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# Green Synthesis and Characterization of Metal Oxide Nanoparticles Using *Nelumbo Nucifera* Flower and Its Antimicrobial Activity

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**ABSTRACT :**The green synthesis of well-stabilized metal oxide nanoparticles is a promising development because of the lack of toxic chemicals usage and excessive energy consumption. In this study a simple approach was applied for synthesizing metal oxide nanoparticles (CuO, CoO&FeO) using aqueous extract of *Nelumbo nucifera* flower. The initial formation of nanoparticles was confirmed using UV-Visible spectrophotometer which showed characteristic peaks around 260 – 320 nm. The various phytochemicals responsible for reduction and capping of nanoparticles were identified using FTIR spectroscopy. The SEM image shows the morphology of CuO and FeO as spherical shape whereas CoO NPs had rock like structure. The size of nanoparticle calculated using XRD was 21nm for CuO, 32nm for CoO and 23 nm for FeO NPs respectively. The sample purity was analysed using EDX spectra. Further these biosynthesized metal oxide nanoparticles were found effective against 8 different microorganisms. This is for the first time aqueous *Nelumbo nucifera* flower extract has been used for the synthesis of metal oxide nanoparticles.

**KEY WORDS:** NELUMBO NUCIFERA, BIOSYNTHESIS, METAL OXIDE NANOPARTICLES

## I. INTRODUCTION

Metal oxide nanoparticles are of great technological relevance because of their applications in photonics, fuel cells, sensors, catalysis and drug delivery system. Oxide nanoparticles can exhibit unique physical or chemical properties due to their limited size and high density of corner or edges surfaces sites [1]. Among all the functional materials designed at the nanoscale, metal oxides especially copper oxide (CuO), cobalt oxide (CoO) and iron oxide (FeO) are particularly attractive from scientific as well as a technological point of view. In the present scenario, the development of biocompatible nanomaterials with non-toxic nature through eco-friendly approaches is one of the needs of the hour. In this context, green synthetic methods can be looked upon which the most is commonly preferred eco-friendly approaches. Green synthesis of metal oxide nanoparticles using plant extract is a one-pot or single-step eco-friendly bio reduction method that requires relatively low energy to initiate the reaction [2].

Green synthesis of CuO, CoO and FeO nanoparticles by various plant extracts like *Syzygiumalternifolium*, *Ixorococcinea*, *CatharanthusRoseus*, *Sageretiathea*, *Calotropisprocera*, *Helianthus annus*, *Camellia sinensis*, *Avicennia marina*, *Skimmialaureola Brassica oleracea var. italic* has been reported in the literature so far [3-12]. *Nelumbo nucifera* (lotus) is a potential aquatic crop grown and consumed throughout Asia. These flowers are traditionally used to treat diarrhoea, cholera, fever, hepatopathy, hyperdipsia and many bleeding disorders [13]. Extracts of flower was shown to exhibit potent antioxidant, antipyretic and antiviral activity. In this study, we report for the first time synthesis of 3 metal oxide nanoparticles using the aqueous extract of *Nelumbo nucifera* flower and its antimicrobial activity against 8 different pathogens including 4 bacteria and four fungi were also investigated.



## II. MATERIALS AND METHODS

### 2.1 Preparation of plant extract

Fresh and healthy flowers of *Nelumbo nucifera* was collected from Madurai district, Tamil Nadu. The flowers are washed thoroughly using tap water and then with distilled water to remove any unwanted dust particles. About 10g is weighed and transferred into 250ml beakers containing 100ml distilled water and boiled for about 20 min. The extract was filtered with normal filter paper and then using whatmann No1 filter paper to remove particulate matter and to get clear solution. It is refrigerated (4°C) in 250ml Erlenmeyer flasks for further experiments [14].

### 2.2 Phytochemical screening

Preliminary phytochemical screening of the fresh aqueous plant extract of *Nelumbo nucifera* was done using standard procedure to identify the presence of possible phytochemicals responsible for reduction and capping of metal oxide nanoparticles.

