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Effect of Zeolite/ Hydroxyapetite Nanofertilizer on Soil Quality, Nutrients Status and Plant Productivity in Solanum *melongena*

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Abstract

In the modern era of nanotechnology, nanomaterial-based nanofertilizers are emerging as promising alternatives for sustainable agricultural practices. The development of Zeolites and hydroxyapetites-based nanofertilizers have attracted great attention due to their high impact and benefits in agricultural production benefits in agricultural production. In this study, a hybrid nanocomposite of Zeolite/ hydroxyapatite (ZHNC) was used as a nanofertilizer and the impacts of ZHNC on soil physico-chemical quality, soil water retention potency and corresponding response on plant growth and productivity was observed. Scanning electron microscopy (SEM), energy dispersive X-ray (EDX) analysis, FTIR Spectroscopy were used to charecterise nanoparticle by examining their morphology, elemental nature and chemical composition. SEM results detect the presence of needle-like hydroxyapetite crystals on the amorphous zeolite matrix. Its chemical composition and elemental nature were confirmed by FTIR and EDX. Soil studies were performed to assess the impacts of ZHNC tretatmens on soil quality and nutrient potency. Results revealed that ZHNC possesses the great potential to improve soil quality via influencing different parameters i.e., higher CEC, lower bulk density, higher porosity, retains of good moisture as well and maintains higher nutrients Along with, it also promotes plant growth and productivity, coincident with increased nutrients using *Solanum melongena* as an experimental plant. Hence, ZHNC can be used as a suitable substitute for fertilization in agricultural practices. Overall remark, ZHNC is proven to be highly efficient nanofertilizers in improving soil conditioning and in turn boosting plant growth and productivity, retaining good levels of organic nutrients (Organic P significantly). Hence, ZHNC can be used as a suitable substitute for fertilization in agricultural practices.

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Introduction

Global food security is a highly challenging task that initiates the agriculture sector to be revolutionized on time. The overgrowing world population and limited arable land have forced new agro-technological approaches to sustain agricultural production. The use of fertilizers to increase crop production is a common agricultural practice since past periods. However, conventional fertilizers are now less preferable due to their major drawbacks as lesser efficiency of nutrient delivery to soil and plant systems, major contents (50-70%) are washed away from the soil through leaching before being used.[1] Overuse of these fertilizers resulted in environmental pollution and disruption of soil quality and agro-ecosystem.[2] The adoption of sustainable agricultural approaches are more viable solution to ensure food security without compromising the soil fertility as well as preserving environment and natural resources. In the recent eras, nanotechnology- based fertilization processing has shown high efficiency to resolve the food scarcity problem.3 Fertilizers in the nano-dimension (Nanofertilizers) can be efficiently utilized by soil and plants to deliver adequate nutrient requirements in a slow and sustained way, improving in vivo nutrient delivery and ensure the distribution of nutrients precisely.[4,5,6] Nanofertilizers improve soil properties viz. optimized availability of macro and micronutrients in soil, long- term nutrient accessibility in soil and avoiding nutrient loss due to leaching.[3] Several studies reported on novel nano-assisted fertilizer developments, such as chitosan-based nanofertilizer,[7] urea–formaldehyde polymer nanocomposite,[8] Urea-Kaolinite Nanocomposite, thermoplastic starch/urea (TPSUr),[9] nanozeolite/ Hydroxyapatite,[10] chitosan-montmorillonite (MMT) nano-composite hydrogel.[11]

