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RESEARCH ARTICLE

BIO-ASSISTED SYNTHESIS OF POTASSIUM DOPED FERRIC SULPHIDE NANOPARTICLES FOR AGRICULTURAL APPLICATIONS

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ABSTRACT

A simple and cost effective green synthesis (Bio-Assisted) method was used to prepare Potassium doped ferric sulphide nanoparticles. The FTIR spectrum of potassium doped ferric sulphide shows characteristic peak at 617.2 cm^{-1} indicating M-O bond and shows the presence of K and Fe. The XRD analysis revealed the crystalline nature of the NPs. The average crystallite size was found to be 7.02 nm. The observed FESEM images showed the agglomeration of nanoparticles and are sponge like structure. The study revealed that potassium doped ferric sulphide nanoparticles could be used for high yield in agriculture.

Keywords: Green synthesis, Ferric sulphide, Morphology.

1. INTRODUCTION

There is a tremendous research interest in the area of nanotechnology to develop reliable processes for the synthesis of nanomaterials over a range of sizes and chemical composition. Although the conventional methods of synthesis of metal sols, known since the times of Michael Faraday, continue to be used for generating metal nanoparticles, there have been several improvements and modifications in the methods which provide a better control over the size, shape, and other characteristics of the nanoparticles. Nanoparticles (NPs) having one of the dimension in the range of 1–100 nm act as a bridge between bulk materials and atomic or molecular structures [1]. They possess remarkable and interesting properties owing to their small sizes, large surface area with free dangling bonds and higher reactivity over their bulk cousins. Since the nineteenth century scientists have been well aware of the ability of biological entities to reduce metal precursors but the mechanisms are still unexplored.

Synthesis and assembly strategies of nanoparticles mostly accommodate precursors from liquid, solid or gas phase; employ chemical or physical deposition approaches; and similarly rely on either chemical reactivity or physical compaction to integrate the nanostructure building blocks within the final material structure. The variety of techniques can be classified in to top-down and bottom up approaches. These techniques are further classified into three categories namely physical methods, chemical methods and bio-assisted methods. Physical methods are like inert gas condensation, physical vapour deposition, laser pyrolysis, flame spray pyrolysis, electro spraying techniques, melt mixing. Chemical methods are like co-precipitation synthesis, micro emulsion technique, hydrothermal synthesis, polyol synthesis, bio-assisted methods

Bio-Assisted method for the synthesis of nanoparticles:

The NPs synthesized following physico-chemical methods are expensive with many problems including use of toxic solvents, generation of hazardous by-products and the imperfection of the surface structure. Chemical methods are generally composed by more than one chemical species or molecules that could increase the particle reactivity and toxicity and might harm human health and the environment due to the composition ambiguity and lack of predictability [2,3].

The particles produced by green synthesis differ from those using physico -chemical approaches. Green synthesis, a bottom up approach, is similar to chemical reduction where an expensive chemical reducing agent is replaced by extract of a natural product such as leaves of trees/crops or fruits for the synthesis of metal or metal oxide NPs. Biological entities possess a huge potential for the production of NPs. Biogenic reduction of metal

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precursors to corresponding NPs is eco-friendly [4], sustainable [5], free of chemical contamination [6], less expensive and can be used for mass production [7]. Bio-assisted methods, biosynthesis or green synthesis provides an environmentally benign, low-toxic, cost-effective and efficient protocol to synthesize and fabricate nanoparticles. These methods employ biological systems like bacteria, fungi, viruses, yeast, actinomycetes, plant extracts, etc. for the synthesis of metal and metal oxide nanoparticles [8].

Bio-assisted methods can be broadly divided into three categories: i) Biogenic synthesis using microorganisms ii) Biogenic synthesis using bio-molecules as the templates iii) Biogenic synthesis using plant extracts. The progress of efficient green synthesis utilizing natural reducing, capping and stabilizing agents without the use of toxic, expensive chemicals and high energy consumption have attracted researchers towards biological methods [9-11]. Rapid industrialization, urbanization and population explosion are resulting in deterioration of earth is atmosphere and a huge amount of hazardous and unwanted substances are being released. It is now high time to learn about the secrets that are present in the nature and its natural products which lead to advancements in the synthesis processes of NPs. Furthermore, NPs are widely applied to human contact areas and there is a growing need to develop processes for synthesis that do not use harsh toxic chemicals. Therefore, green/biological synthesis of NPs is a possible alternative to chemical and physical methods. The unique properties of the NPs synthesized by biological methods are preferred over nanomaterials produced from physico-chemical methods.