### 2.3 Preparation of metal oxide nanoparticles

About 80 ml of 1mM solution of metal precursor is taken in 250 ml beaker and it was heated around 60 – 70°C and stirred for 30 minutes. To the above solution 10ml of plant extract (1:8 ratio) was added and stirred for about 1hr. Nanoparticles formed was centrifuged at 10,000 rpm and dried in muffle furnace.

### 2.4 Characterisation of nanoparticles

UV-Visible spectrometer (Beckman-Model No.DU-50, Fullerton), between the wave lengths of 180 -700nm having a resolution of 1nm was used to confirm the formation of nanoparticles. FTIR spectroscopy was recorded with a Perkin Elmer Spectrum Express version 10,300. The Crystal structure of the produced NPs was determined and confirmed by using X-ray diffraction meter (Model PW 1710 control unit Philips Anode Materials Cu, 40 kv, 30 MA, optics Automatic divergence slit). The Quanta FEG-250 Scanning Electron Microscope was used for high-resolution imaging of surface morphology. The elements present in metal oxide NPs were characterized using TESCAN S9000.

### 2.5 Disc diffusion method for antimicrobial activity

The antimicrobial activity of synthesized metal oxide NPs was done using standard disc diffusion method. The inoculums for the disc diffusion method were prepared using a suitable broth (agar agar) and the medium was dried at 30 – 35°C prior to the application. The standardized inoculums are transferred in to the sterilized petri dish prepared earlier. After removing excess of inoculums the petri dish was dried at room temperature for 24 hours with lids closed. The petri dish was divided into parts in which the samples were placed with the help of sterile forceps. DMSO solvent is used as control. Then petri dishes were placed at room temperature for one hour for diffusion and incubated at 37°C for 24 hours. Finally, the zone of inhibition produced by different samples was measured using a standard scale [15].

## III. RESULTS AND DISCUSSION

### 3.1 Phytochemical screening

The result of phytochemical screening was shown in table 3.1. It can be seen that the aqueous extract is rich in flavonoids and glycosides and hence these compounds are responsible for reduction and capping of metal oxide nanoparticles.



Table 3.1 Phytochemical screening of *Nelumbo nucifera* flower

Phytochemical	Test used	Aqueous extract
Tannins	Ferric chloride test	-
Saponins	Frothing test	-
Terpenoids	Salkowski test	-
Glycosides	Keller-killani test	+
Carbohydrates	Fehling’s test	+
Flavonoids	Magnesium and HCl	+
Alkaloids	Mayer’s test	-
Steroids	Acetic anhydride and Con.H <sub>2</sub> SO <sub>4</sub>	-