Among different nanomaterials, nanozeolite and Hydroxyapetite based fertilizers are considered high potential nanofertilzer for the optimized delivery of essential nutrients to the soil and plant system in a slow and sustained way. Its nano pore size, high ionic exchange capacity, high rehydration capacity, high nutrient absorbance capacity make them highly applicable as nanofertilizer.[12,13,14] Zeolites, aluminosilicate compounds are one of the most promising materials for slow- release nano-fertilizer (SRF) because of its high specific surface areas, ion-exchange abilities, nano-porous structure.[12] Zeolite NaP1 is a synthetic form of nanozeolite, synthesized from various wastes using an alkaline hydrothermal treatment.[15] It acts efficiently in enhancing the nutrient retention capacity of soil up to longer periods time because of its slow disintegration and decomposition rate in soil.[16] Zeolite NaP1 actively mobilizes the ammonium and potassium ions in plant- available form, due to its higher affinity with cationic groups. However, the immobilization of phosphate ions is difficult for zeolite structures due to its lesser affinity for anionic groups.[12] To resolve this problem, various studies suggested the fabrication of zeolite with hydroxyapetite nanoparticles (HAP, [Ca_{10} (PO_4) 6 H_2O] crystal calcium phosphate. HAP aids the better efficacy for phosphorus ion mobilization in soil and plant system.[17,18,19] Hybrid nanocomposite zeolite NaP1/hydroxyapatite(ZHNC) is proven as high potential nano-fertilizer having a strong affinity to bind both cationic and anionic nutrients and released over a long time.[9,12] However in the past, studies have mainly concern with its synthesis and physico-chemical characterization of zeolite NaP1/hydroxyapatite(ZHNC).[12,15] For sustainable applicability of ZHNC as a nano-fertilizer in the field it is important to examine its accessibility to soil and plant systems.

This study aims to investigate the effects of ZHNC on the soil physico-chemical properties, water retention capacity, soil nutrient status and on the other hand to analyze the corresponding response on plant morphological traits, growth, biomass production and nutrient uptake using the vegetable crop *Solanum melongena* as an experimental plant.

Materials and Methods

Zeolite NaP1/ hydroxyapatite nanocomposite (ZHNC)

The Zeolite NaP1/ hydroxyapatite nanocomposite (ZHNC) used in this study was was purchased from a nano-product company NanoLab, Jamshedpur, Jharkhand, India.

ZHNC is a hybrid nanocomposite of zeolite and nanohydroxyapetite components. Zeolites are three dimensional, crystalline, porous hydrated aluminosilicates compounds containing the constituent SiO_4^{4-}, AlO_4^{5} and various other elements (sodium, potassium, calcium, magnesium). Zeolite NaP1 ($Na_6Al_6Si_6O_{32}.12H_2O$) is a synthetic zeolite form, hydrothermally synthesized from fly ash treating with sodium hydroxide at atmospheric pressure.[12] Zeolites are having nanoporous structure, unique physico-chemical properties, high specific surface areas, good ion-exchange abilities and environmental friendly catalytic properties which make them suitable for use in slow-release fertilizers.[20] Hydroxyapatite $[Ca_{10}(PO_4)6(OH)_2]$ HA is a mineral composed of phosphate ions and calcium ion and found naturally in teeth and bones. Its synthetic nano-form (Nano-hydroxyapatite) has shown high potency as phosphate fertilizer due to its lower solubility and slow-release of phosphate ions.[14] Hybrid nanocomposite of zeolite NaP1 and HA nanocomposite are reported to be high potential nanofertilizer for slow-release of multi-nutrients including phosphate ion.[12,14]

Zeolite / hydroxyapetite nanocomposite characterization

The zeolite / hydroxyapetite based nanocomposite was characterized via different techniques i.e., scanning electron microscopy (SEM), energy dispersive x-ray spectrometer (EDX) and Fourier transform infrared spectroscopy (FTIR).

