Sugarcane Bagasse and Nano-zeolite based Slow Release Fe Fertiliser Hydrogel: Its Synthesis and Characterization

C. Bharaani Sri¹, R. Shanmugasundaram¹, S. Marimuthu²,

T. Chitdeshwari¹, A. Senthil³, T. Kalaiselvi⁴

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ABSTRACT

Background: Iron (Fe) is an essential micronutrient that plays numerous vital functions in crop growth and development. This study was carried out to synthesise and characterize sugarcane bagasse and nano-zeolite based slow release Fe fertiliser hydrogel. **Methods:** At the Tamil Nadu Agricultural University in Coimbatore, a laboratory experiment was carried out to synthesize and characterise a slow release Fe fertiliser (SR Fe) hydrogel encompassing sugarcane bagasse and nano-zeolite. A slow-release Fe fertiliser was successfully created using the graft co-polymerization method. The chemical composition and surface morphology of the synthesised slow-release Fe fertiliser were characterised using scanning electron microscopy (SEM), FTIR and thermogravimetric analysis (TGA).

Result: SEM results show the evenly distributed pore space and EDX data show that the superabsorbent nanocomposite was successfully loaded with fertiliser Fe. The grafting of acrylate-based monomers onto starch molecules, the incorporation of nano zeolite into the polymeric matrix and the loading of Fe nutrition into the hydrogel network were all effective, according to the results of the FTIR analysis. The superabsorbent nanocomposite's temperature stability was demonstrated by the TGA research findings. The current study's findings demonstrated that the SR Fe fertiliser was successfully synthesised and may be regarded as the most promising option for slowly delivering nutrients to crops in order to meet their nutrient needs and improve nutrient use efficiency.

Key words: Fe fertiliser, Nano-zeolite, Slow release, Sugarcane bagasse.

INTRODUCTION

Utilizing fertiliser and water effectively is a crucial issue because these essential inputs limit the production of agricultural crops (Vejan et al., 2021). Depending on the method of application and the soil condition, between 30 and 70 percent of the nutrients from conventional fertilisers applied are lost without any benefit to the environment and unable to get to the intended plant, causing significant resource loss as well as serious environmental pollution (Anas et al., 2020). A successful strategy for minimising nutrient loss is the creation of slow-release fertiliser (SRF) compositions, which keep fertiliser concentration at an active level in soil by releasing nutrients in accordance with plant needs (Olad et al., 2016). These SRF formulations supersede conventional ones in a number of ways, including decreased application frequency, reduced fertiliser loss, sustainable nutrient supply and reduced environmental pollution concerns (Murawski et al., 2019). Superabsorbent hydrogel is among the most significant components served as an intricate polymer matrix in the creation of SRF compositions such as superabsorbent hydrogel (SAH). Water or solution that is aqueous can be absorbed and held in substantial quantities by SAHs, which are hydrophilic polymers having a three-dimensional cross-linked chains (Firmanda et al., 2022). Therefore, using them in agriculture can have favourable impacts like enhancing the retention of fertiliser in the soil, stimulating crop development while minimising the negative ecological impact posed by watersoluble fertiliser products. The main obstacles to their

¹Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore-641 003, Tamil Nadu, India. ²Centre for Agricultural Nano Technology, Tamil Nadu Agricultural University, Coimbatore-641 003, Tamil Nadu, India.

³Department of Crop Physiology, Tamil Nadu Agricultural University, Coimbatore-641 003, Tamil Nadu, India.

⁴Department of Agricultural Microbiology, Tamil Nadu Agricultural University, Coimbatore-641 003, Tamil Nadu, India.

Corresponding Author: C. Bharaani Sri, Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore-641 003, Tamil Nadu, India. Email: bharaanisri@gmail.com

