

RESEARCH ARTICLE

Green Synthesis of Copper Oxide Nanoparticles using Aloe Vera Extract

D. Hemalatha¹, S. Saraswathi¹**ABSTRACT**

In material science, green method for synthesis of nanomaterials is feasible, cheaper and eco-friendly protocol. To accomplish this phenomenon, present study was aimed to synthesize Copper oxide nanoparticles using leaf extract of Aloe vera with two different precursors $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Cupric chloride) and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Cupric sulfate). The extraction of Aloe vera is employed as reducing and stabilizing agent for this synthesis. Copper oxide Nanoparticles is effective use of biomedical application due to their antibacterial function. The synthesized Copper oxide nanoparticles were characterized by X-ray Diffraction Spectroscopy (XRD), Energy Dispersive Spectroscopy (EDX), Fourier-Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscope (SEM). XRD studies reveal the crystallographic nature of Copper oxide nanoparticles. Furthermore the Copper oxide nanoparticles have good Antibacterial activity against both gram negative (*E. Coli*, *Klebsiella pneumoniae*) and gram positive (*Bacillus cereus*, *Staphylococcus aureus*) bacteria.

Keywords: Green synthesis, Aloe vera, SEM, XRD, FTIR, Antibacterial activity

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1. Introduction

The field of nanotechnology is one of the most active areas of research in modern materials science. Metal nanoparticles found many applications in different fields because of their unique optical, electronic, mechanical, magnetic, and chemical properties.^[1] The various processes available for the synthesis of nanoparticles, biosynthetic process plays a very important role in nanotechnology as it is cost-effective, is eco-friendly and is a better alternative to chemical and physical methods.^[2] As metal nanoparticles are widely used in the areas of human contact, the necessity to develop eco-friendly methods for nanoparticles synthesis that do not use toxic chemicals has been constantly growing. Green synthesis of copper nanoparticles is of great interest because of many advantages: copper is highly conductive and also cheaper than silver and gold.^[1] Copper oxide (CuO) nanoparticles are important due to their applications as antimicrobials and in gas sensors,^[2-9] batteries, high temperature superconductors, solar energy conversion tools and so on. Nowadays, CuO nanoparticles are utilized as heterogeneous catalysts, antioxidants, drug delivery agents, and imaging agents in field of biomedicine.^[2] Biosynthesis of copper oxide

nanoparticles CuO NPs using microorganisms such as bacteria, fungi, yeast have been reported in the literature.^[4] In this paper we synthesized, CuO Nanoparticles using Aloe vera extract. Aloe vera (*Aloe barbadensis* Mill.) is a well-known medicinal herb, which comprises nearly 75 potentially active ingredients having immune modulatory, antiparasitic, and wound healing and antimicrobial potential.^[5] The synthesized CuO Nanoparticles were characterized by XRD, FTIR, and SEM with EDX.

2. Experimental Procedure

2.1 Aloe Vera Extraction

Aloe vera leaves were collected from a nearby garden and cleanly washed with tap water. Then the leaves were cut into small pieces and dried in sunlight for two days. After two days, the dried leaves were grained into a fine powder. 12g of dried Aloe vera powdered was taken and mixed with 200 ml of Deionised water. Then this solution is kept in oven at 100°C when it turns into mild yellow color this aqueous solution is stirred continuously for 20 mins at 100°C . Finally the solution was filtered using whatmann filter paper.

2.2 Synthesis of copper oxide nanoparticles

In this present study, two copper precursors were applied: ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$), ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$). Aloe vera extract was added to the aqueous solution of copper precursors. This combined solution was stirred at 100°C for 30 mins and then kept at 100°C for 45mins in oven. The color of the solution is changed as green. Afterwards the aqueous solution of NaOH was added drop by drop to change the color of the prepared solution into brown. Finally after centrifuging for 10 mins the precipitation is taken and then placed in the oven to dry it completely. The dried sample was grained with mortar to obtain fine CuO Nanoparticles.

Table 1: Condition to form CuO Nanoparticles

Sample	Aloe vera Extraction (ml)	Precursors Concentration (ml) (salt+ DI water)	NaOH (ml)
A	15	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (8.5g+50ml)	50
B	15	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (12.48g + 50ml)	50

3. RESULTS AND DISCUSSION

3.1 X-ray diffraction analysis

The crystallography analysis was carried out by X-ray diffraction method. X-ray diffraction pattern of the prepared CuO nanoparticles are shown in Fig. 1. The major peaks located for sample-A at $2\theta=38.71^\circ$, 35.49° , 67.98° and for sample-B at $2\theta=38.81^\circ$, 35.59° , 48.92° . The straight line and sharp peaks shows that the synthesized powder containing crystalline in nature. The average crystalline size is determined using Scherrer formula, $D = K\lambda/\beta \cos\theta$.

