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SYNTHESIS AND CHARACTERISATION OF HYBRID NANO FE-NPK FERTILIZER FOR ENHANCED NUTRIENT UPTAKE IN PLANTS

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Abstract

Nanotechnology offers innovative solutions to the limitations of conventional fertilizers, particularly regarding nutrient availability and uptake efficiency. This study presents the synthesis and characterization of a hybrid nano Fe-NPK fertilizer designed to address iron deficiency and enhance nutrient absorption. The fertilizer was prepared by thermally reacting calcium phosphate, potassium chloride, and urea, combined with nano-iron synthesized via chemical reduction of iron(III) tetraoxosulfate heptahydrate using sodium borohydride. The final formulation achieved an optimized nutrient ratio of 1:2:1:2 (N:P:K:Fe). Characterization techniques including AAS, EDS, XRD, FTIR, UV-Vis, and SEM were employed to verify composition and structure. AAS confirmed elemental presence, while EDS revealed uniform iron distribution. XRD patterns showed peaks at $2\theta = 26°$, $37°$, and $48°$, indicating iron oxide phases. FTIR peaks at 1429.20 cm^{-1} and 3481.60 cm^{-1} suggested carbonyl and amine functional groups, and UV-Vis absorption at 210 nm and 350 nm further validated the presence of iron and amine species. SEM imaging revealed a core-shell morphology typical of nanostructured materials. The successful synthesis and structural integrity of the nano Fe-NPK fertilizer highlight its potential for improving iron bioavailability and nutrient uptake in crops, representing a step forward in sustainable metallo-agriculture.

Keywords: Nanotechnology in Agriculture, Nutrient Bioavailability, Nano-Formulated Fertilizers, Iron-Fortified NPK, Sustainable Crop Nutrition

Introduction

Iron is an essential micronutrient for plant growth and development, playing a central role in photosynthesis, respiration, and DNA synthesis (Kobayashi & Nishizawa, 2012; Hider & Khodr, 2020). Although soils typically contain between 10,000 and 100,000 mg/kg of total iron, only 1–100 mg/kg is found in soluble forms readily available for plant uptake (USDA). Plants require 2–100 mg/kg of iron in their tissues, and deficiencies often manifest as chlorosis, especially under conditions of high pH or waterlogging, both of which limit iron solubility (Khan, 2006). Despite its importance, most commercial fertilizers contain iron in quantities of less than 1% by weight, which is inadequate for the needs of high-yielding crops. Furthermore, the efficiency of primary macronutrients—nitrogen (N), phosphorus (P), and potassium (K)—is frequently reduced by poor solubility, leaching, and fixation in the soil.

Nanotechnology offers promising solutions to these challenges. Nano fertilizers, with particle sizes between 1–100 nm, possess high surface area, improved solubility, and controlled nutrient release, thereby enhancing nutrient use efficiency (NUE) and minimizing environmental losses (DeRosa, 2010; Subramanian et al., 2015). Recent advancements have included nano-based delivery systems and microbial nanosensors capable of monitoring micronutrient availability in real time (Maderova & Paton, 2013). However, much of the existing research has concentrated on single nutrients or bulk NPK formulations, with relatively little focus on integrating micronutrients such as iron into multifunctional nano-formulations. This leaves a gap in the development of hybrid fertilizers that combine N, P, K, and Fe in a controlled-release form optimized for plant uptake.

The present study aims to synthesize and characterize a hybrid nano Fe-NPK fertilizer with an optimized nutrient ratio, using nano-iron produced via chemical reduction. The goal is to improve iron bioavailability and overall nutrient uptake in crops, thereby promoting sustainable and high-efficiency agricultural practices.

