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Green Synthesis and Applications of Copper Oxide Nanoparticles- A Review

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ABSTRACT : Copper oxide nanoparticles have garnered a lot of attention among the metal oxide nanoparticles, especially because of their many characteristics and applications in many disciplines, notably nanomedicine and biomedical sciences. We have covered all of the conceivable green production techniques of copper/copper oxide nanoparticles in this review. This manuscript also diagrammatically depicts the exact mechanism of all conceivable biosynthetic routes. We also look at the antibacterial, antifungal, antiviral, and anticancer properties of biosynthesized copper oxide nanoparticles, as well as their effects on plant growth, nutrition, and defence mechanism.

KEYWORDS - antibacterial, characterization, green synthesis, nanocluster, reduction.

I. INTRODUCTION

In the sphere of research, nanotechnology is a relatively new approach. This technology is now widely used in diversified fields. The smaller dimension of nano materials, ranges from 1–100 nano meters (nm), alters their physicochemical properties like shape, size, and chemical composition. In the twenty-first century, a more in-depth investigation of metallic NPs was carried out by several researchers. Bio nanotechnology is a rapidly growing field of nano technology in which bio-organisms are extensively used to synthesize nano materials and the synthesized nanomaterials are simultaneously used to improve the quality of life of the organisms. Biological synthesis uses the bio logical principle of oxidation and reduction by microbial enzymes or plant phytochemicals. In recent times physical and chemical methods are mainly used for the synthesis of inorganic NPs. Both physical and chemical methods have some disadvantages like low-productivity, non-eco-friendly, toxic, and capital intensive. For these reasons, biological synthesis is trying to replace the chemical methods of producing NPs.

Due to their intriguing features, copper oxide (CuO) nanoparticles are one of the most significant transition metal oxides in the developing area of nanotechnology. Due to its simplicity, environmental friendliness, and economic viability, its synthesis using green chemistry principles is becoming more significant as a next-generation antibiotic [1]. Considering the context, the purpose of this review paper is to investigate the potential of different plant extracts for the environmentally friendly synthesis of CuO nanoparticles and their antibacterial properties. In addition to these uses, copper has also been determined to be harmless to mammals [2]. In the visible spectrum, copper oxide (CuO), a narrow bandgap p-type semiconductor (1.2–1.8 eV), has significant light absorption characteristics [3]. The green synthesis of CuO NPs offers several advantages over traditional chemical synthetic methods. It eliminates hazardous chemicals and reduces toxic waste production, making it an eco-friendly approach [4,5]. Additionally, a green synthetic approach often utilizes natural extracts derived from plants, algae, or microorganisms, which are abundant, renewable, and cost-effective [6].

These natural extracts contain various bioactive compounds, such as polyphenols, flavonoids, and proteins, which can serve as reducing and stabilizing agents for the synthesis of CuO nanoparticles [7]. Thus, eco-friendly methods for synthesizing metal oxide nanoparticles, such as using plant extract, microorganisms, and algae, attracted attention. However, among biological methods, plant extract was more favored due to its easy handling, easy availability, cost- effectiveness, and compatibility with biomedical applications such as drug delivery, cancer treatment, antibacterial and antifungal agents, and insecticide treatment [8]. This review aims to provide a comprehensive guide on the green synthesis, characterization, and applicationss of CuO nanoparticles, which may be useful to researchers aiming to develop new or hybrid fabrication techniques. In addition, this review also makes an effort to discuss the methods used to characterize the CuO NPs' characteristics. The synthesis, characterization, and uses of CuO nanoparticles utilizing plant extracts are summarized in this review paper.

Green Synthesis Of Copper Oxide Nanoparticles : Copper oxide nanoparticles have been synthesized using physical, chemical, and biological techniques. However, this review focused on the green synthesis of CuO nanoparticles using plant extracts. The reduction of copper salts is brought about by the electrons produced by the plant extract. The phytochemicals' interaction with the copper ion results in a reduction, which is followed by the formation of copper oxide nanoparticles.

Characterization of Cu/Cuonps : To establish the demand for the generation of nano particles one researcher should go through a series of characterization. After the synthesis of NPs, the crystal structure and chemical composition are the initial stage in the characterization process. The size and morphology of the Cu/CuONPs were investigated using scanning electron microscopy, transmission electron microscopy, dynamic light scattering, particle analyzers, and field emission scanning electron microscopy, while UV–visible spectroscopy, X-ray diffraction, Fourier transform infra-red spectroscopy, surface plasmon resonance, and energy-dispersive X-ray spectroscopy were used to analyze the elemental chemical compositions of Cu/CuONPs (Figure 1).

