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A Review on Green Synthesis of Metal and Metal Oxide Nanoparticles

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ABSTRACT

Metal oxide nanoparticles have captivated scrupulous research interest because of its major relevance in the field of medicine, catalysis, pigment, electronics, biotechnology, sensors, optical devices, adsorption, DNA labelling, drug delivery, kinetics, spintronics and piezoelectricity. Nanoparticles (NPs) became more significant for its reasonable property as a heterogeneous non-toxic catalyst with environmental reimbursement. The biogenic innovation of metal oxide NPs is an enhanced alternative owing to eco-friendliness. In the biological field, the probable efficacy of NPs has been reported by scores of scholars in the treatment of cancer. Owed to munificent returns, NPs explored as a powerful catalyst for several organic transformations. This section unlocks with a short course on to synthesize metal oxide NPs on a natural scale.

INTRODUCTION

Nanotechnology is the greatest dynamic area of exploration in modern material science and has established as the great innovation of things at the nanoscale of 1 to 100 nm. Nano is a Greek word symbolizing "dwarf" in the one-billionth scale (10^{-9}). To synthesis nanomaterial of various shapes and dimensions, two different methods of approaches have been widely used namely bottom-up and top-down approaches (Fig. 1).

Building up of nanomaterials from an atom by atom is a bottom-up approach while trimming down bulk material to smaller nano size is a top-down approach. Some methods of bottom-up approaches are spray pyrolysis, laser pyrolysis chemical vapour deposition, atomic/molecular condensation and sol-gel processes. Mechanical milling, hydrothermal synthesis, photolithographic processing, electron beam lithography, laser ablation, micromachining, electron beam machining, etching and sputtering are the methods of top-down approaches in exploitation. The physical and chemical methods of synthesis utilize high reactive agents, high temperature, pressure, hazardous chemical vapours and defile environment. In general, different reducing agents such as sodium citrate, ascorbate, sodium borohydride, elemental hydrogen, polyol, Tollen's reagent, N, N-dimethylformamide (DMF) and poly (ethylene glycol)-block copolymers are used in NPs synthesis. The main factor about NPs is its surface to volume ratio, which makes NPs very primitive in the field of technology with specific applications in respective fields like catalysis, adsorption, drug delivery, biotechnology and DNA modelling. NPs are virtualized in its application by its dimension, shape, morphology and size (Vijayaraghavan et al. 2012, Khin et al. 2012, Dimkpa et al. 2012 and Ain et al. 2013) It can be one dimensional (1D), 2D or 3D. NPs used in electronic gadgets and sensing devices are thin-film 1D. 2D carbon nanotubes (CNTs) have more application in the field of catalysis because of its stableness and a high degree of adsorption. Quantum dots and clusters are grouped as 3D NPs. Metal NPs like Ag, Au, Pd, Pt, Zn, Fe are mainly formed from its salt solutions like $AgNO_3$, $AuCl_4$, $PdCl_4$, $PtCl_4$, $ZnSO_4$ by physical and chemical methods. Based on its chemical nature NPs are grouped as metals, metal oxides, silicates, non-oxide ceramics, polymers, organics, carbon and biomolecules. Correct exploitation of ecologically benevolent solvents and nontoxic chemicals are some of the core subjects in the green synthesis approach contemplations. This review implies the importance of green synthesis of metal NPs in a benign greener way following the 12 principles of green chemistry without defiling the environment (Fig. 2).

MATERIALS AND METHODS

Characterization of Metal and Metal Oxide NPs

Techniques used for structural characterization (size, shape, lattice constants and crystallinity) of NPs are X-ray diffraction technique, electron microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), high resolution transmission electron microscopy

