SYNTHESIS OF IRON OXIDE NANOPARTICLES USING PLANT MATERIAL

Project submitted to University of Kerala in partial fulfilment of the Requirement for the Degree of Bachelor of Science in Chemistry

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DECLARATION

We hereby declare that this project titled '**Synthesis of iron oxide nanoparticles using plant material**'is the bonafide record of the project work carried out by us under the supervision and guidance of Dr.Beena Kumari KS ,Assistant Professor ,Department of Chemistry ,All Saints' College ,Thiruvananthapuram ,and that no part of the report has been submitted by us for any other degree, diploma or similar titles of any other university.

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This is to certify that this project report titled **"Synthesis of Iron Oxide Nanopartiles Using Plant Material"** submitted by Sreedevi K M, Sreelekshmi P, Viji Nicotious, Abhirami P V and Alia Ayoob S is a bonafide record of the project work carried out by them under my supervision and guidance and that no part of the report has been presented for any other degree, diploma or any other similar title of any other University.

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SYNTHESIS OF IRON OXIDE NANOPARTICLES USING PLANT MATERIALS

1. INTRODUCTION

Nanomaterials are the keystone of nanotechnology and a nano science. Nano science and nanotechnology are the wide area of research and development activity that has been growing explosively worldwide from the last few years. It has the potential for developing the way in which material and products are generated and range and nature of functionality that can be accessed. Nanomaterial can be defined as a set of substance where one dimension is at least less than around hundred nanometres. The prefix "nano" has found in last decade. An ever-increasing application to different field of knowledge. Nano means extremely small or dwarf. Nanoparticle are particle which lie in the dimension between one to 100 nm. Nanomaterials are of interest because at this scale unique magnetic, electric and other properties. The emergent properties have the great impact in industry, machinery and in other fields.

We cannot see the nanoparticles through our naked eye. Bulk materials are particles that have their size above 100 nm in all dimensions. We can see their particles through naked eyes. The material properties of nano structure are different from the bulk due to high surface area over volume ratio and the possible appearance of quantum effects at the nano scale. Nano materials have several advantages over bulk materials, such as the huge surface to volume ratio, very high porosity and the completely different physiochemical properties. The physical and chemical properties of nano material are typically differed significantly from the corresponding coarser bulk material. These nanomaterials have various shapes and structures such as spherical, needle like, platelets etc. Chemical composition is another important parameter for the characteristics of nanomaterials, which comprise nearly all substance examples metals,polymers, metal oxide, compounds as well as biomolecules. Under ambient conditions nanoparticles tend to stick together and form aggregates or agglomerates.

Nanomaterials have a number of advantages due to its physical, chemical, mechanical and other properties due to fine grain size, superior form ability and potential super plasticity, possess high strength, toughness, and ductility, possess enhanced activity, reduced thermal conductivity, dispersion can be utilized further increase strength, reduce energy costs etc.

Metal nanoparticles (NPs) are widespread used in various scientific fields including biochemistry, optics, and plasmonics, but specially employed in heterogeneous catalysis for maximizing the exposed area of a metal catalyst, typically a rare, expensive late transition metal. In the metal NPs, chemical reactivity is intimately linked to their elementary composition, atomic arrangement, shape, and size, a variety of features whose role is often intertwined. Theoretical simulations on proper NP models, employing efficient computational chemistry algorithms, and parallel computer codes provide the necessary detailed atomistic knowledge of the specific structure and reactivity of metal NPs, paving the way for the design of NPs with a desired chemistry. Metal nanoparticles are nano materials that are made up of only one element. Individual atoms or groups of numerous atoms can exist. Au, Ag, Pt, Cu, Pd, Re, Zn, Ru, Co, Cd, Al, Ni, and Fe are some of the most commonly produced nanoparticles.

Simple approaches, such as bio-assisted method, hydrothermal method, and microwave-assisted method, are used to make metal nanoparticles in the form of colloidal fluids or solid nanoparticles. They have remarkable properties including localized surface plasmon resonance (LSPR), high reactivity, and broad electromagnetic spectrum absorption. Metal nanoparticles are very fascinating materials for a variety of practical applications due to their improved optical, optoelectrical, catalytic, antimicrobial/cancer/viral characteristics.

