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A Short Review on Green Synthesis of Iron Oxide Nanoparticles by Neem Plant (Azadirachta Indica) Leaves Extract

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Abstract:

Several researches were conducted to perform synthesisation of metal oxides for nanoparticles by using green synthesis method, shows the essence of using extracts of plants for reduction the metal into their nanoparticles and determined their application in different research domains. NEEM also known as Azadirachta Indica is an evergreen tree throughout the year, member of Meliaceous family. The presence of several phytochemicals such as glycosides, terpenes, triterpenes and flavonoids were reported in AI leaves. This study focuses on application of AI leave extract are used for metal oxide NPs fabrication. The commonly used metal oxides for this purpose are chromic oxide, cobalt oxide, molybdate oxide, titanium oxide, zinc oxide, manganese oxide, calcium oxide and iron oxide. In this study the respective research attempts were included to better understand the applications of nanoparticles like anticancer activity, antibiotic and antibacterial and photocatalytic activity. The use of extract of AI leave is to indicate the involvement of phytocompounds for the reduction of metal oxides and also help in achieving stabilized nanoparticles. The study concluded that NPs of metal oxide have more heterotrophic activity and also better catalysis potential over liquid extract of AI leaves.

Keywords— Azadirachta Indica, Phytochemicals, Metal Oxide nanoparticles, Anti-microbial potential, Photocatalytic activity

1. Introduction

The scientific domains such as medicine, biomedical, environment, energy, sensors, IT, electronics, agriculture and material chemistry are continuously working of exploring nanostructured metal oxides for their promising benefits and uses, [1] these advantageous applications of nanoparticle metal oxides attract researchers to develop more advance methods and techniques of fabrication with simple processing and cost effectiveness[2]. The metal oxides are unstable compounds then rare earth elements such as Au, Ag, Pt and so on. To achieve their crystalline nanoparticles and remove oxygen, some processes such as annealing and calcination are used. Metal oxides are heat treated at high temperature, sometimes beyond 300 °C. By the help of several method of fabrication, the nano metal oxides are modified. Then after these metal oxides are further classified for different parameters with the help of advance techniques. The characterization of these oxides is also helpful to modify their antibacterial and anticancer effects as per the requirement in biological applications. A study over size of nanoparticles suggested that, the antibacterial property increases with decrease in particle size [3].

The green approaches for extracting metals and metal oxides nanoparticles were basically used because they provide metal colloidal dispersions by reducing the complex metals in dilute solutions. The most commonly used reducing agents were $NaBH_4$ (Sodium borohydride) and H_6N_2O (Hydrazine hydrate), but they are not preferably used for synthesisation of nanoparticles because they infuse toxicity in nanoparticles [4]. There are some particular chemicals presents in green and natural materials which were non-toxic at all and have significant potential to be used as reducing agents for synthesisation of metal oxides. The green approaches use these microbial or biological extracts and mix them with metal salt solutions [5]. These biomolecules are used to reduce metal salts to the state of zero oxidation from positively oxidised state. They are also helpful in stabilization of the formed nanoparticles and also acts as in situ reducer agents and recovery medium [6]. It also averts the formation of cluster of particles which forms due to physical and chemical reactions between the nanoparticles. The agglomeration or cluster formation is prohibited due to the formation of a layer that surrounds the nanoparticles. So that the energy of surface of nanoparticles should be unchanged [7]. The size of nanoparticles is highly affected by the natural components present in nanoparticles. The high reduction rate depends on the stronger reductant available in the extract and that affects the size of nanoparticles. The synthesised nanoparticles are smaller in size and have narrowed particle size distribution [8]. There are several applications of nanoparticles, in which the most important is the antimicrobial action against different microbes. Antimicrobial action increases with decrease in particle size. It is observed that the antimicrobial properties of final nanoparticle in improved, if the coated natural compound also have antimicrobial characteristics. [9, 10].