In plant nutrition, nano fertilizers are valued for their solubility, stability, controlled release, and targeted delivery, which together enhance nutrient uptake and transport within the soil-plant system. They stimulate growth, activate metabolic processes, and reduce ecological toxicity while being relatively easy to apply and manage. Fertilizers, whether natural or synthetic, provide essential nutrients to plants, and inorganic formulations have been a central focus of agricultural science for decades (Smil et al., 2001). The need for fertilizers is driven by global food security concerns, population growth, and economic development (Cakmak et al., 2002). Among macronutrients, nitrogen is the most widely used, playing a fundamental role in amino acids, proteins, enzymes, and nucleic acids (Scherer et al., 2009). Plants take up nitrogen mainly as nitrate (NO_3^-) or ammonium (NH_4^+), and its presence enhances yields, improves food quality, and promotes leaf area development (Ahmad et al., 2009; Arshadullah, 2010). Deficiency is marked by pale leaves, stunted growth, and reduced productivity (Leghari et al., 2016).

Phosphorus, another vital macronutrient, is a component of nucleic acids and phospholipids and plays a critical role in ATP synthesis, which drives energy transfer in plants (Scherer et al., 2009). Adequate phosphorus supports photosynthesis, root development, and biomass accumulation, while deficiencies cause slow growth and grey-green foliage (Reich et al., 2010). Potassium, the most absorbed macronutrient, regulates osmotic balance, stomatal function, and enzyme activation. It enhances drought resistance, salt tolerance, and disease resistance while contributing to fruit quality and yield (Wang et al., 2013; Zekri & Obreza, 2020).

Iron, a key micronutrient, is taken up by roots primarily as Fe^{2+} or Fe^{3+}, with optimal concentrations in plant tissue ranging from 50 to 250 ppm. It is a crucial component of cytochromes, heme proteins, Fe-S proteins, and leghaemoglobin, all of which participate in redox reactions during respiration and photosynthesis. Iron acts as a catalyst in chlorophyll synthesis and is part of nitrogenase, an enzyme vital for nitrogen fixation in certain bacteria. Deficiency, more likely when tissue concentrations drop below 50 ppm, typically appears first in young leaves due to the immobility of iron within plants. This manifests as interveinal chlorosis that can progress to complete whitening and necrosis in severe cases.

Nanotechnology offers new avenues for crop improvement by merging nanoscale engineering with biotechnology. Nanoparticles, nanofibers, and nanocapsules can be used to deliver DNA, biomolecules, or nutrients directly into plant cells, enabling precise control in genetic engineering and trait development (Friends of the Earth, 2008). These advancements also open opportunities for innovative mutation studies, further broadening the scope of agricultural improvement.

Materials and Methods

Chemicals

Calcium Triphosphate $Ca_3(PO_4)_2$, Iron (III) tetra oxosulphate (IV) hepta hydrate, Urea $(COCNH_2)_2)$, Potassium Chloride (KCl), Tetra oxo Manganese VII. $Kmno_4$, Sodium Borohydrate $NaBH_4$, Distilled water and Ethanol

Equipment/Instrument/Apparatus

Analytical weighing balance, Beakers, Measuring Cylinder, Conical flask, Spatula, Oven, Whattman filter paper, Magnetic stirrer with a hot plate, atomic absorption spectroscopy (AAS), Ultra Violet UV, Powder X-ray, FTIR and Electrical conductivity meter

Synthetic Method of Nano Zero Valent Iron (NZVI)

Nano Zero Valent Iron (NZVI) was synthesized via the chemical reduction method. Initially, 8.34 g of $FeSO_4·7H_2O$ was dissolved in a mixture of 24 mL ethanol and 6 mL distilled water. Separately, a reducing solution was prepared by dissolving 3.78 g of $NaBH_4$ in 100 mL of purified water.

The iron solution was placed on a magnetic stirrer and heated to 300°C using a hot plate. While stirring was maintained, 90 mL of the $NaBH_4$ solution was gradually added dropwise from a burette into the $FeSO_4$ solution. During the reaction, black particles were formed, indicating the reduction of iron.

The resulting particles were filtered using Whatman filter paper and rinsed with ethanol to remove any residual impurities. After filtration, the black particles were dried overnight at 500°C in a hot air oven. Upon drying, the particles changed from black to brown, confirming their transformation into nano zero valent iron.

