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Biosynthesis of Cobalt Oxide Nanoparticles - A Short Review

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ABSTRACT

This short review article presents a summary of ecofriendly ways to synthesize cobalt oxide (Co_3O_4) nanoparticles using biomaterials like plant extract, bacterium and fungus. Currently, researchers have focused their concentration on the biosynthesis of nanoparticles using biomaterials containing phenols, carbonyls, amines, carbohydrates, proteins and amino acids which act as bio-templates, reducing agents, stabilizing agents, capping agents and chelating agents for nanoparticles. This article also discussed the reports of characterization of cobalt oxide nanoparticles by UV-Vis spectroscopy, diffuse reflectance spectroscopy (DRS), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), selected area electron diffraction (SAED), scanning electron microscopy (SEM), high resolution scanning electron microscopy (HRSEM), transmission electron microscopy (TEM), high resolution transmission electron microscopy (HRTEM), atomic force microscopy (AFM) and Brunauer - Emmett -Teller (BET) analyzer which were carried out by various researchers. The applications of these biosynthesized cobalt oxide nanoparticles in a wide range of potential zones are listed including antibacterial activity, photo catalyst, sensor and supercapacitor.

1. Introduction

Due to the astonishing physical, chemical, optical, magnetic, electrical, catalytic and electronic properties of Co_3O_4 nanoparticles, it has enormous applications in fuel cells, lithium-ion batteries, supercapacitors, field-emission materials, solar selective absorbers, pigments, electrochromic devices, gas sensors and catalytic applications [1]. However, most of the synthesis routes have been connected with a number of drawbacks to the use of complex synthesis steps, expensive equipment, prolonged reaction time, high cost, and high temperature of synthesis. In addition, some of the stabilizers and surfactants used in the synthesis procedure tend to be unsafe with cytotoxic and carcinogenic effects making the synthesized nanoparticles inappropriate for various applications. As a result, present review interest is wandering towards biosynthesis involving the use of environmentally friendly biological materials in the synthesis of nanoparticles [2]. These biomaterials contain phenols, carbonyls, amines, carbohydrates, proteins and amino acids which act as bio-templates, reducing agents, stabilizing agents, capping agents and chelating agents for nanoparticles.

In this article, we have reported the various pattern of biosynthesis of Co_3O_4 nanoparticles, their characterization findings with respect to UV-vis, DRS, FTIR, XRD, SAED, SEM, HRSEM, TEM, HRTEM, AFM, BET and its reported applications.

2. Biosynthesis of Co_3O_4 Nanoparticles

Synthesis of Co_3O_4 nanoparticles can be done by phytochemicals present in the plant extract, bacterium and fungus. These biological materials are not only act as reducing agents but also as capping agents which help to reduce the agglomeration of nanoparticles and thereby controlling the morphology and helping to stabilize the nanoparticles.

Table 1 represent the Co_3O_4 nanoparticles synthesize using *Aspalathus linearis* leaf extract, *Azadirachta indica* leaf extract, *Calotropis gigantea* leaf extract, *Calotropis procera* latex, *Ginkgo biloba* leaf extract, *Manihot esculenta Crantz* root extract, *Moringa Oleifera* leaf extract, *Punica granatum* peel extract, *Sageretia thea* (Osbeck.) leaf extract and *Sechium edule* fruit extract, Bacterium like *Bacillus pasteurii, Bacillus subtilis, Brevibacterium casei and Micrococcus lylae* were used for the synthesis of greener Co_3O_4 nanoparticles. Also biosynthesis of Co_3O_4 nanoparticles by *Aspergillus nidulans and* yeast were listed in Table 1.

In general, cobaltous nitrate hexahydrate, cobalt(II) acetate tetrahydrate, cobalt chloride hexahydrate and cobalt (II) acetyl acetonate were used as precursors for the green synthesis of Co_3O_4 nanoparticles.

