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Research Article

BIO SYNTHESIS AND CHARACTERIZATION OF COBALT OXIDE NANOPARTICLES USING AQUEOUS EXTRACT OF *MORINGA OLEIFERA* LEAVES

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ABSTRACT

Recently the green synthesis of nanoparticles involving plant extract has attracted the attention of researchers. In this work we present an environmentally friendly, one step, ultra-fast, cost-efficient method for producing cobalt oxide nanoparticles using readily available *Moringa Oleifera* (Drumstick) extracts as reducing and capping agent. The bio-reduced cobalt nanoparticles were appropriately characterized. The cobalt oxide nanoparticles are characterized by UV-DRS, IR, powder XRD, SEM and EDX. To characterize the crystal phase identification of the nanoparticle, X-ray Powder Diffraction (Powder XRD) is used. Surface morphology was studied by SEM and EDX to determine the amount of cobalt present. The unique physicochemical characteristics of Cobalt oxide are believed to have increased medical applications when synthesized via environmentally benign methods free of toxic byproducts.

Keywords: Plant extract, Bio- reduced, XRD, *Moringa Oleifera.*

INTRODUCTION

As the global scenario is now changing towards the use of non- toxic and eco-friendly development of nanoparticles using plant extract is gaining importance. In this study, we report for the first time, the use of the *Moringa oleifera leaves* natural extract as an effective chelating agent for the facile and rapid biosynthesis of pure cobalt single-phase nanoparticles at a room temperature. The fact that there was no use of inorganic/organic solvents neither surfactants nor high temperature makes this synthesis an effective green and eco-friendly process (Guo *et al*., 2002). In the present approach, H_2O is utilized as the environmentally benign solvent throughout the preparation. The second concern in a green nanoparticle preparation method is the choice of the reducing agent (Singh *et al*., 2022). The cobalt oxide nanoparticles are characterized by UV-DRS, IR, powder XRD, SEM and EDX (Koyyathi *et al*., 2016). To characterize the crystal phase identification of the nanoparticle, X-ray Powder Diffraction (Powder XRD) is used. Surface morphology was studied by SEM and EDX to determine the amount of cobalt present (Ahamed *et al*., 2021). Our environment, which is endowed by nature, needs to be protected from ever substantial improvement in our lifestyles and chemistry has been contributing significantly to this evolution.

MATERIALS AND METHODS

Materials

Cobalt nitrate (99%purity) used as the introductory material was supplied by Merck- chemicals.

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Preparation of cobalt nitrate solution

In a preparation process cobalt nitrate solution was prepared by dissolving cobalt nitrate (0.01M) in 100 ml deionized water.

Collection of Extract

Moringa Oleifera leaves were collected from the local region. They were washed and cleaned with distilled water and dried with water absorbent paper. Then it was crushed with mortar and pestle, soaked in water, and dispensed in 100 ml of sterile distilled water and kept for 2-3 minutes. The extract was then filtered using Whatman's No.1 filter paper. The filtrate was collected in a clean and dried conical flask. The fresh extract is used for the nanoparticle preparation.

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Preparation of nickel nanoparticles

10 ml of 0.1M Aqueous cobalt nitrate was added under constant stirring to 50 ml of aqueous extract of *leaves of Moringa Oleifera* at 40^{o} C for 20 min. The pale-yellow solution was slowly turned brown. The precipitated cobalt oxide nanoparticles were filtered and washed over and over again with distilled water followed by ethanol to get free of the impurities. The precipitate was dried in an air oven at 90^{o}C for 90 min and was submitted to annealing for 2 h in air at 500°C to get cobalt oxide nanoparticles.

Preliminary phytochemical investigation

Aqueous extracts of leaves of *Moringa Oleifera* were subjected to preliminary phytochemical screening for the detection of various plant constituents. The preliminary phytochemical investigations were done by the standard chemical tests

Characterization

The UV-DRS spectrum was recorded in a SHIMADZU UV-Vis Spectrophotometer (Model V550) at the range of 200-800 nm. FT-IR spectra (Fourier Transform Infrared Spectrometer) were recorded on SHIMADZU FT-IR 5300 model spectrophotometer in KBr pellets in the range of 4000- 400 cm^{-1}. Crystal phase identification of the samples were characterized by powder X-Ray Diffractometer (XRD, PW 3040/60 Philips X'Pert, Holland) with Cu (Kα) radiation (λ=0.15416 nm) operating at 40 kv and 30 mA with 2θ ranging from 10- 90°. SEM and EDX were also recorded. Energy Dispersive X-ray Spectroscopy (EDX) technique is applied to determine the particle size and quantitative distribution of elements on those composites