Synthesis of Iron-Enriched NPK Nano Fertilizer

To synthesize the nano Fe-NPK fertilizer, a stepwise thermal reaction process was employed. First, 3.10 g of calcium phosphate $[Ca_3(PO_4)_2]$ was dissolved in 20 mL of distilled water in a beaker. Then, 0.056 g of synthesized NZVI, previously dissolved in 20 mL of distilled water, was added to the solution. The mixture was stirred and heated for 4 minutes on a magnetic stirrer.

Afterward, 0.75 g of potassium chloride (KCl), dissolved in 20 mL of distilled water, was introduced into the mixture, followed by another 4 minutes of stirring and heating. Finally, 6 g of urea, dissolved in 20 mL of distilled water, was added. The complete mixture was then stirred and maintained at 90°C for 2 hours to ensure uniform reaction and product formation.

Results and Discussions

Energy Dispersive X-Ray Spectroscopy (EDX)

EDS analysis was conducted in triplicate to determine the elemental composition of the synthesized Fe-NPK fertilizer. The EDS spectrum (Fig. 1) consistently showed a strong peak at 6.3 keV, corresponding to the Fe Kα region, which confirms the presence of elemental iron. Additional elements detected across all replicates included carbon (C), calcium (Ca), nitrogen (N), potassium (K), magnesium (Mg), silicon (Si), and sodium (Na), suggesting successful integration of macronutrients and supporting materials in the fertilizer matrix. The results align with earlier reports by Subramanian et al. (2015), who documented similar elemental distribution in nano-formulated fertilizers.

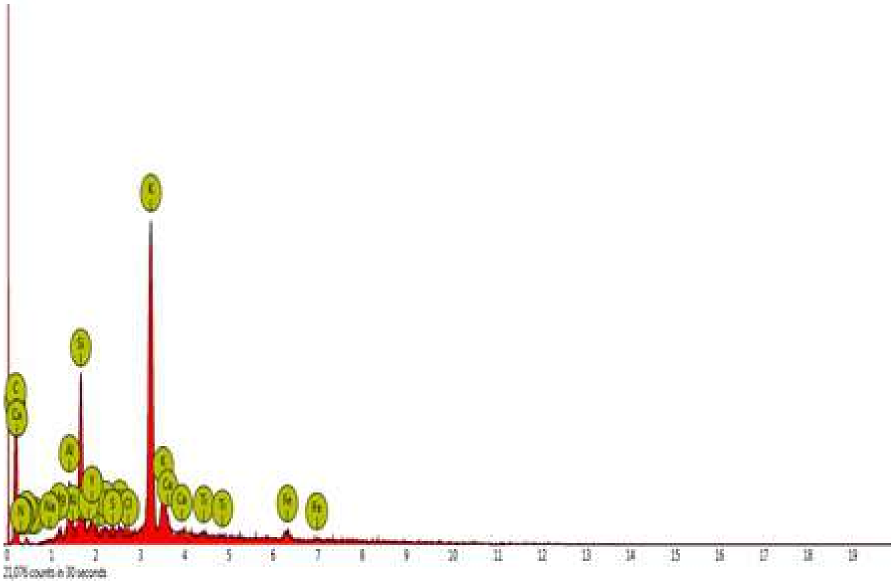


Fig 1. EDX image of Fe-NPK Spectra
Fe-NPK has a core-shell structure.