Structural and Chemical Composition of Zeolite/ Hydroxyapetite Nanocomposite (ZHNC)

SEM images reveal the surface morphology of $Ca2^+$ –zeolite NaP1/ hydroxyapetite nanocomposite (Fig.1 a, b). As shown in Fig. 1. Ca^{2+}–zeolite NaP1/ hydroxyapetite nanocomposites are amorphous structure showing zeolite NaP1 with needle-like hydroxyapetite crystals on them. Elemental nature of ZHNC nanocomposite was confirmed by EDX spectra, where peaks hikes in regions of 0.25, 0.50, 1.30, 1.50 and 1.75 and 2.0 keV represent the binding energies of Ca, K, O, Fe, Mg, Al, Si, P (Fig. 2). FTIR spectra (Fig. 3) exhibited distinct absorption peaks of ZHNC at 3694.4, 3441.4, 1651, 1032.1, 912.8, 797, 518.2 and 469 cm^{-1}. Major characteristic FT-IR peaks were present at 1032 and 912 cm^{-1}. The other major absorption peaks occur in the regions 3620 –3433 cm-1 and 1640 – 1024 cm^{-1} and the region below 800 cm-1 (Fig. 3).

Site Description and Soil Properties

The experiment site was Department of Botany, Langat Singh College, Muzaffarpur, located at 25°54' N and 84°53' E. Maximum and minimum temperatures during the growing season are 35°C -20 °C. Experimental soil is new alluvium type. Its main characteristics were 0.86% organic matter, 0.49% organic carbon, pH 7.8, 162.84 dS m^{-1} CEC 54.80 meq Kg^{-1} soil). The available nitrogen, phosphorus and potassium values were 21.4 mg N Kg^{-1}, 4.63 mg P Kg^{-1}, and 49.4 mg K Kg^{-1}. Other details are mentioned in Table 2.

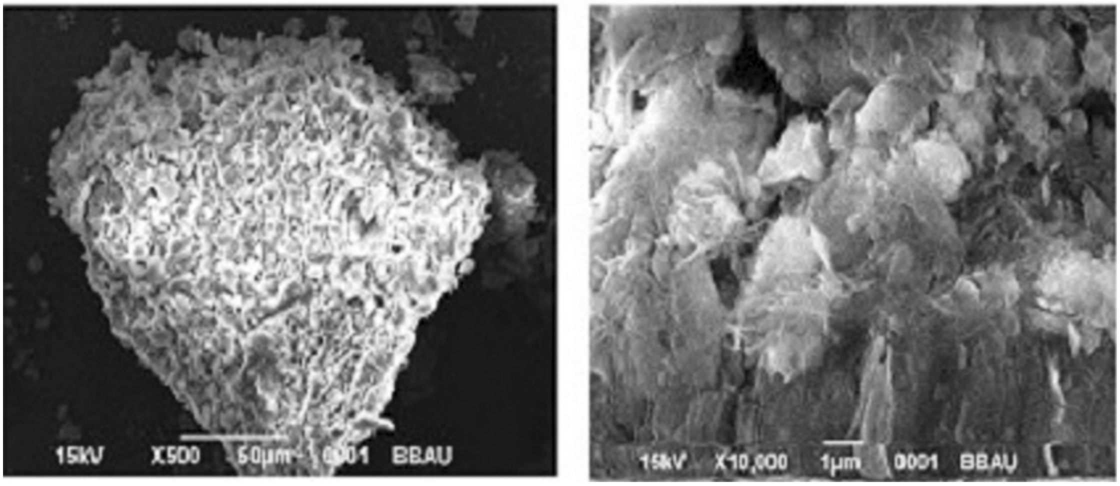

Fig. 1: SEM image of zeolite NaP1/Hydroxyapetite nanocomposite showing structural morphology

