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Synthesis and Characterization of Molybdenum Oxide Nanoparticles by Green Method

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Abstract

In the present study, the synthesis of molybdenum oxide nanoparticles is performed by the reduction of Ammonium Molybdate with Citrus Sinensis leaves extract. An absorption peak of the molybdenum oxide in the range of 700 - 800 nm range using UV – Vis spectroscopy. FTIR study reveals the presence of flavonoids which acts as a reducing agent. The diffraction studies reveal its crystallinity with a cubical shape and 16 nm size. The zeta potential which is used to characterize the metal nanoparticles has been studied. The antifungal property of the nanoparticles has been studied and a plot for disease index has been discussed. Thus, this natural method of synthesizing the molybdenum oxide can go a long way in biophysics application.

Keywords: Green synthesis; molybdenum oxide; nanoparticle; flavonoids; capping agent; reducing agent, antifungal.

Introduction

Nano science and nanotechnology has become powerful tool in various sectors of science and engineering. One interesting phenomenon is the modification of the semiconducting optoelectronic materials in size, shape, morphology and dimensions, has resulted in synthesizing nanoparticles almost equal to Bohr radius [1]. Green method is one of the simple, inexpensive and non-polluting method of synthesizing nanoparticles, when compared to other methods such as spray pyrolysis, sputtering etc. Thus, plant mediated synthesis of metal nanoparticles is of utmost importance which is gaining popularity.

Heavy metals which are naturally present in earth‟s crust as metals and metalloids having greater density are toxic when present in higher concentration. But, the synthesis of binary chalcogenides of the group II –VI semiconductor in the nano is important due to their applications in optoelectronics. Due to the presence of biomolecules such as alkaloids, terpenoids, phenols, flavonoids, tannins, quinines etc, plants are used as the medium for synthesizing nanoparticles. Also, this method, lead to capping agent groups that are biocompatible, supporting light exchange. The phytochemicals has its importance in controlling the size of nanoparticles by inducing surface activity [2]. The nanoparticles produced by green synthesis have more bioactivity and catalytic activity. Hence, the green synthesis obeys redox reaction, in which metal ions are reduced to nanoparticles by the extract from the plants. Stabilization also is accompanied with the synthesis.

Colletotrichum gloeosporioides is one of the most common fungi in plants or a plant pathogen. This fungus is heavily dependent on stagnant water around plants and air that can promote spread of pathogen. Thus, it spreads either through soil or air to plants. The enzymes produced in the fungi can damage the seed tissues during germination or can cause necrosis or abnormal pattern and colour in leaves. These microbial infections can be prevented by proper treatment of certain selective micronutrient. Present work reports the green synthesis of molybdenum oxide nanoparticles using Citrus Sinensis plant leaves extract and a vegetable oil as a capping agent and also its antifungal property in treating leaf necrosis or anthracnose [3].

Role of flavonoids as a secondary metabolite

Flavonoids are natural polyphenolic compounds that include flavone, flavaal, flavone, and flavanol and inoflavone derivatives. It has been a skeleton of 2 phenyl rings connected by an oxygenated heterocycle ring and tannins. Many flavonoids chelate metal ions by forming stable complex through their hydroxyl (OH-) groups and the carbonyl moiety. Flavonoids are produced in cytosol of plant and then sent to vacuole for storage. The number of hydroxyl groups and the structure of flavonoids are important for metal binding activity. The metabolites which are adsorbed onto the surface of the nanoparticles can be removed by elution and magnetization. When the metallic salt dissociates into cation and anion, cation will be saturated to form hydroxyl complex. This results in the formation of some crystalline planes with different energy levels. Capping agent helps in arresting the growth of high energy atomic growth planes in some direction, which lead to specific type of nanoparticles [4].

Significance of Molybdenum in plant growth

Reducing agents from plant extract donate electrons to metal ions and convert them to nanoparticles. Thus, due to the presence of higher amount of reducing agent and stabilizing agent, biosynthesis of metal nanoparticles seems promising. Molybdenum is an interesting candidate as; it is a transition metal in group 6 of periodic table, which is less toxic when compared to other metals in the group. Also, Mo can exist in variety oxidation states that are important for applications. Plants absorb micro and macro nutrients from the soil. Thus, Mo nanoparticles can be made to absorb, as many deficiency disorders can be cured by the absorption of the same. The deficiency may some time lead to disruption of enzyme activity which may be involved in nitrogen metabolism. These enzymes include Nitrogenase, Xanthiase dehydrogenase and Nitrate reductase. Without sufficient Mo, an

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essential micro nutrient, Nitrate gets accumulated in the leaves and cannot be used to make proteins for normal growth. Plants become stunted and leaves become pale green or yellowish in colour between the veins [5]. This leads to necrosis in plant. Leaves of Citrus Sinensis commonly called as orange which comes in the family of Rutaceae is taken for study. The deficiency of Mo can enhance a fungal infection of