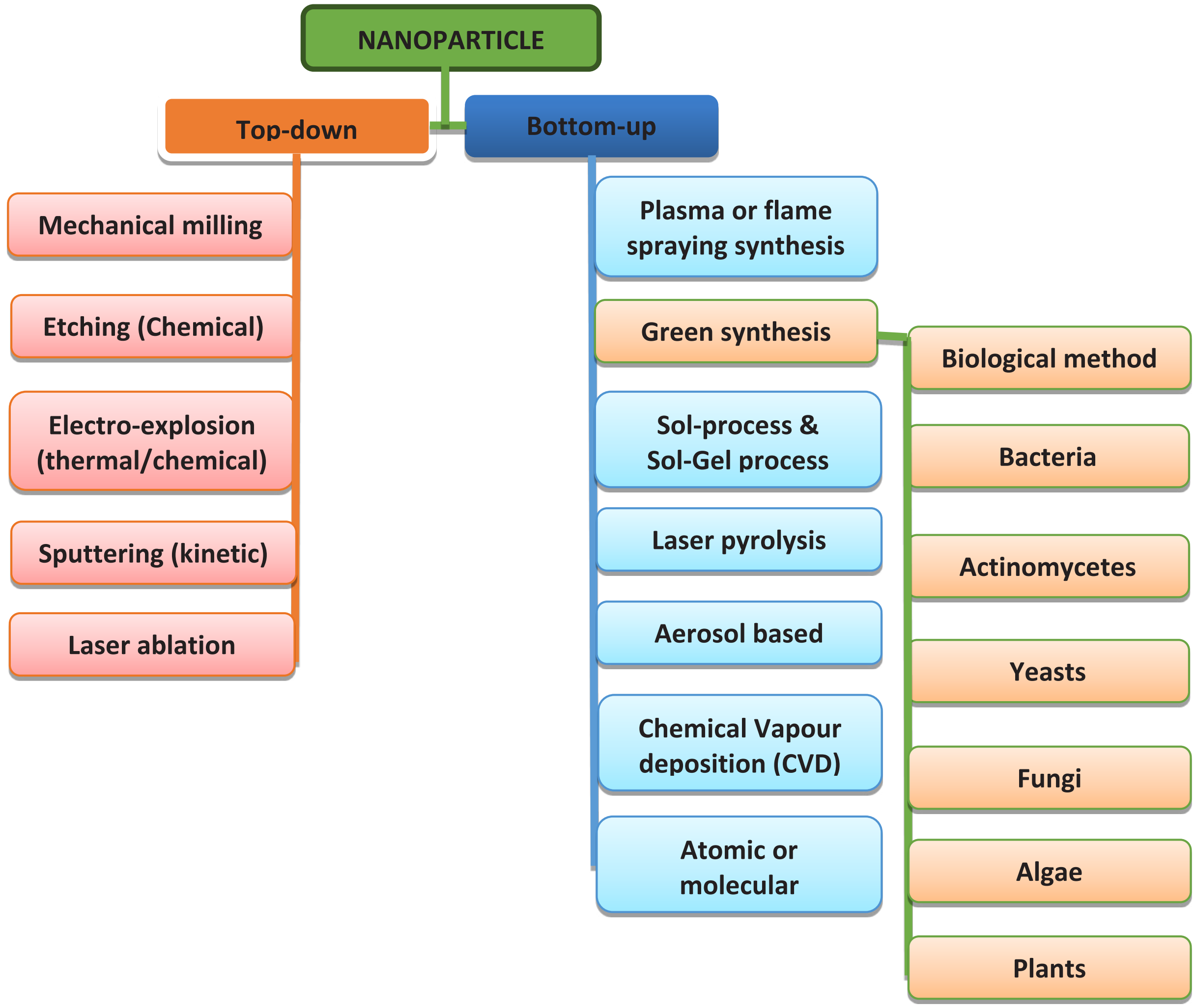

Fig. 1: Various methods of nanoparticles (NPs) synthesis.

Fig. 2: Progression of green chemistry principles over environment deflation.

(HR-TEM), environmental transmission electron microscopy (ETEM) and scanning probe microscopy (SPM). Some internal chemical characterisation techniques used to detect internal chemical constituents of NPs are Fourier transform infrared spectroscopy (FT-IR), energy dispersive x-ray spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS), auger electron spectroscopy (AES) and ultraviolet photoelectron spectroscopy (UPS). Two characterisation techniques for structural and internal chemical characterisation is briefed in this paper.

X-Ray Diffraction Method (XRD)

XRD technique is a handy analytical method to detect crystalline forms of the samples using the equation (1)

$$n\lambda = 2d\sin\theta \qquad \ldots(1)$$

λ = wavelength of X-ray

d = interplaner spacing

θ = diffraction angle

n = 0, 1, 2, 3, ….

X-ray diffractometer operating (Fig. 3) at 40kV and 30mA with 2θ ranging from 20° - 90° with Cu (Kα) radiation (λ = 1.5416 A°). The Kα doublets were well resolved. From Scherrer's formula (2), the crystallite size of the NPs can be deduced.

$$D = k\lambda/\beta \cos\theta \qquad \ldots(2)$$

Where,

k = is a constant

λ = is the X-ray wavelength which equals to 0.15416 nm

β = the full width half maximum intensity (FWHM)

θ = the half diffraction angle

Scanning Electron Microscope (SEM)

SEM is a multipurpose instrument and provides qualitative information of the samples like its topography, morphology, composition and crystallographic arrangements with high resolution (Fig. 4) and produces three-dimensional black and white images with magnification ranging from 20X to approximately 60,000X, spatial resolution of 50 to 100 nm

Fundamental Principles of Scanning Electron Microscopy (SEM)

Incident electron beam sample interactions produce secondary electrons (SE), backscattered electrons (BSE), diffracted backscattered electrons (EBSD), photons, visible light and heat. Secondary electrons are most valuable for showing morphology and topography of samples, backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples to determine crystal structures of orientations and photons are used for elemental analysis (Fig. 5).

Energy Dispersive X-Ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for the elemental analysis of a sample. A high energy beam of electrons or protons or a beam of X-rays is focused into the sample, the number and energy of the X-rays emitted from a specimen can be measured by an energy dispersive spectrometer (EDS).

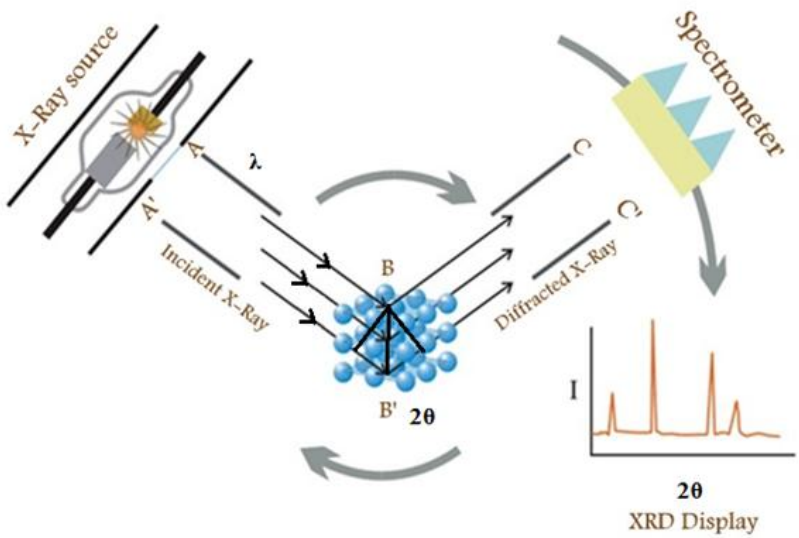

Fig. 3: Bragg's diffraction with XRD display.