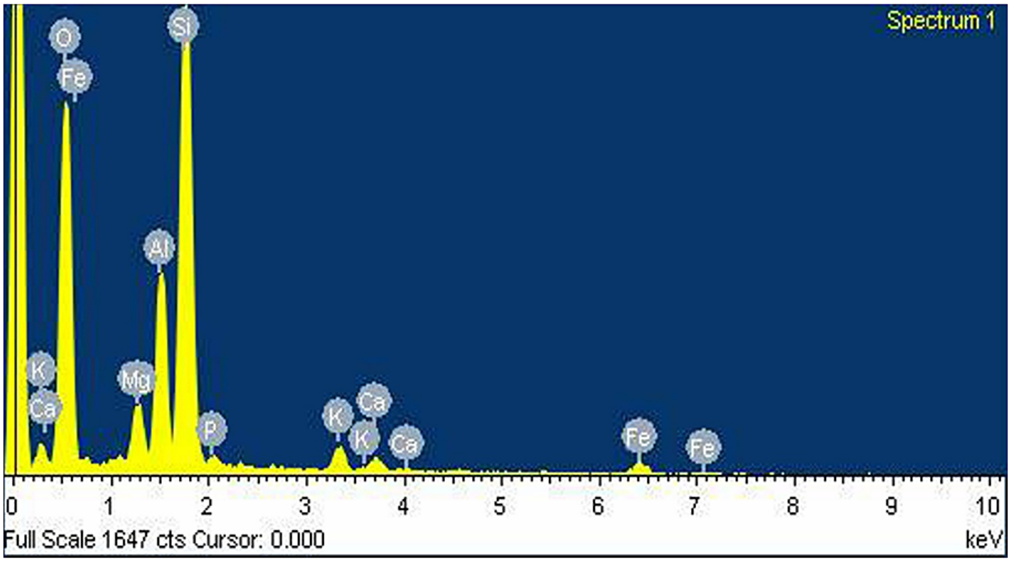

Fig. 2: EDX analysis of Zeolite NaP1/ hydroxyapetite nano-composite showing elemental nature

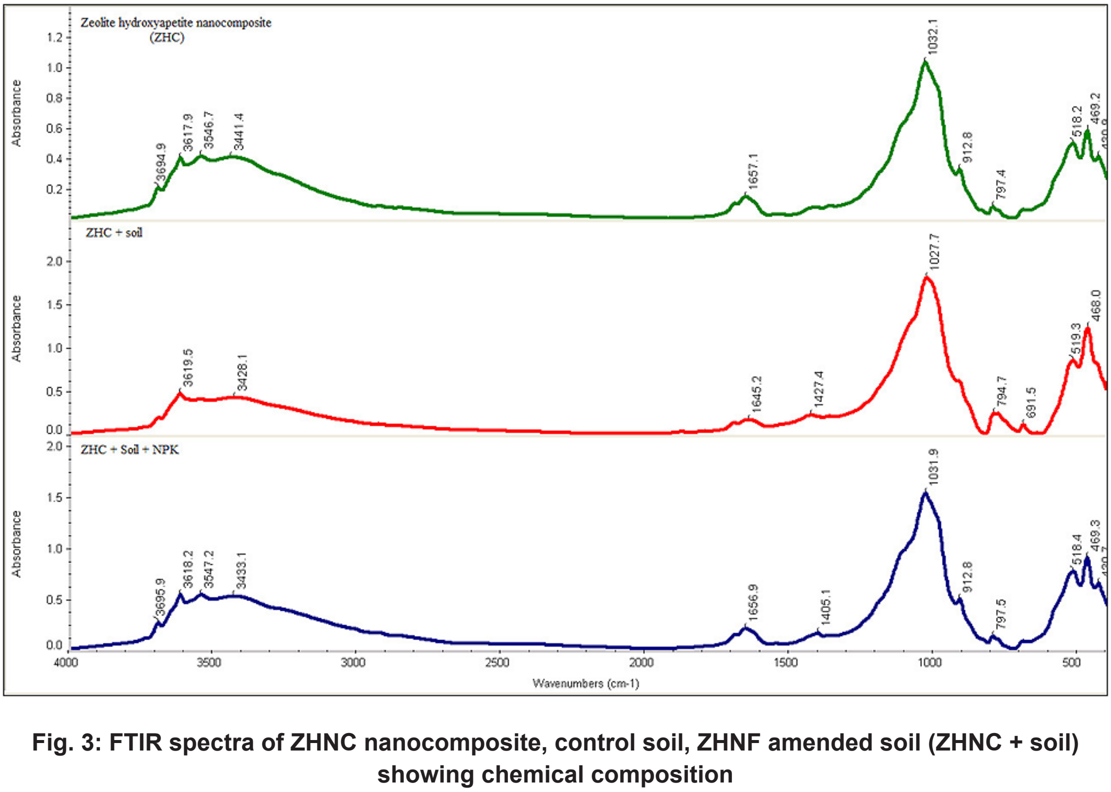

Fig. 3: FTIR spectra of ZHNC nanocomposite, control soil, ZHNF amended soil (ZHNC + soil) showing chemical composition

