International Journal of Research Publication and Reviews

Journal homepage: www.ijrpr.com ISSN 2582-7421

Green Synthesis of TiO_2 Nanoparticles using Onion Extract

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ABSTRACT

Green nanotechnology is a new developed branch in the bionanotechnology filed. It provides an alternative method for synthesis of NPs with the economic and environmental benefits compared with chemical and physical methods. Titanium dioxide TiO_2 NPs is one of the most common materials in the biomedical field due to its unique photocatalytic properties, excellent biocompatibility, high chemical stability, and low toxicity. The physicochemical characteristics Of TiO_2 NPs synthesized using onion extract was characterized by XRD, , FTIR and HRTEM. In this manuscript we investigate the role of green synthesis of TiO_2 NPs from plant extract (Onion) on the on its phytochemical properties.

Keywords: Green Nanotechnology, Onion, structural analysis, TiO_2

1.Introduction

Titanium dioxide (TiO_2) is a distinguished semiconductor with a very interesting properties, which enabled it to be one of the leader NPs in the all daily life applications. TiO_2 NPs have a band gaps 3.2 eV for anatase, 3.02 eV for a rutile and 2.96 eV for a brookite phases (Rufai et al., 2020a). Because of its high stability, low cost and non-toxicity , TiO_2 is used as a photovoltaic cells in dye sensitized solar cells(Said and Mar, 2021; Venkatesh et al., 2015), hydrogen production in photocatalytic splitting of water, hydrogen production from natural seawater, separate evolution of H_2 and O_2 from water under visible light irradiation(Said and Mar, 2021), hydrogen storage in reversible storage of H_2 on nanotubular TiO_2 arrays(Said and Mar, 2021). In the biomedical field it used in Cancer prevention and treatment, Photodynamic therapy, drug delivery system, cell imaging, biosensing , genetic engineering and medical implants (Ziental et al., 2020). TiO_2 is considered as effective antimicrobial agent due to the ROS generated from photoactivated TiO_2, ROS lead to kill many pathogenic organisms such as bacteria and fungi. Also, when TiO_2 illuminated by UV it can targeted cancer cells by accumulation the chemotherapeutic drug (Hashem and Al-Karagoly, 2021).

There are different methods for synthesis of TiO_2 NPs such as vapor deposition, solvothermal ,electrochemical approaches ,solution combustion , microemulsion technique, micelle and inverse micelle combustion flame–chemical ,vapor condensation process , sonochemical reactions, plasma evaporation ,hydrothermal processing ,sol–gel technology (Rufai et al., 2020b). Green synthesis of NPs using microorganisms and plants is an alternative method to produce NPs with a promising characteristic(Jadoun et al., 2021a; Nadeem et al., 2018a). The using of plant extracts for synthesis of NPs have the advantages of being easier than using bacteria or fungi, more disciplined, safer to handle and available but fungi and bacteria need a longer incubation period in growth media to reduce metal ions. Bacteria, fungi, algae and plants were used in the biological preparation of metal NPs(Caputo et al., 2014; Manivasagan and Kim, 2015). They possess properties such as reduced or antioxidant responsible for reducing metal nanoparticles , nanoparticles are synthesized by plants (the easy scale-up process) more often than microorganisms are not applicable for large-scale production and need to high sterilization condition (Gour and Jain, 2019). Nanoparticles are produced intracellular inside live plants (Phytomining) or extracellular using plant extracts, bioactive compounds, latex and gum in a controlled approach related to their size, dispersity, and shape. plant is suitably scaled up for large-scale synthesis of nanoparticles (Tarafdar et al., 2013). Different parts of the plant such as seeds, leaves, bark and fruit to obtain extract then it was mixed with a solution of the metal salt , it takes minutes to a few hours for the reaction completes at ordinary room temperature to reduce a metallic salt solution into nanoparticles (Arya et al., 2021). Plant extracts used as a reducing agent for the reduction of metal ions because it contains carbohydrates and proteins with functional amino groups (Baker et al., 2013).

The aim of the presented study is to synthesize and study the structural properties of TiO_2 NPs using onion plant extract.

2.Materials and method

2.1 Green synthesis of TiO_2 NPs by Onion extract:

Onion extract was prepared by cutting (30 g) of onion leaves from the local market and mixed with (100 ml) of deionized water. The mixture was left to boil for 20 minutes, the produced extract is then filtered 2 times using filter sheets. TiO_2 precursor solution was prepared by adding of (10 ml) of

TTIP in (150 ml) of distilled water under vigorous stirring. 7 ml of fresh onion extract were added during the preparation of TiO_2 precursor solution under continuous stirring for 2h. The formed NPs were then dried at room temperature overnight. Finally, the sample was crushed into fine powder using agate mortar and pestle and calcinated at 400 °C for 3h.

