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### Synthesis and Characterization of Magnesium Doped Ferric Sulphate Nanoparticles (Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs) for Agriculture Applications

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#### KEYWORDS

Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs

XRD

FESEM

Seed germination

Cowpea seed

*Vigna unguiculata*

#### ABSTRACT

The present study aimed to synthesize the magnesium doped ferric sulphate nanoparticles (Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs) and investigate their seed germination efficacy. Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs were prepared by a simple and cost-effective method and subjected to characterization. The X-ray Diffraction (XRD) spectrum revealed the crystalline nature of Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs with an average crystallite size of 36.41 nm. The field emission scanning electron microscope (FESEM) image displayed the agglomeration of Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs with the shape of the grains appeared like starfish which has limbs grown from a common cluster. The energy dispersive X-ray spectroscopy (EDS) demonstrated the existence of C (10.5%), O (49.14%), Fe (26.67%), Mg (0.78%) and S (13.35%) elements in Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs. It also revealed the absence of impurities in the synthesized NPs. Through Fourier transform infrared spectroscopy (FTIR), Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs showed the characteristic peaks at 615.29cm<sup>-1</sup>, 1130.29cm<sup>-1</sup>, 1400.32 cm<sup>-1</sup> and 1633.71cm<sup>-1</sup> which corresponded to Fe-O, C-N, O-H and N-H vibration respectively. Further, the seed germination study revealed that the Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs treatment caused a significant increase in seedling growth of cowpea (*Vigna unguiculata*) seeds compared to the untreated samples.

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## 1 Introduction

Presently, the agriculture sector has been fronting extensive challenges like unpredictable climate change (due to different types of pollution), utilization of several harmful chemicals in agricultural land, and toxic industry outlets (Pouratashi and Iravani 2012; Fraceto et al. 2016; Sun 2019; Mittal et al. 2020; Yilin et al. 2020). The United Nations Project reports that, in the year 2030 the world population will become 8.5 billion, which gives an alert of food demand in the future (Mittal et al. 2020). Nano-size materials display different physicochemical properties such as chemical, optical, physical, and biomedical characteristics when compared to bulk particles, because of the quantum properties of the materials with large surface area (Thakkar et al. 2010; Jeevanandam et al. 2018; Khan et al. 2019). These new characteristics of nano-size particles allow us to focus on unique output in different sectors such as biomedical, electronics, solar cell, water purification, agriculture, etc., (Yata et al. 2018; Zhu and Zhou 2019; Mittal et al. 2020). Nanomaterials, at the cutting-edge technology of nanoscience, are combined into a diversity of commercial goods due to their formidable properties including antimicrobial, antiviral, anticancer, growth promotor, etc., compared to their bulk materials. Hence, the novel and emerging characteristics of these nano-size particles have gained more attention in the agronomics sector. With the assistance of nano-size particles, the current agriculture practice can be transformed into precision cultivation with the existing resources to meet the food demands (Somenath et al. 2020; Weitao et al. 2020).

Multiple shapes of nano-size particles, including nanotubes, nanowires, and nanoclays possess unique physicochemical, magnetic, and electrical characteristics with unique applications in different sectors (Imada et al. 2016; Kumbhakar et al. 2014; Yaqoob et al. 2020). Fertilizers act as key molecules in the agriculture system and are necessary to improve crop production, but on the other side, the chemical fertilizers disturb the soil mineral balance and decrease the fertility of the soil. Currently, nanomaterials play a vital role in the agriculture system to create sustainable farming and increase soil fertility (An and Zhong 2019; Kumari and Singh 2020). Several factors such as size, charge, and shape are responsible for the interaction of nanomaterials with plants. Previously, different metals and metal oxides nanomaterials such as aluminum (Al), Cerium (Ce), Molybdenum (Mo), carbon (C), Iron (Fe), Titanium (Ti), Magnesium (Mg), Carbon (C), Gold (Au), Zinc (Zn), Silicon (Si), Copper (Cu), Silver (Ag), etc., were used in agriculture applications (Partila 2019; Sadak 2019; Anthony et al. 2020; Fuad et al. 2021). These nano-size particles are introduced into the agronomics sector to enhance seed treatments, and soil and foliar applications (Deshpande et al. 2017; Khan et al. 2019).