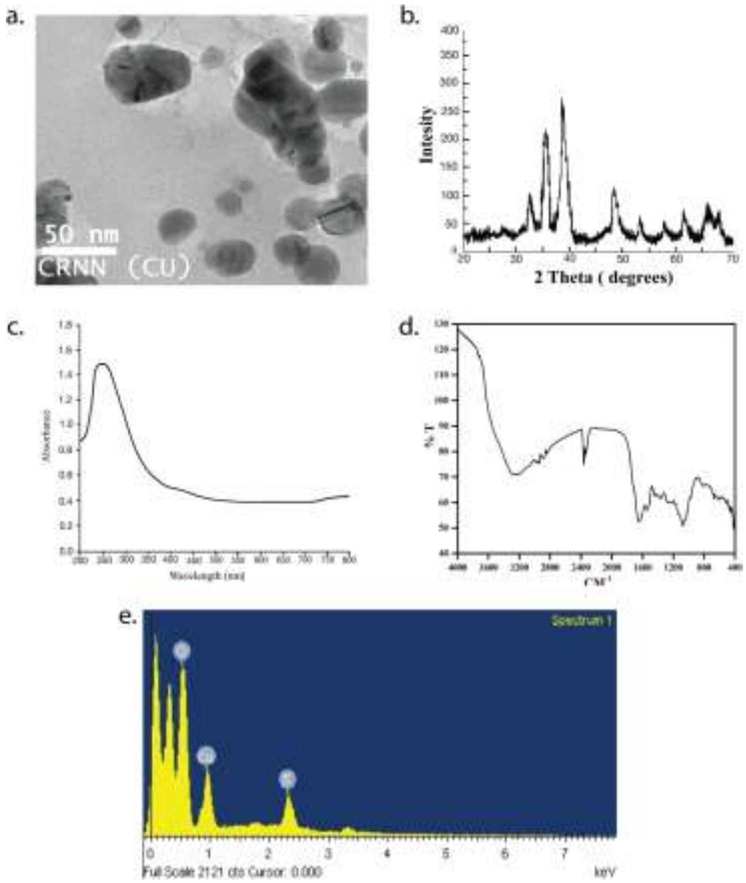

Figure 1. Characterization of synthesized copper oxide nanoparticles (a) TEM images (b) XRD analysis (c) UV-Visible Spectroscopic (d) FTIR analysis (e) EDX analysis of CuONPs.

II. LITERATURE SURVEY ON CHARACTERIZATION AND SYNTHESIS OF COPPER OXIDE NANOPARTICLES

From the review of the literature, it is found that the copper oxide (CuO) nanoparticles (NPs) have been researched as potential candidates for several important technological fields namely, as magnetic storage devices, solar energy transfer, gas/ organic solvent vapour sensors, and super capacitors and especially it acts as a good catalyst in some of the chemical reactions. Also, most researchers have focused their research in preparing these particles by simple, economic and ecofriendly methods such as aqueous precipitation methods, simple Sol-Gel, followed by spray pyrolysis method, carbon nanotube templating methods, reverse-micelle methods or green methods using leaf extracts. The following Table 1 provides a summary of several reports on the synthesis of copper oxiden nanoparticles and their nanoclusters utilising plant extracts and chemicals.Huaming Yang et. al 2014 [9] successfully synthesized Cuprous oxide (Cu_2O) nanoparticles of 35nm in crystal size via electrochemical method. The obtained NPS were characterized by means of UV–vis, XRD, XPS, FT-IR, FE-SEM and EDS.

Photocatalytic degradation of methyl orange (MeO) in aqueous Cu_2O NPs solution was investigated under either ultraviolet (UV) light or sunlight. Recycling use of the catalyst revealed that Cu_2O still has a high photocatalytic efficiency when repeatedly used for four times. Renu Sankar et.al 2014 [10] synthesized copper oxide (CuO) nanoparticles by treating 5mM cupric sulphate with *Carica papaya* leaves extract. The kinetics of the reaction was studied using UV–visible spectrophotometry. An intense surface Plasmon resonance between 250–300nm in the UV–vis spectrum clearly reveals the formation of copper oxide nanoparticles. The results of (SEM) and (DLS) exhibited that the green synthesized copper oxide nanoparticles are rod like shape and having a mean particle size of 140nm, Furthermore, colloidal copper oxide nanoparticles effectively degrade the Coomassie brilliant blue R-250 dye beneath the sunlight.