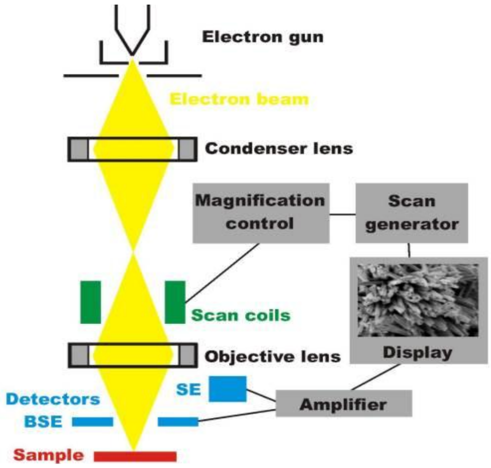

Fig. 4: Instrumentation of scanning electron microscope.

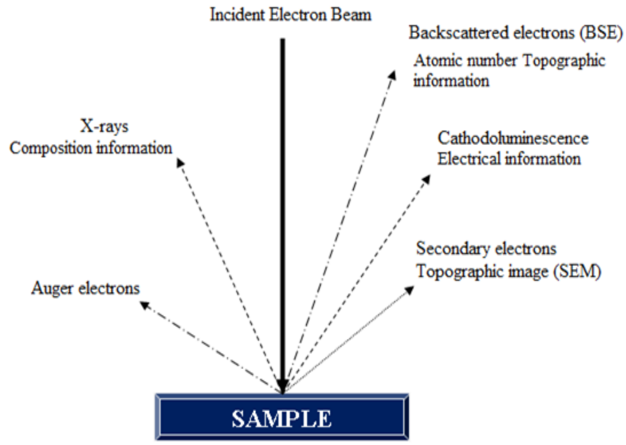

Fig. 5: Fundamental principles of scanning electron microscopy.

Fourier Transform Infrared Spectroscopy (FT-IR)

Sample for FT-IR analysis is mixed with solid KBr pellets uniformly and compressed on a thin transparent film. To determine the functional group of the sample, the compressed thin film is kept in the chamber of the instrument in the scanning range of 4000-400cm^{-1} at a resolution of 4cm^{-1}.

Working principle of FT-IR: Fig. 6 represents the working principle of FT-IR. The instrumentation is as follows.

***The source*:** Glowing black body source emits the infrared energy. This radiation is then allowed to pass through the aperture and then to the sample and detector. The resulted beam undergoes special encoding in the interferometer. This exists as interferogram signals.

***The sample*:** Depending on the type of analysis, the sample may transmit or reflect the interferogram signals in the sample compartment.

***The detector* :** The detectors are specially designed in a way to detect the interferogram signals from the sample compartment.

***The computer*:** The computer converts the measured digitalized signals as spectral lines (Fig. 6). It is then observed by the observer.

RESULTS AND DISCUSSION

Green Synthesis of Metal and Metal Oxide NPs

NPs produced by plants are more steadfast, cost-effective and quiet novel and have fascinated researchers in the field of production of engineered nanomaterials with application in catalysis, adsorption, drug delivery, biotechnology, DNA modelling and have a progression over chemical procedures. In this method, there is no need to use high pressure, energy, temperature and toxic chemicals. Even though bacteria and fungi are used for the synthesis of NPs but the use of plant extracts trim down the cost and do not require any special culture preparation and isolation techniques. Though there are many chemical and physical methods, green synthesis of

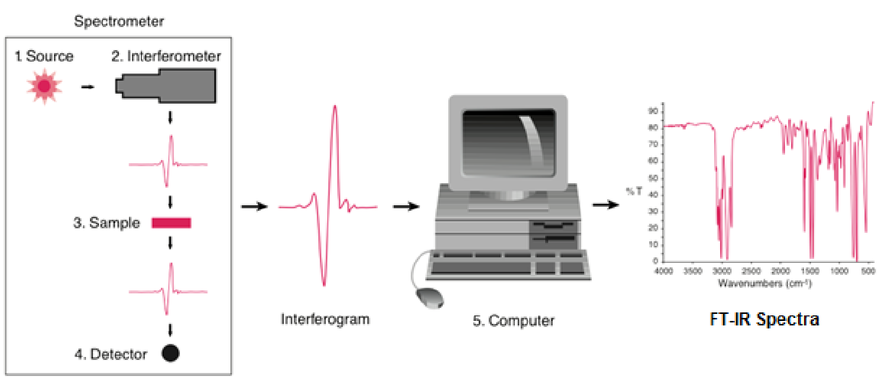

Fig. 6: The interferometer to detect functional group.

nanomaterial is a most promising method depends only on the plant source and organic compound in the crude leaf extract. Green synthesis offers novel challenges for the reduction of environmental pollution. Plants are potent biochemists and have components of phytochemicals acting as natural antioxidants since time immemorial. Presently there has been an augmented interest globally to identify antioxidant compound from plant-derived secondary metabolites which include phenols, tannins, carbohydrates, saponins, flavonoids, amino acids, proteins and polysaccharides (Subbarao et al. 2013, Vijayaraghavan et al. 2010). These molecules prevent agglomeration by acting as reducing and stabilizing agents to synthesis nanoparticle in a benign eco-friendly method that are potent and have no side effect in synthesizing NPs (Table 1).

The metal ions in the salt solution are reduced by the antioxidants or phytochemicals present in the plant extract to make the first phase of formation of NPs more active and leads to the growth and stabilization of NPs by nucleation in the second phase. After nucleation, the so formed NPs are capped and stabilized by the phytochemicals to exhibit various shapes like cubes, spheres, triangles, hexagons, pentagons, rods and wires (Fig. 7). It has also been reported that the amount of extract, pH, time taken and concentration of metabolites gives distinct morphology to NPs.