3.2 UV-Visible spectroscopy

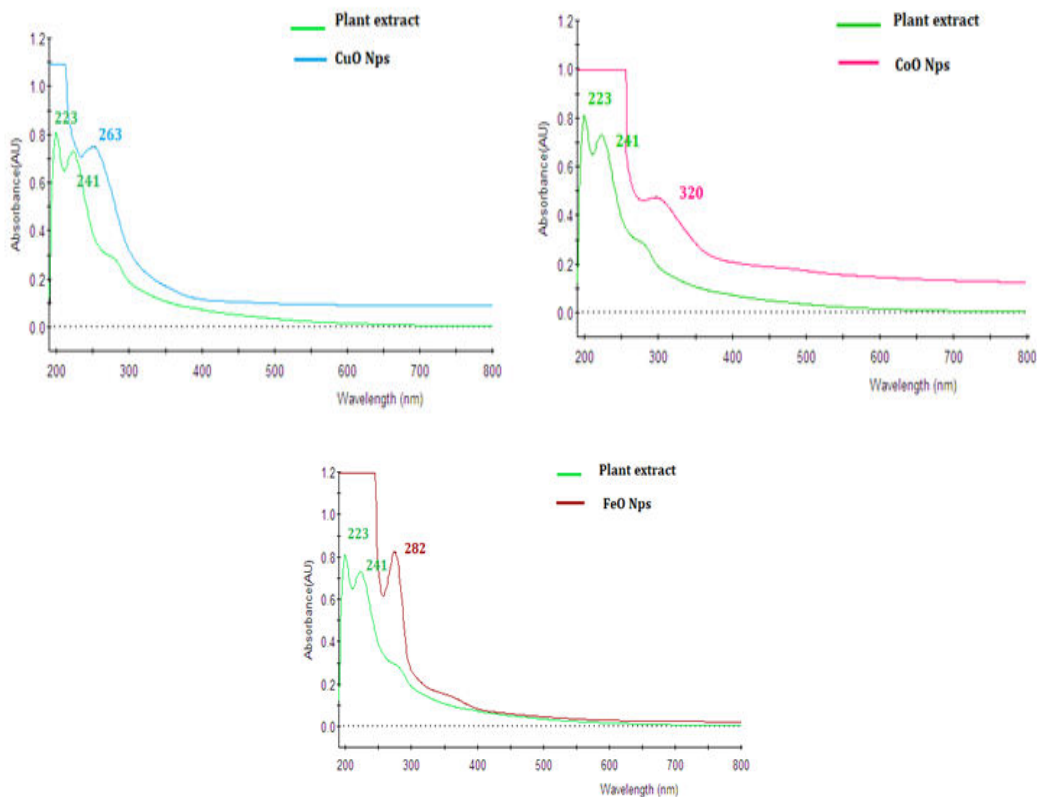


Figure.3.1 UV – Visible spectra of metal oxide NPs and plant extract

The plant extract shows two absorption peaks at 223.05 nm and 241.23 nm. The peak observed at 263.52nm 320 nm and 282nm is attributed to the formation of cuprous oxide, cobalt oxide and iron oxide nanoparticle respectively [16-18]. This is due to the surface plasmon effect of free electrons which exist in the conduction band of metals that oscillate around the particle surface when exposed to electromagnetic radiation.

### 3.3 FTIR spectroscopy

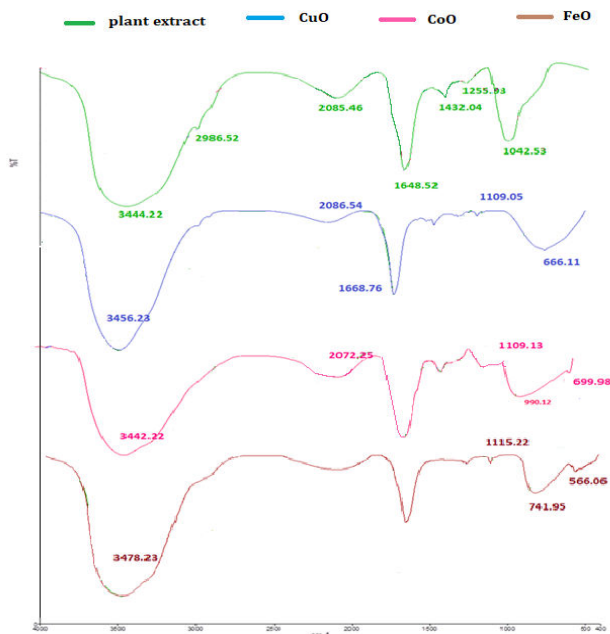


Figure.3.2 FTIR Spectra of plant extract and metal oxide nanoparticles

To identify the biomolecules responsible for reduction and capping of copper oxide nanoparticles FT-IR spectroscopy was recorded. The plant extract shows bands at 3444, 2986, 1648, 1432 and 1042  $\text{cm}^{-1}$  corresponding to the frequencies of O-H stretching or N-H stretching, C-H stretching, C≡C stretching, C-O stretching and C-N stretching of amines respectively. After the formation of metal oxide nanoparticles the peaks are being shifted which confirms that the reduction and stabilization of nanoparticles proceeds via these groups.