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widespread development are their high production costs and non-biodegradability because the majority of SAHs are based on synthetic acrylate monomers (Kabir *et al.*, 2018). Starch, chitosan, cellulose and alginate are only some of the naturally occurring polysaccharides that have been put to use as beneficial SAH materials due to their abundance, lack of toxicity, biocompatibility and sustainability (Shariatinia, 2020). However, the use of sugarcane bagasse (SB), low cost industrial by product and the most practical source of starch for the synthesis of SAHs from an economic standpoint has gained little consideration (Kumar, 2022). In addition to providing novel perspectives on how to utilise agricultural residues effectively, using SB as SAH substrate fosters the biodegradability of SAHs (Arafa et al., 2022). Despite the unique benefits of hydrogels in the field of agrochemistry, their practical deployment is unrealistic due to the high production costs (Paswan et al., 2022). To address this challenge, considerable effort has been invested into formulating nanocomposite hydrogels employing clay minerals like zeolite, particularly nanozeolite. Farming applications have drawn a great deal of interest in nano-zeolite, a clay mineral with favourable physicochemical characteristics like strong ion exchange capacity, dehydration activity, a huge surface area and relatively low cost. Incorporating nano-zeolite within a hydrogel matrix lowers production costs while also enhancing the hydrogel nanocomposite's swelling properties, mechanical durability and thermal stability (Pimsen et al., 2021; Chakraborty et al., 2023). Iron (Fe) is an essential micronutrient that plays numerous vital functions in crop growth and development, which includes chloroplast development, chlorophyll synthesis and RNA synthesis. To produce crops with a good yield and quality, the soil must contain an adequate amount of iron in addition to primary nutrients (Arif et al., 2022; Kaur et al., 2022). The poor solubility of Fe within the pH range of 7.4 to 8.5, on the other hand, dramatically limits its availability for plants cultivated in calcareous soils, resulting in a serious micronutrient disorder known as lime induced iron deficiency chlorosis (Kalyan et al., 2021). Low fertiliser micronutrient use efficiency by crops (< 5%) is caused by a lack of synchronisation between the fertiliser micronutrient release and the crop requirement during the growing season (Jariwala et al., 2022). In the light of above points, developing suitable products as an alternative to conventional fertilisers would help the farmers to manage iron chlorosis in a cost effective way and eventually improve the crop and oil yield and returns to the farmers. The proposed project focuses on creating a new slow-release nanocomposite iron fertiliser based on hydrogels with superabsorbent properties that may efficiently enhance plants iron nutrition, increase use efficiency, improve crop quality and growth rate and decrease fertiliser loss. By grafting acrylic acid (AA) and acrylamide (AAm) monomers onto the SB backbone in the presence of zeolite and a Fe fertiliser component, a new kind of slow release fertiliser formulation was developed and characterised.

MATERIALS AND METHODS Materials

Sugarane bagasse (SB), ammonium persulfate (APS), ethanol, nano-zeolite, acrylamide (AAm), acrylic acid (AA), N, N'-methylene bisacrylamide (MBA), 1000 mL flatbottomed three-neck flask, reflux condenser, magnetic stirrer with hot plate, thermometer and nitrogen line. In this experiment, only analytical-grade chemicals were used.

Methods

Preparation of slow release Fe fertiliser hydrogel

An appropriate amount of pulverised sugarcane bagasse (particle size range = 40-80 mesh), a versatile and lucrative byproduct of the sugar industry, was mixed in 50 mL of Milli-Q® water, after which nano zeolite (99 nm) (10 weight per cent with regard to sugarcane bagasse) was added while stirring continuously. After this, the ensuing suspension was sonicated using a probe sonicator set to 50 W for 5 minutes with a 10 second on, 10 second off cycle, with the temperature held steady at roughly 32°C and the amplitude kept at 35%. The liquid was subsequent transferred to a 1000 mL flat-bottom, three-neck flask equipped with a reflux condenser, magnetic stirrer with hot plate, thermometer and nitrogen line. Starch macroradicals were formed by adding APS (10 weight percent) after the mixture was heated to the required temperature gradually and purged with nitrogen to remove dissolved oxygen and keep the environment anaerobic. After 10 minutes, a solution containing 70 per cent neutralised AA, 10% MBA and 2g AAm was added to the mixture. Following the inclusion of APS, FeSO, fertiliser was introduced. The polymerization procedure required a constant temperature of 60°C during the reaction time frame. Pieces of the resulting hydrogel were then cut out, treated with ethanol to remove any inert species and dried in a vacuum oven at 40°C until their weight remained constant.