Cinnamomum zeylanicum leaf extract was used to synthesize crystalline palladium NPs of 15 to 20 nm (Smitha et al. 2009). Cubic shaped silver, nanoparticles of 15 to 500nm, have been premediated from the leaves of *Diospyros kaki* (Song et al. 2010), *Magnolia kobu*, *Carica papaya*. Fruits of *Tanacetum vulgare*, *Emblica officinalis*, *Psidium guajava* produces spherical silver, gold NPs of 16nm, 15 to 25nm, 25 to 30nm. There have been reports (Fig. 8) in the synthesis of silver, gold, zinc, platinum, palladium, indium oxide and zinc oxide NPs using leaf, stem, stem bark, latex, fruit, pulp, seed, root and rhizome extracts of *Aloe barbendis* (Sangeetha et al. 2011), *Azadirachta indica*, *Stevia rebaudiana*, *Calotropis procera*, *Pelargonium graveolens*, *Cuminum cyminum* (Kumar et al. 2010), *Emblica officinalis*, *Tanacetum vulgare*, *Magnolia kobus*, *Coriandrum sativum*, *Jatrapha curcas*, *Cycas, Eucalyptus camaldulensis*, *Cymbopogon flexuosus*, *Oscimum sanctum, Rhizopus nigricans*, *Acalypha indica*, *Zingiber offcinale* and *Musa paradisiaca*.

Table 1: The Taxonomy and phytoconstituents of plants.

S. No	Plant Species	Botanical Name	Family	Vernacular Name	Stabilising and Complexing Phyto-constituents	Plant parts	Traditional use	Earlier Reported NPs & References
1.		*Ocimum sanctum*	Lamiaceae	Tulsi	Flavonoid, Amino acid, Steroid, Protein and Alkaloid	L	Controls diabetes, blood cholesterol, effective in Fever, Cough and Bronchitis.	Silver and Zinc Oxide (Singhal et al. 2011 & Gnana et al. 2015)
2.		*Acalypha Indica*	Euphorbiacea	Kuppaimenii	Terpenoid, Amino acid, Glycoside, Steroid, Fixed oil, Protein and Alkaloid	E	Antibacterial Emetic, Expectorant used in Bronchitis, Asthma, Pneumonia	Silver and Zinc Oxide (Krishnaraj et al. 2010 & Gnana et al. 2014)
3.		*Emblica officinalis*	Euphorbiacea	Amla	Steroid, Flavonoid, Glycoside, Saponnin, Fixed oil, Protein and Alkaloid	A	Restorative, Stomachic, Alterative, Anti-inflammatory used to treat Peptic ulcer	Silver Gold and Zinc Oxide (Ankamwar et al. 2005 & Gnana et al. 2014)
4.		*Azadirachta indica*	Meliaceae	Neem	Amino acid, Terpenoid, Flavonoid, Glycoside, Steroid, Protein and Alkaloid	V	Anthelmintic, Antifungal, Anti-Diabetic, Antibacterial, Antiviral and Sedative.	Silver, Zinc Oxide and Gold (Gnana et al. 2015 & Shiv et al. 2004)
5.		*Corriandrum sativum*	Apiaceae	Corri-an-der	Alkaloid, Terpenoid, Flavonoid, Amino acid, Glycoside, Steroid, Fixed oil and Protein	E S	Analgesic, Carminative, Digestive, Diuretic and Sedative	Silver and Zinc Oxide (Gnan et al. 2014)

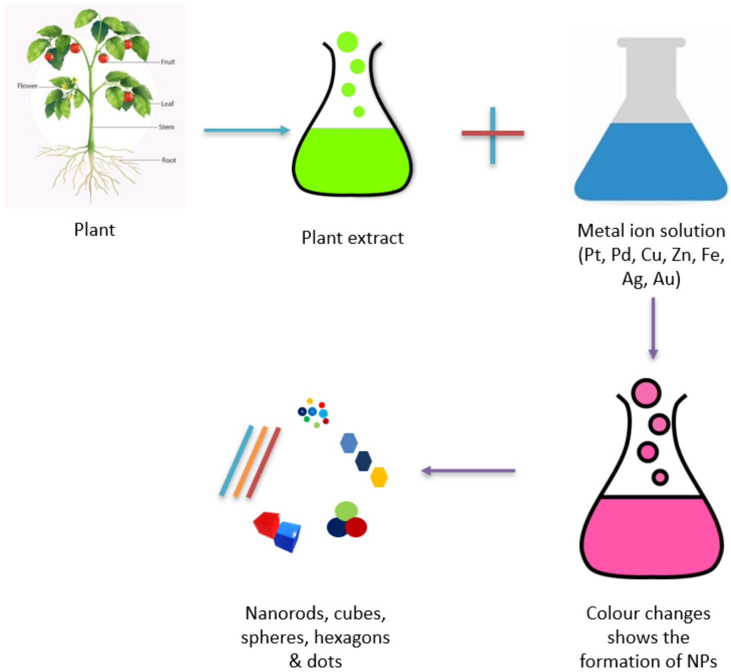

Fig. 7: Schematic representation of green synthesis of NPs.

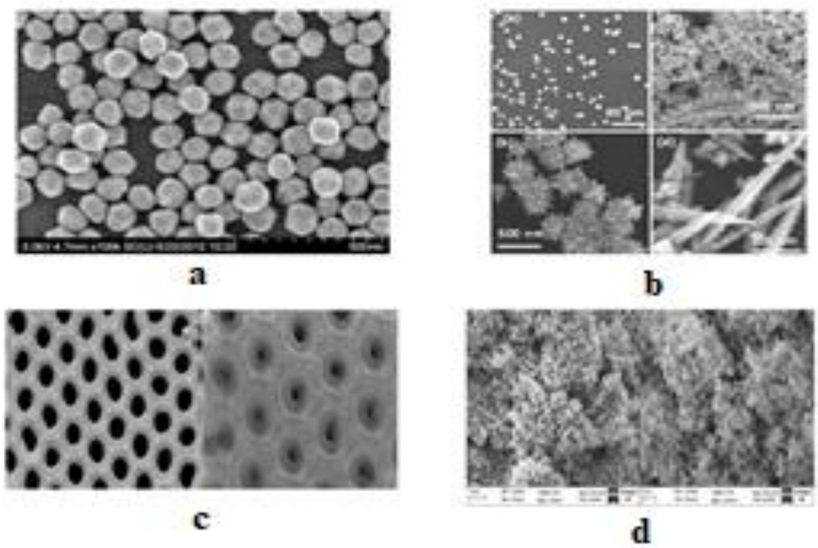

Fig. 8 a, b, c and d: SEM image of zinc oxide, gold and palladium NPs.