Table 1: Treatment set ups of different fertilizer- amended soil

	Treatments	Soil application with Base Fertilizer
i).	C	Control soil (without any treatment)
ii).	F	inorganic NPK fertilizer
iii).	NZF	Nanozeolite based fertilizer
iv).	ZHNF	Nano Zeolite/ Hydroxyapetite based nanofertilizer

Experimental design and Treatment conditions

In the ontrol set up, no fertilizer treatments were provided to the soil. For F treatment (ii) recommended dose of NPK as per Agriculture Research Institute, Pusa was amended to soil. In Treatments (iii & iv); 5% of nutrients (N, P, K) in the form of their salts were embedded with a respective dose of 2% (v/w) of NZ and ZHNF aqueous suspension res. The plant experiment was conducted in the soil pot culture, grown under the recommended doses of fertilizers amended soil.

Test for Soil Physico-Chemical Property

Control and different fertilizer amended soil samples were subjected to following physico-chemical analyses by the methods indicated below.

Soil pH

The pH of the soil was determined electrometrically in aquous suspension of soil using electrode on a digital pH meter.[21]

Electrical Conductivity

Electrical conductivity of the soil sample was measured in aquous soil suspension at 250 C using conductivity meter.[22]

Bulk Density

Soil samples were oven dried and weighed and bulk density (g cm^{-3}) was calculated from the known weight and volume of the soil mass as determined by the Black (1965) method.[23]

The formula for the calculation of Bulk density (B.D) is as follows.

B.D. = Oven dry weight of soil /Volume of soil

Organic Carbon

Organic carbon in the soil samples was determined according to Walkely-Black chromic acid wet oxidation method.[24]

Water Holding Capacity of Soil

To describe the water status in a soil, the water holding capacity (WHC) of the soil was analyzed by methods of Keen box method.[25] To determine the water holding capacity by mass, the following equation was used.

Water Hilding Capacity (WHC) % = Total quantity of water present in the wet soil / Oven dry weight of total soil X 100

Soil N, P,K Analysis

Available nitrogen in soil was estimated using the alkaline potassium permanganate by the Kjeldahl method.[26] Available phosphorus by the method of Olsen *et al.* (1954)[27] and soluble potassium was also estimated by the method described by Jackson (1958).[28]

Plant Analysis

Experimental Plant

Eggplant (*Solanum melongena* L.; *Solanaceae* family) is chosen as an eperimental plant to evaluate the impacts of different fertilization conditions on plant systems. Eggplant fruits are high in fiber contents, vitamins and minerals and rich sources of iron and manganese). It is an agronomical and economically valuable vegetable crop.

Parameters Assessed

Eggplants grown under different fertilizer- amended soil were examined for its morphological traits and biomass productivity. Different parameters i.e., seed germination %, Plant height, thickness, plant weight, fruit length and fruit weight was assessed at the harvest stage of plants (60 DAG)

Statistical Analysis

The data was analyzed using Microsoft Excel and Sigma Plot 12.5 software. All the readings are reported as an average of three repeats.