Characterization

Morphological analysis

Scanning electron microscope (SEM)

To evaluate the exterior structure of the polymer, scanning electron microscope visualisation of the superabsorbent nanocomposite iron fertiliser was carried out. Emitech's SC 7620 Basic Magnetron Sputter Coater was used to sputter coat dried hydrogels with 10 nm gold after they had been placed in the carbon-coated stubs. The sputter-coated superabsorbent nanocomposite iron fertiliser hydrogel was examined using an energy-dispersive X-ray analyzer and a field emission scanning electron microscope (MIRA 3 TESCAN).

Structural characterization

Fourier Transfer-infrared spectroscopy

The molecular "fingerprint" method, also known as Fourier Transfer-infrared spectroscopy, is a potent instrument for identifying chemical bonds in a sample's molecules. The superabsorbent hydrogel's fertiliser loading and polymer crosslinking were both verified by the FTIR analysis. ATR PRO ONE was used to do an ATR (Attenuated Total Reflectance) scan on the small part of dried sample that was placed in the sample contact region of the spectrometer (JASCO FT IR 6800 Spectrometer). Samples were scanned with a resolution of 4cm⁻¹ and 64 times with 400-4000 cm⁻¹.

Thermal gravimetric analysis (TGA)

Using a PerkinElmer Pyris 1 TGA (United States) and a flowing nitrogen environment, thermal gravimetric analysis

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(TGA) was performed from 25°C to 1000°C at a heating rate of 10°C min⁻¹.

RESULTS AND DISCUSSION

Preparation of SH/zeolite superabsorbent nanocomposite Fe fertiliser hydrogel

In the presence of APS (an initiator), MBA (a crosslinking agent) and Nano-zeolite (a filler), graft copolymerization as well as crosslinking reactions of SB with AA and AAm monomers are depicted in Fig 1 as potential processes. APS molecules are initially thermally dissociated to produce sulphate radical anions (Czarnecka and Nowaczyk, 2021). These radicals convert the hydroxyl groups that are part of the starch molecules in SB into starch-based alkoxy radicals by eliminating hydrogen atoms (Olad et al., 2016). By forming these active radical sites on starch molecules, graft copolymer chains can propagate by transferring radicals to nearby AA and AAm monomers (Soliman et al., 2016). As the graft copolymer chains lengthen, the vinyl end groups of MBA molecules may interact synchronously with the copolymer chains to form a crosslinked hydrogel network. Zeolite, which may function as a physical crosslinking agent, can also inhibit the growth of polymer chains through a chain transfer process (Mushtaq et al., 2022). Direct loading of the Fe fertiliser was proceeded during synthesis. Using an AAS analysis approach, it was discovered that the total soluble Fe content of the FeSO added had 21% Fe (g of Fe/100 g of the product). Around 6.4% Fe with a 40% loading efficiency in terms of nutritional Fe loading was found within the hydrogel network. Due to the incorporated nano-zeolite's hydrophilic nature, SH/ Nano-zeolite has a greater potential to absorb water. This characteristic enables SH/ Nano-zeolite to absorb and hold more nutrients inside its hydrogel network (Rashidzadeh *et al.*, 2014). Moreover, the superabsorbent nanocomposite may have had a larger nutritional loading percentage due to some of the nutrients that zeolite absorbed to make up for its charge deficit.

Characterization

Morphological analysis

Scanning electron microscope (SEM)

Scanning electron microscopy was used to study the surface morphology of superabsorbent nanocomposite iron fertiliser. The electron microscopy images are given in Fig 2. The images show the well distributed pore space. The presence of porous structures justifies the crosslinking of polymer which might be due to generation of starch macroradicals from sugarcane bagasse. Numerous porous structures in the image confirms the high rate of cross-linking efficiency.

Additionally, an EDX study on an SR Fe was carried out in order to learn more specifics about the chemical makeup of the multinutrient fertiliser formulation. The weight proportion of each element in the SR Fe fertiliser is shown in Fig 3. The SR Fe fertiliser was successfully incorporated in the superabsorbent nanocomposite network, as shown by the acquired EDX diffractogram, which also shows the existence of peaks corresponding to the element Fe. The percentage of Fe loading was discovered to be 6.4%.



Fig 1: Proposed reaction mechanism for synthesis of superabsorbent nanocompositive Fe fertiliser.

