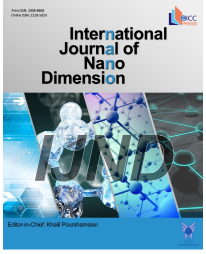

Volume 15, Issue 4, 152430 (1-8)

International Journal of Nano Dimension (IJND)

https://dx.doi.org/10.57647/j.ijnd.2024.1504.30

Sol gel synthesis of undoped and nitrogen doped Titanium Dioxide nanoparticles as nano fertilizer for plant growth

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Original Research

Received:
4 June 2024
Revised:
2 July 2024
Accepted:
10 July 2024
Published online:
10 October 2024

Abstract:

The present study organized the different concentrations of undoped and nitrogen doped titanium dioxide nanoparticles have synthesized during agriculture as Nano fertilizer as well as the physiological impact on the seeds of green gram and Fenugreek. X-Ray Diffraction analysis confirms the presence of anatase and brookite structure of undoped and N doped TiO_2 nanoparticles. The Fourier transform infrared spectroscopy shows the presence of functional groups of the prepared nanoparticles. The Scanning Electron Microscopy was carried out to determine the morphology of prepared nitrogen doped titanium dioxide nanoparticles. The elemental composition and atomic percentage of the prepared samples were confirmed by energy dispersive X-Ray analysis. The band gap energies of the prepared nitrogen doped titanium dioxide nanoparticles were studied using UV-vis Diffusion Reflectance Spectroscopy. The pH value and the total NPK availability were analysed for the undoped and nitrogen doped titanium dioxide nanoparticles at different ratio 1:1 and 1:2 and the biological growth of the plants were observed.

Keywords: 2^{nd} orientations; Nitrogen; Plant growth; Sol gel method; Titanium dioxide; W-H Plot; XRD analysis

1. Introduction

Nanotechnology is an emerging field in 21^{st} century, which aims to acquire the Nano materials with novel, significant characteristics and improved features at Nano scale level. Nano technology is capturing more interest in research area because of its significant applications in various fields such as cosmetics, agriculture [1, 2] healthcare, textiles [3, 4] electric and electronic fields, medical fields, aerospace and in semiconducting devices worldwide. In the recent years, the nano materials have been gained more attention in the field of sustainable agriculture, and have high level of potential applications. Nano materials are used as Nano sensors, Nano pesticides for sensing the environmental stress, setting of crops, biotic resistance and also used as Nano fertilizers to enhance the growth of plants and crop yield [5–9].
The method of preparing nanoparticles using Sol-gel, have recently gained more attention because of its Non-toxic, cost effectiveness and environmental friendly. Nutrient availability plays an important role in the field of agriculture. Crop productivity, high yield and quality of crops are highly dependent on soil nutrients. Fertilizers play a significant role for enhancing the production of food and their quality subsequently the initiation of high-yield with fertilizer perceptive varieties. The nano fertilizers have been improved the contribution towards the growth and evolution of crops, for substantial absorbance and high reactivity. Herewith the Nano fertilizers plays an important role for controlling and modifying the availability of nutrient will leads the nutritive value of crops. Application of Nano fertilizer enriches the nutrient content in soil and improves the crop yield. Hence, the productivity of the crops are highly depends on the usage of fertilizers in soil. In recent years, the Titanium dioxide (TiO_2) nanoparticles were used as Nano fertilizer which helps the plants to uptake the presence of nutrients from the soil. Titanium dioxide (TiO_2) nanoparticles are used as nutrient for crop growth and development. Titanium dioxide (TiO_2) nanoparticles also help to enhance the presence of photosynthetic pigments in plants such as chlorophyll (a&b), anthocyanin contents and carotenoids. Herewith, the Titanium dioxide (TiO_2) nanoparticles provide more posi-

tive effects which leads to increase the growth of the plant and yield acquired from crops [10]. Other than fertilizers, the Titanium dioxide (TiO_2) nanoparticles are also used as Nano pesticides and as Nano sensors in sustainable agriculture.

