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Green Synthesis of Co_3O_4 Nanoparticles using *Mappia foetida* leaf extract and its Antimicrobial Potential

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ABSTRACT

In this paper the novel green synthesis of cobalt oxide nanoparticles (Co_3O_4NPs) from cobalt chloride ($CoCl_2$) using *Mappia foetida* leaf extract was investigated. The characterization of the Co_3O_4NPs was done by using UV-Vis spectroscopy, EDX, XRD and SEM analysis techniques. Comparative antibacterial study was done against *Gram-positive* and *Gram-negative* bacteria by well diffusion method in which results revealed that the biologically synthesized Co_3O_4NPs showed relatively similar antibacterial potential as chemically synthesized Co_3O_4NPs and higher antibacterial potential than that of positive control.

Keywords: Antibacterial potential, EDX, FRET, Green synthesis, *Mappia foetida*, SEM, XRD.

INTRODUCTION

As cobalt oxide nanoparticles (Co_3O_4NPs) are antiferromagnetic p-type semiconductor they have great interest of researchers due to their various applications in different fields such as semiconductors[1], sensors[1], batteries[1], catalysis[1], storage devices[1] and capacitors[1]. In Co_3O_4NPs Co^{3+} occupy the octahedral position and Co^{2+} occupy the tetrahedral position at cubic close packed arrangement of oxide ions in regular spinel structure[1].

Various chemical, physical and electrochemical methods have been reported for the synthesis of Co_3O_4NPs, but these methods are not eco-friendly as hazardous chemicals are used hence an alternative approach of green chemistry with minimum toxic chemicals and eco-friendly materials was used[1]. Microorganisms[2-4] and plant extract[5] can be used in green chemistry but use of plant extract is beneficial as use of microorganisms requires biohazards and elaborate process of maintaining the cell culture.

Mappia foetida or *Nothapodytes nimmoniana* is an Indian indigenous tree commonly

known as Amruta, Kalgur or Narkya, belonging to the family Icacinaceae with anticancer, antiviral as well as anti HIV properties[6]. *Mappia foetida* contains various biomolecules and among these alkaloid Camptothecin (CPT) shows efficiency in animal tumour models but CPT showed cytotoxic nature and hence it is not used clinically but its water soluble derivatives are used in the treatment of cancer[7] such as Topotecan, Irinotecan etc.

As per literature survey on the green synthesis of Co_3O_4NPs various plant extracts such as leaf extracts (*Calotropis gigantea*[8], *Aspalathus linearis*[5], *Sageretia thea*[1], *Euphorbia heterophylla* L.[9], *Helianthus annuus*[10], *Moringa oleifera*[11]), peel extracts (*Punica granatum*[12]) and fruit extracts (*Terminalia chebula*[13] and *Manihot esculenta crantz*[14]) were used for the synthesis of these particles but the green synthesis of Co_3O_4NPs using *Mappia foetida* leaf extract have not been reported which encouraged us to use it as a stabilizing agent for Co_3O_4NPs synthesis.

In the present study the green synthesis of Co_3O_4NPs by *Mappia foetida* leaf extract and its antimicrobial activity was studied and the structural and morphological properties were investigated by Ultraviolet-Visible (UV-Vis) spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis techniques.

MATERIALS AND METHOD

Sample collection was done from the forest department of Shirala Tehsil (Sangli, Maharashtra, India). Green leaves were shed dried and crushed in mortar and pestel. The powder was stored in a desiccator at room temperature. Cobalt chloride ($CoCl_2$) was purchased from Merck specialities private limited, Mumbai. The solutions were prepared by using distilled water. All spectroscopic measurements were done at room temperature.

Preparation of nanoparticles

Brown coloured leaf extract of *Mappia foetida* was prepared by heating dried leaves powder into 100 mL of distilled water for 20 minute. at 80^0C which was filtered through Whatman filter no.1 and stored at 5^0C. This leaf extract was then added to 0.01M $CoCl_2$ with constant stirring and heating at 60^0C, which was further boiled and allowed to cool down before centrifuged, then after washing a black powder was obtained that was scraped out and dried in Muffle Furness for the further study.

Chemically synthesized Co_3O_4NPs were prepared as per literature[15].

Characterization of Co_3O_4NPs

UV-Vis double beam spectrophotometer of Equip-tronics UV-Visible spectrophotometer (EQ-826) was used for the UV-Visible spectral analysis, and the baseline was adjusted by distilled water. EDX analysis was done on the EDX, EM912 model. X-ray diffractometer (Bruker, D2-Phaser) embedded with CuKα radiation at 30 mA current and 40 kV voltage was used for XRD analysis by using 2θ in the range of 0-900. The JSM-6360 JEOL model was used for Scanning Electron Microscopy (SEM) analysis. Carbon coated copper grid was used to prepare sample film in which a small amount of sample was dropped on the grid and mercury lamp was used to dry this film on the grid after removal of extra solution by blotting paper.