Magnesium (Mg) is a crucial micro component for all the crops to utilize photo energy for the photosynthesis process, as Mg is the central atom of the chlorophyll molecule, responsible for energy utilization. In general, Mg also plays significant responsibility in triggering enzymes, which are involved in photosynthesis, respiration, and nucleic acid synthesis. Seed germination is the first step (seed into the plant) and the most sensitive stage with great significance for improving crop yield and quality. Owing to this, the present study aimed to synthesize and characterize the magnesium doped ferric sulphate nanoparticles (Mg-Fe<sub>2</sub>SO<sub>3</sub> NPs) to investigate the effect of these nanoparticles on the germination property of cowpea (*Vigna unguiculata*). It is reported that the deficiency of Mg and Fe in plant systems resulted in a decrease in the production of chlorophyll (Balakrishnan et al. 2000). This study was planned to synthesize the nanoparticles with the help of Mg and Fe for agriculture applications through green synthesis approach for getting additional benefit of incorporating phytochemicals into the nanoparticles for the better yield. To the best of our knowledge, this is the first report of utilizing the leaves extract of *Aegle marmelosby* in synthesizing Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs. The *A. Marmelosby* contains bioactive molecules in various parts of the plants especially the leaves contain different types of phytochemicals (Pathirana et al. 2020). The leaves extract of *A. Marmelosby* was used as an effective reduction agent in the green synthesis of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs for seed germination applications.

## 2 Materials and Methods

### 2.1 Materials and preparation of plant extract

Ferric chloride (Fe<sub>2</sub>Cl<sub>3</sub>), sodium sulphide (Na<sub>2</sub>S), and magnesium chloride (MgCl<sub>2</sub>) were obtained from Himedia, Mumbai, India. The leaves of *A. marmelosby* (Vilvam) were collected from the local area nearby the college campus and authenticated by a plant taxonomist. The collected leaves were washed with distilled water thoroughly and these leaves were dried for 10 days at room temperature. To prepare aqueous leaves extract of *A. marmelosby*, about 20g of leaves (fine powder form) was well mixed with 100mL of sterile water, and the reaction vessel was kept in a Soxhlet extractor system to obtain the secondary phytochemicals from *A. marmelosby*. Obtained aqueous leaf extract was used for the synthesis of Mg doped Fe<sub>2</sub>SO<sub>3</sub>NPs.

### 2.2 Preparation of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs

Mg doped Fe<sub>2</sub>SO<sub>3</sub> nanoparticles were synthesized via green synthesis by adding Fe<sub>2</sub>Cl<sub>3</sub>, Na<sub>2</sub>S, and MgCl<sub>2</sub> to the plant extract *A. marmelosby*. Initially, about 5mL of plant extract was added to 20mL deionized water and kept in stirred condition for 15 minutes using a magnetic stirrer. About 6.48g of Fe<sub>2</sub>Cl<sub>3</sub> was mixed in 40mL of deionized water and this was added drop by drop into the

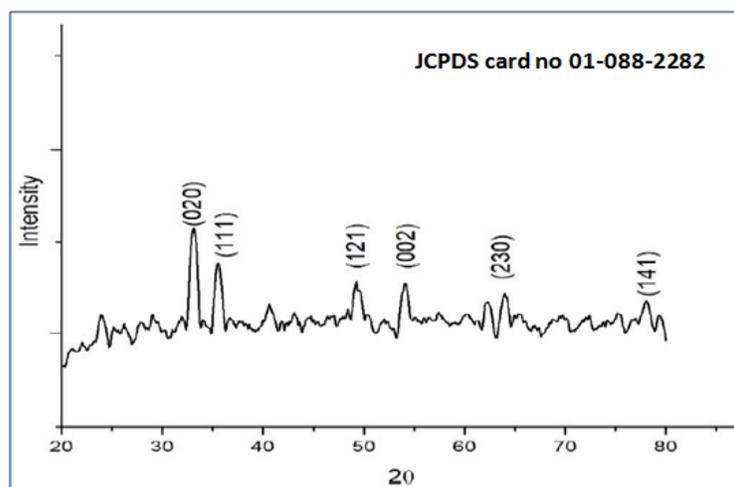


Figure 1 XRD spectrum of Mg doped  $\text{Fe}_2\text{SO}_3$  NPs

prepared extract solution for 15 minutes. Then  $\text{Na}_2\text{S}$  (1.56g) was added slowly into the reaction mixture under stirred conditions. Finally,  $\text{MgCl}_2$  solution dissolved in 40mL of deionized  $\text{H}_2\text{O}$  was added into a reaction vessel and continuously stirred for 1 hour. After that, the obtained reaction mixture was centrifuged for 30 minutes at 5000 rpm, then the obtained pellet was washed with distilled water followed by calcined at  $600^\circ\text{C}$  for 4 hours.