As the demand of metal oxides increases, the researchers are using eco-friendly methods for synthesisation of metal oxides, in which the plant extracts used for the purpose of synthesis are reported as the prominent ones [11]. Several research works were conducted to study the synthesis of oxides and dioxides of zinc, copper, iron, titanium, cerium and selenium due to vast field of application in different industries [12]. The present paper includes the requirement of materials, method and process of green synthesisation of metal oxide NPs with the help of neem leaves extracts and use of those NPs in different scientific domains.

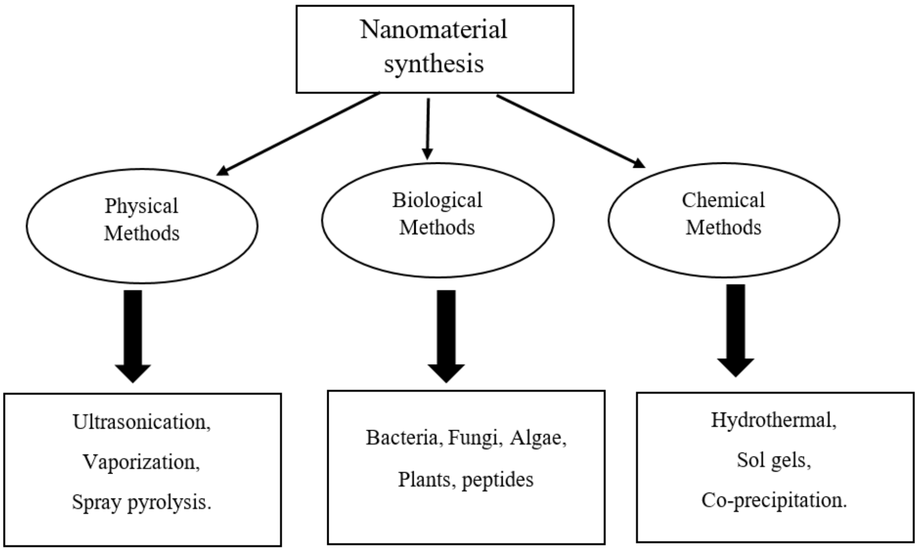

Figure 1: - Methods of Nanoparticle Synthesis

2. Morphology of Azadirachta Indica leaves

Azadirachta Indica leave have green petiole that connects leaf to the stem, odd-pinnate combination of 7 to 19 foliole. The foliole are acuminate apex, serrate margins, lanceolate and opposite with asymmetric base. The foliole is botanical name for leaflets. The foliole have dark green upper surface whereas lower surface is lighter green [13-15]. The lower surface has more important midrib with some scales on reticulate venation. The length of the foliole is about 3-9 cm, width of 1 to 3 cm with papery texture. The measuring diameter is 0.01 to 0.05 cm and length with 0.3 to 0.5 cm and each leaflet has cylindrical green petiole. Leaflets have astringent bitter in taste and indefinite odour when it crushed [16].

3. Presence of Phytochemicals in Azadirachta Indica leaves

The demand of AI extract for conventional uses is very high, the varieties of specialised metabolites such as bioflavonoids, isoprenoids, having large amounts of oils are also present in the extract; and are separated from Neem leaves and structurally elucidated [17]. In 1992, Siddiqui Mahmoodin and Naheedin Hossain's, worked for the detailed analysis of phytochemical which are present in extract of AI leave. The analysis shows hexane-extract having $C_{20}H_{40}O$, $C_{19}H_{36}O_2$, $C_{20}H_{400}$, $C_{18}H_{36}O_2$, $C_{16}H_{48}O_8Si_8$, $C_{20}H_{40}O_2$, $C_{19}H_{40}$, $C_{22}H_{44}O_2$, $C_2H_2O_4$, $C_{24}H_{46}O_4$, $C_{27}H_{56}$; chloro- form extract contains $C_{20}H_{40}O$, $C_{17}H_{34}O_2$, $C_{18}H_{31}ClO$, $C_{20}H_{400}$, $C_{18}H_{36}O_2$, $C_{29}H_{60}$; while methanolic-extract having C_8H_8O, $C_{17}H_{34}O_2$, $C_{18}H_{36}O_2$, $C_{18}H_{31}ClO$ [18]. In 2014, Banerjee et al. observed that AI leaves glycoprotein (or NLGP) having immunity boosting properties when mammalian subjects is tested [19].