Titanium dioxide (TiO_2) nanoparticles are regulating the activity of enzymes which involved in nitrogen metabolism such as nitrate reductase, glutamate dehydrogenase, and glutamine synthase and glutamic pyruvic transaminase. These activities help the plants to absorb nitrate and also make the conversion between the inorganic nitrogen and organic nitrogen to form a protein and chlorophyll which increases the biomass of plant. Titanium dioxide (TiO_2) nanoparticles also used as photo catalyst which induces the oxidation – reduction reaction [11–15]. Herein, this reaction promote the formation of chlorophyll, stimulate Ribulose 1, 5-bisphosphate carboxylase (Rubisco) activity and increases the process of photosynthesis in plants which induces the marker gene Rubisco activity (RCA) mRNA. Furthermore, Titanium dioxide (TiO_2) nanoparticles increase the rate of photosynthesis in plants and also enhance the growth of plants and crop yield by increasing nutrient uptake efficiency. Herewith, Titanium dioxide (TiO_2) nanoparticles are used to increase the germination rate, and length of root with an improved growth rate.

The application of Anatase Titanium dioxide (TiO_2) nanoparticles, will be increase an whole chain of electron transport, evolving O_2, photophosphorylation activity of chlorophyll under both visible and ultraviolet light and photo reduction activity of photosynthesis II. Moreover, the application of Titanium dioxide (TiO_2) nanoparticles (in low concentration) via roots and leaves will increase crop enzyme activity, enhancing chlorophyll content and photosynthesis, promoting mobilization of nutrients, biotic and abiotic stress tolerance and by improving the crop yield and quality.

Sol-gel method is a simple, sophisticated and promising method for the synthesis of metallic oxide nanoparticles [16]. In the present work, nitrogen-doped Titanium dioxide (TiO_2) nanoparticles were prepared by sol-gel method by using urea and organometallic precursor TTIP Titanium isopropoxide. Then the prepared nanoparticles were characterized using XRD, FTIR, SEM and also applied to crops for testing the crop yield.

2. Materials and methods

The synthesis of Titanium dioxide nanoparticles was prepared using Titanium isopropoxide ($C_{12}H_{28}O_4Ti$) and Glacial acetic acid was used as precursors. Glacial Acetic Acid was dissolved with de-ionized water in the molar ratio of 10:200 M to form a precursor solution under continuous stirring using a magnetic stirrer. 1 M of Titanium (IV) isopropoxide $C_{12}H_{28}O_4Ti$ was added into the solution and continuously stirrer using a magnetic stirrer until the colour of the solution changed to white in colour. Then the gel was separated and subjected to hot air oven for about 7 hours at 80 °C. Further, the dried crystal gel was grinded using motor and pestle to get the nanoparticles. Herewith the prepared nanoparticles were calcinated using a Muffle furnace at 500 °C for 5 hours. The prepared pure TiO_2 Nano Particles were collected in a vial for further characterizations.

The Nitrogen-doped Titanium dioxide (N-TiO_2) Nanoparticles were prepared by adding the Urea into the precursor solution [taking 10 M of Glacial Acetic Acid, dissolved with 200 M of De ionized water and, 1 M of titanium (IV) isopropoxide $C_{12}H_{28}O_4Ti$] under continuous stirring using magnetic stirrer. For dopant, the nitrogen with TiO_2 and Urea of different molar ratios 1:1, 1:2 (i.e) urea of 6.006 g for 1:1 ratio and urea of 12.012 g for 1:2 ratio were added into the precursor solution. Herein, after the gelation period formation of White solution is obtained. Then the gel was separated and dried in Hot air oven 7 hours at 80 °C. The dried gel (which is in crystal form) was grinded to get a powder and calcinated using Muffle furnace at 500 °C for 5 hours.

2.1 Plant growth and soil test

Step 1: Two varieties of seeds as Green gram and Fenugreek were collected and washed several times with distilled water. The soil was collected which is in Good quality. Furthermore the collected soil was weighing as 250 grams and placed in each of the three containers. Further, the Green gram and fenugreek seeds were soaked in water for 12 hours.