3. Crystallography, Optical and Vibrational Properties

Table 2 provides detailed reports of the characterization responses of Co_3O_4 nanoparticles which were derived from different routes as mentioned in Table 1. Fourier-transform infrared (FTIR) analysis had performed to confirm the formation of Co–O bond and possible functional groups of biomaterials involved in the biosynthesis of Co_3O_4 nanoparticles. Authors studied the crystallographic properties by using X-ray diffraction (XRD) and optical properties using UV–vis spectroscopy and diffuse reflectance spectroscopy (DRS). In Fourier-transform infrared (FTIR) analysis, two peaks related to Co-O bond were reported by researchers. The first one is due to Co^{3+} in octahedral site and the second one is attributed to Co^{2+} in tetrahedral site. They are related to ligand–metal charge transfer $O^{2-} \rightarrow Co^{2+}$ and ligand–metal charge transfer $O^{2-} \rightarrow Co^{3+}$.

4. Size, Morphology, Crystallinity and Surface Area

To calculate the crystallite size of the biosynthesized Co_3O_4 nanoparticles based on XRD data, most of the authors used the Debye–Scherer formula, $D = K\lambda/\beta\cos\theta$, where, K is a constant equal to 0.9, λ is the wavelength of Cu-Kα radiation, β is the full width at half maximum (FWHM) of the diffraction peak in radiant and θ is the Bragg angles of the main planes.

As mentioned in Table 3, researchers observed the surface morphology and size of the synthesized Co_3O_4 nanoparticles by scanning electron microscopy (SEM), high-resolution scanning electron microscope (HRSEM), transmission electron microscopy (TEM), High resolution transmission electron microscopy (HRTEM), atomic force microscopy (AFM). Crystallinities were confirmed by selected area electron diffraction

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(SAED). The specific surface area of the Co_3O_4 nanoparticles were measured by the gas adsorption technique based on the Brunauer–Emmett–Teller (BET) method. Quasi-spherical, irregular, cubic, granular, spherical, hollow-rod, flower-like, unclear lattice structure, relatively distinct and dispersed mixed shape morphologies were reported for biosynthesized Co_3O_4 nanoparticles. The size of the Co_3O_4 nanoparticles synthesized by bacterium and fungus were reported in the range of 2 to 31 nm. Thus, bacterium and fungus can be used to synthesize very small size nanoparticles. Shim et al. reported highest surface area (73.3 m^2/g) of *Bacillus subtilis* mediated Co_3O_4 NPs when comparing with others.