Smitha et al. (2009), reported gold nanoprisms with fcc (111) at low concentrations and nanospheres at high concentration using *Cinnamomum zeylanicum* leaf broth as the reducing agent. Crystallinity and morphology of the foresaid nanoprisms of fcc (111) and spheres were characterized by XRD, TEM and SEM. Silver NPs synthesized using leaf extract of *Acalypha indica* by Krishnaraj et al. (2010), shows the formation of NPs within 30 min. The size of the silver NPs was confirmed by high-resolution transmission electron microscopy as 20-30 nm. Song et al. (2009), testified Au, Ag and Au-Ag NPs with fcc (111) of spherical and hexagonal morphology with 20-150 nm in water using the extract of *Volvariella volvacea* by extracellular synthesis method. FT-IR proves (Fig. 9) that biomolecules responsible for capping and efficient stabilization for Au, Ag and Au-Ag nano prisms are proteins and amino acids.

In addition, Ravindra et al. (2011), reported zinc oxide NPs of 5-40 nm with spherical and granular nature using *Calotropis procera* latex. Petla et al. (2012), reported that TEM images of the palladium NPs of 15 nm confirmed that proteins and amino acids of soybean extract reduced palladium ions. Owing to the excitations of surface plasmon vibrations, steady change in colour from transparent orange (Fig. 10b) to dark brown (Fig. 10c) occurs when soybean leaf extract is added into palladium ion solution. The appearance of brown colour indicates the formation of palladium NPs.

Mallikarjuna et al. (2011) synthesised silver NPs of 20-3nm using leaf broth of *Ocimum sanctum* as reductant and stabilizer with aqueous silver nitrate. Titanium dioxide NPs of 100 to 150nm from titanium isopropoxide solution using N*yctanthes* leaves extract was focused by Sundrarajan et al. (2013). Gnana et al. (2013), fabricated ZnO nanosized flakes from *Ocimum sanctum* with a crystalline size of 81nm. Padil et al. (2013), reported CuO nanoparticles with gum karaya and discovered its probable antibacterial application. Synthesised palladium NPs by Kalaiselvi et al. (2015) at 60°C with *Catharanthus* plant leaves and (PdOAc) have good wastewater remediation property. The shape of the NPs is spherical with a size of 38nm. Additionally, a researcher reported that aqueous solution of *Cochlospermum gossypium* (Gum kodagogu) is used in the development of platinum NPs which was very stable in its synthesis and utility. Aqueous flower extract of *Calotropis gigantea* is used to synthesise TiO_2 NPs with $Ti(OH)_2$ as a predecessor. The mixture of the foresaid was kept in magnetic agitation for a period of 6 h. The resulted mixture is ultrasonicated for 30 min to pull out the agglomerates. Particle size variation of 160 to 220nm is confirmed by SEM micrographs, EDX and XRD (Fig. 11 and Fig. 12).

Hematite (a-Fe2O3) NPs synthesized from green tea (*Camellia sinensis*) leaf extract shows four times higher