RESULTS AND DISCUSSION

The recent development and implementation of new technologies have led to a new era, the nano-revolution which unfolds the role of plants in bio and green synthesis of nanoparticles which seem to have drawn quite an unequivocal attention with a view of synthesizing stable nanoparticles (Matinise *et al*., 2018) Employing plants towards synthesis of nanoparticles are emerging as advantageous compared to microbes with the presence of broad variability of bio-molecules in plants can act as capping and reducing agents and thus increases the rate of reduction and stabilization of nanoparticles. Biological synthesized nanoparticles have upsurge applications in various sectors (Ju *et al*., 2016)**.** Green synthesis of cobalt oxide nanoparticles was carried out using the aqueous extract of *M. oleifera* leaves for the ecofriendly development of novel technologies. The aqueous leaf extract was chosen as a reducing agent and stabilizing agent because triterpenoids, Vitamin C, polyphenols, flavonoids, gallic tannins, catechol tannins, anthraquinones and carbohydrates are biodegradable, non-toxic and water soluble at room temperature, unlike other polymers. Second, they can form complexes with metal ions and thereafter can reduce the metals (Younis *et al*., 2020). Third, extract contains molecules bearing alcoholic functional groups which can be exploited for reduction as well as stabilization of the nanoparticles.

The colour change was noted by virtual observation in fresh aqueous extract of *Moringa Oleifera* leaves incubated with aqueous solution of cobalt nitrate. It started to change color from colorless to light green and to brownish red due to the cobalt oxide nanoparticles. The intensity of colour increases with increase in time and after one hour there is no significant change in colour observed due to the completion of reaction. As the synthesize cobalt oxide nanoparticles by green process are generally amorphous, an annealing was necessary to induce an effective crystallization. In this case, the as obtained centrifuged precipitate was heated at 90°C for 90 min to remove bio-compounds in excess. Following such a phase, the Co-based precipitate was nano submitted to annealing during 2 h in air at 500°C. The strong co relationship exists between particle size and shape in inorganic materials for their potential application as sensors. Particle properties can be tuned after optimization with the help of reaction time, temperature, pH, type of precursor used, and its concentration (Iravani *et al*., 2020). An optical property of cobalt oxide nanoparticle was recorded with respect to high purity, and its crystallinity was confirmed with the help of UV-DRS by observing an excitonic absorbance band at 300 nm with a tail extending towards a longer wavelength due to their quantum size effects as shown in Figure 1. The absorption peaks exhibit a broad peak due to the particle size. The stability of Co_3O_4 nanoparticles can be attributed to symmetrical-polarity structure which depends on the weak interaction of VanderWaals forces within the particle regime.

Table 1. Not detected; ++: present in moderate concentration; +++ present in high concentrations

S.No	Phytochemicals	Aqueous extract
1	Gallic tannins	++
2	Catechol tennins	++
3	Coumarins	-
4	Steroids and triterpenoids	++
5	Flavonoids	++
6	Saponins	++
7	Anthraquinones	+++
8	Alkaloids	++
9	Reducing sugars	++
10	Cardiac glycosides	-

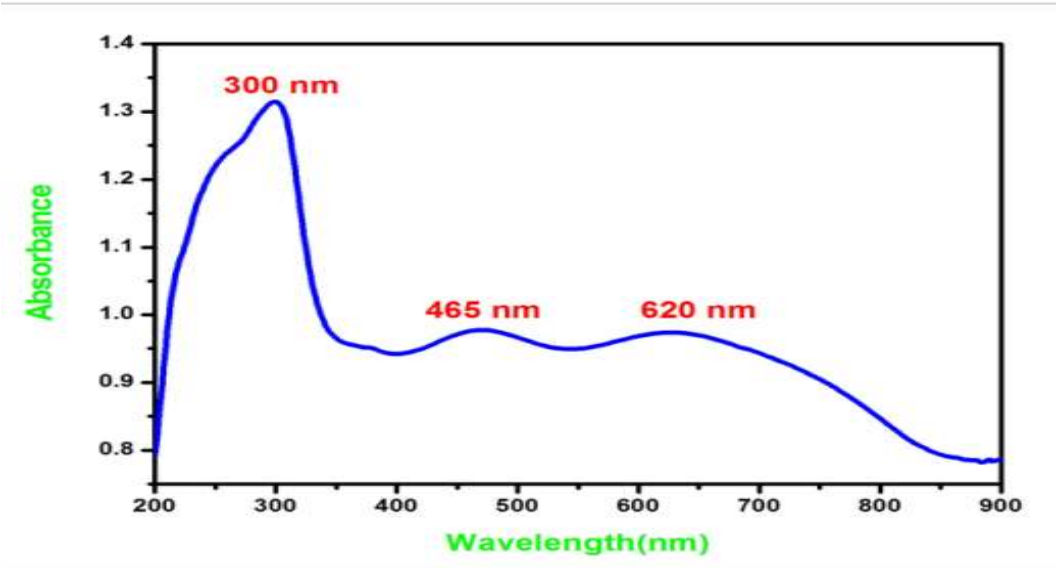

Figure 1. UV- DRS of Co_3O_4 nanoparticles.