### 3.4 XRD Analysis

The XRD pattern of synthesised copper oxide NPs is shown in the figure 3.4A. The values of  $2\theta$  showed peaks at 29.6, 36.4, 41.3, 61.4, 73.5 and 77.2 corresponding to the crystal planes 110, 111, 200, 220, 311 and 222. These peaks are indexed to the cubic phase of  $\text{Cu}_2\text{O}$  as per JCPDS 05 – 0667. The crystal size of the nanoparticle was calculated using the Scherrer formula was found to be 21 nm. The cobalt oxide nanoparticles (figure 3B) shows peaks at 32.5 (220), 37.3 (311), 39.6 (222), 45.1 (400), 56.2 (422), 59.4 (511), 65.2 (440) and 79.8 (622) can be assigned to the cubic symmetry of cobalt oxide NPs according to the JCPDS file 74 – 1657. The size of the cobalt oxide nanoparticle was calculated to be 32nm. The diffraction patterns of iron oxide nanoparticles are shown in the figure 3C. There exists strong diffraction peaks with  $2\theta$  values 30.5, 36.6, 42.9, 53.7, 57.2 and 63.2 corresponding to the crystal planes (220), (311), (222), (400), (422), (511) and (440) respectively. The results show the cubic phase of  $\text{Fe}_3\text{O}_4$  in accordance with JCPDS – 65 – 3107. Using the Scherrer equation the size was calculated to be 23nm.