Table 1 Biosynthesis of Co_3O_4 nanoparticles

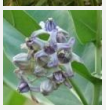
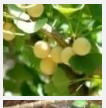
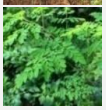
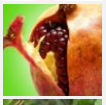
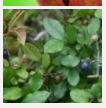
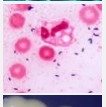
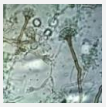
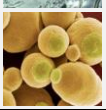
Biomaterial	Image	Role of biomaterial	Precursor	Reaction Temp.	Drying Temp.	Annealing Temp.	Bioactive components	Ref.
Plant extract								
Aspalathus linearis (Leaf)		Reducing agent and chelating agent	$Co(NO_3)_2{\cdot}6H_2O$	75 °C to 85 °C for 2–3 h	90 °C for 90 min	100 °C, 300 °C, 400 °C, and 500 °C for 2 h	Phenolic compounds	[3]
Azadirachta indica (leaf)		-	$Co(NO_3)_2{\cdot}6H_2O$	250 °C for 15 min	100 °C	300 °C for 2 h	-	[4]
Calotropis gigantea (leaf)		Reducing agent and capping agent	$Co(NO_3)_2{\cdot}6H_2O$	60–80 °C	-	400 °C for 2 h	Triterpenoids, flavonoinds (polyphenols), steroids, cardenolides, alkaloids	[5]
Calotropis procera (latex)		Reducing and stabilizing agents	$Co(CH_3COO)_2.4H_2O$	Room Temp. (RT) for 2h	60 °C for 24 h	450 °C for 3 h	Carbohydrates, proteins, amino acids, vitamins, lipids, enzymes, resins, alkaloids, and tannins, etc.	[6]
Ginkgo biloba (leaf)		-	$Co(CH_3COO)_2$	RT for overnight	60 °C for overnight	400 °C for 3 h	Celluloses, lignins and proteins	[7]
Manihot esculenta Crantz (root)		Chelating agent and stabilizing agents	$CoCl_2$	24 h under ambient condition	60°C for 24 h	500 °C for 2 h	-	[8]
Moringa oleifera (leaf)		Chelating agent	Cobalt chloride hexahydrate	RT for 18 h	100 °C for 24 h	500 °C for 2 h	-	[9]
Punica granatum (peel)		Stabilizing and reducing agent	$Co(NO_3)_2.6H_2O$ Precipitating agent : NaOH	RT for 3h	Over night	500 °C for 5 h	Alkaloids	[10]
Punica granatum (peel)		Reducing, stabilizing and capping agents	$Co(NO_3)_2.6H_2O$	60 °C for 90 min	60°C for 8–9 h	-	Gallic acid, punicalagins A and B, Ellagic acid and gallotannins	[11]
Sageretia thea (Osbeck.) (leaf)		Stabilizing and chelating agent	Cobalt acetate	2 h at ~ 60°C	100 °C for 2 h	500 °C	Taraxerol, Quercetin, Syringic acid, Myricetrin, Kaempferol, Daucosterol	[12]
Sechium edule (fruit)		Capping agent	$Co(NO_3)_2.6H_2O$ Precipitating agent : NaOH	65-70 °C for 48 h	95 °C	500 and 700 °C for 6 and 12 h	Ascorbic acid	[13]
Bacterium								
Bacillus pasteurii		Microbial induced precipitation	$Co(NO_3)_2$ Precipitating agent : urea	35 °C for 24h	50 °C	400 °C, 550 °C, 700 °C, and 850 °C for 6h	-	[14]
Bacillus subtilis		Soft templates	$CoCl_2{\cdot}6H_2O$ Precipitating agent : $NaBH_4$	24 h at RT	60 °C for 6 h	300°C for 12 h	Proteins, teichoic acids, and polysaccharides,	[15]
Brevibacterium casei		-	Cobalt acetate	RT for 24 h	-	200 °C for 2 h	Proteins	[16]
Micrococcus lylae		Bio-templates	$CoCl_2{\cdot}6H_2O$ Precipitating agent : $NaBH_4$	12 h at RT	60 °C for 6 h	-	Teichoic acid and lipoteichoic acids	[17]
Fungus								
Aspergillus nidulans		Capping agent	Cobalt (II) acetyl acetonate	RT for 5 days	-	-	Sulfur-bearing proteins like cysteine and methionine	[18]
Yeast		Bio-templates	$CoCl_2{\cdot}6H_2O$	60 °C for 3 h	60 °C	400 °C for 3 h	Carbonyls, amines and hydroxyls	[19]

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Table 2 Crystallography, optical and vibrational properties