Step 2: The next day, 10 grams of soaked seeds were taken and sowed in the soil equally in three soil containing containers. Furthermore, the prepared undoped TiO_2 and Nitrogen-doped TiO_2 at different ratios of 1:1 and 1:2 were applied to the soil in the respective boxes.

Step 3: The growth of the plants was monitored every day.

Step 4: After 6 days of plant growth, the soil samples were collected and tested for their pH value and its NPK availability (i.e.) total Nitrogen Phosphorous and Potassium content presented in the samples.

3. Results and discussion

3.1 XRD analysis

X-ray powder Diffraction (XRD) analysis was carried out to determine the Crystallinity of the prepared nanoparticles using X-ray diffractometer with Cu kα radiation ($\lambda = 1.5418$ $\AA$) were recorded in the range of $10° - 90°$. Fig. 1(A, B, C, D, E & F) represents the comparative diffraction pattern, W-H plot and 2θ orientations of undoped and Nitrogen-doped - TiO_2 (1:1 ratio and 1:2 ratio) nanoparticles. The diffraction pattern of as prepared TiO_2 sample were observed at 25.32°, 37.87°, 48.07°, 53.89°, 62.849°, 68.77°, 75.11° and 82.69° corresponds to the miller indices (101), (103), (200), (105), (204), (116), (301), and (224) respectively. The prepared nanoparticles exhibited tetragonal structures are well matched with the JCPDS card number 78–2486. Herewith these characteristics revealed the Anatase structure of TiO_2 nanoparticles and the rutile phase may also appeared at 54.18° and 70.18° corresponds to the h k l planes (211) and (320) respectively [17].

For doping of nitrogen in TiO_2 nanoparticles in the ratio of 1:1 the peak intensity of anatase phase will decreases and disappears the rutile phase. This suggests that the doping concentration of N in TiO_2 nanoparticles on lower con-

