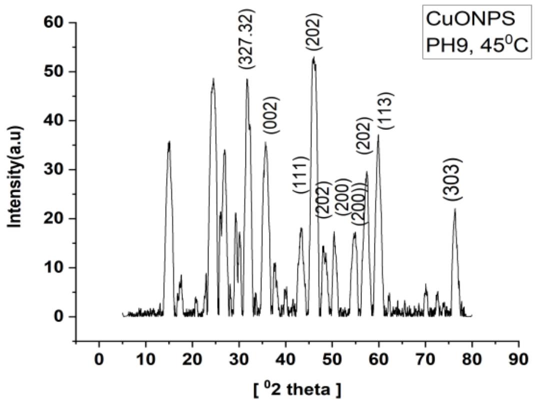

Fig .8: XRD spectrum of copper oxide nanoparticles at pH9, 45 ℃

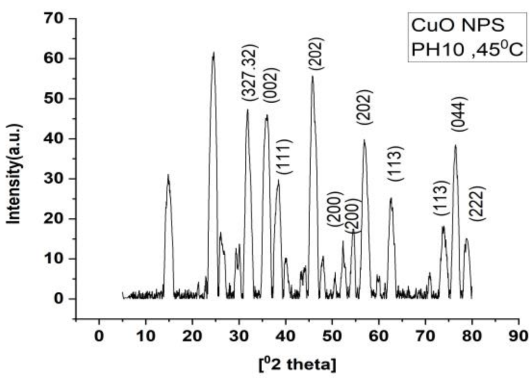

Fig .9: XRD spectrum of copper oxide nanoparticles pH9, 45 ℃

E. scanning electron microscope

A scanning electron microscope (SEM) was used to measure the sizes of the nanoparticles and their surface characteristics [23]. Copper oxide nanoparticles made from plant extract Typha domingensis are depicted in the image (1-4). The creation of copper oxide nanoparticles from the plant extract is caused by hydrogen bonds and electrostatic interactions between the organic molecules. The copper oxide nanoparticles' comparatively consistent spherical shape [24]. Ranging from 1 to 100 nm, this study verified this. At 70 ℃ and pH 10 and 9, the nanoparticle sizes were (37-81) and (32-62) nm, respectively; at 45 ℃ and pH 10 and 9, the sizes were (40 -65) and (63-82) nm, respectively. In the current study, the best copper oxide nanoparticles were obtained at pH 9,70 ℃, pH 10,45 ℃. The images 1-4 show.