Colletotrichum gloeosporioides called Anthracnose leads to dropping of leaves prematurely and black streaks are found to develop. It is a group of fungal disease added by excess water on leaves, stems and fruit. An attempt has been made to reduce Mo ions by orange leaves extract and they were found to be stable. Moreover, the antimicrobial properties especially antifungal properties of Mo nanoparticles were studied which shows enhancement in activity due to synergise effect. Capping agents (organic ligand, surfactant etc) are basic component for the synthesize of metal nano particles of definite shape and size. The metal nanoparticle which is in the high dispersed state tries agglomerate and crystallizes. The stabilization of the nanoparticles can be achieved by wrapping them in an organic ligand shell. The secondary metabolites including alkaloids, saponins, flavonoids, reducing sugar and phenolic content present in the almond seeds are used as the essential criteria for the capping action A capping agent acts as an amphilic molecule consisting of a polar head and a nonpolar hydrocarbon. The efficiency of capping agent depends on both the part. The nonpolar head interacts with the medium and the polar head with the metal nanoparticles. It acts as the barrier which avoids further aggregation of nanoparticles and hence helps in stability.

Materials Needed

In the current work leaves of citrus sinensis extract is taken as the reducing agent of ammonium moly date, along with few drops of almond oil acting as a capping agent. Ammonium moly date $[(NH_4)_6Mo_7O_{24}]$ has been procured from Sigma Aldrich Pvt. Ltd. India, almond oil was purchased from local market. Citrus sinensis (Orange tree) under investigation is grown in a pot in a temperature range of 25°C - 35°C.

Preparation of Bio-extract: Bio extract can be prepared after washing 20 gm of fresh leaves of Citrus Sinensis with distilled water and then, can be boiled in water for 5 to 10 minutes and then filtered through Watt man filter paper. This fresh bio extract as seen can be utilized for the synthesis of molybdenum oxide nanoparticles.

Preparation of molybdenum oxide nanoparticles using bio-extract : In this experiment, 10 ml of a 0.01 M aqueous solution of $(NH_4)_6Mo_7O_{24}$ was added to 50 ml of the above extract and 4 ml of almond oil was finally added at room temperature. This is the solution for reaction. After 2 hours formation of molybdenum oxide nanoparticles, can be seen to appear in the flask, which is a black precipitate. The mixture is centrifuged for 20 minutes at 5000 rpm and the redispersed solution is washed with deionized water. This is repeated for three times and black colour molybdenum nanoparticles have been synthesized after final drying at 20 ± 2°C. This results in the synthesis of stabilized molybdenum nanoparticles with cubical shape and 16 nm size. High percentage of carboxylic acid is concerned with acting as the capping agent in the formation of nanoparticles. An idea of mechanism of this green synthesis Citrus Sinensis consists of flavanones, flavones, flavonols hydroxycinnamic acid and coumarins. Also, it contains antioxidants including flavonoids, carotenoids, vitamin C etc. The ability of plant secondary metabolite is to chelate metal ions and form stable complexes through hydroxyl and carboxyl moiety. The accumulation of metal ions and phenolic compounds can lead to the formation of nanoparticles. The reducing agents donate electrons to the metal ions and convert them into nanoparticles. Thus the presence of higher reducing agents and stabilizing agents results in smaller nanoparticles. Bioaccumulation helps providing the ability to detoxify metal ions. When the plants absorb metal ions faster than the catabolism, they can be accumulated in the plant tissues. Even the boiled bio extract helps in bioaccumulation interestingly, which can be rather extracted separately.

Characterization studies of Mo Nanoparticles

Optical studies

UV-Vis is used to identify the biomolecules and capping agents in Citrus Sinensis leaves that may be responsible for reduction of Mo ions. It is observed that, the absorption occurs between 700 – 800 nm. Because of the their carrier density, Surface Plasmon effect Resonance (SPR) is observed at The SPR effect is the resonance effect due to the interaction of conduction electrons of metal nanoparticles with incident photon. This depends on the size, shape, nature and composition of the medium of nanoparticles. As the size of the nanoparticles increases, extinction magnitude also increases by Mie theory, which plays an important role in the shape of the spectrum. The number of hydroxyl groups and the structure of flavonoids are important for metal binding activity. The influence of capping agent can be confirmed by the FTIR spectra. The spectra were compared with that of the palin almond oil. It was observed that, in the plain almond oil case, -OH stretching peaks were found to be low when compared to the final solution from reaction. This is due to the presence of different aliphatic compounds in oils. Also a shift of 18 cm-1 has been observed due to the capping influence. Similarly, there is a shift in C = O stretching bond also which is shown. It can be confirmed that, effective control over growth of molybdenum oxide nanoparticles through homogenous capping by edible oil lead to the formation of smaller nanoparticles.