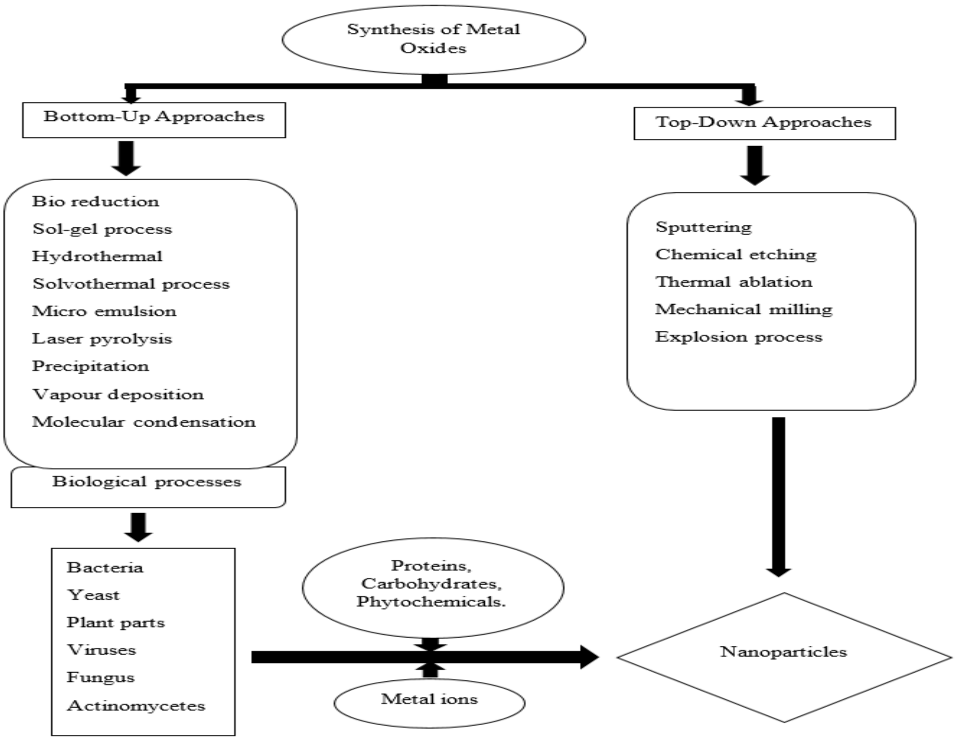

Figure 2: - Scientific Approaches for Synthesis of Metal Oxide.

Table 1: - Classification of AI (Neem) Leaves [20].

Common Name	Neem
Botanical Name	Azadirachta Indica
Kingdom	Plantae
Class	Magnoliopsida
Division	Magnoliophyta
Tribe	Melieae
Order	Rutales
Genus	Azadirachta
Species	Indica
Family	Meliaceae

Table 2: - Presence of phytochemicals found in NEEM leaves [21].

Phytochemical	Plant Extract
Alkaloids	Present
Glycosides	Present
Saponins	Present
Steroids	Present
Volatile Oils	Present
Flavonoids	Present
Fatty Acids	Present
Phenol and Tannins	Present
Carotenoids	Present

4. Pharmacological benefits of AI leaves

In AI leaves, many phytochemicals are commonly found. Since long decade of time, the AI leaves are used for treatment of many diseases and disorders. The pharmacological and medical aspects related to AI leaves were scientifically tested through various pharmacological models and the results were acceptable as well as appropriate [22-24].