Antimicrobial assay

Pure cultures of four human pathogenic bacteria in which *Staphylococcus aureus*, *Bacillus subtilis* as *Gram-positive* and *Escherichia coli*, *Pseudomonas vulgaris* as *Gram-negative* bacteria were produced from the Microbiology Department of "New Arts, Commerce and Science College" Ahmednagar, Maharashtra for investigation of the antibacterial activities of Co_3O_4NPs in triplicates by well diffusion method[16].

RESULT AND DISCUSSION

In biological synthesis of Co_3O_4NPs black precipitate obtained after addition of brown coloured Mappia foetida leaf extract acting as capping and reducing agent confirms the formation of nanoparticles.

The UV-Vis absorption spectrum of the solution was observed in the range of 200-800 nm where the characteristic absorption peak or surface plasmon resonance (SPR) peak was observed at 425 nm which was due to absorption of metal oxide. The SPR peaks were dependent on the size and

shape of the particles and the type of solvent used for particle synthesis[17].

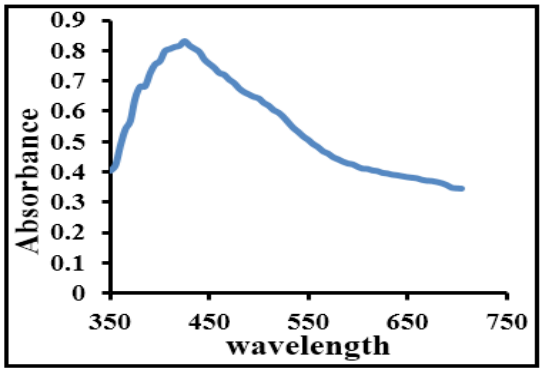


Fig. 1. UV-VIS spectra of biologically synthesized Co_3O_4NPs

Chemical purity, stoichiometry and elemental phase was determined by Energy Dispersive X-ray[18] (EDX) as indicated in Fig. 6 in between 0 to 10 keV. Obtained results shows strong signals at 0.8 keV, 7.0 keV and 7.6 keV were for Co and intense signal between 0.0-0.5 keV for O suggesting that Co and O were the major elements and formation of synthesis of cobalt oxide arise from the sample and other unexpected weak signals at 0.3 keV, 1.3 keV, 1.5 keV, 1.8 keV, 2.0 keV, 2.4 keV, 3.8 keV were from bio-compounds present in the leaf extract.

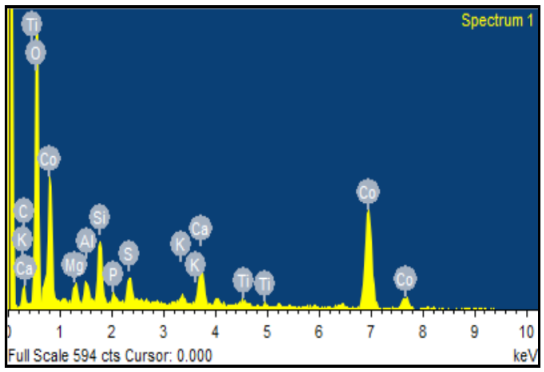


Fig. 2. EDX spectra of biologically synthesized Co_3O_4NPs

X-ray diffraction (XRD) technique was used to determine the purity and phase of the powdered Co_3O_4NPs. Fig. 3 represented the typical diffraction pattern in which the peaks at 2θ were 31.28, 36.76, 59.28 A^0 corresponds to Co_3O_4 having spinel structure and cubic close packed phase [JCPDS card no.–01-073-1701]. Insignificant peaks observed could be attributed to organic substances[19]. A shift in some peaks was due to the presence of impurities owing to the biomass residue[20]. The presence of broad peaks suggests the synthesized particles to be very small in size in the nano dimensional state and amorphous in nature[21]. The average crystallite size determined by the Scherrer formula, $D = 0.9\lambda/\beta \cos\theta$ using the half-width of the intense peak in the powder pattern. Where D is the crystallite size, λ is X-Ray wavelength which is 1.54 A0, β is full width at half maxima (FWHM) and θ is Bragg's angle. The crystallite size of biologically synthesized Co_3O_4 NPs corresponding to the highest peak observed in XRD pattern was approximately 5 nm.