The present work focuses on the synthesis of K doped Ferric sulphide using a plant extract Simarouba glauca for plant growth applications. The characterization of nanoparticles were carried out by using spectroscopy methods like Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction and field emission scanning electron microscopy (FESEM).

2. **Experimental:**

2.1. Synthesis procedure of Potassium doped Ferric sulphide nanoparticles using plant extract:

Potassium doped Ferric sulphide nanoparticles were prepared via green synthesis by adding ferric chloride (99.99 % pure), Na_2S (99.99 % pure) and KCl (99.9 % pure) to the plant extract Simarouba glauca. Initially, Simarouba glauca leaves collected from Coimbatore region were washed and cleaned with double distilled water and then dried with absorbent paper. Then it was cut into small pieces with an ethanol sterilized knife and crushed with mortar and pestle. The crushed leaves 20 g was dispensed in 100 ml of double distilled water and heated for 1 hour at 80 °C. The extract was then filtered using Whatman filter paper. The filtrate was collected in a clean and dried conical flask by standard sterilized filtration method and was stored. The mother solution was prepared by using 5 ml of plant extract which was added to 20 ml of deionized water. Then, the solution was stirred for 15 minutes using magnetic stirrer. Followed by stirring 1 mol % of ferric chloride was mixed with 40 ml of deionized water which was added drop wise to the mother solution and stirred again for 15 minutes. Then, Na_2S (1 mol %) was added drop wise to the mother solution and stirred for 15 minutes. Finally, 0.125 mol % of KCl dissolved with 40 ml of deionized water was added to the mother solution and continuously stirred for 1 h to get homogenous mixture. The obtained solution was centrifuged and dried in a hot air oven for 1 h. The resultant powder was crushed well to get fine particles and was shown in Fig. 1.

Fig. 1. As prepared Potassium doped Ferric sulphide nanoparticles

The prepared potassium doped Ferric sulfide nanoparticles were characterized by X-ray diffraction analysis, field emission scanning electron microscopy and Fourier transform infrared spectrum analysis.

3. RESULTS AND DISCUSSION

The prepared potassium doped ferric sulphide nanoparticles were then subjected to FTIR spectroscopy measurements. It was recorded by using a Thermo Scientific, Nicolet 10 using a KBr pellet technique. The FTIR spectrum of potassium doped ferric sulphide nanoparticle is shown in Fig. 2. The FTIR spectrum of potassium doped ferric sulphide shows the characteristic peaks at 617.2 cm^{-1} due to the M-O bond and shows the presence of K and Fe. The peak at 1120.64 cm^{-1} shows the presence of amide or amine. The peak at 3130.40 cm^{-1} is due to C-H stretching. The peak observed at 1402.25 cm^{-1} is due to O-H bond. The peak 1631.78 cm^{-1} is due to the unsaturated nitrogen (N-H bend) compounds from the leaf extract [12,13].

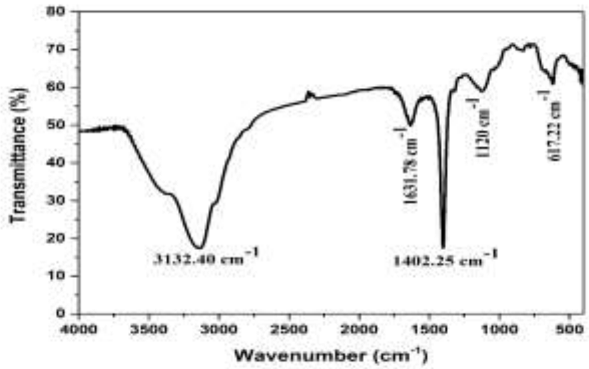

Fig. 2. FTIR spectrum

To understand the structural information of the prepared potassium doped ferric sulphide nanoparticles, the powder X-ray diffraction analysis was done. The powder X-ray diffraction study was carried out in Panalytical X-Pert Pro (Netherlands) X-ray diffractometer using Cu-Kα radiation source (λ=1.5406 Å) operated at 40 kV, and a scan rate of 10°/min.