From the above details we concluded that the crystalline size of copper oxide Nanoparticles for Sample-A was

calculated by scherer's formula as 11.7nm. Similarly for Sample-B was 11.3nm. The average crystallite size of CuO nanoparticles was estimated in the range of 9-23nm with reference.^[10] Depending on the Copper precursor and NaOH concentration, the intensity for Sample A increased with high crystalline size rather than Sample B.

3.2 Fourier transform- infrared spectral analysis [FTIR] studies

The functional group analysis has been performed by Fourier transform infrared spectrum. The FTIR spectrum of copper oxide nanoparticles for sample-A and sample-B were synthesized by green method. The wavelength regions were recorded in the range of $400\text{--}4000\text{cm}^{-1}$.

FTIR analysis of green synthesized CuO nanoparticles for sample A and sample B compared, since A and B have similar peaks at 3450.65 , 1325.10 , 1637.57 cm^{-1} show the presence of $-\text{OH}$, C-N, C-C stretching of hydroxyl, sulfone and alkenes^[2] respectively. For the synthesis of CuO-NPs, in the sample – A cupric chloride is used as precursor and it attains the CuO stretching at 529cm^{-1} . In the sample – B cupric sulfate as precursor and it confirms the CuO vibration at 1147 cm^{-1} .^[11] So, cupric chloride precursor is more suitable for synthesizing CuO nanoparticles rather than cupric sulfate. Some of the wave number and corresponding band assignments for both samples are tabulated below.

3.3 Scanning Electron Microscope (SEM) Analysis

Scanning Electron Microscopy was employed to analyze the morphology and the growth features of the prepared nanoparticles. The SEM images of prepared CuO Nanoparticles were shown in Fig. 3 (a to d) SEM micrograph indicates the agglomeration of spherical particles of almost similar size. Based on the analysis of SEM micrograph, it was found that the morphology of CuO nanoparticles is more clearer in Sample-B than Sample-A.

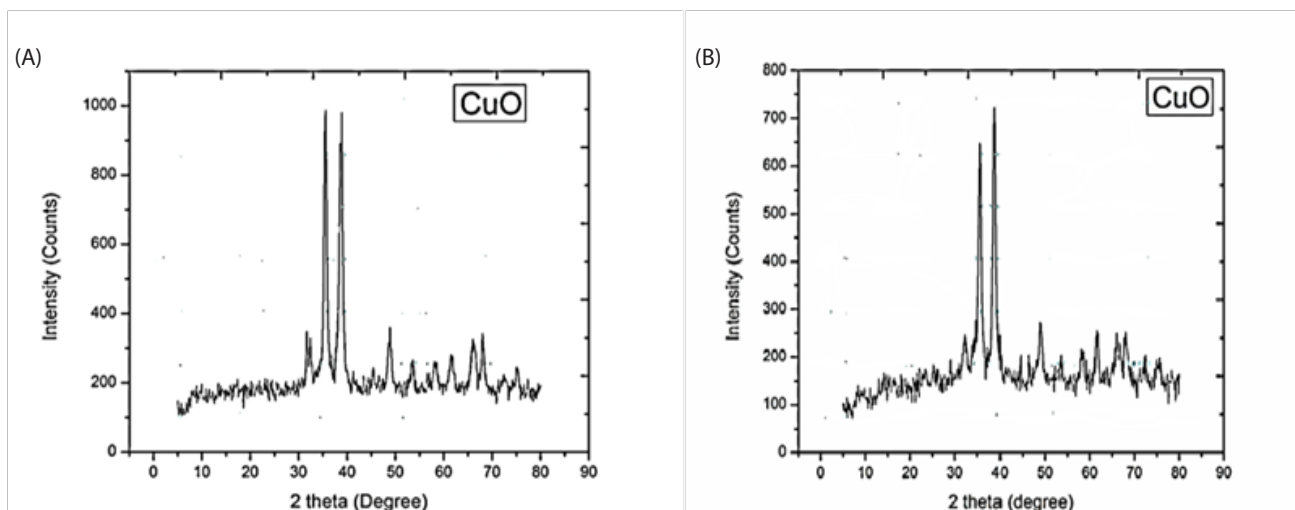
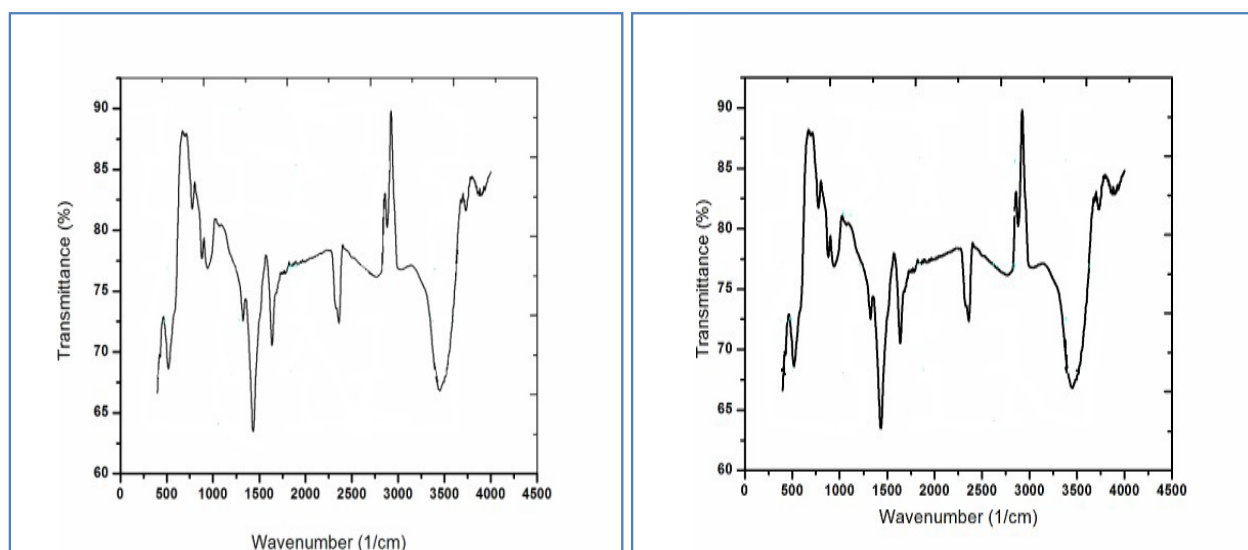