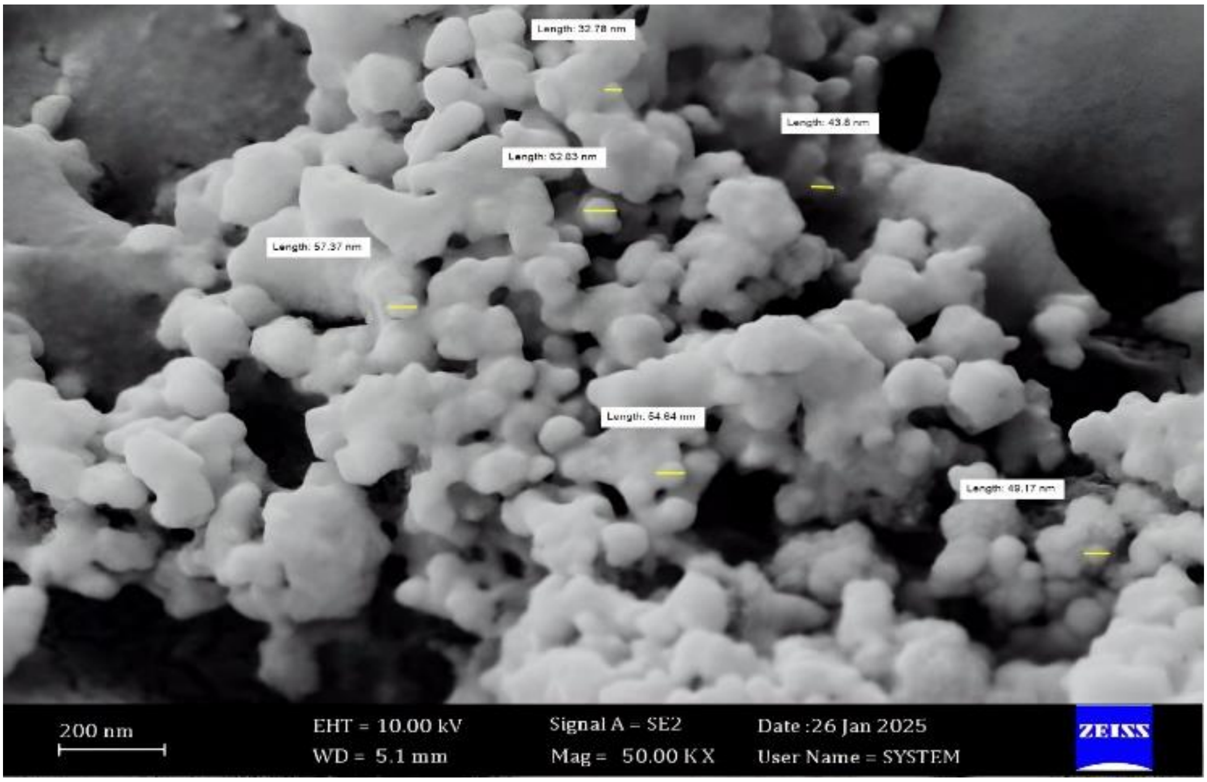

Fig.10: SEM image for copper oxide nanoparticles pH 9,70 ℃

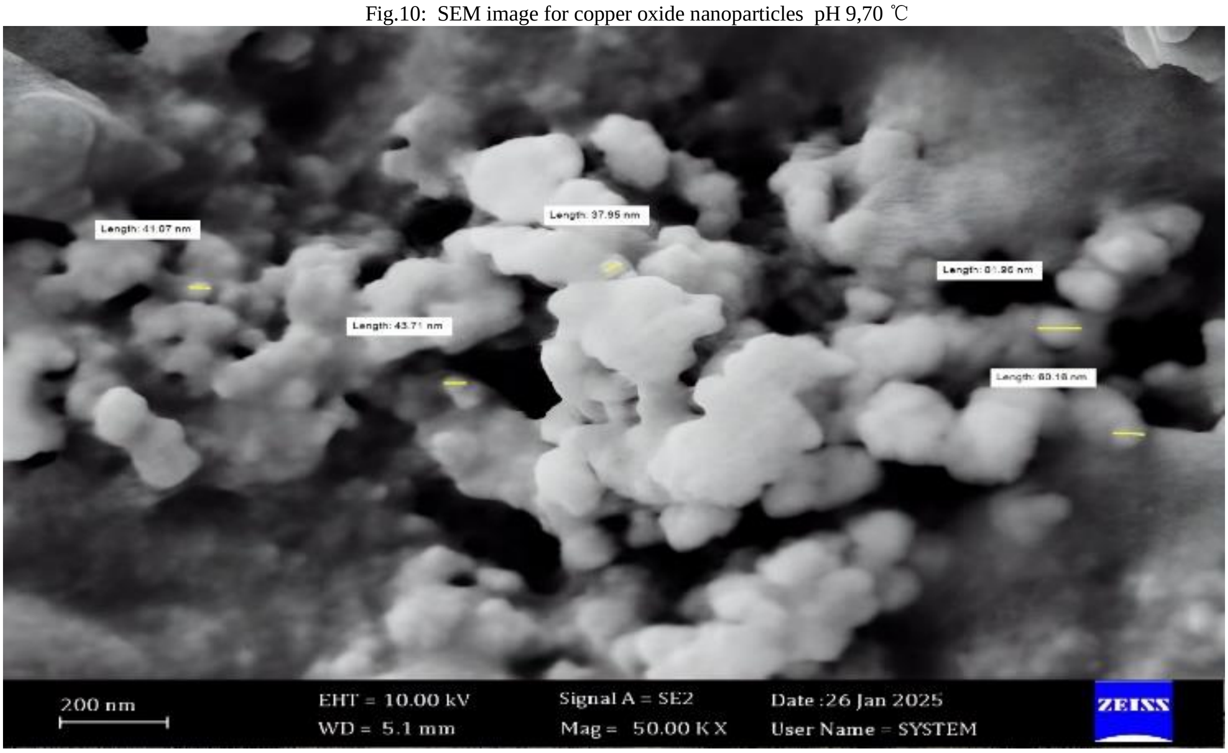

Fig.11: SEM image for copper oxide nanoparticles pH 10,70 ℃

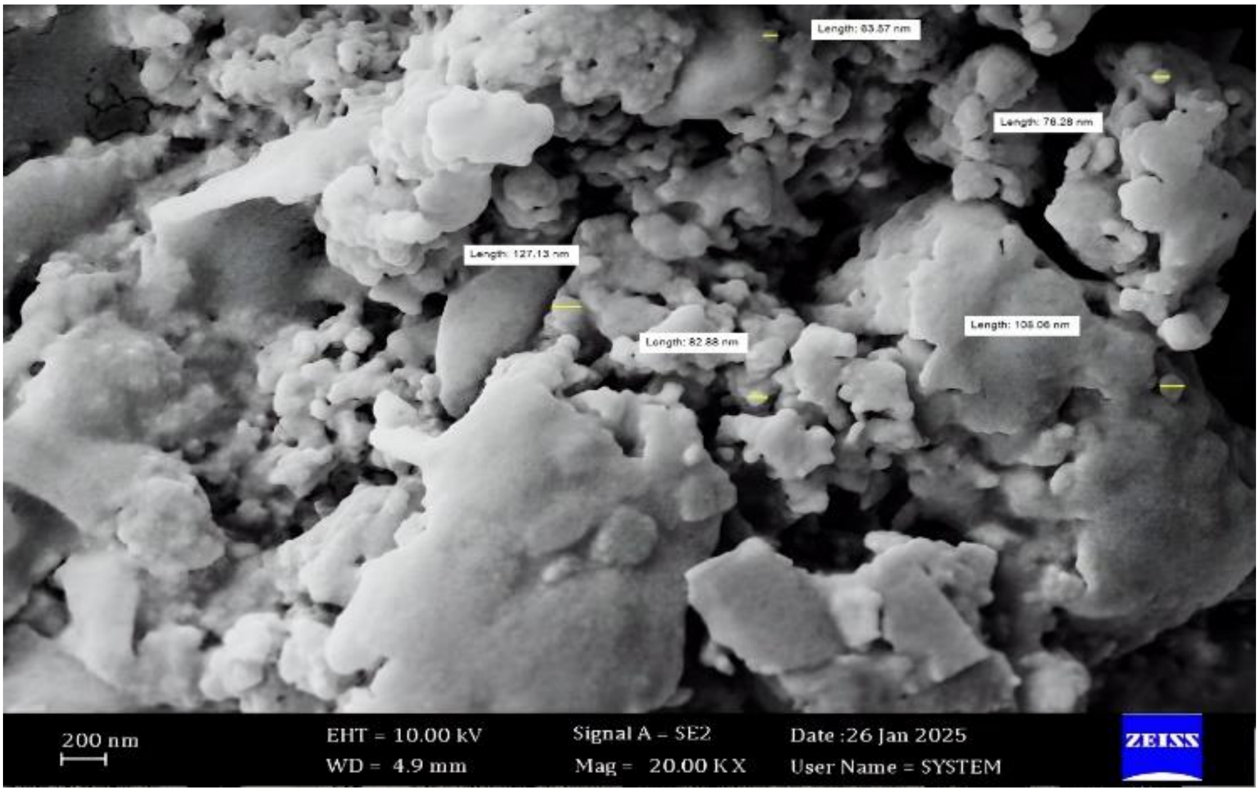

Fig. 12: SEM image for copper oxide nanoparticles pH 9,45 ℃

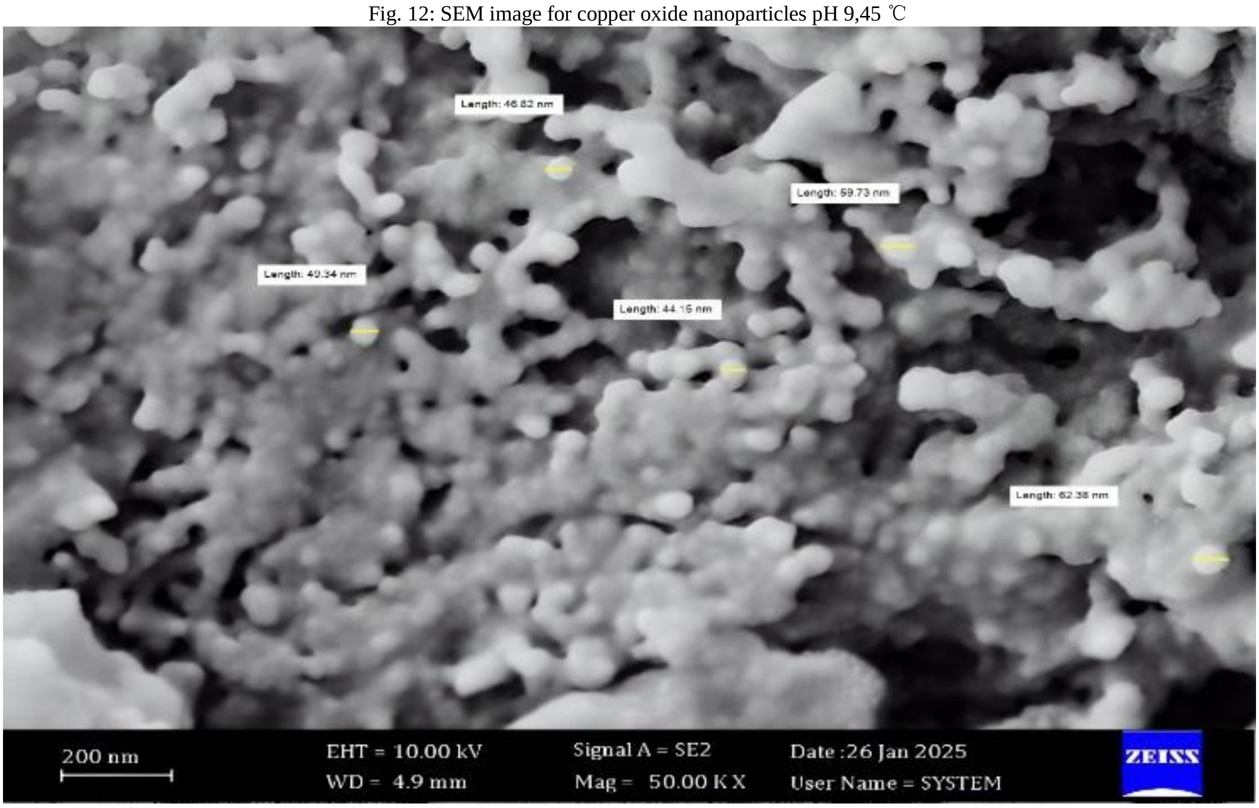

Fig. 13: SEM image for copper oxide nanoparticles pH 10,45 ℃

F. Antibacterial Activity of Copper oxide nanoparticles NPs

The antibacterial activity with microorganisms was detected against Gram-negative bacteria P. aeruginosa and Gram-positive bacteria streptococcus aureus using the agar disk diffusion method and compared with the antibacterial activity of antibiotics (VA, CFM, CAZ, ATM, KF, E, NET, TCC). The antibacterial action of microorganisms was shown using a circle diffusion approach. Copper oxide nanoparticles kill bacteria because they have a high surface-to-volume ratio and are tiny, which lets them get near to microbial membranes. We considered the potential of copper oxide nanoparticles to eradicate bacteria such as against Gram-negative bacteria P. aeruginosa and Gram-positive bacteria streptococcus aureus. We employed the plate dispersion approach. We added the copper oxide NPs to the purified circle and then incubated the samples at 37 °C for 24 hours. Then, they were gently spread out on

Mueller-Hinton agar plates, from the plate concentration point to the right area. The area of confinement was measured in mm, and the results were written down. We employed copper nanoparticles in different amounts: 25%, 50%, and 75% (w/v) [20]. The results indicated a progressive impact on the growth of bacterial strains subjected to elevated concentrations. The bacterial species exhibited varying susceptibility to the CuO NPs. We used different concentrations of copper oxide nanoparticles (25%, 50%, and 75%) (w/v) at pH 10 and 9 and at temperatures of 70°C and 45°C, respectively. They were the diameter of the inhibition zone of bacteria, as shown in the table 2

Table 2. the inhibitory ability of copper oxide nanoparticles against Gram-positive bacteria and Gram- negative bacteria.