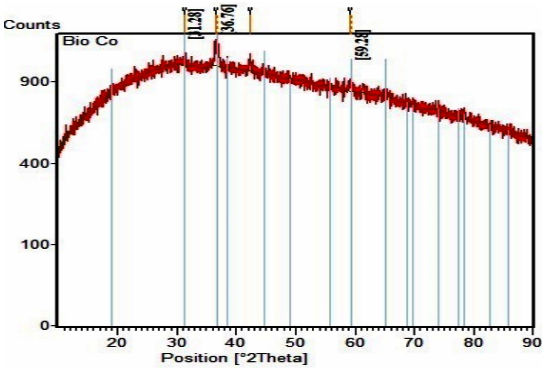


Fig. 3. XRD of biologically synthesized Co_3O_4NPs

The surface morphology of the nanoparticles was determined by analysing the structure by the scanning electron microscopy. SEM images in Fig. 7 showed spherical shaped agglomerated surface morphology of Co_3O_4 NPs. Biomolecules from leaf extract acts as capping and stabilizing agents which forms coating on the individual nanoparticles and contains hydroxyl group which causes intermolecular hydrogen bonding resulting in agglomeration[22]. This agglomeration depends upon the nature and compounds present in the extract[23].

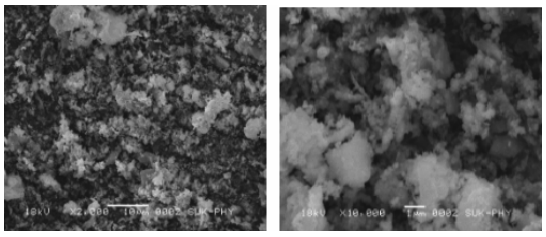
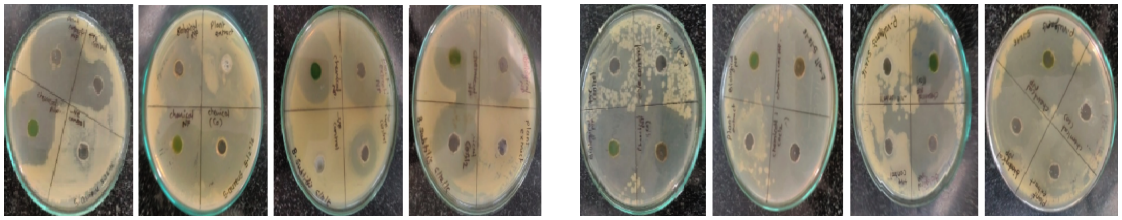


Fig. 4. SEM images of biologically synthesized Co_3O_4NPs

Eco toxic properties of transition metal oxide are due to shape, small size, high chemical reactivity, biological activity and agglomeration tendency which causes threat to the environment and human beings. The well diffusion method was used for antibacterial study against *S. aureus, B. subtilis* as *Gram-positive* bacteria and *E. coli, P. vulgaris* as *Gram-negative* bacteria. Here biologically synthesized Co_3O_4NPs showed relatively similar zone of inhibition as

chemically synthesized Co_3O_4NPs (except for *B. subtilis*) and $CoCl_2$ (except for *E. coli*) and higher ZOI than that of positive control i.e. streptomycin. Hence antimicrobial activity of the biologically synthesized Co_3O_4NPs and chemically synthesized Co_3O_4NPs was significantly higher than that of streptomycin as antibiotics which indicate the development of resistance against the antibiotics. Our study showed different zone of inhibition for test bacteria indicating difference in sensitivity against Co_3O_4NPs due to difference in membrane stability as they belong to different genera and a thick peptidoglycan layer was present in *Gram-positive* bacteria while a rigid lipid and lipoproteins outer membrane is present in *Gram-negative* bacteria[24].



(a) (b) (c) (d) (e) (f) (g) (h)

Fig. 5. Antibacterial activity of Co_3O_4NPs against a), b) *S. aureus*, c), d) *E. coli*, e), f) *B. subtilis*, g) and h) *P. vulgaris*

Table 1: Antimicrobial activity of Co_3O_4NPs (n =3)

Pathogens	Biological NPs	Chemical NPs	Plant extract	$CoCl_2$	Positive Control
S. aureus	33.5	32.5	0	35.5	19.7
B. subtilis	33.5	29	0	32	12
E. coli	38.3	37.6	0	26	22.5
P. vulgaris	37.3	37.6	0	41.5	12

CONCLUSION

In present work biological synthesis of Co_3O_4NPs using *Mappia foetida* leaf extract provides an environmentally friendly route for the synthesis of nanoparticles by avoiding use of harmful and toxic chemicals. Spherical and agglomerated nanoparticles with an average size of 5nm were synthesized. Biologically synthesized Co_3O_4NPs showed relatively similar antimicrobial activity as chemically synthesized Co_3O_4NPs and higher antimicrobial activity than that of streptomycin as positive control and hence can be used as a strong antimicrobial agent.

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