### 2.3 Characterization of Mg doped $\text{Fe}_2\text{SO}_3$ NPs

The crystallite peaks of prepared Mg doped  $\text{Fe}_2\text{SO}_3$  NPs were analyzed by using the X-Ray diffraction (XRD) spectrum (PANalytical-X-pertPro) at the range of  $2\theta = 20^\circ - 80^\circ$ . The surface topology and the elemental composition of the prepared Mg doped  $\text{Fe}_2\text{SO}_3$  NPs were studied by scanning electron microscopy (TESCAN MIRA-3 attached with EDS spectrum). The function groups of the prepared NPs were identified using Fourier transform infra-red (FTIR) spectra (FTIR00585, Perkin-Elmer).

### 2.4 Effect of Mg doped $\text{Fe}_2\text{SO}_3$ NPs on Cowpea seed germination

In 10 mL of distilled water, about 0.005 g of Mg doped  $\text{Fe}_2\text{SO}_3$  nanomaterial was dissolved and used as germination stock solution. Collected seeds were surface sterilized by ethanol to remove unwanted bacteria. In a petri dish, the filter paper was used instead of soil and collected seeds were placed. One of the petri dishes that contain seeds received only water (2mL/day) served as a control for the experiment and other petri dishes with seeds received Mg doped  $\text{Fe}_2\text{SO}_3$  NPs solution (2mL/day). These petri dishes were placed under visible light conditions. The whole experiment was set at lab scale level and the seedling growth of nanoparticles treated and untreated seeds were measured and demonstrated after 5 days. The obtained results were statistically analyzed.

## 3 Results and Discussion

### 3.1 XRD study

The structure of the Mg doped  $\text{Fe}_2\text{SO}_3$  NPs was examined by XRD and measurements were carried out over the diffraction angle  $2\theta = 10^\circ - 90^\circ$ . The XRD results showed diffraction peaks at  $33.05^\circ$ ,  $35.53^\circ$ , and  $49.41^\circ$  which correspond to (020), (111), and (121) orientation planes, respectively, indicating orthorhombic crystallinity (JCPDS card no. 01-088-2282) of the synthesized nanoparticles (Figure 1).

The crystallite size of the Mg doped  $\text{Fe}_2\text{SO}_3$  nanoparticles was estimated using Scherrer's formula (Ning et al. 2020; Madhan et al. 2021).

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where  $D$  = crystallite size,  $K$  = constant (shape),  $\lambda$  = X-ray wavelength,  $\beta$  = Full Width Half Maximum [reflection located at  $2\theta$ ] and  $\theta$  = angle of reflection

The average crystallite size of the prepared nanoparticles was estimated to be around 36.41 nm. The observed peaks confirmed the predominately crystalline nature of the magnesium doped ferric sulphate nanoparticles (Yu et al. 2011).

### 3.2 FESEM analysis

The surface topology of the prepared Mg doped  $\text{Fe}_2\text{SO}_3$  NPs was studied through FESEM analysis. The FESEM image showed the agglomeration of Mg doped  $\text{Fe}_2\text{SO}_3$  NPs with the shape of grains looking like starfish which have limbs grown from a common cluster (Figure 2).

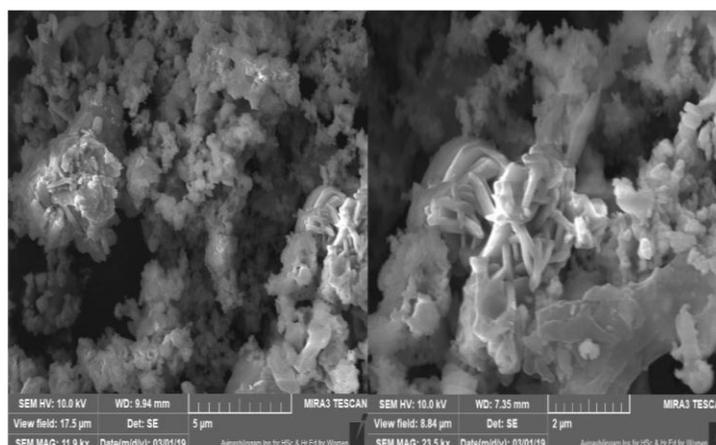


Figure 2 FESEM images Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs at different magnification scale

The different sizes and shapes of the nanomaterials certainly influence the multiple fields of biological applications like clinical, agricultural, and food technology because nanomaterials possess unique chemical and physical properties due to their surface area and nanoscale size. The size of the nanomaterials can influence the physiochemical properties of the substance, which led to novel applications (Ibrahim et al. 2019). Pradeev et al. (2018) described the nanoscale particles with hexagonal crystalline structure of ZnO NPs and Mg doped ZnO NPs around 30-110 nm through the precipitation method. In addition, they reported that the grain size of the final particles inside the ZnO matrix was increased with a high concentration of Mg ions doped ZnO NPs. In this study, the FESEM images of prepared Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs showed that the larger particles cause the particles to aggregate on their surfaces.