Bacteria name	Bacteria strain	Inhibition zone (mm)										
		25%	50%	75%	VA	CFM	CAZ	ATM	TCC	KF	E	DMSO
P. aeruginosa In PH10 ,70^{0}C	_	20	25	26	28	0	0	0	0	0	0	0
P. aeruginosa In PH9 ,45 ^{0}C	_	21	23	25	28	0	0	0	0	0	0	0
s. aureus In PH10 ,700C	+	22	21	23	20	30	0	0	28	0	0	0
s. aureus In PH9 ,450C	+	26	23	21	20	30	0	0	28	0	0	0

The interaction between the nanoparticles and the outer surface of the cell membrane of cultures is the mechanism that underlies their antibacterial capability. In the end, this contact causes pits to form in the membrane, which compromises its integrity and permits the release of lipopolysaccharide molecules or other protein compounds, finally resulting in cell death. Due to its ability to adversely affect a wide range of cell processes, copper is a powerful microbial inhibitor. Copper usually harms microorganisms by binding to or replacing natural cofactors in metalloproteins and creating reactive oxygen species (ROS) [25].

Fig.(A): CuO NPs pseudomonas aeruginosa (25% ,50%, 75%)

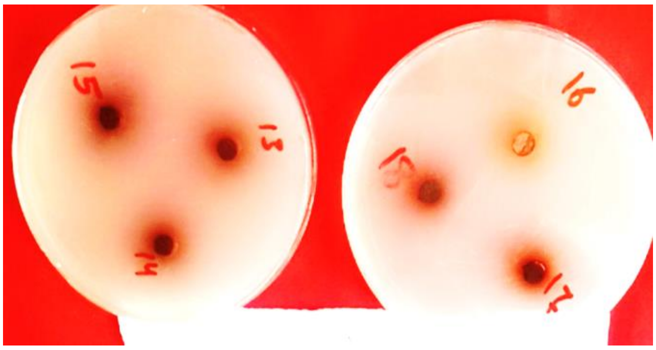

Fig.(B): copper oxide nanoparticles staphylococcus aureus (25% ,50%, 75%)

Figure 14 (A, B). Antibiotics activity of copper oxide nanoparticles against Staphylococcus aureus and Gram-negative Pseudomonas aeruginosa

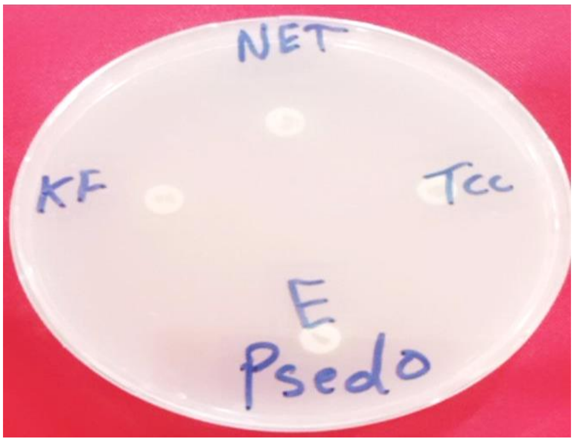

Fig.15:Antibiotics activity against Gram-positive Staphylococcus aureus and Gram-negative Pseudomonas aeruginosa

V. Conclusion

The green preparation of metal nanoparticles (MNPs) and metal oxide nanoparticles (MONPs) is a promising and appropriate subject for a variety of investigations due to the development of nanoformulations and their many uses. A thorough understanding of the characteristics of the kinetics and mechanism of nanoparticle creation, as well as the factors influencing the green synthesis of metal nanoparticles (MNPs) and metal nanoparticles (MONPs) from plant extracts, particularly plant leaf extracts, is currently lacking. They are a popular source for the synthesis of nanoparticles despite their many benefits, including lower toxicity and simplicity in method application. These substances have been employed for many years to successfully manufacture a variety of molecules and organic compounds because they are efficient, highly selective, reasonably priced, conventional, and "historically safe" reducing agents. Other safer, more sustainable, and dependable reducing and capping agents must be used in order to synthesize nanoparticles in a completely environmentally friendly manner. Finally, using dangerous chemicals must be avoided.

Conflict of interest

The authors declare no conflict of interest

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