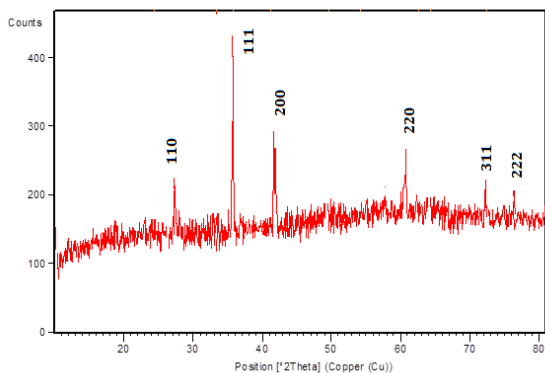


Figure.3.4A XRD of CuO nanoparticles

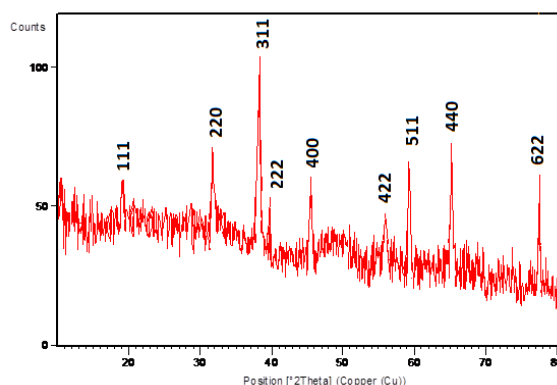


Figure.3.4B XRD of CoO nanoparticles

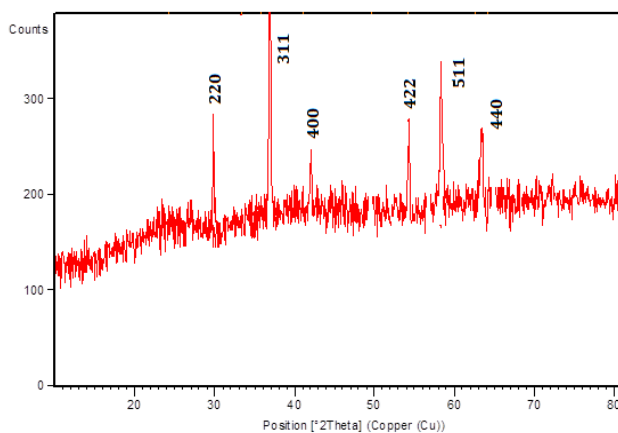


Figure.3.4C XRD of FeO nanoparticles

### 3.5 FESEM – EDX analysis

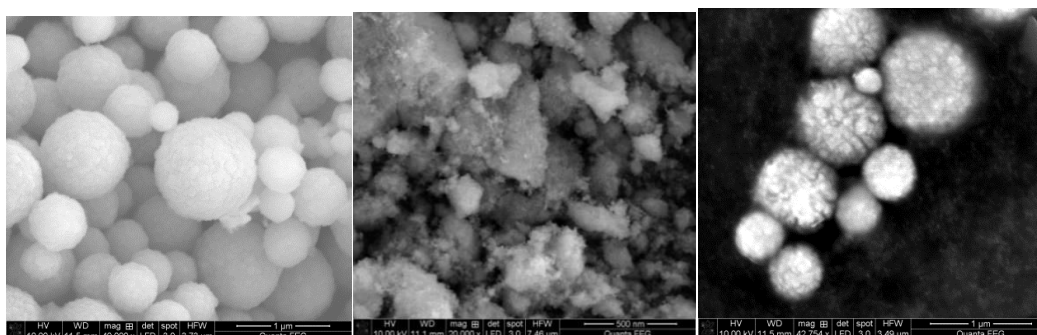


Figure 3.5 FESEM image of nanoparticles a- CuO, b- CoO, c- FeO

FESEM analysis was carried out to determine the size and morphology of metal oxide nanoparticles. The morphology of CuO and FeO was found spherical whereas CoO had rock like structure. Though varying in size the shape of all the nanoparticles are perfectly spherical. This might be due to the reducing ability of phytochemicals present in the

plant extract. However, slight aggregation of particles has arbitrarily arisen in CoO nanoparticles. The aggregation occurs depending on the nature of the metal oxide nanoparticle and reaction condition.

The EDX spectra of synthesized metal oxide nanoparticles was shown in the figure 5. The strong intensity and narrow width of diffraction peaks indicate that the resultant products were highly crystalline in nature. Apart from peaks for metal and oxygen, a carbon peak was observed which could be from plant phytochemicals used during the synthesis. No other impurities are seen indicating that the sample is pure.