Metal nanoparticles such as gold nanoparticles (AuNPs), silver nanoparticles, and cysteine-capped silver nanoparticles (CysAgNPs) were synthesized using the reducing ability of EABs in the presence of the organic substrate such as an electron provider. EAB used oxides of metal as an electron acceptor for different metal nanoparticle synthesis where oxides were used as a precursor. Sodium acetate was bio electrochemically oxidized on a support (stainless steel) which generates electrons to reduce precursors of nanoparticles to form different metal nanoparticles. The presence of a conductive support such as stainless steel provides an electron-rich environment to convert metal ions to metal nanomaterial having zero valences. Furthermore, these synthesized nanoparticles were utilized for different applications such as biohydrogen production and antimicrobial activity .There are several methods for creating nanoparticles, including co-precipitation , hydrothermal synthesis, inert gas condensation, ion sputtering scattering, micro-emulsion, microwave, pulse laser ablation, sol-gel, sonochemical, spark discharge, template synthesis, and biological synthesis. . Co-precipitation technique involves the precipitation of metal in the form of hydroxide from a salt precursor with the help of a base in a solvent. The controlled release of anions and cations assists to regulate the nucleation and particle growth kinetics, which helps to synthesize mono dispersed nanoparticles

Hydrothermal synthesis is one of the most commonly used methods for preparation of nanomaterials. It is basically a solution reaction based approach. In hydrothermal synthesis, the formation of nanomaterials can happen in a wide temperature range from room temperature to very high temperatures. Hydrothermal synthesis is commonly used to grow synthetic quartz, gem sand other single crystals with commercial value.

Biological synthesis involves the synthesis of nanoparticles by using plant extract and microorganisms such as bacteria and fungi. Phytonanotechnology has shown a new field for the synthesis of nanoparticles which is eco-friendly, simple, cost effective. Scalability, bio-compatibilty and use of universal solvent(water) as reducing agents are advantages of phytonanotechnology. Phytonanotechnology use plants for synthesis of nanoparticles .Nanoarticles are synthesizes using of plant such as root, fruit, stem, seed and leaf. The exact mechanism for synthesis of nanoparticles using plant remain to be elucidated. It has been illustrated that organic acid, proteins vitamins and secondary metabolites such as alkaloids, flavonoids, terpenoids, polysaccharides and heterocyclic compound are responsible for synthesis of various types nanoparticles. Microorganism is considered as nano factories that hold wide potential as cost effective tool, which is ecofriendly and avoid toxic reductants. Enzymes present in the microorganism have ability to accumulate and detoxify heavy metals. These reductase enzymes play vital role in reduction of metal salt into the nanoparticles. The present study focused on the preparation of iron oxide nanoparticles from neem leaf extract. Basically, this process involves the precipitation of Fe^{2+} and Fe^{3+} from aqueous salts solutions (e.g., chlorides, sulphates, and nitrates) by addition of a base (e.g., NaOH).

2. OBJECTIVES

Nanotechnology found numerous applications in very important areas of development. Synthesis of a nanomaterial having wide range of applications in a low cost method is of great achievement. In the present work, preparation of a nanomaterial using ecofriendly and economically viable method is applied. Its characterization and application also planned to carried out as part of this work.

3. REVIEW

A brief over view of the antibiotic era,beginning from the discovery of first antibiotics until the present day situation was discussed in literatures [1]. Magnetic nanoparticles exhibit many interesting properties that can be exploited in a variety of applications such as catalysis and in biomedicine [2]. Nanocubes of α Fe_2O_3 were successfully synthesized by aqueous polymer thermolysis method in air [3]. Nanoparticle-metal oxide and gold represents a new class of important materials that are increasingly being developed for use in research and health related activities [4]. The DNA degradation potential and anti-cancer activities of copper nanoparticles of 4-5 nm size are reported [5]. The low rate of resistance development against NPs because of its multiple mode of action has contributed to its increased acceptance in clinical settings

[6]. The effect of the concentration and MW of chitosan were investigated, respectively, and the antimicrobial mechanism was discussed [7].