M. Behera and G. Giri [11] reported a facile green synthesis of Cu_2O nanoparticles (NPs) using copper sulfate as precursor salt and hydrazine hydrate as reducing agent in presence of bio-surfactant at 60 to 70^0 C in an aqueous medium. A broad band centered at 460 nm in absorption spectrum reveals the formation of surfactant stabilized Cu_2O NPs. X-ray diffraction pattern of the surfactant stabilized NPs suggested the formation of only Cu_2O phase in assistance of a bio-surfactant with the crystallite size of ∼8 nm. Red-shift in the vibrational band (Cu–O stretching) of Cu_2O from 637 cm^{-1} to 640 cm^{-1} in presence of bio-surfactant suggests an interfacial interaction between NPs and O-atoms of –OH groups of saponin present in the plant (i.e. *Calotropis gigantea*) extract. From XPS spectra, a decrease in binding energy of both 2p3/2 and 2p1/2 bands in Cu_2O with saponin molecules as compared to bulk Cu atom reveals a charge transfer interaction between NP and saponin surfactant molecules. Transmission electron microscopy images showed crystalline nature of Cu_2O NPs with an fcc lattice.

Udayabhanu et.al. [12] synthesized the copper oxide nanoparticles by a solution combustion method using *Tinospora cordifolia* water extract. The Nps were characterized by XRD, SEM, TEM and UV–visible studies. XRD data indicates the formation of pure monoclinic crystallite structures of CuO Nps. SEM images showed that the particles have sponge like structure and the average crystallite sizes were found to be ~6–8nm. Photocatalytic activity studies of CuO Nps revealed that they act as very good catalyst for the effective degradation of methylene blue (MB) in the presence of UV and Sun light. The Nps found to inhibit the activity of DPPH free radicals effectively. CuO Nps exhibit significant bactericidal activity against Klebsiella aerogenes, Pseudomonas aeruginosa, Escherichia coli and Staphylococcus aureus.

Mahmoud Nasrollahzadeh et.al. [13] synthesized CuO nanoparticles (NPs) using aqueous extract of *Gundelia tournefortii* as a mild, renewable and non-toxic reducing agent. CuO NPs were characterized by SEM, TEM, XRD, EDS, FT-IR and UV–vis spectroscopy. The green synthesized CuO NPs presented excellent catalytic activity for reduction of 4-nitrophenol and synthesis of N-monosubstituted ureas via hydration of cyanamides with the aid of acetaldoxime as an effective water surrogate in ethanol as a green solvent.

Zhihong Zhang et. al. [14] reported a novel nanocomposite of cuprous oxide nanospheres, three-dimensional reduced graphene oxide, and nano-chitosan (Cu_2O@3D-rGO@NCS) with diverse functionalities by a feasible one-step in situ reduction synthesis. The photocatalytic ability of nanocomposite using the degradation of rhodamine B under simulated sunlight irradiation was accessed. The superior photocatalytic ability of Cu_2O@3D-rGO@NCS compared to the pristine Cu_2O nanospheres and Cu_2O@3D-rGO nanocomposite, was attributed to high porosity from 3D-rGO, an efficient charge transfer from Cu_2O to rGO, and high adsorption ability of NCS.

Md. Ataur Rahman and Tajmeri S. A. [15] prepared copper oxide composite nanoparticles from copper (II) acetate. According to the findings of SEM the particle size was within the range from 450 nm to 550 nm. Band gap energy was determined by reflectance measurement and value was found to be 4.21 eV. From the result of EDS, it appears that the composite contains only Cu and O atoms. Result of XRD confirmed that the composite contains CuO, Cu_2O and metallic Cu. Adsorptive and catalytic properties of composite were studied using Methylene blue as adsorbent. Photo-oxidative degradation of MB was investigated in the suspension of composite under visible light.