Biomaterial	FTIR vibration of Co –O bond		Crystallography (XRD)		Optical properties (UV / UV –DRS)			Ref.
	Co^{3+} in octa hedral site	Co^{2+} in tetra hedral site	Planes	JCPDS card	ligand–metal charge transfer $O^{2-} \rightarrow Co^{2+}$	ligand–metal charge transfer $O^{2-} \rightarrow Co^{3+}$	Band gap	
Plant extract								
Aspalathus linearis (Leaf)	570 cm^{-1}	668 cm^{-1}	(220) (311) (222) (400) (422) (511) (440)	00-42-1467	-	-	-	[3]
Azadirachta indica (leaf)	-	-	(220) (311) (222) (400) (422) (511) (440) & (620)	43-1003	520 nm	492 nm	1.89 eV & 2.52 eV	[4]
Calotropis gigantea (leaf)	-	-	(220) (311) (222) (400) (422) (511) (440) & (553)	-	250-350 nm	400-600 nm	1.59 eV & 2.53 eV	[5]
Calotropis procera (latex)	570 cm^{-1}	660 cm^{-1}	(220) (311) (222) (400) (511) & (440)	65-3103	528 nm	785 nm	-	[6]
Ginkgo biloba (leaf)	-	-	(111) (220) (331) (222) (440) (422) (511) (440)(531) & (620)	42-1467	-	-	2.86 eV & 2.54 eV	[7]
Manihot esculenta Crantz (root)	561 cm^{-1}	665 cm^{-1}	(111) (220) (311) (222) (400) (511) (440) & (620)	00-042-1467	-	-	2.54 eV & 2.86 eV	[8]
Moringa oleifera (leaf)	-	-	(220) (311) (222) (400) (422) (511) & (440)	-	-	-	-	[9]
Punica granatum (peel)	-	-	(111) (311) & (200)	-	-	-	1.46 eV.	[10]
Punica granatum (peel)	618 cm^{-1}	628 cm^{-1}	(111) (220) (311) & (400)	073–1701	508 nm (SPR)	-	-	[11]
Sageretia thea (leaf)	576 cm^{-1}	674 cm^{-1}	(111) (220) (331) (400) (422) (511) (440) (620) (533) & (711)	00-042-1467	-	-	-	[12]
Sechium edule (fruit)	571.16 cm^{-1}	664.68 cm^{-1}	(111) (220) (311) (400) (422) (511) & (440) (Co3O4/700°C/6h)	94715	350 -600 nm	600 - 800 nm	1.42 eV & 2.53 eV	[13]
Bacterium								
Bacillus pasteurii	-	-	(111)(220) (311) (400) (422) (511) (440) & (533)	42-1467	-	-	-	[14]
Bacillus subtilis	-	-	-	42-1467	-	-	-	[15]
Brevi bacterium casei	-	-	(200) (400) (440)	43-1003	-	-	-	[16]
Micrococcus lylae	-	-	(111) (220) (311) (400) (422) (511) & (440)	42-1467	-	-	-	[17]
Fungus								
Aspergillus nidulans	-	-	(311) (400) (440)	01-080-1536	-	315 nm (SPR)	3.63 eV	[18]
Yeast	-	-		76-1802	-	-	-	[19]

Table 3 Size, morphology, crystallinity and surface area

Biomaterial	Size	Morphology	Crystallinity	BET Surface area (m^2/g)	Ref.
Plant extract					
Aspalathus linearis (leaf)	2 - 7 nm (histogram)	Quasi-spherical (HRTEM)	Amorphous (SAED)	-	[3]
Azadirachta indica (leaf)	1 - 7 nm (histogram); 0.24 nm (XRD)	Nearly quasi-spherical (HRTEM)	Polycrystalline (SAED)	-	[4]
Calotropis gigantea (leaf)	~60-80 nm(HRTEM); 50 - 60 nm (TEM)	Nearly spherical shaped (HRTEM) Spherical (TEM)	-	46.7	[5]
Calotropis procera (latex)	5–19 nm (HRTEM) ; 3 - 5 nm (XRD)	Quasi-spherical (TEM)	Amorphous (XRD)	-	[6]
Gingko (leaf)	30–100 nm (SEM)	Irregular (TEM)	-	-	[7]
Manihot esculenta Crantz (root)	36 nm (XRD)	Defect free prism liked-anchored octahedron (SEM)	Crystalline (SAED)	-	[8]
Moringa Oleifera (leaf)	20 - 50 nm (HRTEM); 38 nm (XRD)	Cubic (HRTEM)	Crystalline (SAED)	-	[9]
Punica granatum (peel)	~46 nm (SEM);	Granular (SEM)	-	40.548	[10]
Punica granatum (peel)	43.78–73.10 nm (XRD) ; 40–80 nm (AFM); < 80 nm (SEM)	Spherical (SEM)	-	-	[11]
Sageretia thea (Osbeck.) (leaf)	20.03 nm (XRD)	Cubic (HRTEM)	Crystalline (XRD& SAED)	-	[12]
Sechium edule (fruit)	31.79 nm (TEM); 21.4 nm (AFM) 39 nm(XRD)	Co_3O_4/500°C/6h NPs Irregular (SEM); Unclear lattice structure (TEM); Co_3O_4/500°C/12h NPs Relatively distinct (TEM); Co_3O_4/700°C/6h NPs Sphere(SEM); Dispersed mixed shape (TEM)	-	15.784	[13]
Bacterium					
Bacillus pasteurii	10-31 nm (SEM & TEM)	Non-specific shape (HRTEM)	Crystalline (XRD)	-	[14]
Bacillus subtilis	6.6 nm (XRD)	Hollow-rod (TEM)	Polycrystalline (SAED)	73.3	[15]
Brevibacterium casei	6 nm (TEM)	Quasi-spherical (TEM)	Single Crystalline (XRD & SAED)	-	[16]
Micrococcus lylae	~ 8 nm (XRD); ~ 2–10-nm (HR-TEM)	Flower-like (FE-SEM)	Poly crystalline (SAED)	-	[17]
Fungus					
Aspergillus nidulans	20.29 nm (histogram); 41.97 nm (XRD)	Spherical (TEM)	Polycrystalline (SAED)	-	[18]
Yeast	24 nm (XRD); 20nm -30 nm (SEM)	Spherical (SEM)	-	-	[19]