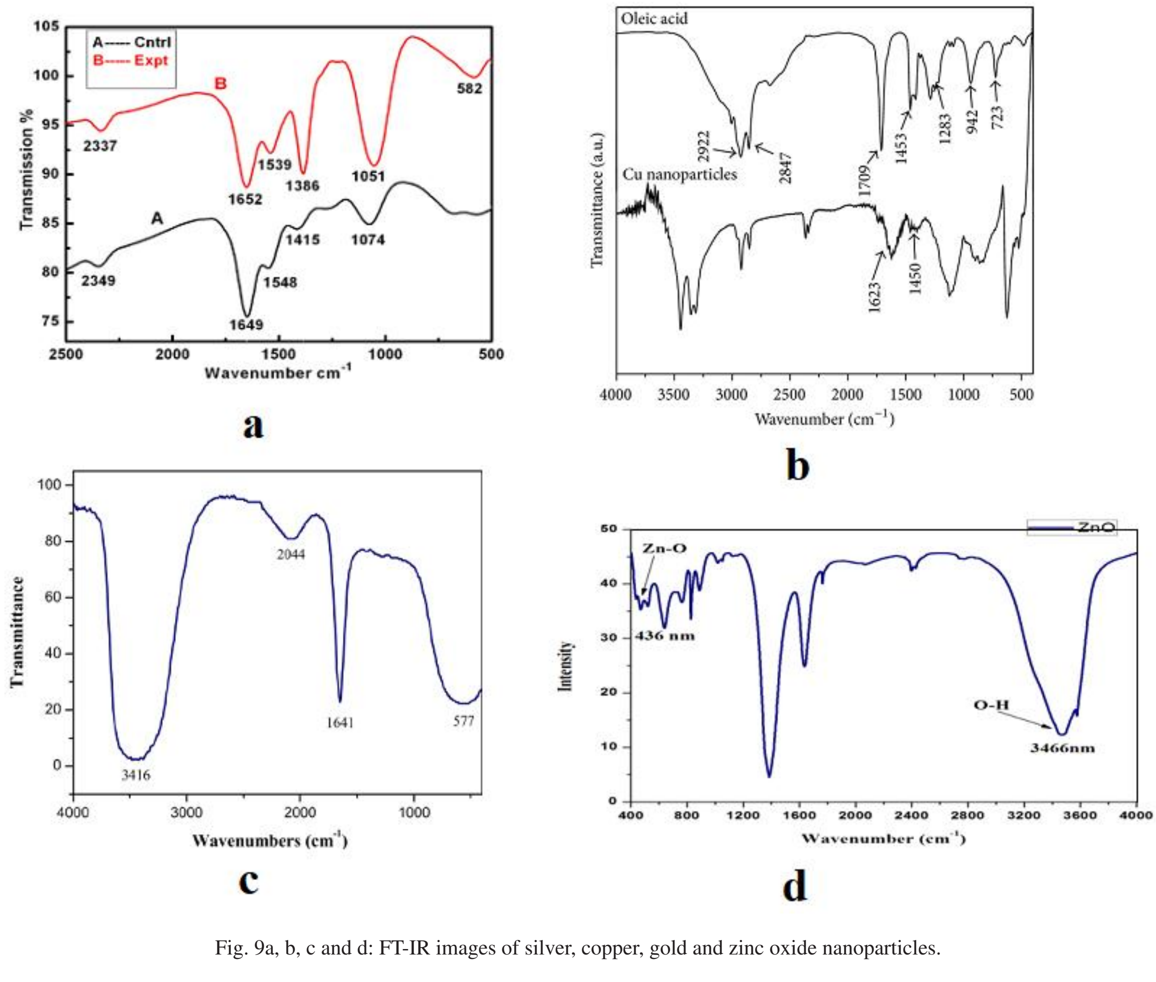

Fig. 9a, b, c and d: FT-IR images of silver, copper, gold and zinc oxide nanoparticles.

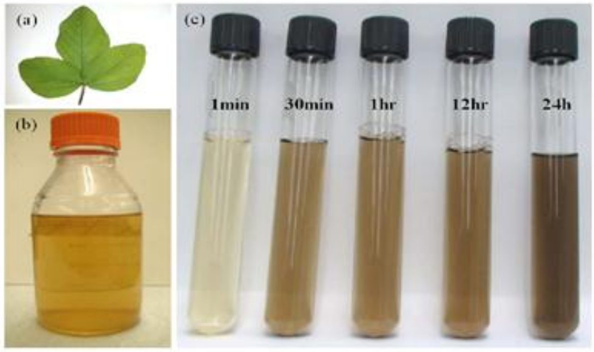

Fig. 10: Photograph of (a) soy leaf; (b) soy leaf extract; and (c) reaction mixture at different time intervals.

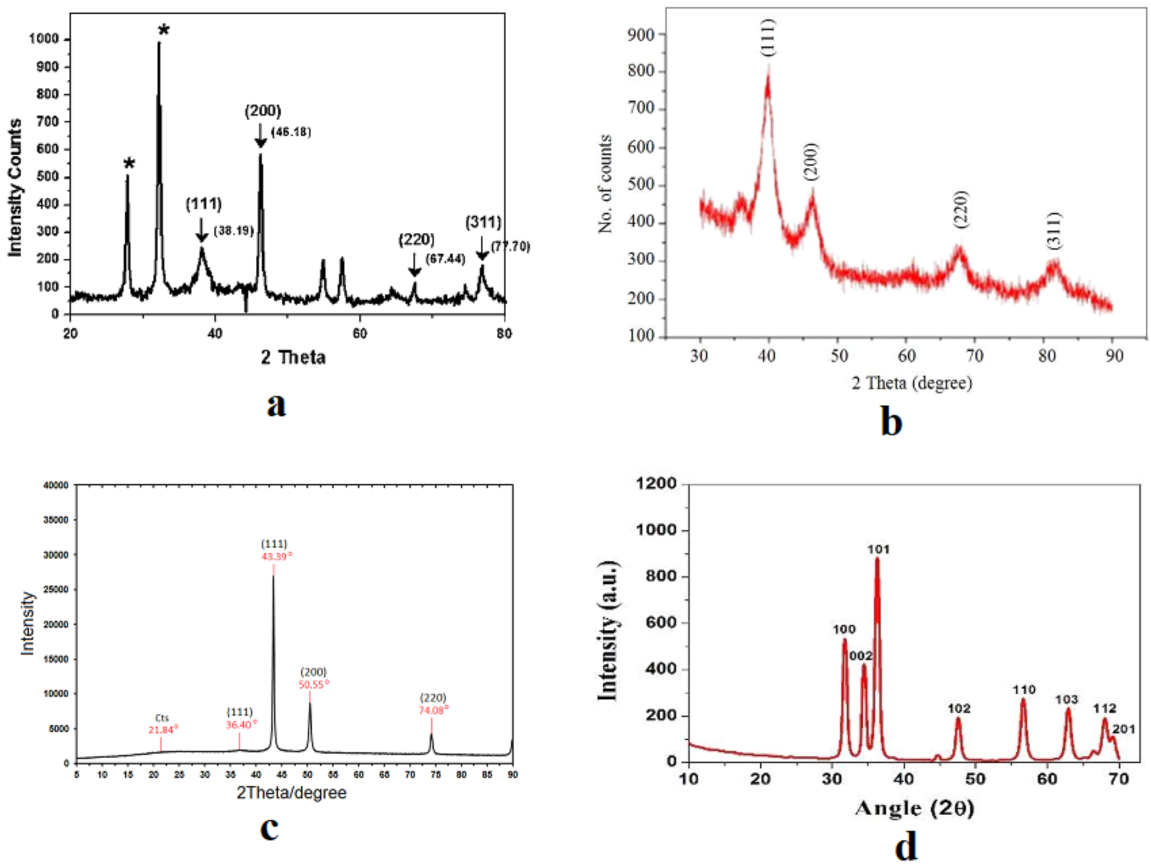

Fig. 11a, b, c and d: XRD images of silver, platinum, copper and zinc nanoparticle.