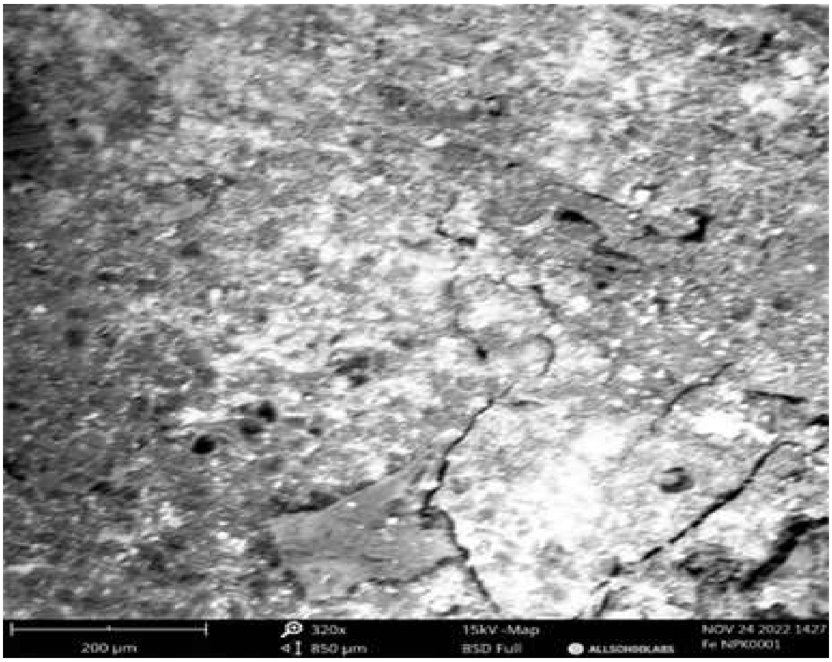
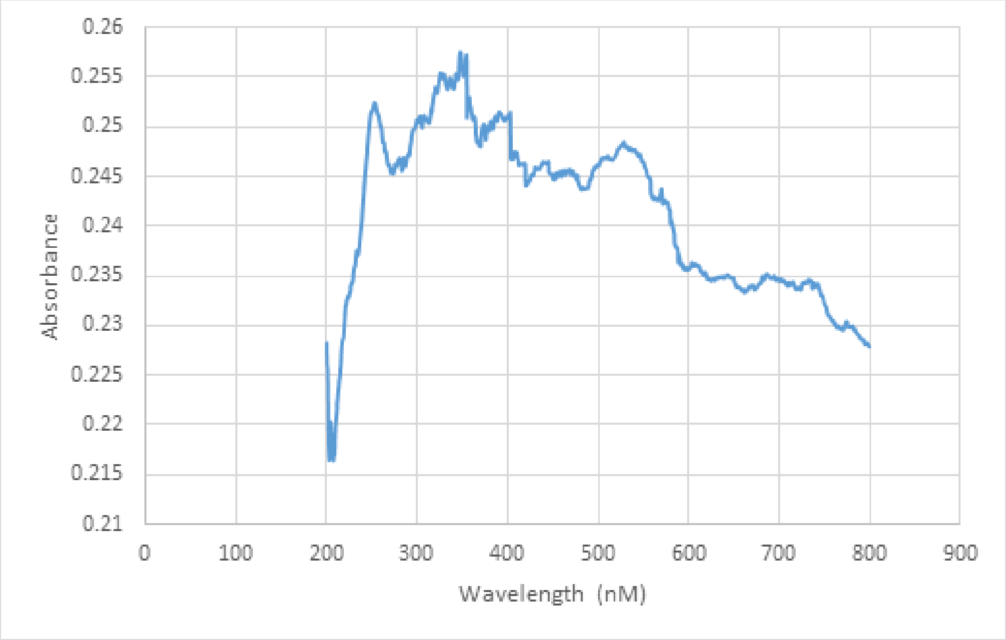


Fig 2. SEM image of Fe-NPK fertilizer

Scanning Electron Microscopy (SEM)

SEM imaging was performed at multiple magnifications, and consistent results were obtained across three independently synthesized batches. As shown in Fig. 2, the Fe-NPK particles exhibit a core–shell nanostructure, where the core is composed of metallic Fe^0 and the shell represents oxidized iron species (Fe_2O_3/Fe_3O_4). This morphology is consistent with findings by Mollah et al. (2012), who demonstrated that core–shell structures enhance stability and slow oxidation of nano iron particles. The nanostructure supports the sustained release behavior anticipated in controlled nutrient delivery systems.

UV-Visible Spectroscopy



UV-Visible spectra of Nano Fe-NPK

UV-Vis spectroscopy was conducted on three independently prepared samples to confirm optical properties. Spectra were recorded in the 200–800 nm range using a 1 cm quartz cuvette, and mean values were used with <5% variation observed. Two distinct absorption peaks were consistently observed at 210 nm and 350 nm (Fig. 3). The 210 nm peak is associated with amine groups, while the 350 nm peak corresponds to iron-related d–d electronic transitions. These results are in agreement with the findings of DeRosa et al. (2010), supporting the successful synthesis of iron-integrated nano fertilizers.