An in vitro demonstration of gold nanorods as novel contrast agents for both molecular imaging and photothermal cancer therapy was shown in literature [8]. Stable Ag nanoparticles were prepared and their shape and size distribution characterized by particle characterizer and transmission electron microscopic study [9]. The following study examines the feasibility of nano shell assisted photo-thermal therapy (NAPT) [10]. The synthesis of metal oxide nanoparticles is described in terms of precursor formation, nucleation, growth, and aging processes [11]. One approach that shows immense potential is based on the biosynthesis of nanoparticles using biological microorganisms such as bacteria [12]. The development of reliable, eco-friendly processes for the synthesis of nanomaterials is an important aspect of nanotechnology today [13]. This important book focuses on the synthesis and fabrication of nanostructures and nanomaterials, but also includes properties and applications of nanostructures and nanomaterials, particularly inorganic nanomaterials [14]. Chemical processes provide a diverse array of valuable products and materials used in applications ranging from health care to transportation and food processing [15]. Recent advances in a variety of biomedical applications of nanostructured materials was shown in literature [16]. Biosynthesis of a large variety of nanomaterials using different biological resources among which algae-based entities have been gaining much more attention within the community of material scientists worldwide [17]. Green synthesis of nanoparticles that have environmentally acceptable solvent systems and eco-friendly reducing agents is of great importance [18]. Selenium is an essential trace element and is an essential component of many enzymes without which they become inactive [19]. green reduction of graphene oxide (GRO) using various natural materials, including plant extracts, has drawn significant attention among the scientific community [20]. The ability of selected strains of cyanobacteria and micro algae to biosynthesize silver nanoparticles (Ag-NPs) by using two procedures; (i) suspending the live and washed biomass of micro algae and cyanobacteria into the $AgNO_3$ solution and (ii) by adding $AgNO_3$ into a cell-free culture liquid was studied [21]. Silicon nanowire possesses great potential as the material for renewable energy harvesting and conversion [22]. Selenium nanoparticles (SeNPs) are gaining importance in the field of medicine owing to their antibacterial and anticancer properties [22]. Intracellular synthesis of gold nanoparticles, as well as extracellular formation of nanoparticles in the presence of fungal cell extract has been successfully demonstrated [23]. Synthesis of NPs constantly forming an impute alternative for conventional chemical and physical methods [24]. over the past several years, plants, algae, fungi, bacteria, and viruses have been used for production of low-cost, energy-efficient, and non toxic metallic nanoparticles [25]. In situ generated Ru NPs in [DAMI][NTf_2] ionic liquid was found highly active in terms of formic acid formed during the CO_2 hydrogenation reaction to other ionic liquid immobilized standing Ru NPs [26].

In recent years, the development of efficient green chemistry methods for synthesis of metal nanoparticles has become a major focus of researchers. They have investigated in order to find an eco-friendly technique for production of well-characterized nanoparticles. One of the most considered methods is production of metal nanoparticles using organisms. Among these organisms plants seem to be the best candidates and they are suitable for large-scale biosynthesis of nanoparticles[27].

The SCF possesses both gaseous and liquid properties. Whereas gas-like diffusivities and low viscosity coefficients allow the fluid to permeate through porous solids more quickly than a pure liquid could, liquid-like densities and dissolving powers allow the SCF to function as an effective reaction solvent.[28]

Present study focuses on the green synthesis of ZnO nanoparticles by zinc nitrate and utilizing the bio components of leaves extract of Calotropis gigantea. The ZnO nano crystallites of average size range of 30-35 nm have been synthesized by rapid, simple and ecofriendly method. Zinc nanoparticles were characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The particles obtained are spherical in nature and are agglomerates of nanocrystallite.[29]

Zinc oxide nanoparticles have fascinated meticulous research interest because of its significant applications in the field of medicine, pigment electronics, spintronics and piezoelectricity. The biogenic invention of zinc oxide nanoparticle is a better option due to ecofriendliness. Aqueous leaf extract of Acalypha indica were used to synthesis zinc oxide nanoparticles not only in the laboratory scale, but also in their natural environs. This green synthesis approach shows that the environmentally benign and renewable aqueous leaf extract of Acalypha.[30]

This work is to report an environmental benign route for the fabrication of copper oxide nanoparticles using Centella asiatica (L.) leaves extracts at room temperature. This method is completely a green method, free from toxic and harmful solvent.

These NPs can reduce methyl orange to its leuco form in aqueous medium in the absent of reducing agents. It is more economy as compare to other methods. This catalytic effect of copper oxide nanoparticles can be contributed to its small size.[31]

Nanoparticles have many active sites as compared to the bulk materials because of its large surface to volume ratio. Copper oxide nanoparticles such prepared has good catalytic properties. Indium oxide (In_2O_3) nanoparticles using Aloevera plant extract: Synthesis and optical properties. Recently, investigations on preparation of In2O3 nanostructures with various forms such as nanotubes [15], nanobelts [16-18], nanofibers [19, 20], wires [21-28], and nanoparticles [29-31] have been widely emphasized to extend their technological applications. Among these nanostructures, In_2O_3 with nanoparticulate form has been intensively studied to be used as a promising material for gassens or applications.[32]