### 3.3 EDAX analysis

EDAX is an analytical technique and standard method for identifying chemical characterization or elemental composition using a very small quantity of samples. The elemental analysis of the prepared Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs was carried out by EDAX and confirmed that the prepared sample was free from impurities as the

peaks showed the existence of only C, O, Fe, Mg and S elements (Figure 3) (Pradeev et al.2018).

### 3.4 Fourier Transformation Infrared Spectroscopy (FTIR)

The prepared Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs was subjected to FTIR spectrum to investigate the vibration of the functional molecules through infrared absorption spectrum. Figure 4 displays the FTIR spectrum of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs.

The FTIR spectrum of prepared Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs showed characteristic peaks at 3142.04 cm<sup>-1</sup>, 1633.71 cm<sup>-1</sup>, 1400.32 cm<sup>-1</sup>, 1130.29 cm<sup>-1</sup> and 615.29 cm<sup>-1</sup>. The band appeared at a low frequency of 615.29 cm<sup>-1</sup> corresponds to the stretching vibration of Fe-O bonding (Bharathi et al. 2019; Piyush et al. 2021; Win et al. 2021). The obtained band at 1633.71 cm<sup>-1</sup> corresponds to the vibration mode of N-H bending and the band vibration at 1130.29 cm<sup>-1</sup> was due to the vibration of C-N stretching. These vibration modes of N-H and C-N indicated amines and aromatic amine groups. The characteristic band at 1400.32 cm<sup>-1</sup> was assigned to the vibration mode of C-F stretching of fluoro compounds. The strong peak at 3142.04 cm<sup>-1</sup> was assigned to the vibration of the hydroxyl group due to the O-H vibration of stretching (Kumuth and Alias 2006; Pradeev et al. 2008; Anam et al. 2018; Lesiak et al. 2019).