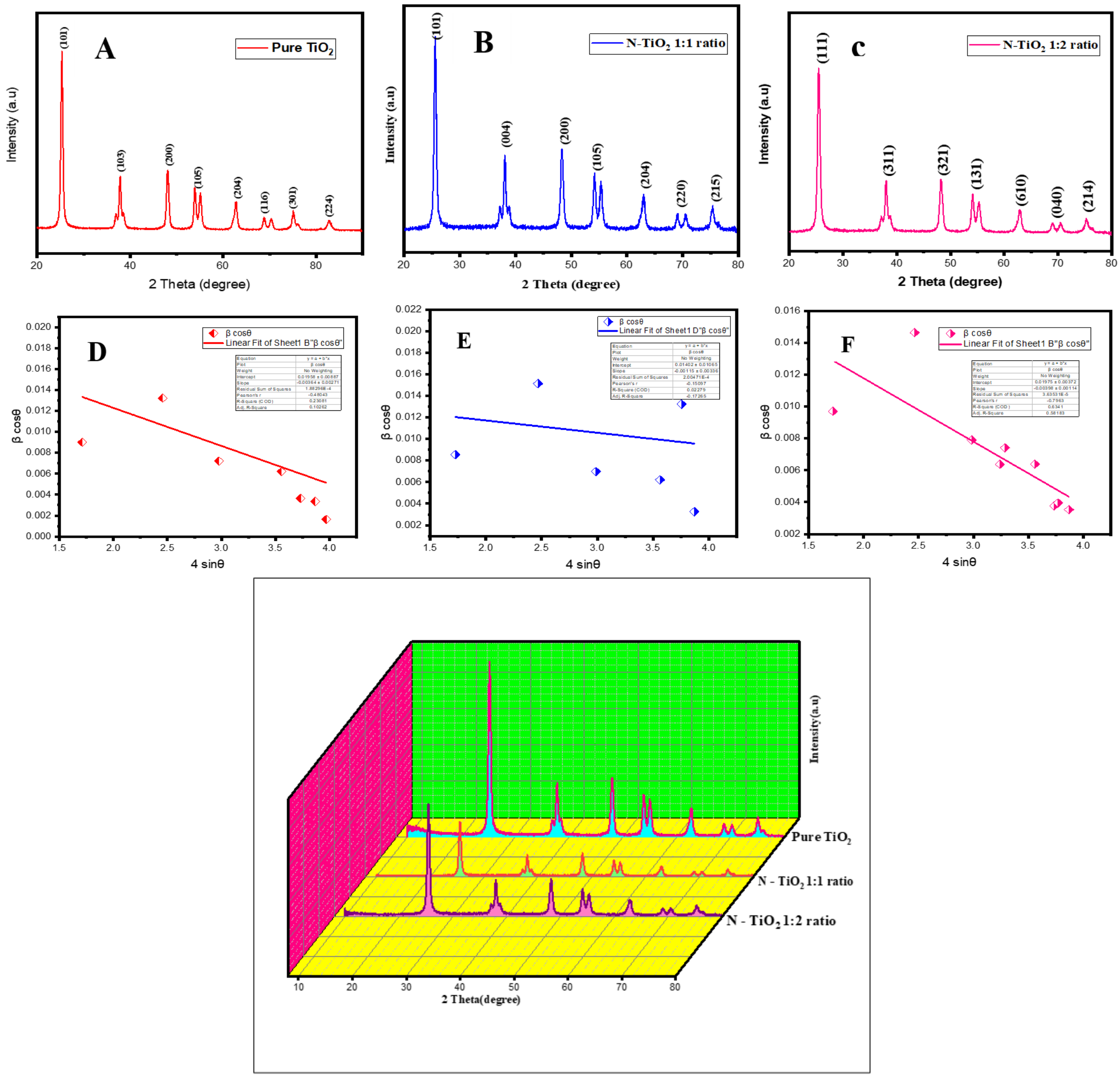

Figure 1. (A, B, C) XRD spectra, (D, E, F) W-H plot and 2θ orientations for the comparison of pure and nitrogen doped TiO_2 nanoparticle.

centration. As the concentration of TiO_2 nanoparticles increases in the ratio of 1:2 will increases the peak intensity of anatase phase which gradually increases the peak intensity of rutile phase. The occurrence of shift in XRD diffraction pattern may due incorporation of N at O site in TiO_2 lattice. The most intense peak of the diffraction pattern shifting to Non–monotonous variation may due to the variance between the ionic size effect between the host ions and dopant, defects in structural states, oxygen ionic vacancies etc. Furthermore the anatase peaks are become weak while doping N in TiO_2 nanoparticles in the ratio of 1:1 may due to the reorientation effect and disappears the rutile phase. The average crystallite size is calculated using Debye Scherer's equation

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

where λ is the wavelength of X-ray radiation, β is the Full Width at Half Maximum and θ is the Bragg's angle. D is the average crystalline size of the particles. The average crystallite size of synthesized nanoparticles is calculated using the Debye Scherer formula and also W-H plot method (Table 1).

Table 1. XRD parameters of TiO_2 nanoparticles.

sample TiO_2	a ($\AA$)	C ($\AA$)	crystallite size (nm) (Debye Scherrer formula)	crystallite size (nm) (W-H plot)
TiO_2	6.20960	5.5389	20.6114	15.6317
N- TiO_2 (1:1)	6.2937	5.55334	25.99847	13.0191
N- TiO_2 (1:2)	6.67000	5.5623	34.47518	7.0204

The lattice parameters of the prepared nanoparticles are a = b = 6.203 Å and c = 5.53 Å. The average crystallite size of the prepared nanoparticles was found to be 25.99 nm for nitrogen-doped TiO_2 (1:1 ratio) and 34.475 nm for nitrogen-doped TiO_2 (1:2 ratio) nanoparticles respectively. The position of the Bragg's angle and broadening analysis was carried out to determine using Williamson Hall Plot. The W-H Plot was plotted using $\beta \cos\theta$ versus $\sin\theta$ revealed the strain and linear profile. The y-intercept and the slope linear fit showed the strain and particle size. The occurrence of elastic deformation may due to thermal stress was revealed from distribution of dots in the linear plot. Furthermore the non-linearity's showed in the linear plot revealed the prepared nanoparticles are in multiphase [18–20].