Table 2: Comparative change in physicochemical characteristics of soil after ZHNC and other fertilizer amendment after 14 days incubation period

Soil parameters	Control soil	Inorganic fertilizer amended soil	Nanozeolite amended soil	Nanozeolite/ Hydroxyapetite amended soil
pH	7.84 ± 0.59	8.19±0.43	7.23±0.58	7.52±0.63
Bulk density (g cm^{-3})	1.28 ± 0.11	0.98±0.04	0.69±0.03	0.64±0.03
Pore density (g cm^{-3})	2.43 ± 0.11	3.61± 0.21	4.58± 0.34	5.76± 0.43
Water-soluble salts (meq Kg^{-1} soil)	47.85 ± 2.36	57.04 ± 4.38	72.31 ± 5.23	69.58 ± 4.87
Water holding capacity of soil (%)	42.62± 3.97	59.17± 4.3	68.23±4.7	73.76±5.1
EC (µS cm^{-1})	162.84 ± 9.8	217.23±23.4	286.23±17.6	314.23±15.9
Cation exchange capacity (meq Kg^{-1} soil)	54.80 ± 0.36	72.68±3.28	94.73±6.82	112±10.5
Soil Organic carbon (%)	0.49 ± 0.015	0.73±0.02	0.95±0.03	1.13±0.45
Organic matter (%)	0.86 ± 0.25	1.37±0.13	1.84±0.61	2.17±0.83
Total Nitrogen (g kg^{-1})	8.6±1.1	12.3±0.92	15.8±1.0	18.2±0.83
Nitrogen (avail) (mg/ Kg)	21.41±0.12	38.26±0.21	56.61±0.98	64.87±0.86
Phosphorous (avail) (mg/Kg)	4.63 ± 0.25	7.06±0.28	15.9±1.1	19.85±2.34
Potassium (mg/Kg)	59.4 ± 15.3	78.6±0.11	94.8±0.36	109.6±0.42

Values are mean ± S.E. of three replicates.

Results

Effect of ZHNF on Soil Physicochemical Characteristics

Soil analysis results interpret that experimental soil is sandy loam in texture with a pH range of 7.2-8.4, organic matter (SOM) approx. 0.86 %, total organic carbon (SOC) 0.43%, total N (8.6 g Kg^{-1}), available N (21.41 mg Kg^{-1}), available P (4.63 mg Kg^{-1}), total K (59.4 mg Kg^{-1}) dry weight. Amendments of different fertilization in soil resulted in elevated values for these parameters (Table 2). Total Organic carbon (TOC) and organic matter (OM) showed a significant hike of approx. 2 to 2.5 fold after NZ & ZHNF relatively when compared with untreated (control) soil. Next on, soil bulk density found to decrease from 1.28 to 0.64 g cm^{-3}, whereas poring density in reversal increased from 2.43 to 5.76 g cm^{-3}), cation exchange ability (CEC); from 54.80 meq Kg^{-1} to 112.68 meq Kg^{-1} after ZHNF treatments in respective to control (Table 2). Among soil nutrients, significantly the highest increase in N, P, K values were observed in ZHNF treatment with respect to both other treatments (NZ & F), when compared with untreated control soil. NZ and ZHNF by induced 2.6 & 3 fold increments in the soil available N. Potassium availability in the soil also maintained at a higher level with relative increase of 59% and 84.5% in NZ and ZHNF treated soil in respective to control. Mean comparison for soil available phosphorous content also follows a similar trend with a maximum increase (almost 4.3 fold) in ZHNF, followed by NZ (2 times).

ZHNF Effect on Water Holding Capacity of Soil

In the present study, the addition of different fertilizer treatments significantly increased the WHC of the soil. Importantly, the addition of ZHNF shows the maximum increase in the value of WHC of soil (73%), followed by NZ (60%), whereas F treatment showed only up to 20% hike when compared with untreated control soil (Fig 4).