Zinc oxide nanoparticles are known to be one of the multifunctional inorganic nanoparticles with effective antibacterial activity. This study aims to determine the antimicrobial efficacy of green and chemical synthesized ZnO nanoparticle against various bacterial and fungal pathogens. In addition,the current study has clearly demonstrated that the particle size variation and surface area to volume ratio of green ZnO nanoparticle are responsible for significant higher antimicrobial activity. From the results obtained it is suggested that green ZnO NPs could be used effectively in agricultural and food safety applications and also can address future medical concerns.[33]

In recent years, the development of efficient green chemistry methods for synthesis of metal nanoparticles has become a major focus of researchers. One of the most considered methods is production of metal nanoparticles using organisms. Among these organisms plants seem to be the best candidates and they are suitable for large-scale biosynthesis of nanoparticles. The advantages of using plant and plant-derived materials for biosynthesis of metal nanoparticles have interested researchers to investigate mechanisms of metal ions uptake and bioreduction by plants, and to understand the possible mechanism of metal nanoparticle formation in plants. In this review, most of the plants used in metal nanoparticle synthesis are shown.[34]

Current advancements in green synthesis of materials especially nanoparticles have led to conservation of natural and non-renewable resources along with reduction in environmental pollution. Development of cost-effective, simple and eco-friendly routes for the synthesis of nanoparticles is very important. All over the world, a wide variety of biogenic sources have been put to trial as a source of green agents to facilitate synthesis process. In addition to this, environmentally benign solvents are also being used these days in order to promote green synthesis. In this review, an attempt has been made to familiarise the readers with the different greenroutes for the synthesis of nanoparticles.[35]

A novel one step synthesis of water soluble Au and Ag nanoparticles has been reported at room temperature using a naturally occurring bifunctional molecule, namely, gallic acid. On the basis of the experimental and theoretical studies, a tentative structure of the supramolecular complex leading to a strong interparticle interaction is provided.[36]

The femtosecond laser ablation of a gold target in aqueous solutions has been used to produce colloidal Au nanoparticles with controlled surface chemistry. A detailed chemical analysis showed that the nanoparticles formed were partially oxidized by the oxygen present in solution. The results obtained help to develop methodologies for the control of laserablation-based nanoparticle growth and the functionalisation of nanoparticle surfaces by specific interactions.[37]

Silver nanoparticles (AgNPs) were prepared from ionic liquids (ILs) of silver(I)N-alkyl ethylenediamine (N-alkyl = N-2-ethylhexyl, N-octyl, and N-dodecyl) complexes (=AgILs) and from protic ionic liquids (PILs) of N-alkylethylene diamines containing dissolved silver(I) nitrate (=AgNO3-PILs) and the two methods were compared. The effect of the ethyl-branch of the alkyl chainson the physicochemical properties of the AgILsis drastic. The AgILs generally provided a more favorable reaction field for AgNP production than the AgNO3-PIL. The preparation of AgNPs depends on the concentration of AgNO3 in the PILs; at approximately 0.01 mol kg− 1 of AgNO3 in the PILs, the PILs were capable of preventing the aggregation of silver(0) particles to allow the formation of AgNPs,while at higher AgNO3 concentrations(0.05 mol kg− 1), even in the long-chained dodecyl-PIL system, larger continuous-shaped aggregates were detected. The bis(fluorosulfonyl)amide (FSA) anion is effective on decreasing the transition temperature for the formation of the liquid states, but not appropriate for the preparation of the AgNPs when combined with the N-2-ethylhexyl ethylenediamine-AgIL cationic unit, compared to theNO3andtrifluoroacetate (TFA) salts.[38]

Stable chromium, molybdenum, tungsten, manganese, rhenium, ruthenium, osmium, cobalt,rhodium, and iridium metal nanoparticles (M-NPs) have been reproducibly obtained by facile, rapid(3 min), and energy-saving 10 W microwave irradiation (MWI) under an argon atmosphere from their metal– carbonyl precursors [Mx(CO)y] in the ionic liquid (IL) 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIm][BF4]). The MWI-obtained nanoparticles have a very small (<5 nm) and uniform size and are prepared without any additional stabilizers or capping molecules as long-term stable M-NP/IL dispersions (characterization by transmission electron microscopy (TEM), transmission electron diffraction (TED), and dynamic light scattering(DLS))[39] hot resulting mixture. Subsequently, Prepared 1 M 20 mL of sodium hydroxide (NaOH) was added in to the reaction mixture drop by drop from burette with vigorous stirring. The instantaneous black colour appearance indicated the formation of Fe_3O_4 nanoparticles .The resulting mixture was cooled at room temperature. After sometime, the intense black precipitate was filtered using whatman filter paper and dried in air. The obtained black powder was collected and used for further characterisations. All the chemicals used in the study were of analytical grade.