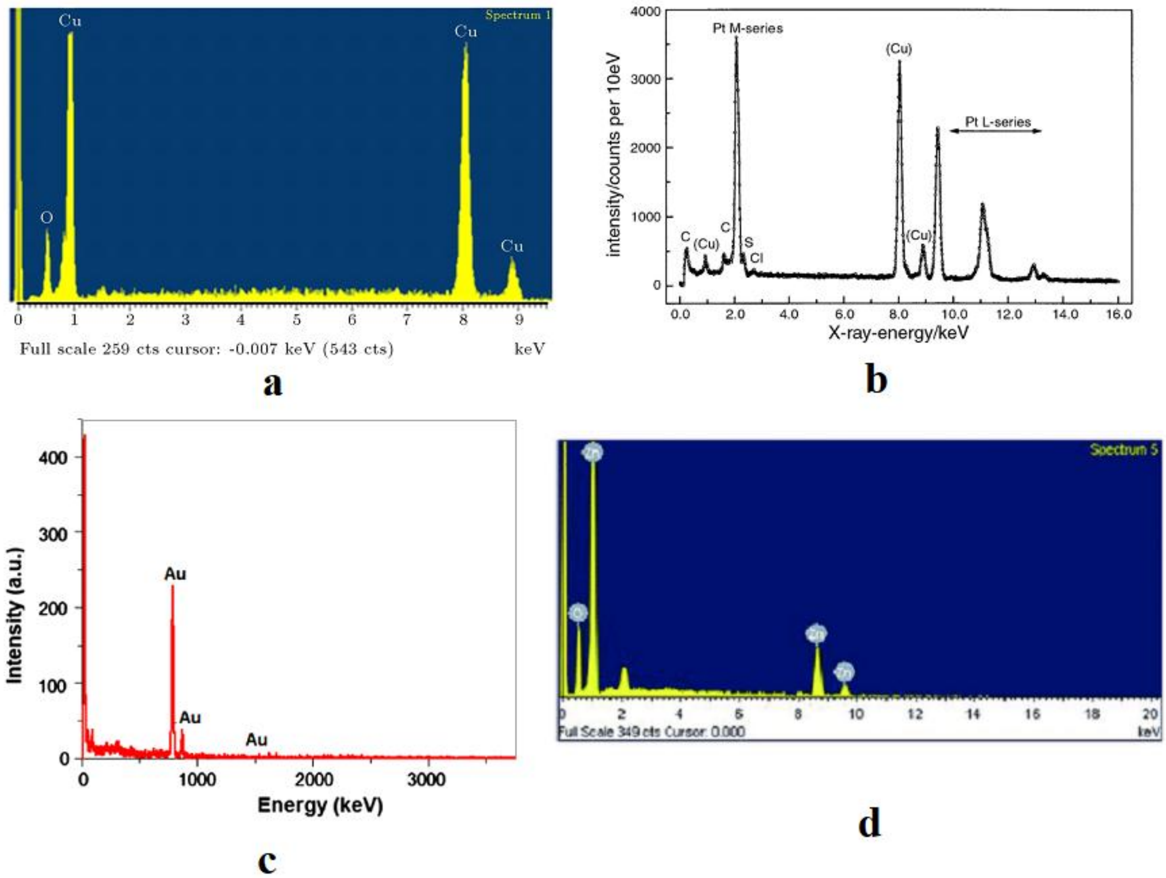

Fig. 12a, b, c and d: EDX images of copper, platinum, gold and zinc oxide nanoparticles.

surface area and two times higher photocatalytic activities (Santhoshkumar et al. (2014)). When applied in a wet-type solar cell the same has enhanced photoelectrochemical activity. Sheny et al. (2012) synthesised PdNPs from dried leaf powder of *Anacardium occidentale*. So formed PdNPs is confirmed with UV-Vis spectrophotometer. Proteins bound to PdNPs are confirmed by FT-IR. The crystallinity, irregularity and rod shape has been confirmed by TEM images. Size of the synthesized nanoparticles was influenced by the quantity of dry leaf powder used. PdNPs exhibited good catalytic activity in the reduction of aromatic nitro-compound. Pb NPs of 47 nm synthesised by a researcher using *Cocos nucifera* with $Pb(COOH)_2$ at 37°C shows the great ability for the absorption of the carcinogenic dye. Mohammed Rafi Shaik et al. (2017) reported palladium NPs using aqueous extract of aerial parts of *Origanum vulgare* L. (OV) which is well known as a rich source of phenolic components and the same is accountable not only for the reduction and progression of NPs, but also to stabilize it. Synthesised ZnO NPs of 9 nm to 18 nm by Saad S M Hassan et al. (2015) show high photocatalytic activity for the degradation of anthracene using zinc acetate as a precursor with *Coriandrum sativum* leaf extract. In a research, Barbosa et al. (2020) authenticated that zinc oxide NPs prepared using madar (*Calotropis procera*) latex and *Ipomoea pes-caprae* showed 40 nm and 20 nm of spherical shape. Table 2 summarizes the abundance of NPs from plant extracts.

CONCLUSIONS

Rigorous agricultural practices, swift development and progressively erudite existences have loaded chemicals into the environment in all forms. Green NPs due to their special features scored many researchers worldwide as it is benign and have extensive applications in the field of medicine agriculture and water remediation. In this work metal and metal oxide nanoparticles like silver, gold, copper, platinum, titanium, zinc and palladium from various parts of leaves, fruits, seeds, barks, root, stem, pulp and rhizome were reported. As benign NPs are nontoxic, it can be used as a novel material to degrade environmental pollutants in multiple ways. These

Table 2: Green NPs from plant leaves, seeds, fruits, barks and roots.