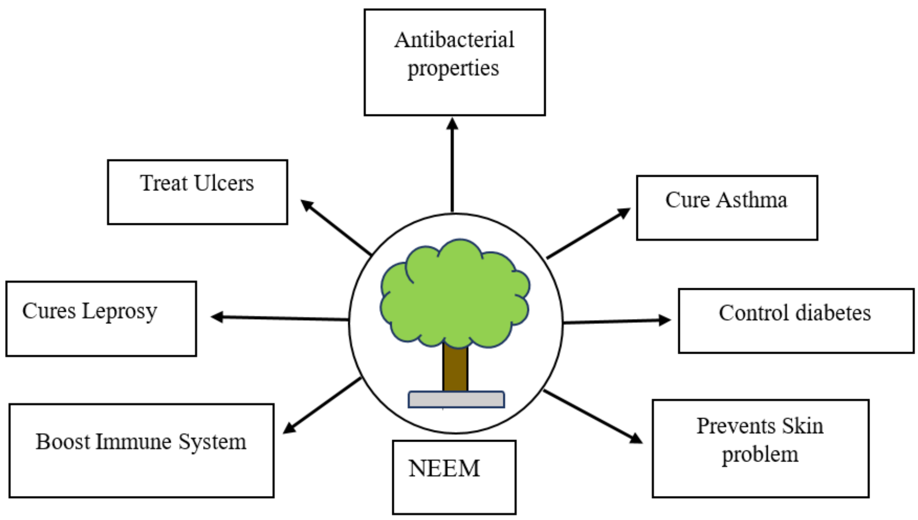

Figure 3: - Pharmacological benefits of AI leaves and it's extracts.

5. Green Synthesized Metal Oxide NPs using AI leaves

5.1 S-CaO nanoparticles: -

The several applications of calcium oxide (CaO) commonly used for making sensors, solar cells, ceramics, beauty products and catalysts; also convenient for purification of gases having high temperature [25]. CaO have anti corrosive property and can be handled easily and economically benignant. Due to no passivation and protection over the surface, aggregation takes place over CaO NPs [26].

The dangling bond density and the defect sites are required to be passivate by the help of modification of the surface through suitable capping agent. With concern to this, Dani et al. 2016 determined antibacterial properties of capped and uncapped S-CaO-NPs and report green synthesis on capped S-CaO nanoparticles by the help of an AI leaf aqueous extract and $C_{12}H_{22}O_{11}$, $Ca(NO_2)_2$ and NaOH [27]. Only the presence carbon, calcium and oxygen were detected through EDX spectrum. The fine crystalline appearance of nanoparticles was analyzed by XRD. Images of TEM shows that the sucrose capped CaO-NPs have star shape. Uncapped and capped S-CaO nanoparticles shows antibacterial activity against bacillus bacteria. The study says that, uncapped S-CaO-NPs have lower exhibition zone than inhibited by capped S-CaO-NPs. This is due to amphoteric nature of polyhydroxylated molecules, they act as acceptor as well as donor of hydrogen bond [28]. These hydroxyl groups cause a high electrostatic interaction between Bacillus bacteria and S-CaO nanoparticles. Antimicrobial activities can be achieved by the nanoparticles having large amount of free radicals. Hydroxyl radicals, superoxide and peroxide anions are some free radicals which are generated by inducing oxidative stress, responsible for damaging primary intercellular biomolecules [29].