Prakash et al., 2018 [16] have synthesized CuO nanoparticles using *Cordia sebestena* aqueous flower extract. The CuO nanoparticle size is in the range of 20 to 35 nm. The flower extract contains the phytochemical constituents like flavonoid, tannin, phytosterol, coumarin and fumaric used as stabilizing agent in CuO NPs synthesis. So, the synthesized CuO NPs showed improved antibacterial activity on both gram positive and gram-negative bacterial species and photocatalytic dye degradation of Bromothymol blue.

The CuO nanoparticles of 22 to 40 nm size was synthesized by Sharmila et al., [17] using *Bauhinia tomentosa* aqueous leaf extract. Presence of phytochemicals like phenolics, tannins and proteins were confirmed by FTIR analysis, which involved in the synthesis of CuO NPs. The antibacterial activity of the CuO NPs were tested against gram positive and gram-negative bacteria, from the results it has been observed that the antibacterial activity of gram-negative bacteria was higher than the gram-positive bacteria. It acts as good antibacterial agent and used in biomedical applications.

Gebremedhn et al., 2019 [18] were synthesized CuO nanoparticles using aqueous leaf extract of *Catha edulis*. Secondary metabolites like phenols, flavonoids, carbohydrate and alkaloids present in the leaves extract were involved in the nanoparticle synthesis. CuO NPs showed significant antibacterial activity against Salmonella typhimurium and Escherichia coli. Copper oxide nanoparticles of 80 nm size was derived from *Juglans regia* (walnut) aqueous leaf extract by Asemani et al., 2019 [19]. The bioactive compounds like malic acid, 3-O-caffeoylquinic acids and quercetin O-pentoside were present in the leaves extract, which act as effective stabilizing and reducing agents in the synthesis of CuO NPs. The effective antibacterial and antioxidant activity were found in CuO NPs.

Sarkar et al., 2020 [20] have prepared CuO nanoparticles using whole plant extract of *Adiantum lunulatum*. The plant extract contains chemical constituents like terpenoids, carbohydrates and phenols. They have involved in the CuO NPs synthesis. The diameter of synthesized CuO NPs is in the range of 6.5±1.5 nm. They were employed in the seed germination of Vigna radiata with various concentrations, optimum concentration of CuO NPs improved the immunity of plants. M. Muthukumaran et.al. [21] emphasized on the green synthesis of cuprous oxide (Cu_2O) nanoparticles (NPs) by Fehling's route using banana (*Musa acuminate*) fruits where the formation of products by the involvement of natural ingredients is considered to be the solvent-free route. On testing of the photocatalytic activity, the material is investigated to be p-type Cu_2O and exhibited highly efficient photocatalytic behavior due to the interfacial structure that tends to reduce the recombination rate of a photogenerated electron (e^-) - hole (h^+) pairs. Further, they have explored this property for the degradation of organic contaminants, methylene blue and found that the material is very effective towards the photochemical degradation of pollutants.

Dhara et.al. [22] focused on the synthesis of simple, cost-effective and environmentally friendly biofunctionalized cuprous oxide nanoparticles using root extract of Withania somnifera. The average size of the nanoparticles was found to be 410 nm with an energy band gap of 4.63 eV as confirmed by scanning electron microscopy and UV–visible spectroscopy, respectively. FT-IR gave a distinctive absorption band at 626 cm^{-1}. The bactericidal effects of synthesized nanoparticles are attributed to different bioactive compounds, present in plant extract.

Masresha Tefera et.al. [23] have reported an environmentally friendly, and cost-effective synthesis of visible light active cuprous oxide (Cu_2O) using *prunus cerasifera* plant extract. The phytochemicals from *Prunus cerasifera* extract are used as surface modifying and reducing agents for the synthesis of Cu_2O nanoparticles (NPs). The powder XRD, FT-IR spectroscopy, SEM and EDX instruments were used to confirm the crystal structure, morphology and composition of Cu_2O NPs. Photocatalytic activity of Cu_2O NPs was investigated via the reduction of Cr (VI) under visible light irradiation.

Atul Verma et.al [24] synthesized a Cu-cuprous/cupric oxide-based family of nanoparticle catalysts via simple hydrothermal technique, and investigated the effect of copper oxide phases on the application of interest. These catalysts are applied towards dual application namely 4-nitrophenol (4-NP) conversion and electrochemical hydrogen evolution reaction (HER).