Fig. 1: XRD pattern of CuO-NPs for sample A & B

Table 2: Crystallographic parameters of CuO-NPs for sample – A & B

Sample	2 θ	d(Å)	FWHM (deg)	Crystalline size, D (nm)	Micro strain ϵ	Dislocation density $6 \times 10^{15} \text{m}$
A	38.7148	2.32396	0.74930	11.7460	0.30822	7.2479
	35.4944	2.52708	0.69400	12.5598	0.28824	6.3390
	67.9848	1.37779	0.63040	10.9941	0.22788	0.9905
B	38.8126	2.31833	0.75860	11.6027	0.31202	7.4280
	35.5948	2.51998	0.74480	11.7060	0.30926	7.2968
	48.9291	1.86004	0.85830	10.6264	0.34069	8.8557

**Fig. 2:** FTIR spectra of CuO- NPs for sample- A and B**Table 3:** FTIR data of CuO-NPs for sample A&B

Sample	Wavenumber (cm ⁻¹)	Band Assignments
A	2763.90	C-H stretching of aldehyde
	879.54	C=C bending of alkene
B	2752.42	C-H stretching of aldehyde
	1435.04	O-H bending of carboxylic acid

3.4 Energy Dispersive X-Ray Analysis (EDX) EDX Analysis of sample A

The elemental composition analysis of copper oxide nanoparticles reveals the presence of Cu (copper), O (oxygen), Cl (chlorine). The EDX confirmed the presence of CuO in the sample. From the below Fig.4.1 shows the EDX spectrum confirmed formation of CuO-NPs with extract of Aloe vera.

The element weight of Cu, O and Cl for sample-A was 68.89%, 28.41% and 2.70%.

3.5 EDX Analysis of sample B

The elemental composition analysis of copper oxide nanoparticles reveals the presence of Cu (copper), O (oxygen), S (sulphur), Si (silicon). The EDX confirmed the presence of CuO in the sample. From the below Fig.4.1 shows the EDX