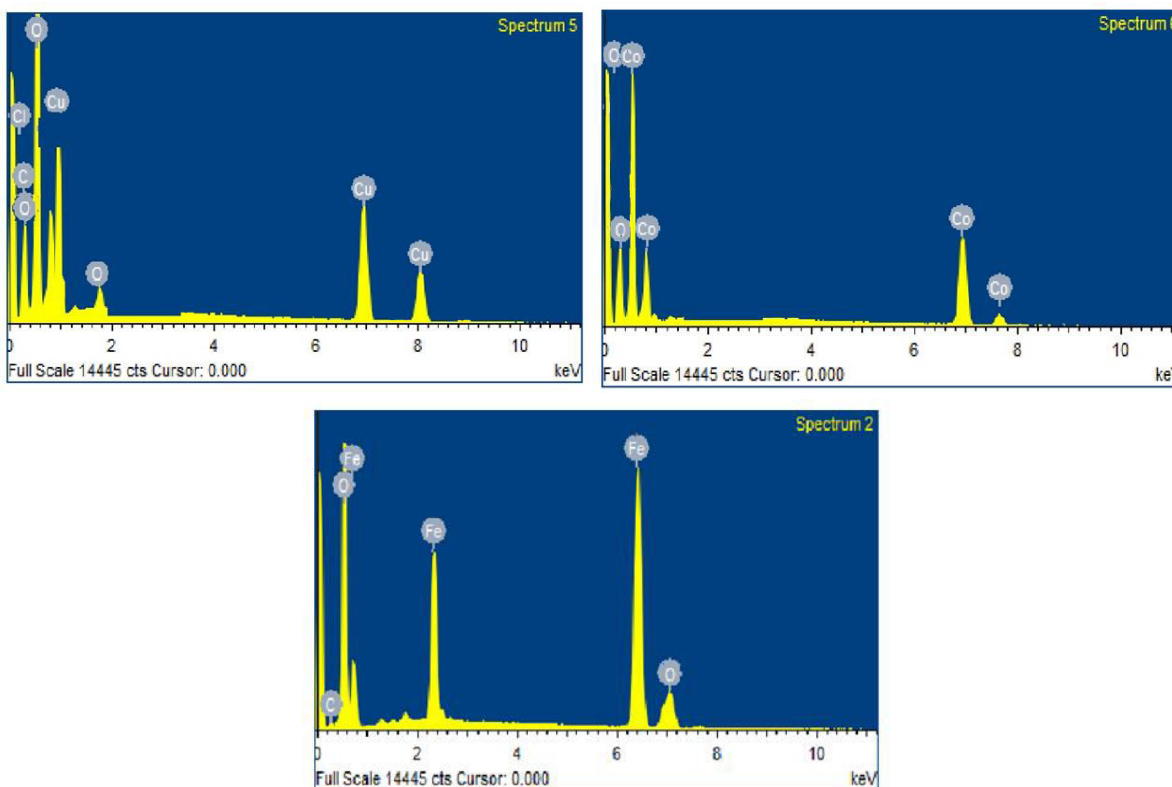


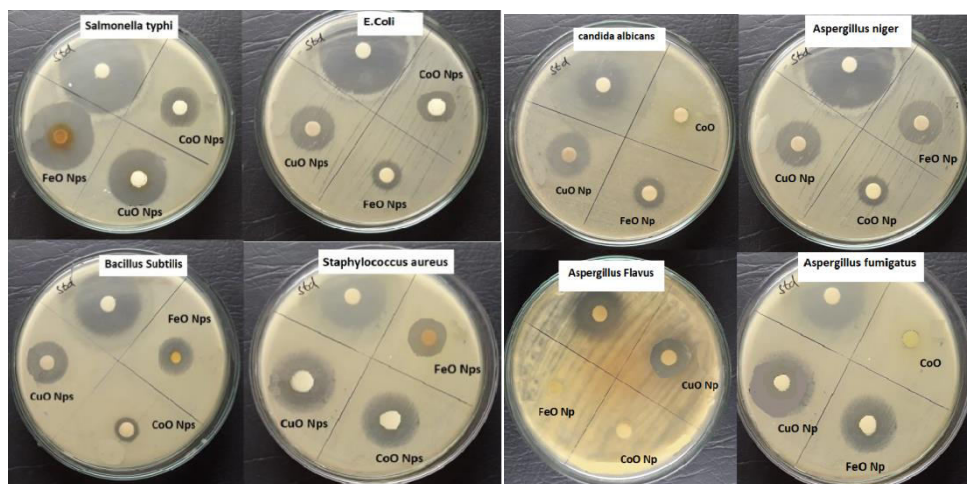
Figure.3.6 EDX spectra of metal oxide nanoparticles

### 3.6 Antimicrobial activity

The metal oxide nanoparticles synthesised using *Nelumbo nucifera* extract was used to evaluate the antibacterial activity against Gram-positive (*Staphylococcus aureus* and *Bacillus subtilis*), Gram-negative (*Escherichia coli* and *salmonella typhi*) bacteria and four different fungi (*Candida albicans*, *Aspergillusniger*, *Aspergillusflavus*, *Aspergillusfumigatus*). Among the three metal oxide, CuO showed highest antimicrobial activity against all the 8 pathogens and CoO showed least activity. From the XRD and SEM studies the metal oxides synthesized showed different size and shapes. It can be concluded that apart from size of the nanoparticle, various other factors like shape, nature of metal and the type of microbial stain are also responsible for the antimicrobial activity. The zone of inhibition was shown in table 3.2.

**Table. 3.2. Zone of inhibition of metal oxide nanoparticles**

Pathogens	Zone of inhibition (mm)			
	Standard	Copper oxide Nano Particles	Cobalt oxide Nano Particles	Iron oxide Nano Particles
<i>Salmonella typhi</i>	21	14	8	17
<i>E.coli</i>	22	9	8	4
<i>Bacillus subtilis</i>	16	10	4	7
<i>Staphylococcus aureus</i>	18	16	13	9
<i>Candida albicans</i>	16	10	-	6
<i>Aspergillusniger</i>	18	8	5	8
<i>Aspergillusflavus</i>	10	7	-	-
<i>Aspergillusfumigatus</i>	13	12	-	7



**Figure.3.7. Antimicrobial activity of metal oxide nanoparticles**

#### IV. CONCLUSION

Three metal oxide nanoparticles namely copper oxide, cobalt oxide and iron oxide was successfully synthesized using *Nelumbo nucifera* flower extract using water as green solvent. The biosynthesized nanoparticles were characterized using different techniques. Various factors including the nature of phytochemicals responsible for reduction and capping of nanoparticles greatly influence the shape, morphology and antimicrobial activity. The fact that plant extract exhibits activity is of interest, but it is only a preliminary idea and should be followed by the identification of the active phytochemicals responsible for reduction and capping of metal oxide NPs.





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