3.2 Scanning electron microscopy analysis

Scanning electron microscopy (SEM) revealed the morphology and growth features of the as-prepared and N-doped TiO_2 nanoparticles with different magnifications. In Fig 2 (A, B, C, D) & 3 (A, B, C, D) showed the morphology of undoped and nitrogen-doped TiO_2 nanoparticles. The prepared nanoparticles showed that TiO_2 particles are spherical like shape to rough cube-shaped nanoclusters. The agglomeration and the clusters of the prepared nanoparticles may due to the metal precursors during the synthesize route. The elemental composition of the prepared nanoparticles was determined using Energy dispersive X-ray analyses are shown in Fig 4. The EDAX spectrum confirms the presence of Ti and O in the prepared nanoparticles. The atomic percentage of titanium and oxygen of the prepared nanoparticles as provided 30.31% and 69.69% respectively and tabulated in the Table 2 [21, 22].

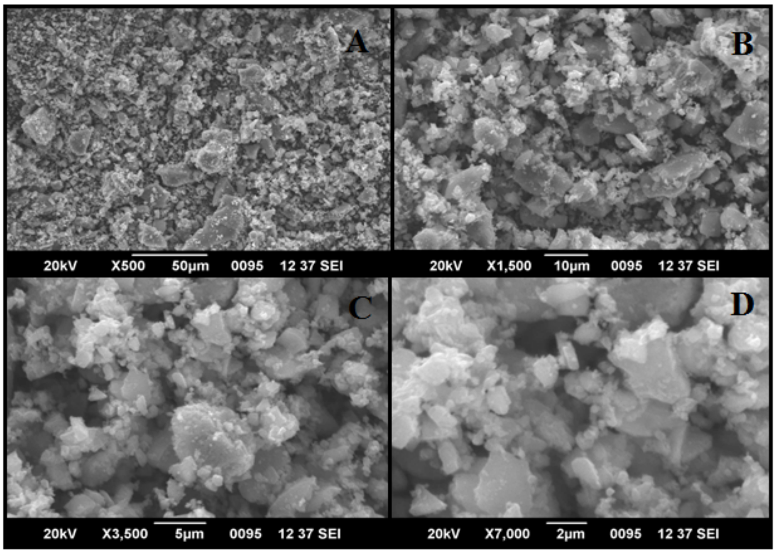

Figure 2. (A, B, C, D) SEM images of pure TiO_2 nanoparticles.

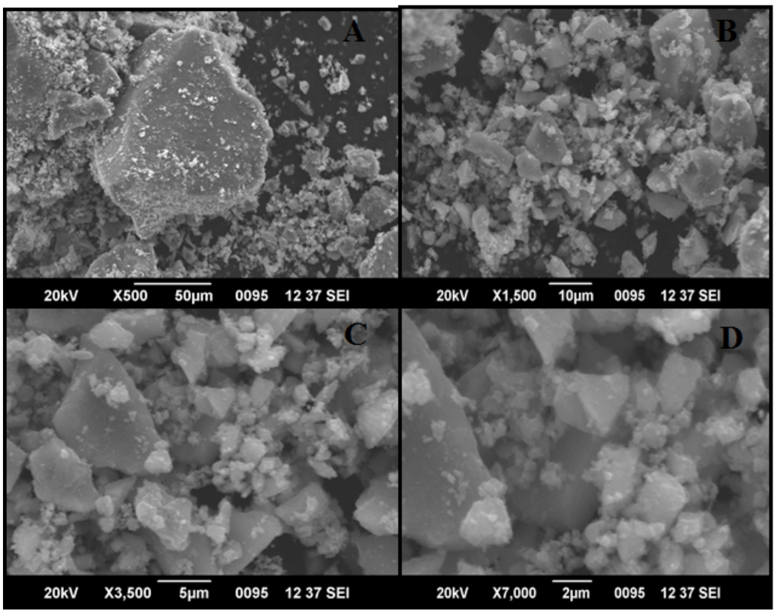

Figure 3. (A, B, C, D) SEM images of nitrogen doped TiO_2 nanoparticles (1:2 ratio).