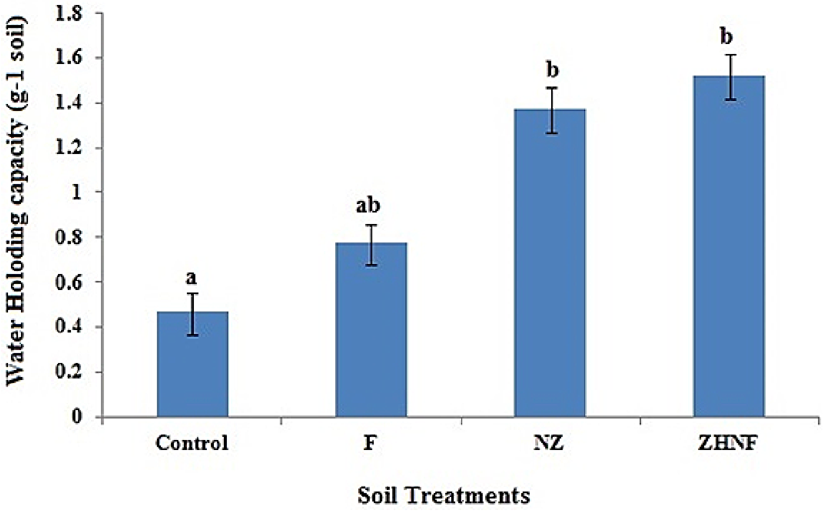

Fig. 4: Water holding capacity (WHC) of the soil under ZHNC and other fertilizer treatments

Effects of ZHNF on Plant Growth Traits and Productivity

At the stage of harvest (60 DAG), eggplants grown in ZHNF- amended soil are significantly taller (91.3 cm) and thicker (2.86 cm) when compared with control and other treatments (Table 3). In addition, the plant biomass and fruit weight also increased significantly ($p < 0.05$) by 1.5 and 2.4 fold (Table 3, 4).

Nutrient analysis results in fruit parts also follow the trends of maximal increase in N, P and K values (86, 148 & 74% respectively) in ZHNF- treated ones, followed by NZ and F respectively (Table 3). The significant ($p < 0.05$) higher increase in organic phosphorus (almost 2.5 fold) in plants grown under ZHNC- treated soil.

Table 3: Comparative change in seed germination potency, plant morphology, plant biomass in purple eggplant *Solanum melongena* grown under ZHNC and other fertilizer- amended soil

Soil treatments	Plant observation			
	Seed germination (%)	Plant height (cm)	Stem diameter (cm)	Plant dry wt. (g)
Control	45%	65.6±9.6	0.85 ±0.05	24.32±0.11
Inorganic fertilizer	63%	73.4±8.4	1.27 ±0.12	31.6±0.13
Nanozeolite	85%	83.7±8.7	2.58 ±0.14	52.9±0.12
Nanozeolite/ Hydroxyapetite	89%	91.3±10.1	2.86 ±0.11	60.2±0.14

Results are mean ± S.E. of three replicates

Table 4 Comparative analysis of eggplant fruit size, biomass and nutrient contents in plants grown under ZHNF and other treatment soils at stage of 60 DAG.

Parameters		Treatments			
		Control (C)	NPK fertilizer (F)	Nanozeolite (NF)	Nanozeolite/ Hydroxyapetite (ZHNF)
Eggplant fruit observation	fruit height (cm)	16.29±1.1 a	19.13±1.ab	21.21±2.1b	21.99±1.6b
	Fruit diameter (cm)	3.26±0.2a	4.2±.0.2b	5.16±0.3c	5.93±0.3bc
	No. of fruit per plant	5.2±0.2a	7.6±0.4b	10.8±0.9b	11.5±1.0b
	fruit dry weight (g)	9. 29±8.6a	12.8±1.1b	14.35±9.8b	17.8±12.2b
Macro-nutrients in fruit (% DW)	Nitrogen	3.6±0.12a	5.8±0.26b	6.1±0.18b	6.7±0.34b
	Phosphorus	0.29±0.02a	0.35±0.01ab	0.42±0.03b	0.72±0.03c
	Potassium	4.7±0.23a	6.3±0.27b	7.8±0.32c	8.2±0.28c

Results are mean ± S.E. of three replicates. Values with different letters in the same column differ significantly at $p<0.05$.