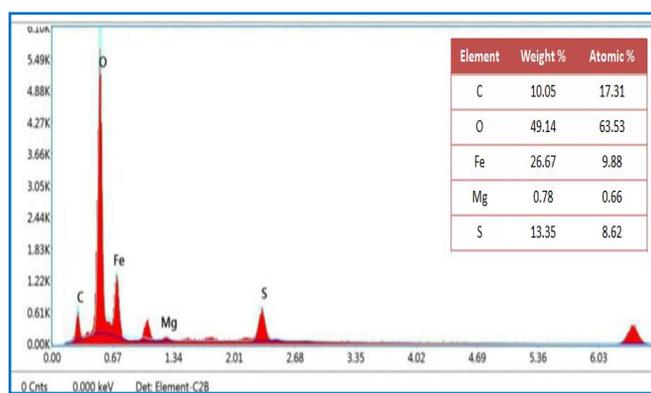


Figure 3 EDAX spectrum of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs

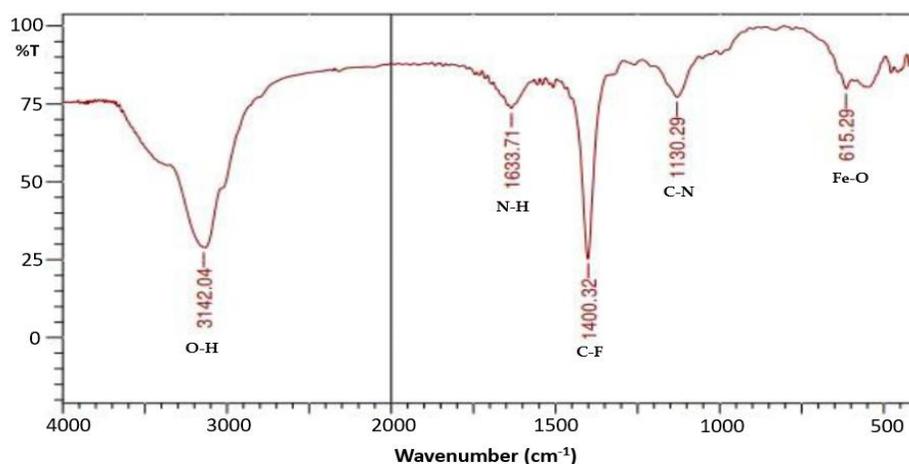


Figure 4 FTIR spectrum of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs

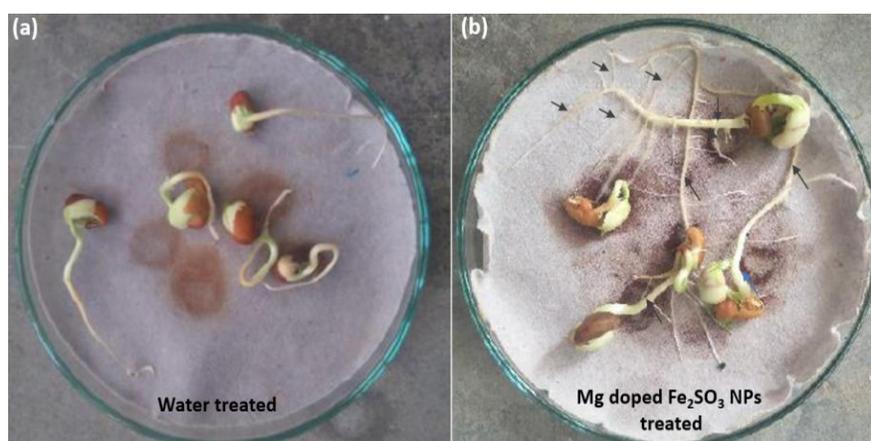


Figure 5 Effect of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs on cowpea seed germination

Table 1 Effect of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs on cowpea seed germination

Parameters	Cowpea seed treated with water (cm)	Cowpea seed treated Mg doped Fe <sub>2</sub> SO <sub>3</sub> NPs (cm)
Shoot length	2	4.5
Root length	2.5	6
Leaves length	Nil	1

### 3.5 Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs on Cowpea Seed Germination

The data presented in Table 1 shows the effect of the Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs treatment of @ 2 mL/day on cowpea seeds in comparison with double distilled water treated as the control at day 5. At the end of day 5, of the experimental period, the shoot and root lengths were measured for both the treatment and control. The water treated seeds showed a maximum shoot length of around 2 cm, and a root length of around 2.5 cm, whereas, the nanoparticles treated seeds showed a maximum shoot and root length of 4.5 cm and 6 cm, respectively. In addition, the nanoparticles treated seeds showed 2 leaves with a length of 1 cm, while no leaf growth was

noticed in the control (Figure 5). The results indicate that the treatment of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs has brought a significant effect on the seed germination process.

Currently, many industries focus on applying nano-size particles to the agriculture sector, with applications such as nanopesticides and nanofertilizers to increase the productivity and health of the agriculture crops (Prasad et al. 2017). Yi et al. (2016) first time studied the effect of Fe<sub>2</sub>O<sub>3</sub> nanocubes, Fe<sub>2</sub>O<sub>3</sub> long nanorods, and Fe<sub>2</sub>O<sub>3</sub> short nanorods on the germination of rice and noted that these nanomaterials promoted shoots growth and stimulated roots elongation at all concentrations. According to the recent report by

Asma et al. (2019), AgNPs also have a significant effect on the various growth parameters such as height of the plant, yields of the crop, fresh biomass, dry biomass, and number of roots and shoot in *Solanum lycopersicum*. Likewise, in the present study, the prepared Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs showed a substantial seedling growth promoter activity.

### Conclusion

The present study has successfully synthesized and characterized the Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs using the aqueous leaves extract of *A. marmelosby*. The synthesized nanoparticles exhibited an average crystallinity of 36.41nm according to XRD spectrum analysis. The shape of grains looked like starfish which has limbs grown from a common cluster as appeared under FESEM analysis. The elemental composition of the prepared nanomaterial by EDAX analysis indicated that the formulated nanomaterial was free from impurities as it was composed of only C, O, Fe, Mg, and S elements. The FTIR spectrum of the nanomaterial showed the stretching vibration of Fe-O bonding, N-H bending, C-N was stretching, the C-F stretching and O-H stretching. Further, the seed germination study demonstrated a significant seedling growth promoter activity of the synthesized Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs on cowpea seeds, indicating the potential of Mg doped Fe<sub>2</sub>SO<sub>3</sub> NPs to be utilized as a plant growth promoter in the agriculture industry.

### Conflicts of interest

The authors affirm that they do not have any conflict of interest.

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