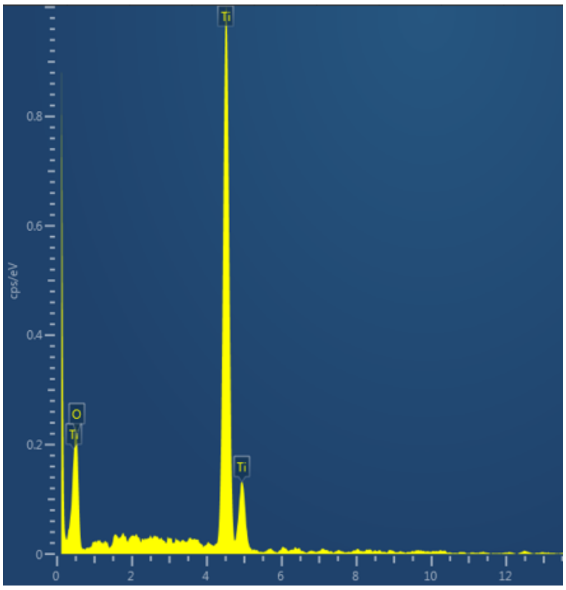

Figure 4. EDAX analysis of TiO_2 nanoparticles.

Table 2. Elementary composition of TiO_2 nanoparticles.

element	line type	Wt %	atomic %
O	K series	43.44	69.69
Ti	K series	56.56	30.31
total:		100	100

3.3 FTIR analysis

The Fourier Transform infrared spectroscopy was used to record the functional groups of undoped and nitrogen-doped TiO_2 nanoparticles are shown in Fig 5. The strong peak at 657.607 cm^{-1} confirms the presence of TiO_2 nanoparticles. The broad adsorption band at 3458.71 cm^{-1} and 3731.58 cm^{-1} attributed O-H stretching of polysaccharide from the aqueous solution during the preparation of nanoparticles. In addition, the adsorption band at 1443.46 cm^{-1} was attributed to the vibrations of the C-N stretching. The peak at 1737.55 cm^{-1} reveals the H_2O bending vibration. The weak peak at 2353.69 cm^{-1} shows the presence of C=N bond or the spectra represents neither TiO_2 nor from urea. No peak at 2900 cm^{-1} showed the removal of all organic compounds by calcination [23, 24].

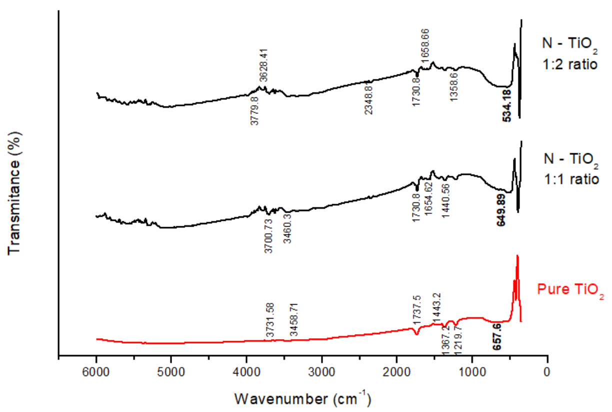

Figure 5. FT-IR spectra of pure and nitrogen doped TiO_2 nanoparticles.

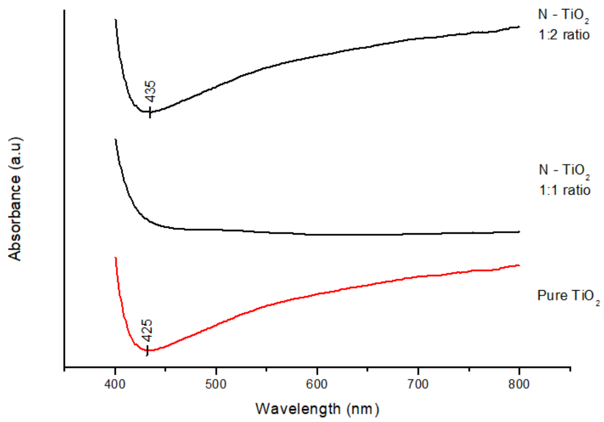

Figure 6. UV-spectra of pure and nitrogen doped TiO_2 nanoparticles.