FT-IR absorption bands in the range of 4000-400 cm-1 are usually assigned to vibration of ions in the crystal lattice were recorded using KBr disc method (Figure 2) Shows the FTIR spectra of Co_3O_4 shows peak at 570.93 cm^{-1} corresponds to vibration in the Co^{3+} in octahedral hole and 663.51 cm^{-1} stretching vibrations in the Co^{2+} in tetrahedral hole it shows the presence of single-phase face centered cubic structured nanoparticles. Annealing at 500◦C for two hours leads to the change of amorphous to the crystalline state with narrow particle size distribution with well-defined particle size, shape, and phase purity. A stretching frequency at 3388 cm^{-1} and a weak asymmetric band at 1443 cm^{-1} support the presence of OH-group due to the absorption of water by nanoparticles during sample preparation. The presence of two strong M–O stretching and bending frequencies at 663.51cm^{-1} and 570.93 cm^{-1}, respectively, supports the presence of phase purity with monodispersed in the face centered cubic structure. A stretching frequency at 3788cm^{-1} assigned to the presence of amino group of proteins and stretching frequency at 1788 cm^{-1} assigned to the presence of carbonyl group of flavonoids and other biomolecules.

Figure 2 shows the absorbance at 250 to 350 cm^{-1} and 550-700 cm^{-1}, the first absorption at due to O^{2-} to Co^{2+} the ligand to metal charge transfer and the band at 580 cm^{-1} corresponds to O2- to Co^{3+} charge transfer the cubic phase Co_3O_4 behaves as a p-type semiconductor. The spectrum can be explained in terms of charge transfer in transition metal (TM) oxides. X-ray diffraction pattern of the as-synthesized cobalt oxide nanopowder was analyzed to investigate the phase structure along with its crystallinity as illustrated in Figure 3. This shows crystalline structure with 8 peaks. The XRD pattern shows a significant amount of line broadening which is a characteristic of nanoparticles. The XRD pattern exhibits prominent peaks at 19 °, 21.4°, 23.26°, 31.23°. 35.1°, 36.84°, 44. 62° and 65.12°. The peak positions appear at 2θ = 19 °, 23.26°, 31.23°, 35.1°, 36.84°, 44. 62° and 65.12° can be readily indexed as (111), (220), (311), (222), (220), (400) and (440) crystal planes The peaks were indexed to pure phase with a face-centered cubic structure, which corresponds to JCPDS file (76–

1802) after annealing the sample and by matching Bragg reflection peaks. The Co_3O_4 with Co^{2+} ($3d^7$) and Co^{3+} ($3d^6$) located at tetrahedral and octahedral sites, respectively, crystallizes in a spinel configuration The average particle size calculated with the help of Debye-Scherrer equation was found to be 50 nm.

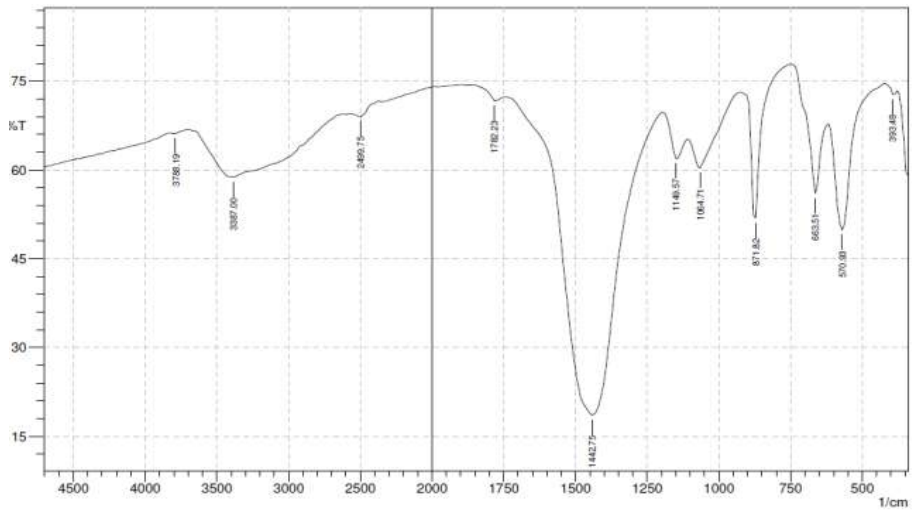

Figure 2. IR spectrum of Co_3O_4 nanoparticles.