Figure 3 shows the powder XRD pattern of potassium doped ferric sulphide nanoparticles prepared by using Simarouba glauca leaf extract.

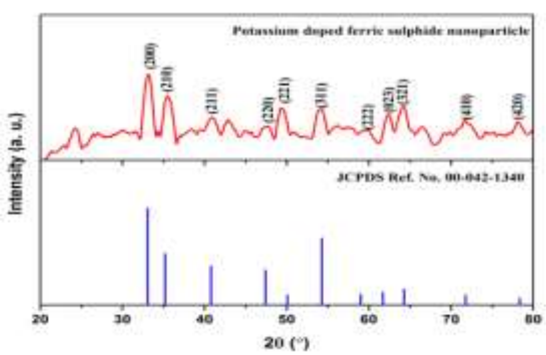

Fig. 3. XRD pattern of potassium doped ferric sulphide nanoparticles

All the diffraction peaks in this pattern were found to be in good agreement with JCPDS card data (Reference code 00-042-1340) which is corresponding to K:Fe_2S_3 in cubic geometry. The sample showed the major characteristic peaks for prepared nanoparticles at 2θ values of 33.13°, 35.64° and 49.50° corresponding to (200), (210) and (221) orientation planes, respectively. The average crystallite size of the prepared nanoparticles was found to be 7.01 nm.

The prepared potassium doped ferric sulphide nanoparticles were analysed by field emission scanning electron microscopy to know the morphology of the sample. The morphology of the samples was analyzed through Quanta-200F SEM. Fig. 4 shows the morphology of K:Fe_2S_3 nanoparticles.

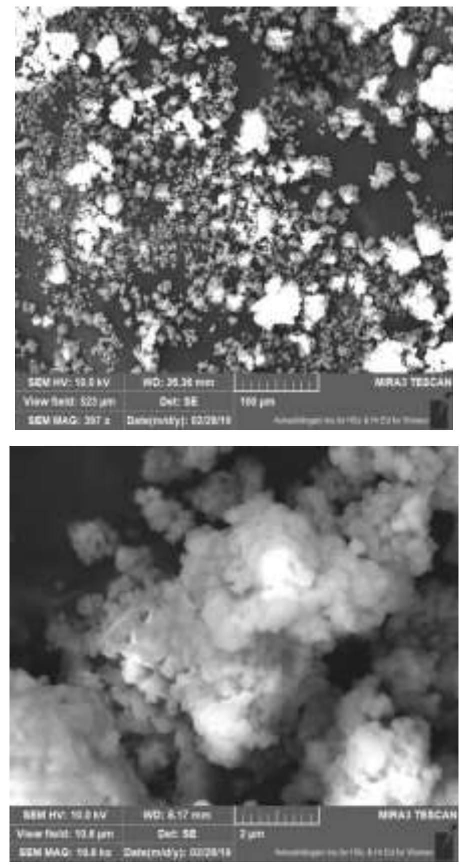

Fig. 4 SEM image a) Magnified at 397 times and b) Magnified at 19600 times

FESEM reveals that the as prepared nanoparticles are agglomerated. The surface seems like almost spongy and sphere shaped. Popcorn like appearance is observed in general.

4. CONCLUSIONS

Potassium doped ferric sulphide nanoparticles were prepared by simple and cost effective Bio assisted co-precipitation (green synthesis) method. The FTIR spectrum of potassium doped ferric sulphide shows the characteristic peaks at 617.2 cm^{-1} which is due to the M-O bond and shows the presence of K and Fe. The peak at 1120.64

cm^{-1} shows the presence of amide or amine. The peak at 3130.40 cm^{-1} is due to the C-H stretching. The peak observed at 1402.25 cm^{-1} due to O-H bond and the peak 1631.78 cm^{-1} is due to the unsaturated Nitrogen (N-H bend) compounds from the leaf extract. The XRD analysis revealed the crystalline nature of the potassium doped ferric sulphide nanoparticles. The structural parameter such as crystalline size was calculated for well resolved XRD peaks. The average crystallite size was found to be 7.02 nm. The observed FESEM images showed the agglomeration of nanoparticles and are sponge like structure. The study revealed that potassium doped ferric sulphide nanoparticles could be used for high yield in agricultural field.

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