4.4.*Characterisation of the prepared Fe_3O_4 nanoparticles*

Elemental composition analysis was carried out using the XRF instrument of Elvatech-Elvax-Maxpro of Ukraine. The XRD analysis was performed using the Rigaku, Japan.

5. RESULTS AND DISCUSSION

5.1. Elemental Analysis

The prepared neem leaf extract was shown in this figure.

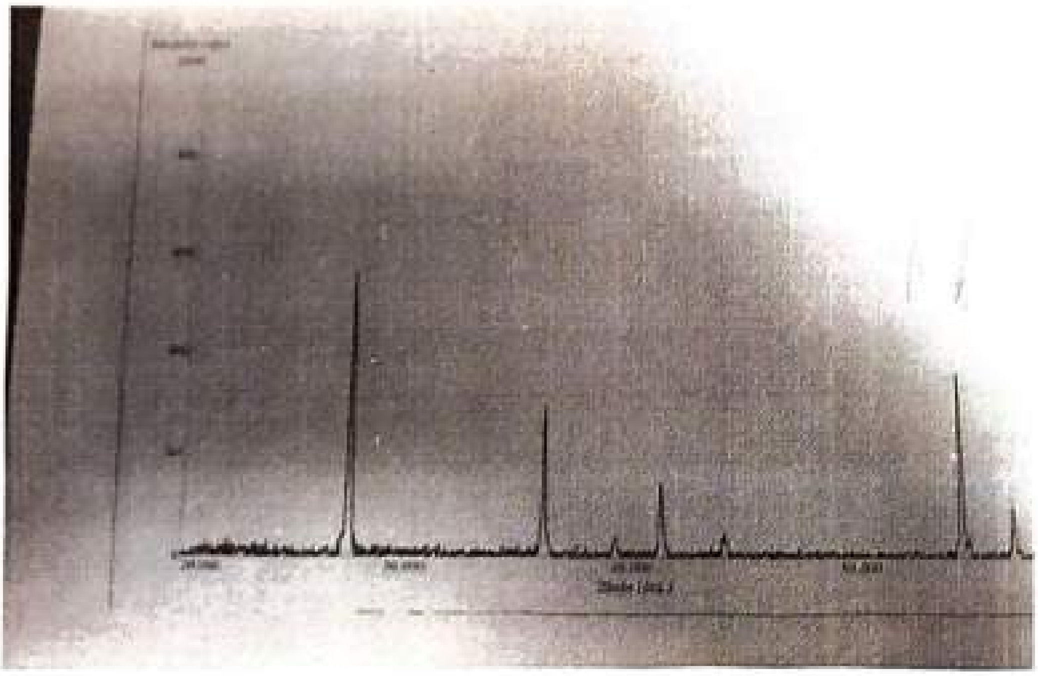
The preparation of iron oxide nanoparticle was revealed from the figure shown below.

Elemental studies revealed the presence of iron particles to about 98%. ie; the prepared nanoparticles contain least impurities.

Elemental Composition

At. Num	Element	Series	Intensity	Concentration
26	Fe	K	4051103	98.171 ± 0.022%
25	Mn	K	16896	0.507 ± 0.010%
14	Si	K	21835	0.418 ± 0.009%
82	Pb	L	8242	0.215 ± 0.006%
13	Al	K	3322	0.195 ± 0.018%
50	Sn	K	1597	0.189 ± 0.041%
24	Cr	K	9443	0.185 ± 0.015%
28	Ni	K	1659	0.106 ± 0.014%
29	Cu	K	368	0.010 ± 0.007%
33	As	K	0	< 0.005%

5.2. XRD Analysis



The peaks were obtained at 2theta values of 29^0, 37^0, 40^0, 42^0, 44^0, 52^0 and 54^0 . These values confirmed the presence of iron oxide particles. The size of the synthesized particles was confirmed from Scherrer equation, $D=K\lambda / \beta\cos\theta$ where, D is the size of the particle, K is Scherrer constant (0.94), λ is the X-ray wavelength (1.54178 A^0, β is the full width at half maximum (FWHM) of the diffraction peak. The particle size obtained was 85nm. This confirmed that the synthesized iron oxide particle was of nanometer in size.

6.CONCLUSION

Nanomaterials are the keystone of nanotechnology and a nano science. The present study focused on the preparation of iron oxide nanoparticles from neem leaf extract. The obtained powder was collected and used for further characterisations. 98% of iron was revealed from the elemental analysis and the particle size was obtained as 85nm.

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