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Structural characterization

Fourier Transfer-infrared spectroscopy

To investigate the chemical structure of the materials, FTIR spectroscopy was used. The FTIR spectra of SB, nanozeolite, Fe fertiliser, superabsorbent hydrogel without Fe (SH/Nano-zeolite) and SH/Nano-zeolite/Fe are displayed in Fig 4a to 4e.

Owing to the FTIR spectra of nano-zeolite (Fig 4b.), the broad absorption band at 3062 cm⁻¹ is attributed to the O-H stretching mode of intramolecular bonded water/ alcohol, whereas the strong peak at 1585 cm⁻¹ is attributed to the O-H bending mode of adsorbed water. The peak that manifested at a frequency of 1299 cm⁻¹ is connected to the stretching vibration mode of the O-H bonds present in the Al–OH and Si–OH groups. Additionally, the distinctive peak that can be found at 550 cm⁻¹ is related to the stretching vibration mode of Si-O-AI and Si-O-Si, whilst the peak that can be seen at 991 cm⁻¹ is linked to the bending vibration mode of Si-O-Si and Si-O-AI.

It may be concluded from a comparison of the FTIR spectra of nano-zeolite, SH + Nano-zeolite and SH + Nano-zeolite + Fe that the zeolite's distinctive absorption band at 3062 cm⁻¹ vanished. Additionally, in the FTIR spectra of SH/ Nano-zeolite and SH/ Nano-zeolite /Fe, the strength of the zeolite peaks at 550 cm⁻¹ and 991 cm⁻¹ reduced. These findings revealed that zeolite's hydroxyl groups (-OH) interacted chemically with AA and AAm monomers and formed bonds with polymer chains.

The typical absorption bands of the starch macromolecule are visible in the FTIR spectra of SB (Fig 4a). The peak at 1031 cm⁻¹ was the result of the stretching vibrations of the ether bonds (-CH-O-CH-) in the backbone of the starch macromolecule. O-H and C-H stretching



Fig 2: SEM image of SR Fe superabsorbent nanocomposite hydrogel.



Fig 3: EDX of SR Fe superabsorbent nanocomposite hydrogel.

vibrations, respectively, were also corresponding to the absorption bands at 3322 cm⁻¹ and 559 cm⁻¹.

It is clear from the FTIR spectra of SH + nano-zeolite (Fig 4e.) and SH + nano-zeolite + Fe (Fig 4d.) that the reaction declined the starch's characteristic hydroxyl absorption band (3322 cm^{-1}). Additionally, in the FTIR spectra of SH + Zeolite (1162 cm⁻¹), the peak associated with the stretching vibration mode of the ether bonds in starch still manifested itself with a slight wavenumber modification. These findings offer crucial proof that starch's hydroxyl groups participated in the grafting reaction along with acrylate-based monomers.

The distinctive absorption band at 3337 cm⁻¹ showed as a peak in the FTIR spectra of $FeSO_4$ fertiliser (Fig 4c), which was caused by the N-H stretching vibration mode. O=C=O and C-N stretching vibration modes, respectively, cause the peak at 2361 cm⁻¹ and 1122 cm⁻¹ to become apparent.

All of the distinctive peaks associated with Fe fertiliser are present in the FTIR spectra of SH + nano-zeolite + Fe and appeared with a small wave number shift. So, it can be said that the process of loading nutrients into the hydrogel network was successful. Some studies found results of a similar nature (Olad *et al.*, 2019; Dos Santos Pereira *et al.*, 2020; Kenawy *et al.*, 2020; Salimi *et al.*, 2020; Pimsen *et al.*, 2021; Mohamed *et al.*, 2022).

Thermal gravimetric analysis (TGA)