5.2 Mn_3O_4 – nanoparticles

Manganese oxide is commonly used for the purpose of catalysis of the waste gases; reduction of nitrobenzene; oxy-dehydrogenation of alcohols and oxidation of CH_4 and CO. With consideration of the uses, in 2016, Sharma et al. use manganous acetate and demineralized extract of neem leaves to fabricate manganese oxide nanoparticles [30]. The characterization of the study found that Mn_3O_4 NPs are crystalline in nature through XRD pattern, Raman spectrum shows hausmannite structure and FESEM found spherical shape with average size (20-30 nm). Through XPS spectral analysis, for $Mn_2p_{3/2}$ and for $Mn_2p_{1/2}$, the Mn shows different binding energies that means Mn have two oxidation state (Mn^{2+}). The hausmannite having both manganic ions and manganous, due to which weak interaction between 2 states of the ions exhibits. For the tetravalent oxide and hydrated structure, three binding energies were assigned [30-31].

5.3 Co_3O_4 - nanoparticles

In various oxidation states Cobalt oxide is a p- type semiconductor and transition metal oxide, like Co, CoO, Co_3O_4, CoO(OH), Co_2O_3 and CoO_2 [32]. Co_3O_4 have a spinel structure of which octahedral sites were occupied by Co^{3+} ions and tetrahedral sites were occupied by Co^{2+} ions. Due to this it becomes anti-ferromagnetic and highly stable [33]. There was wide area of application for Co_3O_4 nanoparticles in several devices which include Li-ion battery, sensors, optical and thermal energy absorber and also in supercapacitors [34]. These nanoparticles are economical to be synthesized and used as catalyst for heterogeneous reactions of chemicals [35]. On behalf of these studies, in 2017, Sivachidambaram et al. perform green synthesis for Co_3O_4 nanoparticles with the help of amino acid glycine, extract of AI leaves, solution of cobalt nitrate by the combustion method using hot plate [36]. Then after the mixture was separated with the help of differential pelleting method and with the help of water the precipitate was washed several times. The polycrystalline nature of Co_3O_4 nanoparticles was observed with the help of XRD patterns. Agglomeration with quasi spherical shape of Co_3O_4 nanoparticles was analysed by HRTEM analysis. Ultraviolet visible spectrum shows to peaks for Co_3O_4 nanoparticles. The internal oxidation and reduction process and IVCT from Co^{2+} to Co^{3+} was indicated by absorption band at λ = 415 nm while on other hand at 500nm LMCT of Co^{3+} to Co^{2+} occurs [37]. It is observed that under magnetic field, the magnetisation curve shows linear behaviour with no coercivity and remanence. The observation was found with the help of magnetic hysteresis (M-H) measurements of Co_3O_4 nanoparticles. The antiferromagnetic barter interaction helps us to observe the tetrahedral sites and octahedral sites occupied by cobalt ions. This causes zero net magnetisation [38,39].

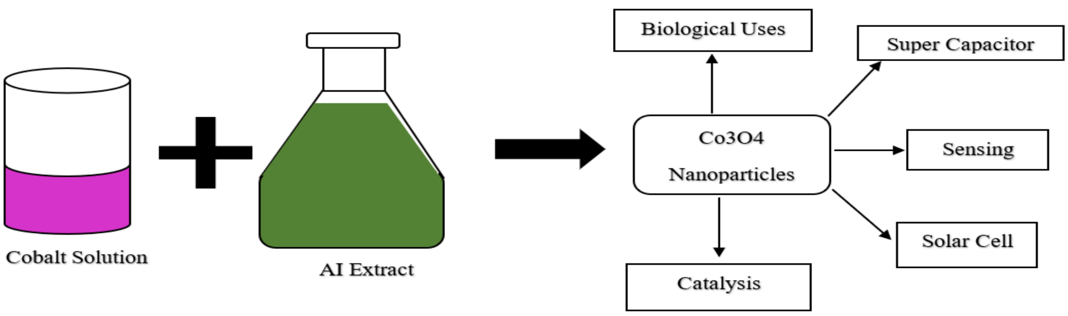

Figure 4: - Synthesis process of Cobalt Nanoparticles.