S. Prabhua et.al. [25] synthesized CuO NPs by reducing copper sulphate using an aqueous flower extract of *Clitoria ternatea*. The UV-visible peak observed at 251 nm confirmed the formation of CuO NPs. The optical bandgap energy value of CuO NPs was found to be 2.16 eV. The presence of Cu-O band at 490 cm^{-1} in the FT-IR spectrum confirms the formation of the CuO NPs. The XRD exhibits monoclinic structure with an average crystallite size of 17.46 nm. The CuO NPs exhibited significant antibacterial activity and photocatalytic degradation of Direct Red (DR) and Crystal Violet (CV) dyes under sunlight.

Badawy et. al. [26] synthesized Copper/Copper oxide (Cu/Cu_2O) nanoparticles by modified chemical reduction method in an aqueous medium using hydrazine as reducing agent and copper sulfate pentahydrate as precursor. The Cu/Cu_2O nanoparticles were characterized by XRD, EDX, SEM and TEM. The analysis revealed the pattern of face-centered cubic (FCC) crystal structure of copper Cu metal and cubic cuprites structure for Cu_2O. The SEM result showed monodispersed and agglomerated particles with two-micron sizes of about 180 nm and 800 nm, respectively. The catalytic activity of Cu/Cu_2O nanoparticles for the decomposition of hydrogen peroxide was investigated and compared with manganese oxide MnO_2. The results showed that the second-order equation provides the best correlation for the catalytic decomposition of H_2O_2 on Cu/Cu_2O.

Table 1: Plant extract mediated synthesis of copper oxide nanoparticles from copper salts.

S.No	Source and plant part	Type of NPs	Shape & Size	Characterizations	Applications	Reference
1	*Syzygium jambos. L* Leaves	Cu_2O	Octahedral / 120 nm	UV-Vis, FTIR, DLS, XRD, SEM, TEM	*Ipso-H*ydroxylation of aryl boronic acids	[27]
2	*Pomegranate* Fruit	Cu_2O	Irregular / 5-20nm	UV-Vis, FTIR, XRD, SEM, TEM	Optical properties	[28]
3	*Lignin* (papermaking waste liquid)	Cu_2O	Irregular / 100-200nm	UV-Vis, FTIR, XPS, XRD, TEM	Antibacterial	[29]
4	*Banana* Peel	Cu_2O	Spherical / 7.33 nm	UV-Vis, FTIR, DLS, XRD, SEM, TEM	H*ydroxylation of aryl boronic acids*	[30]
5	*Bifurcaria bifurcate (Alge)*	Cu_2O, CuO	Spherical / 5-45nm	UV-Vis, FTIR, XRD, HRTEM,	Antibacterial	[31]
6	*Kappaphycus alvarezii*	Cu/ Cu_2O	Spherical / 53nm	UV-Vis, FTIR, DLS, XRD, AFM, TEM	-	[32]
7	*Punica granatum L* peel	$Cu/Cu_2O/$ CuO	Spherical / 29nm	UV-Vis, FTIR, XRD, HRSEM, SAED, TEM	Electrochemical transducer & catalysis	[33]
8	*Pomegranate* marc peels	Cu/Cu_2O	Spherical / 25-30nm	UV-Vis, XRD, SEM, EDX, TEM	photocatalytic degradation of dye	[34]

III. CONCLUSION

The main advantage of green synthesis is the absence of the need for additional agents like reducing, capping, and stabilizing agents in the synthesis and stabilization of nanoparticles. Green synthesis is superior to all other methods since the physical approach takes longer to achieve thermal stability and the chemical approach uses toxic reagents. Green synthesis using a biological approach is significantly easier to carry out since it involves plants and plant parts that are more easily obtained. The phytochemicals also have an impact on the nanoparticles' antibacterial activities. The properties of the nanoparticles generated utilizing diverse plant extracts differed depending on the type of phytochemicals obtained. The size and shape of the particles can be seen using tools such as a scanning electron microscope (SEM) or a transmission electron microscope (TEM). UV–visible spectroscopy was used to analyze the formation of nanoparticles. Numerous studies have noted the biological and catalytic properties of CuO NPs. CuO nanoparticles have special physicochemical characteristics that make them effective nanomaterials for usage in nearly all scientific domains. Essential information about the synthesis and use of CuO NPs in nanomedicine is provided in this review.

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