Fourier transform infrared (FT-IR) analysis of composite

FTIR analysis (Fig. 3) was performed to identify key functional groups. The spectra consistently revealed peaks at 3481.60 cm^{-1}, attributed to N–H stretching (amines), and 1439.20 cm^{-1}, corresponding to C=O stretching (carbonyl groups). These peaks were observed across all three replicates with minimal variation in intensity. The presence of these groups suggests successful incorporation of urea and organic residues during synthesis. These results align with those reported by Chinnamuthu and Boopathi (2009) in their analysis of nano urea carriers.

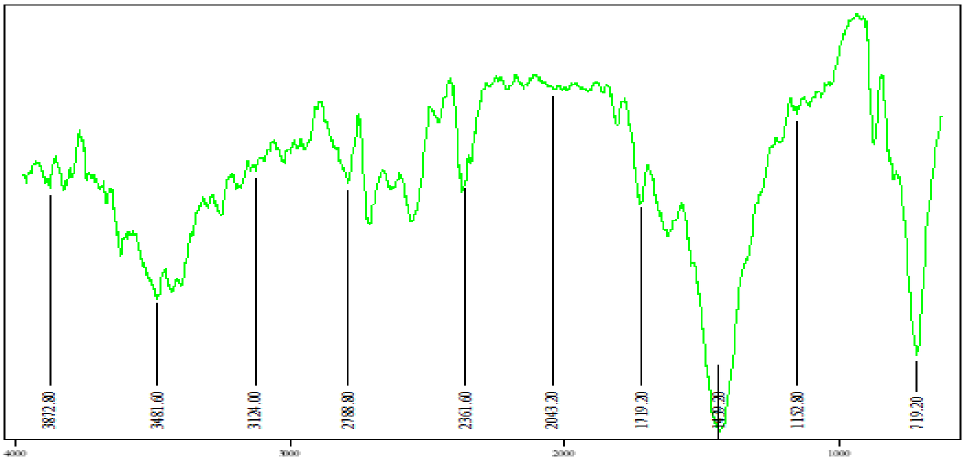


Fig 3. FTIR Spectra of Nano Fe NPK

Powder X-ray diffraction (PXRD) analysis

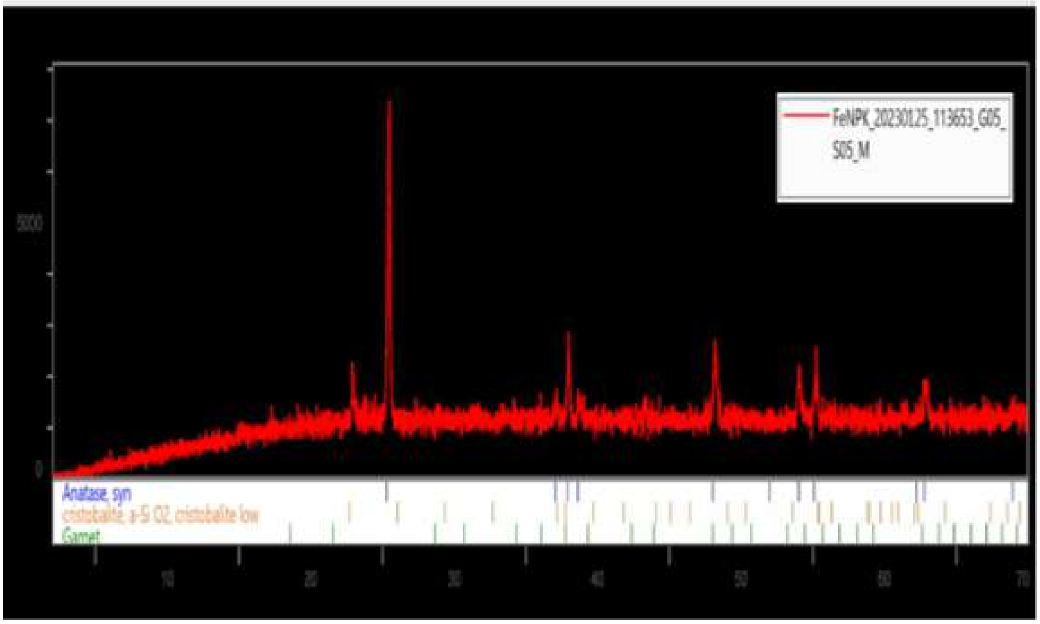


Fig.4a PXRD image of Fe-NPK composites fertilizer

PXRD was used to confirm crystalline phases within the Fe-NPK fertilizer. In all three replicates, the diffraction patterns showed peaks at 2θ = 26°, 38°, and 46° (Fig. 4a). The peak at 46° confirms the presence of zero-valent iron (Fe^0), while those at 26° and 38° are characteristic of iron oxides (Fe_2O_3 or Fe_3O_4). These results were benchmarked against a standard iron XRD pattern (Fig. 5b), which showed peaks at 2θ = 44.81° and 38.01°, confirming phase consistency. This crystalline structure is known to enhance reactivity and slow oxidation in nano-iron fertilizers, as demonstrated by Hwang et al. (2011).

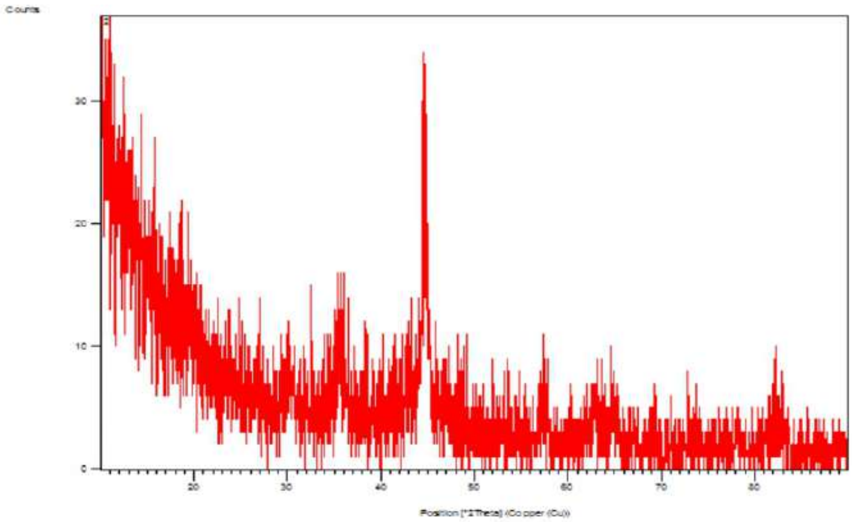


Fig. 4b PXRD Standard of Iron