5.4 MoO_3 nanoparticles

MoO_3 is a type of semiconductor nanomaterial having improved characteristics. The MoO_3 has various application in electronic industry, which includes, material for sensors, superconductors, photoelectron catalyst and in Li-ions battery [40-42]. On the basis of application and demand, in 2018, Karthiga et al. uses liquid AI leave extract and Sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) for biosynthesized MoO_3 nanoparticles [43]. Several advance techniques were used to characterise the synthesized MoO_3 nanoparticles. The crystalline nature of MoO_3 nanoparticles was indicated by XRD pattern, the presence of Mo and O was analysed by EDX analysis, the presence of metabolites of AI such as saladucin, triterpenoids, vlassin, meliacin, flavonoids, geducin, alkaloids, numbidin on the surface of MoO_3 nanoparticle was revealed by FTIR spectrum. These metabolites help nanoparticles to become highly stable without any change in crystal structure. The SEM images found that the microrod-structure sample shows some aggregation.

5.5 α-Fe_2O_3 hexagonal cones

α-Fe_2O_3 is a type of semiconductor which is having the most stable phase of iron oxide (E.g. 2.1eV) [44]. It has an extensive application such as ferrofluids, solar energy conversion, clinical diagnosis, gas sensing, catalysis and corrosion resistance [45]. By considering these applications, α-Fe_2O_3 is fabricated in the form of Nano-fibres, Nano-rods, Nano-belts, Nano-tubes, Nano-cubes, Nano-rings, Nano-wires, Nano-flakes and simple heterogeneity and complex hierarchical structures [46-48].

In 2015, Sharma et al. works on synthesis of α-Fe_2O_3 hexagonal cones by AI leave extract and $FeCl_3$ as ion-donor [49]. HRTEM images and SAED patterns revealed that α-Fe_2O_3 was having rhombohedral phase. Many researchers use α-Fe_2O_3 hexagonal cones as a catalyst of burn rate for thermolysis of NH_4ClO_4 that is found in solid composite propellants and the performance of α-Fe_2O_3 was analysed by the help of thermogravimetric and differential scanning calorimetry analysis. TG-DSC thermal curve for ammonium- perchlorate added with green synthesised α-Fe_2O_3 and pure ammonium-perchlorate extreme decrease in higher temperature decomposition from 445°C to 370°C lower thermolysis from 315 °C to 295 °C. The reduction in temperature shows efficiency of α-Fe_2O_3 hexagonal cones as an AP catalysis. The isoconversional method is used to determine the kinetic parameters. α-Fe_2O_3 hexagonal cones added with AP require lower activation energy then pure AP [49-50].

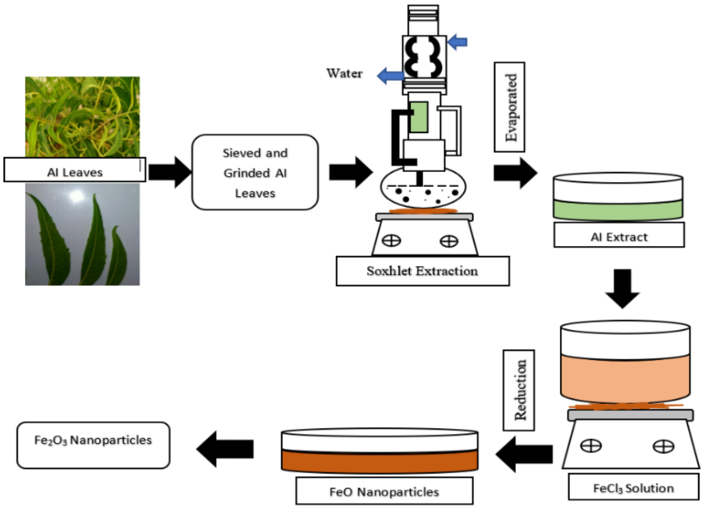

Figure 5: - Synthesis of Iron Oxide using AI leave extract.