Xrd Studies

The XRD pattern provided information for the formation of face centered cubic structure and with an average size of 15 to 17nm. It is treated as the fingerprint for every crystalline material. Full Width Half Maximum gives an estimate of the diffracting domain size in the desired plane. The shape factor is taken to be 0.9. The peak with highest intensity which is considered to be having more periodicity than other directions is taken under consideration. The particle size analysis from XRD analysis obtained for the prepared molybdenum oxide nano particles is revealed in. The XRD analysis shows strong peaks at 35° and 38°. The crystallinity of molybdenum oxide nanoparticles was compared with database with JCPDS file number 01-074-1389.

Also the size of molybdenum oxide nanoparticles can be estimated by Debye – Scherrer equation . $d = K \lambda / \beta \cos \theta$ Where, d- Average crystallite size, k- Scherrer constant (0.9), λ- X ray wave length (0.14nm), β- The full width at half maximum intensity of the peak at the diffracting angle θ. Using the above equation the average crystallite size of molybdenum oxide nanoparticles was calculated to be 16nm. Z (zeta) potential study Zeta potential is important for knowing the surface charges and hence the stability of nano particles. It is the electric potential in colloidal solutions. Studies reveal that a zeta value of ±30 mv is suitable for physically stable and ±20 mv of zeta value for combined electrostatic and stearic state. A zeta potential results for Citrus Sinensis leaves extract is -16.72 mv, while molybdenum oxide nanoparticles is reduced to -19.81mv. This indicates the degree of repulsion between similarly charged adjacent particles.

Antifungal studies

Antifungal assay: invitro Potato Dextrose Agar medium (PDA), is a common growth media for microbes made from the infusion of potato and dextrose. Chloramphenicol present in PDA can act as a selective agent to prevent the bacterial growth but promotes fungal growth. PDA medium prepared is autoclaved and left in petridishes to solidify. A control set is the one, in which the medium is not treated. All the other petridishes were exposed to different concentrations of molybdenum oxide ranging from 1 ppm to 100 ppm. These petridishes was then inoculated with 4 mm of Colletotrichum gloeosporioides and were incubated at 30°C. After the incubation of 24 hours in different concentrations of molybdenum oxide nanoparticles, the samples were studied and are reported.

Disease Index: Inoculation is a process of introduction of a pathogen to a living organism to aggravate the production of antibodies. The effect of molybdenum oxide NPs against anthracnose was observed both, before and after infection to plants. It is observed that a concentration of 100 ppm when applied had inhibited to the maximum percentage. This can be further activated by subjecting to magnetic field. Disease index has been measured for different incubation periods ranging from 1 week to 4 weeks. This has plotted and is shown. This indicates that the inhibition rate increase rapidly and linearly as the treatment with molybdenum oxide.

Optimized size of Nano particle

The effect of Nano particle, size, penetration and transport in organic plant was studied by taking 45 day old plant of Citrus Sinensis. The emulsion of molybdenum oxide nanoparticles was sprayed with 20ppm concentration of 14ml of 16nm size. Plants were observed after 4 days of spraying. The results increased significantly when the leaves were subjected to magnetic field by using a horse shoe magnet. The magnetic field supports diffusion of molecules in cell cytoplasm. Also it is seen from literature that the cubical shape of the nanoparticles promotes greater penetration in the plants. The improvement in the leaves freshly grown can be seen. The diffusion in a biologically active molecule is a ubiquitous process. This can help in controlling many biological mechanics. The rate of diffusion plays an important role in many cell functions as cell mobility, gas exchange, intra cellular transport etc.

Conclusion

The above study gives an insight of the synthesizing molybdenum oxide nanoparticles by an environmentally friendly way. Both the reducing agent and the capping agent are non-polluting and offer a non-toxic way of curing Anthracnose in Citrus Sinensis plant. Also, the bio extract from the same plant has been used to cure the fungal disease in the plant. Optical studies confirm the influence of biomolecules and capping agent. Since extinction magnitude plays an important role in deciding the characteristic of the absorption curve, if the synthesis of molybdenum oxide nanoparticles, is tuned, more novelty can be achieved. XRD provided the information of the average crystal size and the shape factor. Stability of the nanoparticles plays an important role in the applications. Zeta potential had been studied which is necessary for knowing the surface charges. The increase in absorption of nanoparticles when subjected to magnetic field has been observed. Interestingly, the shape of the nanoparticles influences diffusion. Results have lain stress that, the green synthesis of molybdenum oxide nanoparticles by plant extract is an eco-friendly way. The antimicrobial property especially the antifungal effect of the molybdenum oxide nanoparticles studied at 100 ppm concentration during inoculation and the disease index study reveals that molybdenum oxide NPs can be used as a fungicide.

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