Hydrogel samples were subjected to TGA analysis to look into their thermal stability. As presented Fig 5, thermal degradation of FeSO, loaded hydrogel occurred in two steps, between 50°C-300°C, 330°C-665°C. The weight loss percentage was 28.9% when the temperature was raised between 50°C to 300°C and 42.8% of drop in weight was recorded between 330°C to 665°C and a total of 92.9% degradation was measured throughout the analysis. When the temperature was gradually raised from 0°C to 900°C, a slight weight loss after 100°C was attributed to the evaporation of moisture from sugarcane bagasse and the trough past 100°C can also be associated with the elimination of water that has been adsorbed, interlayered, or coordinated to the exchangeable cations in zeolite. The dehydration of zeolite is realized at the temperature between 200°C to 280°C. Zeolite contains 15.72% of water. After the dehydration, 1.32% of water remained in the zeolite structure. According to the structural measurements of zeolite, two types of water that is connected to the nearby atoms by variably long coordination bonds (I. H₂O bond from 0.259 to 0.267 nm; II. H₂O bond from 0.211 to 0.251 nm). This implies the gradual decrease in loss of moisture present in super absorbent nanocomposite. Dehydration of saccharide rings and the cleavage of glycosidic C-O-C bonds in starch chains account for the decomposition of the sample between 250°C and 384°C. Between 235°C to 325°C the vaporisation of bagasse is greater due to thermal degradation of hemicellulose present in the bagasse. In contrast to lignin, which is a more complex macromolecule made up of phenolic hydroxyl, benzylic hydroxyl and



Fig 4a: FTIR spectra of sugarcane bagasse.



Fig 4b: FTIR spectra of nano-zeolite.



Fig 4c: FTIR spectra of FeSO₄ fertiliser.



Fig 4d: FTIR spectra of not loaded hydrogel.

carbonyl groups and connected by straight links, cellulose degrades very quickly above 235°C while lignin present in bagasse continues to exist. If the weight loss of the sugarcane bagasse during pyrolysis can be expressed as the sum of the corresponding weight losses of each of its



Fig 4e: FTIR spectra of FeSO₄ loaded SR Fe fertiliser.

constituents, then at temperatures above 325°C, lignin and cellulose decomposition occur together in the sugarcane bagasse and below 325°C, lignin and hemicellulose decomposition predominate the sugarcane bagasse pyrolysis (Gharekhani *et al.*, 2018; Calcagnile *et al.*, 2019).

The DTG curve, which represents the rate of material weight change during heating, exhibits a peak between 300 and 450°C that is associated with the thermal breakdown of carboxyl and amide groups in co-polymer chains as well as the scission of co-polymer chains, which is associated with the emission of ammonia and CO_2 gases. (Thombare *et al.*, 2021).

The trough in the DTG curve between 472°C and 742°C is accountable for the decomposition of co-polymer chains, the decomposition of cross-linked network structure and the disintegration of water molecules from two adjacent carboxylic groups on polymer chains. This reveals that the heat stability of the super adsorbent hydrogel was increased by zeolite addition to the polymeric matrix. This resulted from the barrier effect of zeolite, which enhanced the thermal





stability of the composite materials by preventing the diffusion of oxygen and volatile thermos-oxidation products. In addition to forming a robust hydrogel framework with great thermal stability, the injected zeolite developed super adsorbent nano composite frameworks with strong physical crosslinks. (Xu *et al.*, 2019; Guo *et al.*, 2021; Rizwan *et al.*, 2022).

CONCLUSION

Graft co-polymerisation mechanism proved successful in synthesising a slow-release Fe fertiliser. The morphological, structural characterization of synthesized SR Fe fertiliser was studied. SEM results depicts the well distributed pore space and EDX results confirms the successful loading of fertiliser Fe into the superabsorbent nanocomposite. According to the findings of the FTIR analysis, the grafting of acrylate-based monomers onto starch molecules, the inclusion of nano zeolite into the polymeric matrix and the loading of Fe nutrition into the hydrogel network were all successfully carried out. The results of TGA analysis indicated the thermal stability of the superabsorbent nanocomposite. The results of the present study revealed that the SR Fe fertiliser was successfully synthesised and can be considered as an excellent candidate for supplying nutrients in slow manner to meet the crops nutrient demand and to enhance the nutrient use efficiency.

Author statement

C. Bharaani Sri, R. Shanmugasundaram, S. Marimuthu: Conceptualization and overall drafting manuscript. C. Bharaani Sri performed the laboratory experiments. T. Chitdeshwari, A. Senthil, T. Kalaiselvi supported the manuscript revision and uploading. C. Bharaani Sri, R. Shanmugasundaram, S. Marimuthu, T. Chitdeshwari, A. Senthil, T. Kalaiselvi reviewed and edited original manuscript to strengthen the scientific discussion.

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Declaration of competing interest

There is no conflict of interest.

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