6. Purification of nanoparticles

The nanoparticles should be free from any type of impurities while using. Before using and evaluating the application of nanoparticle, the nanoparticle must be processed by the step of purification to avoid any unwanted or unpredicted effect over its application [51,52]. In final nanomaterial, there are several reasons responsible for appearing impurities. Impurities are present in nanoparticles be due to the presence of unreduced metal or metal oxide or it may add directly through chemical used for the fabrication purpose [1, 53]. In different cases, for synthesis of metal particles imidazolium-based ionic liquids like BMIM-BF4 are used as versatile solvents. The impurities like water, 1-methylimidazole and chloride ions are commonly present in BMIM-BF4 causes harmful effects on quality of nanoparticles [54-55]. If the sizes of nanoparticles are less, then it exhibited it's uses as pharmacological potential to better extend. Thus, if there is slightly large size of formation of nanoparticle accidently then is seems similar to impurities [55-57]. In 2016, Robertson et al. purify nanoparticles with the help of several technique such as filtration, centrifugation, density gradient centrifugation and gas liquid chromatography on the basis of shapes and sizes of nanoparticles [58]. In 2017, Suthar et al. analysed the presence of impurities while fabricating gold nanoparticle using buffers and gold auric chloride. The nanoparticles were purified by HPLC [59]. The recent research analysed that some impurities such as Fe, Co and Al is commonly present carbon nanotubes allows growth of microbial species without any toxicity but the carbon nanotube with impurities causes depolarisation of cell membrane inhibited esterase activity to a notable extend [60-63].

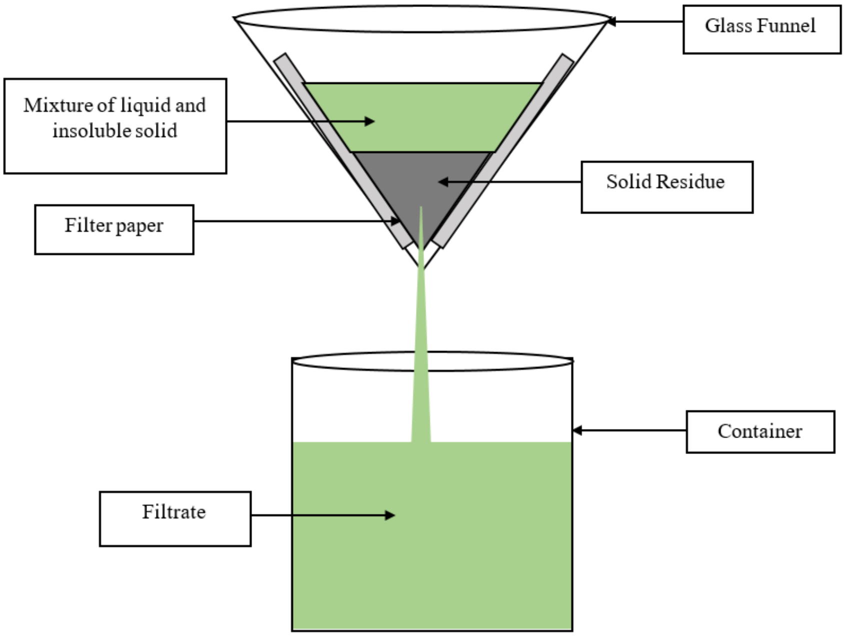

Figure 6: - Filtration process for removal of impurities from metal oxide nanoparticles.

7. Conclusions

Many research works were conducted till now, in which researchers used extracts of AI (Azadirachta indica) leaves to produce metal oxide nanoparticles through an Eco-friendly and Economical methods. The phytochemicals which were present in AI Leaf works as reductants as well as stabilizing agents for the synthesizing of metal oxide nanoparticles. The nanoparticles extracted by these synthesisation shows meaningful roles for various applications in different scientific aspects. This study concluded that, the nanoparticles in form of metal oxides extracted from AI leaf can be synthesized and also be analysed for different applications.

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