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5. Applications of Co_3O_4 Nanoparticles

The biosynthesized Co_3O_4 nanoparticles have been used widely as photocatalyst, sensor, supercapacitor, enzyme inhibitor, antibacterial, antileishmanial and antioxidant applications. Photocatalytic degradation of textile dye waste (73.86% in 150 min), reduction of 4- nitrophenol and 4- nitroaniline and antibacterial activity applications of *Azadirachta indica* mediated Co_3O_4 nanoparticles were reported by Sivachidambaram et al. [4]. Sharma et al. reported electro-catalytic reduction of I_3^- to I^- ions in dye sensitized solar cells and thermal decomposition of ammonium perchlorate applications of *Calotropis gigantea* mediated Co_3O_4 nanoparticles [5]. Efficient antibacterial activity for Co_3O_4 nanoparticles synthesized by *Calotropis procera* latex were reported by Dubey et al. [6].

Green Co_3O_4 nanoparticles synthesized using Gingko leaf extract have been used as non-enzymatic sensor for glucose or H_2O_2 by Han et al. [7]. *Moringa Oleifera* leaf extract mediated Co_3O_4 nanoparticles were reported as an electrode material for supercapacitors by Matinise et al. [9]. Bibi et al. reported photo-catalytic degradion of RBO 3R dye (78.45% in 50 min) using *Punica granatum* peel extract mediated Co_3O_4 nanoparticles [11]. *Sechium edule* fruit extract mediated Co_3O_4 nanoparticles were reported as an electrochemical sensor (H_2O_2 sensing) by Das et al. [13].

Supercapacitor application was studied by Hsu et al. [14] for the Co_3O_4 nanoparticles synthesized using *Bacillus pasteurii.* Electrochemical performance for rechargeable Li-ion batteries of *Bacillus subtilis* mediated Co_3O_4 nanoparticles were reported by Shim et al. [15]. Co_3O_4 nanoparticles synthesized using *Micrococcus lylae* have been reported as an electrode material in high-performance supercapacitors by Shim et al. Solar and thermal applications of *Aspergillus nidulans* mediated Co_3O_4 nanoparticles were suggested by Vijayanandan et al. [18].

4. Conclusion

Biosynthesis of nanoparticles provides progression over chemical synthesis as it is ecofriendly, cost effective, easily scaled up for large scale synthesis and there is no need to use high pressure, temperature, energy and harmful chemicals. The reported data here reveals Co_3O_4 nanoparticles could be effectively synthesized using various biological resources as it satisfies the need of cost effective and ecofriendly. Suitable characterization of the Co_3O_4 nanoparticles were used to understand the prospective implications of biosynthesis and ease of their applications.

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