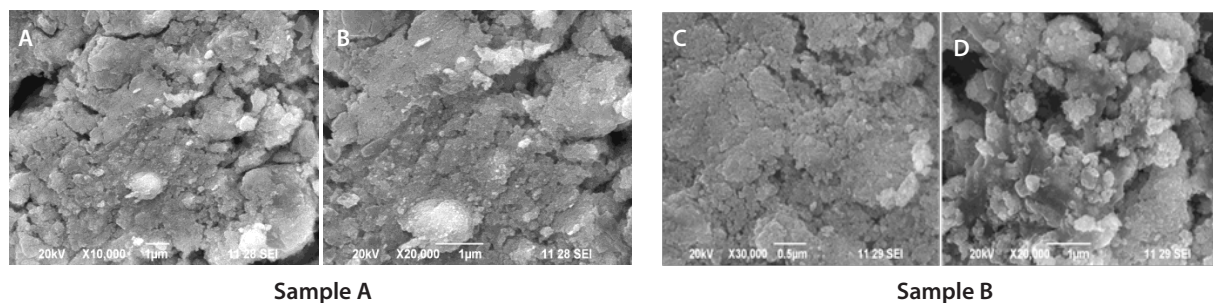


Fig. 3: SEM image of CuO- NPs for sample- A and B

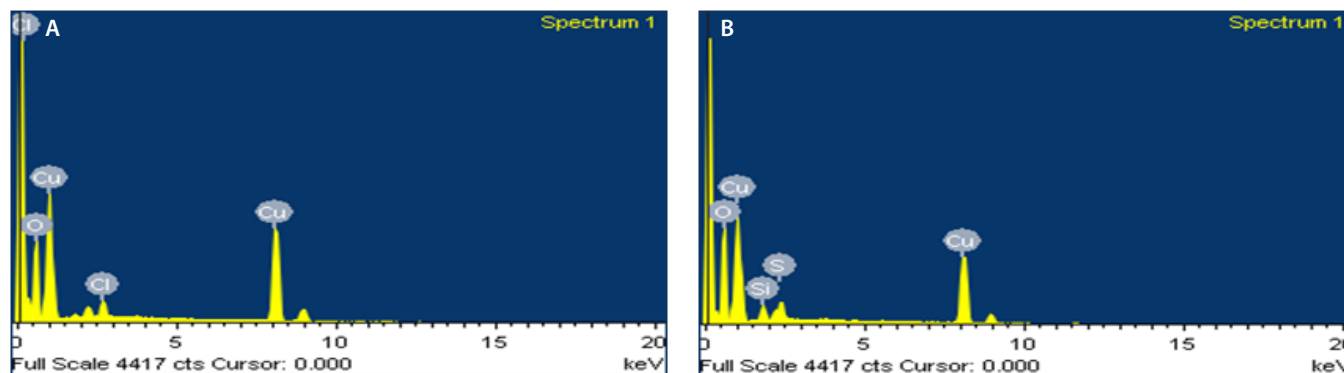
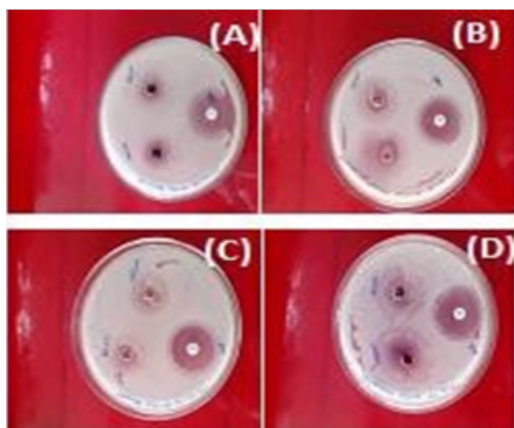
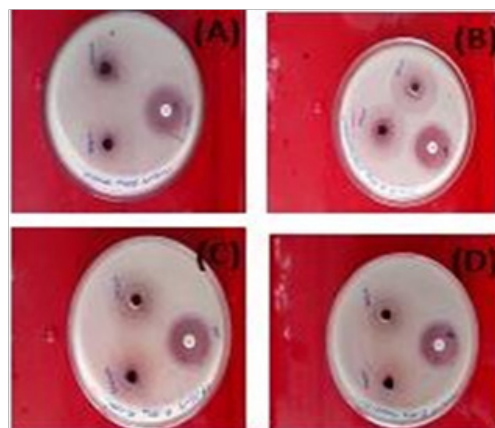


Fig. 4.1A and B: EDX spectra of CuO-NPs

Fig. 5.1A to D: Photographs of antimicrobial results CuO-NPs for sample-A a) *Bacillus cereus* b) *Staphylococcus aureus* c) *Escherichia coli* d) *Klebsiella pneumoniae*Fig. 5.2A to D: Photographs of antimicrobial results CuO-NPs for sample-A a) *Bacillus cereus* (b) *Staphylococcus aureus* c) *Escherichia coli* d) *Klebsiella pneumoniae*

spectrum confirmed formation of CuO-NPs with extract of Aloe vera. The element weight of Cu, O, S and Si for sample-B was 59.23%, 35.81%, 2.11%, 2.84%.