S . No.	Plants	Parts	Nano-particles	Size (nm)	Shapes	References
1.	*Cinnamomumzeylanicum*	Leaves	Au	25	Spherical Prism	Sathishkumar et al. (2009)
2.	*Magnolia kobus*	Leaves	Ag	15-500	Cubic	Song et al. (2009)
3.	*E. coli DH*	E.Coli	Au	8-25	Spherical	Du et al. (2007)
4.	*Glycine max*	Leaves	Pd	15 nm	Spherical	Petla et al. (2012)
5.	*Eucalyptus camaldulensis*	Leaves	Au	5.5–7.5	Crystalline	Ramezani et al. (2008)
6.	*Ocimum sanctum*	Leaves	ZnO	81nm	Flakes	Gnana et al. (2015a)
7.	*Carica papaya*	Fruit	Ag	15 nm	Cubic	Jain et al. (2009)
8.	*Tanacetum vulgare*	Fruit	Ag	I6nm	Spherical	Dubey et al. (2010)
9.	*Jatropha curcas*	Seed	Ag	15-50	Spherical	Bar et al. (2009)
10.	*Rhizopus nigricans*	Black Bread Mold (Fun-gi)	Ag	35-40	Round	Ravindra et al. (2014)
11.	*Pinus resinosa*	Bark	Pt	6–8	Irregular	Coccia et al. (2012)
12.	*Pinus resinosa*	Bark	Pd	16–20	Spherical	Coccia et al. (2012)
13.	*Cinnamomum zeylanicum*	Bark	Pd	15–20	Crystalline	Sathishkumar et al. (2009)
14.	*Zingiber offcinale*	Rhizome	Ag	6–20	Spherical	Kumar et al. (2012)
15.	*Ocimum sanctum*	Root	Ag	5-10	Spherical	Singhal et al. (2011)
16.	*Pelargonium graveolens*	Leaves	Au	20–40	Decahedral, Icosahedral	Shankar et al. (2003)
17.	*Penicillium brecompactum*	Mould Sp.	Ag	23–105	Crystalline Spherical	Shaligram et al. (2009)
18.	*Diopyros kaki*	Leaves	Ag	15-500	Cubic	Song et al. (2010)
19.	*Syzygium cumini*	Seed	Ag	73–92	Spherical	Kumar et al. (2010)
20.	*Acalypha indica*	Leaves	ZnO	80	Cubes	Gnana et al. (2014a)
21.	*Syzygium cumini*	Seed	Ag	3.5	---	Banarjee et al. (2011)
22.	*Emblica officinalis*	Leaves	ZnO	16	Rods	Gnana et al. (2014b)
23.	*Emblica officinalis*	Fruit	Au	15-25	--	Ankamwar et al. (2005)
24.	*Cinnamomum zeylanicum*	Bark Powder	Ag	31–40	Spherical	Sathish kumar et al. (2009)
25.	*Boswellia ovalifoliolata*	Stem Bark	Ag		Spherical	---
26.	*Verticillium*	Fungi	Ag	21–25	Spherical	Mukherjee et al. (2001)
27.	*Shoreatum buggaia*	Stem Bark	Ag		Spherical	Savithramma et al. (2011)
28.	*Stevia rebaudiana*	Leaves	Au	8–20	Octahedral	Mishra et al. (2010)
29.	*Psidium guajava*	Fruit	Au	25-30	Spherical	---
30.	*Azadirachta indica*	Leaves	ZnO	51	Flowers	Shiv et al. (2004)
31.	*Diospyros kaki*	Leaves	Pt	2–12	Crystalline	Song et al. (2010)
32.	*Azadirachta indica*	Leaves	Au	5.5–7.5	Crystalline	Gnana et al. (2015b)
33.	*Tanacetum vulgare*	Fruit	Au Ag	11 16	Triangular & Spherical	Dubey et al. (2010)
34.	*Ginko biloba*	Leaves	Ag	15-500	Cubic	Song et al. (2010)
35.	*Mangifera indica*	Leaves	Ag	20	Spherical, Triangular, Hexagonal	Phillip et al. (2011)
36.	*Aspergillus niger*	Common Mold Fungi	Ag	20	Spherical	Gade et al. (2008)
37.	*Emblica officinalis*	Fruit	Au Ag	20 25	Triangular	Ankamwar et al. (2005)
38.	*Musa paradisiaca*	Fruit	Pd	50	Crystalline Irregular	Bankar et al. (2010)
39.	*Bacillus cereus*	Bacteria	Ag	20-40	Spherical	---

S. No.	Plants	Parts	Nano-particles	Size (nm)	Shapes	References
40.	*Aloe barbadensis* Miller	Leaves	In_2O_3	5-50	Spherical	Maensiri et al. (2008)
41.	*Cymbopogon flexuosus*	Leaves	Au	200-500	Spherical, Triangular	Shankar et al. (2005)
42.	*Garcinia mangostana*	Frut	Ag	35	Spherical	Veerasamy et al. (2010)
43.	*Syzygium aromaticum*	Fruit	Au	5-100	Irregular	---
44.	*Lactobacillus casei*		Ag	20–50	Spherical	Korbekandi et al. (2012)
45.	*Coriandrum sativum*	Leaves	ZnO	66	Composites	Gnana et al. (2015c)
46.	*Cuminum cyminum*	Seed	Au	1–10	Spherical	Krishnamurthy et al. (2011)
47.	*Aloe barbadensis* Miller	Leaves	Au Ag	10-30	Spherical, Triangular	Chandran et al. (2006)

metal oxide NPs also has considerable attention due to its UV filtering properties. So the exploration of the plant systems as the potential nano factories has heightened interest in the biological synthesis of nanoparticles.

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