3.4 UV Vis analysis

The band gap energies of the prepared nanoparticles were determined using UV-Vis spectra were recorded in the wavelength range of 200 nm to 800 nm. The wavelength of the absorbance peak in the UV and VISIBLE region is recorded and shown in Fig. 6. The absorbance peaks are decreases sharply with increasing the wavelength and the absorbance peak was found at 425 nm [25–27]. The band gap energy of TiO_2 nanomaterials may be estimated by using the formula band gap energy (E) = hc/ν where c is the speed of light, $3 * 10^8$ m/s; h is Planks constant, $6.626 * 10^{-34}$ J s; ν = wavelength in nm. The Tauc plot was obtained from $(\alpha h\nu)^2$ verses hν and revealed the band gap energy of the prepared nanoparticles. The bandgap energy of TiO_2 nanoparticles was found to be 2.783 eV. The optical band gap energy value (E) for nitrogen-doped titanium dioxide N-TiO_2 (1:1 ratio) nanoparticles was calculated from Tauc plot and found to be 2.68 eV and for nitrogen-doped titanium dioxide N-TiO_2 (1:2 ratio) nanoparticles was found to be 2.81 eV. The doping concentration of TiO_2 in N revealed the occurrence of shift in absorbance will leads to the narrowing the gaps which decreases the band gap energies for nitrogen-doped titanium dioxide N-TiO_2 (1:1 ratio). The increase in band gap may be attributed to the presence of additional energy levels of nitrogen atoms for improving electrical conduction [20, 23].

3.5 Impact of undoped and nitrogen-doped TiO_2 nanoparticles on plant growth

The effects of undoped and nitrogen-doped TiO_2 nanoparticles on the plants were studied. The common growth of green gram and Fenugreek plants in well-watered was collected and analysed. It was observed that the application of TiO_2 nanoparticles and nitrogen-doped TiO_2 nanoparticles in the ratio 1:1 and 1:2 significantly improved the height and compared to the biomass of plants. The increase in the length of root and shoot by the application of nitrogen-

Table 3. NPK availability in the soil.

sample	Nitrogen content presented (%)	Phosphorus content presented (%)	Potassium content presented (%)
normal value	0.5 – 4	0.5 – 3	0.5 -3
pure TiO_2	0.112	0.625	0.218
Nitrogen TiO_2 (1:1 ratio)	0.192	0.225	0.7
Nitrogen TiO_2 (1:2 ratio)	0.156	0.175	0.953

doped TiO_2 nanoparticles in the ratio 1:1 and 1:2 was 11%, 15% and 16% as compared to plants without any nanoparticles. The height variation is given in the Fig. 7.

3.6 pH test and NPK availability

The pH value of the soil was analysed and compared with the normal values. The normal pH value was known to be 6 – 8.5. The observed pH value was graphically interpreted for undoped and nitrogen-doped TiO_2 nanoparticles in the ratio 1:1 and 1:2 respectively. The graphically interpreted data are shown in Fig. 8. The concentration of potassium was measured in the sample by using a flame photometer. Phosphorus content was measured by the colorimetric method and nitrogen content was analyzed by the Kjeldahl method. The uptake and accumulation of nutrients in the soil including potassium (K), phosphorus (P), and nitrogen (N) was also observed in this experiment and shown in Table 3. The application of pure and nitrogen-doped TiO_2 nanoparticles in the ratio 1:1 and 1:2 gave significant results in the soil. The application of 10 grams of TiO_2 and nitrogen-doped nanoparticles helped the plant to uptake the nutrients of N, P, and K as compared to normal soil. This inferred that the synthesized nanoparticles help the plants to uptake the nitrogen content and help in the conversion of inorganic nitrogen into organic nitrogen. The nutrient uptake efficiency of plants increases the growth rate and quality. The soil application of TiO_2 and nitrogen-doped TiO_2 nanoparticles (ratio 1:1 and 1:2) increased the uptake of these nutrients in plants