3.6 Antibacterial activity

The copper oxide nanoparticles synthesized using aloe vera extract tested for antimicrobial activity by well diffusion method. Liquid Mueller Hinton agar media and the Petri plates were sterilized by autoclaving at 121° C for about 30 minutes at 15 lbs pressure. Under aseptic conditions in the laminar airflow chamber, about 20 ml of the agar medium was dispensed into each Petri plate to yield a uniform depth

of 4mm. After solidification of the media, 18 hrs culture of Gram positive microorganisms such as *Bacillus cereus* (MTCC 430), *Staphylococcus aureus* (MTCC 3160), Gram negative microorganisms such as *E.coli* (MTCC 1698) and *Klebsiella pneumoniae* (MTCC10309) obtained from IMTECH, Chandigarh were swabbed on the surface of the agar plates. Well was prepared by using cork borer followed with loading of 100 µl of each sample to the distinct well with sterile distilled water as negative control and vancomycin (30mcg/disc) as positive control. The sample loaded plates were then incubated at 37° C for 24 hours to observe the zone of inhibition.

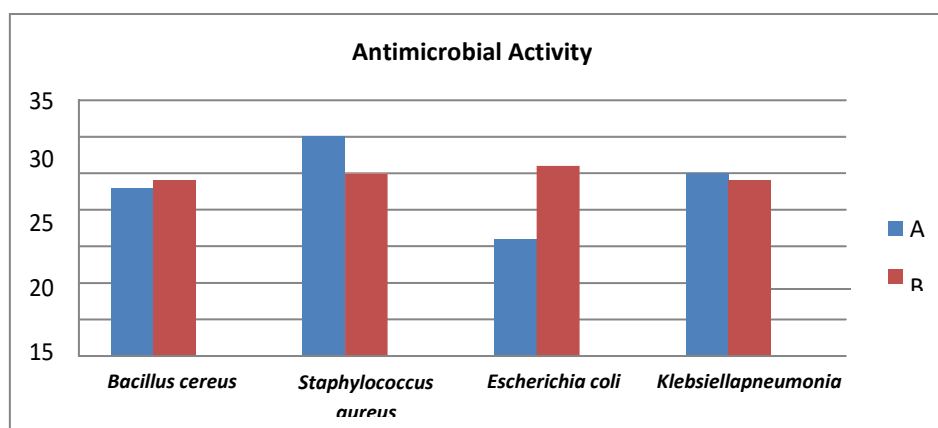


Fig. 6: Graphical representation of Antimicrobial activity of CuO-NP

Table 4: Antimicrobial results of CuO-NPs

S. No	Microorganisms	Zone of Inhibition in Diameter					Std. Antibiotic (vancomycin) 30mcg
		Control (100 µl)	A		B		
			50 µl	100 µl	50 µl	100 µl	
1	Bacillus cereus	Nil	14	23	20	24	30
2	Staphylococcus aureus	Nil	25	30	22	25	22
3	Escherichia coli	Nil	13	16	18	26	25
4	Klebsiellapneumonia	Nil	22	25	21	24	31

The anti-bacterial activities of CuO-NPs were assessed against the growth of two gram negative (*E.Coli*, *Klebsiellapneumonia*) and two gram positive (*Bacillus cereus*, *Staphylococcus aureus*) bacteria. The results of well diffusion test were shown in Table 4. The effective bacteria inhibitory behaviour of the Sample-A and Sample-B were shown in figure. Based on these results Sample-A has highest inhibitory effects on *Staphylococcus aureus*. Least inhibitory effects on *E.Coli*.

4. CONCLUSION

In this study illustrates simple, convenient and eco-friendly method of the synthesis of Copper Oxide Nanoparticles by using the extract of *Aloevera*. According to the results of XRD spectrum, it reveals that the synthesized CuO-NPs of sample – A has average crystalline size of 11.7nm and for Sample –B was 11.3nm. The EDX exhibits elemental composition of CuO- NPs. The spectrum reveals the presence of CuO nanomaterials. FTIR analysis of this study confirms the formation of CuO-NPs. By using the Scanning Electron Microscope technique, it indicates the Agglomeration of spherical particles of similar size for both samples. This Antibacterial activity study confirms the Copper Oxide Nanoparticles synthesized using *Aloevera* has stronger inhibition against both Gram negative and Gram positive bacteria. Furthermore, Sample – A has higher inhibitory effects on *staphylococcus aureus* and least inhibitory effects on *E.Coli*.

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