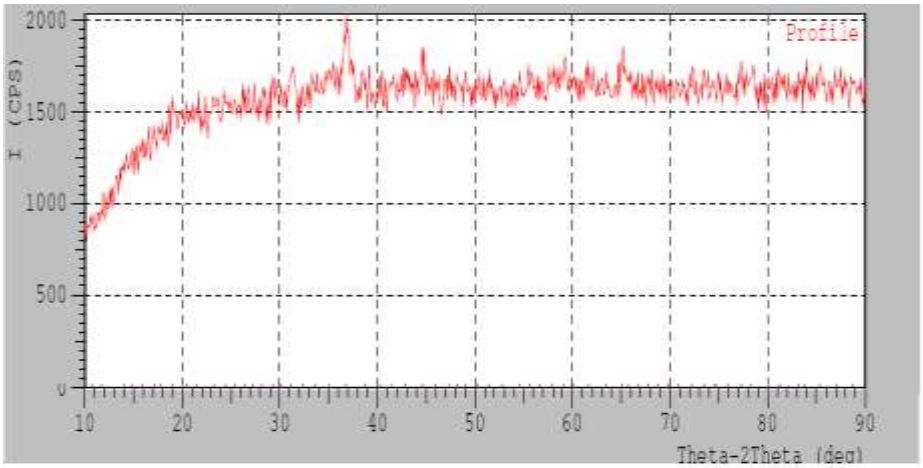

Figure 3. XRD of $Co3O_4$ nanoparticles.

The surface morphological features of synthesized nanoparticles were studied by scanning electron microscopes. Figure 4 supports the microcrystalline nature of the particle after calcinations with least degree of agglomeration. Particles seem to have an irregular shape with chemical homogeneity with uniform morphology due to the presence of interparticle surface connectivity. It was observed that the annealing temperature increases the crystalline nature of the particle that changes due to nucleation. The shape of the cobalt oxide nanoparticles is like needle shaped petals of a flower. These microstructural characterization studies were used to determine the size of nanoparticles and examine the homogeneity and size distribution. Energy Dispersive X-ray Spectroscopy (EDX) technique is applied to determine the particle size and quantitative distribution of elements present shown in Figure 5. The cobalt content of these Co_3O_4 nanocubes in plant extract is found to be 8.64 wt %. Importantly, EDX analysis also indicates that, within the detection limits, Co was the only element present in these particles. Simple green method used to synthesize nanocrystalline Co_3O_4 powder. X-ray diffraction pattern confirmed the formation of single phase Co_3O_4. SEM image indicates the obtained samples have needles like morphology and its grain size will be in the range of 50 nm.

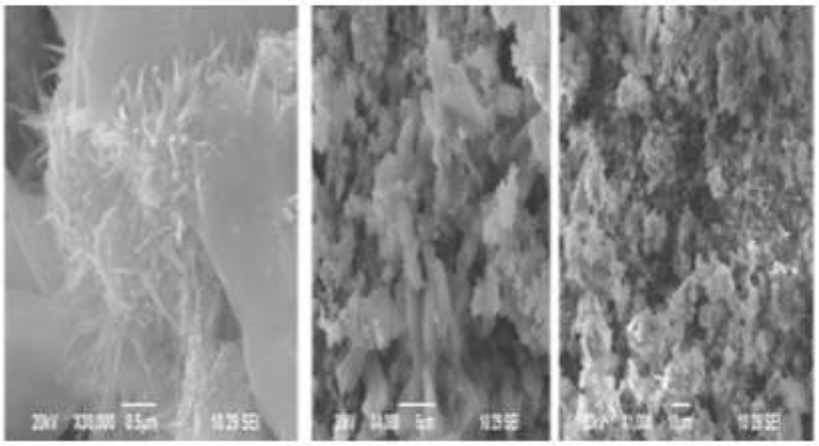

Figure 4. SEM images of Co_3O_4.