The Standard XRD analysis had a peak at 2θ of 44.8096, which indicates the existence of iron nanoparticle, and a peak at 38.0125^0 that indicates the presence of iron oxide (Fe_2O_3 or Fe_3O_4) by comparing the XRD of Nano Fe-NPK fertilizer with that of standard XRD it shows that the synthesis sample contains nano iron.

Physicochemical Analysis

Basic physicochemical parameters were evaluated and are summarized in Table 1. The synthesized Fe-NPK was found to be slightly soluble in water, with a pH of 7.9 (slightly alkaline), and an electrical conductivity of 200 S/m, indicating ionic activity. Replicate measurements showed <3% variation across samples. These results suggest the fertilizer is suitable for controlled nutrient release, which may reduce leaching and improve field efficiency.

Table 1.Physicochemical Parameters of the Synthesized Fertilizers

Sample	Solubility in water	pH	Conductivity (S/m)
Fe-NPK	Slightly Soluble	7.9	200

Conclusion

The successful synthesis and multi-technique characterization of nano Fe-NPK fertilizer confirm the presence of functional iron, nitrogen, phosphorus, and potassium components in a nano-enabled, slow-release matrix. The core–shell nanostructure and associated physicochemical properties offer promising prospects for improving nutrient delivery and minimizing losses. While preliminary results are encouraging, future studies must focus on field validation, toxicity evaluation, and production scalability to realize the full agricultural potential of this nano-formulated fertilizer.

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