Discussion

Structural morphology of Zeolite NaP1/ hydroxyapetite nanocomposite detects the presence of needle-like hydroxyapetite crystals on an amorphous zeolite matrix. Structure well supported with previous related research publications.[29,30, 31] EDX analysis of ZHNC nanocomposite detects the presence of elements Fe, Mg, Al, Si, P. FT-IR peaks assigned to the hydroxyl groups, vibrations of Al-SiO oxide bonds and the PO_4^{2-} group.[30,31] Characteristic peaks at 1032 and 912 cm^{-1} assigned to the asymmetric stretching vibration of the AlO and SieO tetrahedral.[32] Absorption spectra for control and ZHNC amended soil were very different, which refers to a change in the chemical composition of soil after ZHNC nano-composite amendment. The major absorption peaks are attributed to specific chemical constituents including sand, clay, and organic components. The weak broad band at 1640 and 1428 cm^{-1} may be due to the C=O and C-H bending and starching O-H deformation.[31] IR peaks at 1025–800 cm^{-1} and 700–400 cm^{-1} refers to silicate (Si-O) stretching, a major soil component.[33] The region below 1025 cm^{-1} is the fingerprint region of soil, where the absorptions are mainly due to O-H bending N-H bending in the lattice of clay, silt, sand.[34]

Soil analysis results significantly revealed the prominent beneficial aspects of ZHNC treatment in improving soil quality and soil nutrient availability. An increase in soil organic matter and carbon content relates to improved soil health and biological functioning of soils.[35] Whereas ZHNC decreased bulk density, high porosity, CEC of soil consequently interpret to loose, well aerated, porous soil texture.[36] The higher retention of the nutrient elements (especially nitrogen and Phosphorus) in soil upon the nanocomposite treatment may refer to optimized nutrient uptake by soil particles and lesser loss through mineral leaching. Water holding capacity (WHC) of the soil is also one of the most important physical soil factors for sustainable agriculture management. In view, soil analysis results also show a significant enhancement in water holding capacity of the soil indicating the continued water availability in soil for plant requirement during replenishment. Herein, ZHNF acts significantly in improving soil moisture content vis avoids drought tolerance of plants.

Significant positive impacts of ZHNC in improving soil quality in turn results in to increase in plant growth and productivity grown under ZHNC amended soil along with high nutrient assimilation potency. Highest increase in organic P level in ZHNF treated may be due to facilitation in P solubility in plants by hydroxyapetite as supported by previous research literatures.[10]

Conclusion

The results of the present study well interpret that hybrid nanozeolite/hydroxyapetite base fertilizer amendment in the soil acts positively in improving the soil physico-chemical properties, its water retention potency and overall sustaining the water reservoir for the soil-plant system. Further on, plant observation well suggests their positive impact on the plant system, boosting its growth and productivity. ZHNF significantly increased the fruit biomass and the content of bioavailable phosphorus along with Nitrogen and Potassium at higher levels in respective to other treatments, indicating the holistic perspective on the potential high benefits of this nanocomposite as a nanofertilizer.

In an overall remark, Zeolite/ Hydroxyapetite acts as high-value nanofertilizer, helps to facilitate nutrient uptake in soil and water, and accelerates its mobilization, transportation, absorption in plant systems. It also proved its high efficiency in the absorption of phosphate ions along with other nutrients, which is difficult to mobilize in the case of singly zeolite or other fertilizers. Phosphate is the vital nutrient that contributes significantly to crop productivity. So on, ZHNF has proven as a better alternative as a multi-nutrients fertilizer (highly efficient N & P) with obtention of high crop yields and productivity.

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Conflict of Interest

The author declares no competing interests.

Informed Consent Statement

As Rima Kumari is a sole Author of this manuscript and Principal Investigator of concerned research project, informed consent statement is not applicable.

Authors' contributions

As a sole author R. Kumari solely conceptualize the experiment, methodology standardization, and data preparation, experimental analyses, writing the original manuscript and review.

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