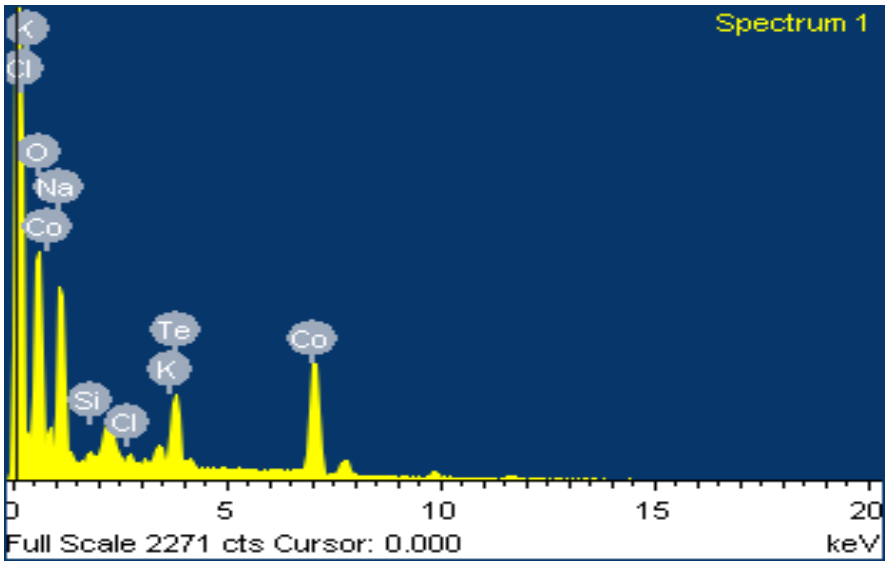

Figure 5. EDX of spectra of Co_3O_4.

CONCLUSION

Cobalt nanoparticles were synthesized in good yield with the help of a green approach with controlled size distribution and phase purity. This methodology is a novel, cheap, and convenient technique suitable for a large-scale production for other metal oxide nanomaterials having monomodal distribution with advanced functional properties for their future use in technological applications. (Putri *et al*., 2021) Co_3O_4 nanoparticles with an average size of 45 nm were prepared and characterized by using XRD, TEM, UV-DRS and FT-IR. The XRD confirms the simple cubic crystal structure of the Co_3O_4. The optical absorption spectrum of cobalt oxide nanoparticles was studied by UV-DRS. The mean particle size is determined by the XRD. We developed an efficient, Quick & simple and low cost, straightforward method, no harmful chemicals, and protect our environment.

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REFERENCE

Guo, F., Zheng, H., Yang, Z., & Qian, Y. (2002). Synthesis of cobalt nanoparticles in ethanol hydrazine alkaline system (EHAS) at room temperature. *Materials Letters*, *56*(6), 906-909.

Singh, A. K. (2022). A review on plant extract-based route for synthesis of cobalt nanoparticles: photocatalytic, electrochemical sensing and antibacterial applications. *Current Research in Green and Sustainable Chemistry*, 100270.

Koyyati, R., Kudle, K. R., &Padigya, P. R. M. (2016). Evaluation of antibacterial and cytotoxic activity of green synthesized cobalt nanoparticles using *Raphanus sativus* var. longipinnatus leaf extract. *International Journal of Pharmtech Research*, *9*(3), 466-472.

Ahmed, K., Tariq, I., & Mudassir, S. U. S. M. (2021). Green synthesis of cobalt nanoparticles by using methanol extract of plant leaf as reducing agent. *Pure and Applied Biology (PAB)*, *5*(3), 453-457.

Matinise, N., Mayedwa, N., Fuku, X. G., Mongwaketsi, N., &Maaza, M. (2018, May). Green synthesis of cobalt (II, III) oxide nanoparticles using *Moringa Oleifera* natural extract as high electrochemical electrode for supercapacitors. In *AIP Conference Proceedings* (Vol. 1962, No. 1, p. 040005). AIP Publishing LLC.

Ju, S., Liu, M., & Yang, Y. (2016). Preconcentration and determination of cadmium, lead, and cobalt in Moringa oleifera (Moringaceae) using magnetic solid-phase extraction and flame atomic absorption spectrometry. *Analytical Letters*, *49*(4), 511-522.

Younis, S., Ijaz, I., Nazir, A., Bukhari, A., Rizwan, A., & Gilani, E. (2020). Synthesis, characterization & bacterial evaluation of cobalt nanoparticles using drumstick leaf extract via green route. *Easy Chair*, *2386*.

Iravani, S., & Varma, R. S. (2020). Sustainable synthesis of cobalt and cobalt oxide nanoparticles and their catalytic and biomedical applications. *Green Chemistry*, *22*(9), 2643-2661.

Putri, G. E., Rilda, Y., Syukri, S., Labanni, A., & Arief, S. (2021). Highly antimicrobial activity of cerium oxide nanoparticles synthesized using *Moringa oleifera* leaf extract by a rapid green precipitation method. *Journal of Materials Research and Technology*, *15*, 2355-2364.