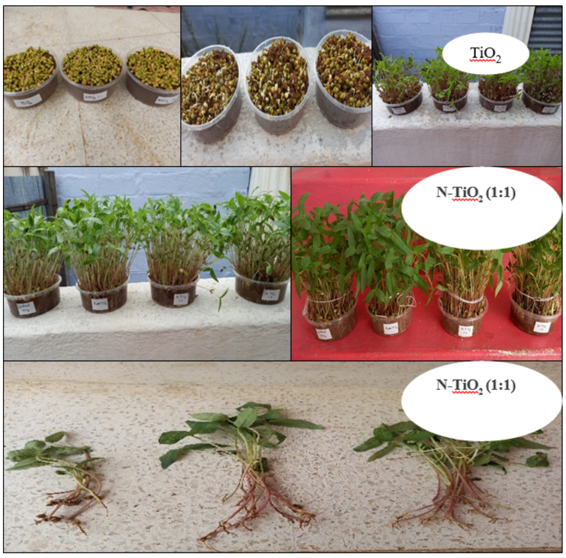

Figure 7. Variation in the height of the plants with TiO_2 nanoparticles.

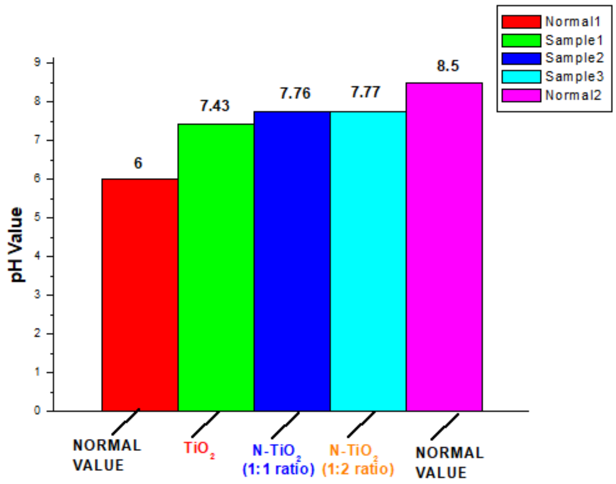

Figure 8. pH value of the soil with TiO_2 nanoparticles.

but the difference among their mean values was significant with each other. The biological analysis of soil confirms the plant growth. Thus, these synthesized nanoparticles were used as nano fertilizer which enriches the nutrients to the plants.

4. Conclusion

Nitrogen-doped titanium dioxide nanoparticles (N-TiO_2) were synthesized using Glacial acetic acid and titanium isopropoxide as precursor solution by Sol-gel method. The average value of crystalline size of TiO_2 calculated from the Debye Scherer formula was found to be 20.614 nm and the average crystalline size of N-TiO_2 1:1 and 1:2 were found to be 25.9 nm and 34.5 nm respectively. The FT-IR analysis showed the sharp peaks in the range of 1400 - 1600 cm^{-1} which confirms the presence of TiO_2. The SEM morphological studies showed the presence of rough cube shaped agglomeration of particles in the synthesized sample. The purity of the prepared nanoparticles and atomic percentage of O and Ti were confirmed by compositional studies energy dispersive X-RAY (EDAX). The optical property of nitrogen-doped TiO_2 nanoparticles revealed the increase of the absorbance value with decreasing the band gap energies for 1:1 ratio. The agricultural analysis inferred that the pH level of the soil was alkali in nature while there was an increase in pH level. The NPK availability in soil was analysed and found to be a fall in NPK level which inferred that the NPK nutrient content is taken up by the plants for plant growth.

Acknowledgment

The authors are grateful to the Secretary, Director, Principal, and Head of the Department of Physics, Sri G.V.G Visalakshi College for Women, Udumalpet for their excellent encouragement and support. The authors thank the Department of Science and Technology (DST) for providing the FTIR Characterization technique.

Authors Contributions

All authors have contributed equally to prepare the paper.

Availability of Data and Materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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