2.2 Physical Characterization

X-ray diffraction (XRD) measurements were performed using X'Pert PRO-PAN analytical diffractometer with Cu-Kα radiation (λ= 1.54056 A°) at 40 kV and 30mA. The crystallites responsible for the Bragg reflection was determined using the well-known Scherer relationship (Patle et al., 2018):

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where D is the crystallite diameter in A°, K is the shape constant (~ 0.9), λ is the wavelength in A°, θ is the Bragg angle in degrees and β is the observed peak width at half-maximum height in rad.

The functional groups in TiO_2 NPs was investigated by Fourier transform infrared spectrometer a typical Jasco Model 4100 – Japan by passing infrared radiation through a sample and determining what fraction of the incident radiation is absorbed at a particular energy, in the wavelength between 400 and 4000 cm^{-1} and a resolution of 4.0 cm^{-1}. A typical high-resolution transmission electron microscope (JEOL, JEM- 2100 – Japan) is employed to study the structural morphology and crystalline properties for all synthesized phtocatalysts NPS it offers an accelerated voltage up to 200 kV.

3. Results and discussion

3.1 X-ray diffraction (XRD)

Onion can be used successfully as a reducing agent to synthesize TiO_2 NPs without changing its structure. XRD pattern of the green synthesized TiO_2 NPs showed various peaks in a wide range of 2θ angle (10 < 2 θ < 80), (see **Fig. 1**). All the characteristic peaks where belong to the anatase phase of the crystalline TiO_2 NPs .The obtained results were closely matched with the Joint Committee on Powder Diffraction Standards (anatase XRD, JCPDS Card No. 21-1272)(Al-Shabib et al., 2020; Arya et al., 2021).
The calculated lattice parameters for our sample was a=b= 3.788 A° and c= 9.73147, which is confirm the purity of our sample. The calculated crystal size from Scherer equation of green synthesized TiO_2 NPs was 37.18 ± 12.96 nm.

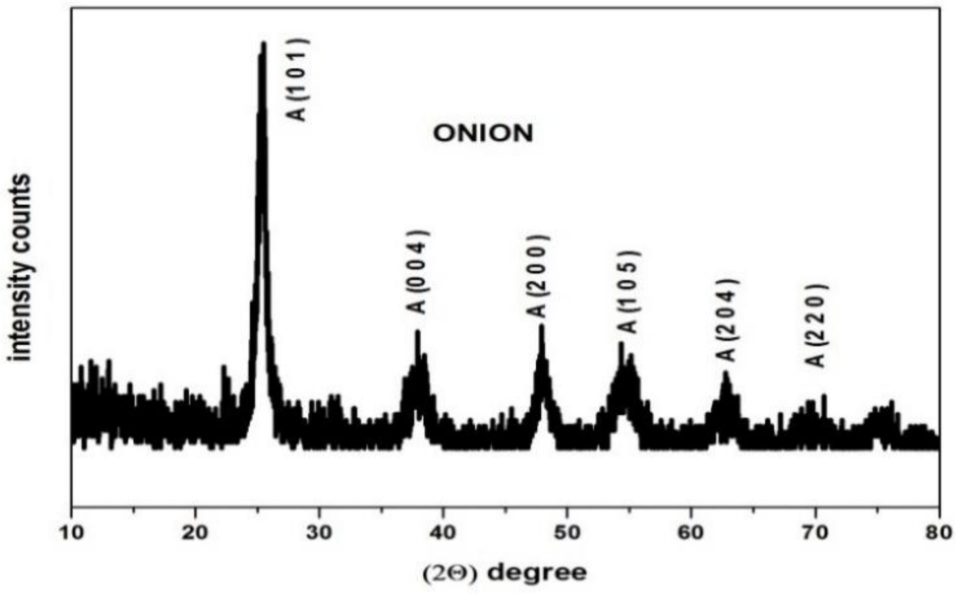

Fig. 1: XRD pattern of green synthesized TiO_2 NPs

3.2 Fourier Transformation IR spectroscopy (FTIR)

FTIR spectrum for TiO_2 NPs is presented in **Fig 2**. The broad band at 3426.2 cm^{-1} assigned for vibration of the hydroxyl group. The vibration band at 1151 cm^{-1} is due to Ti-O- Ti vibrations. The band at 1628 cm^{-1} is described the bending vibration of adsorbed water and Ti-OH. Band at 1376 cm^{-1} attributed to the asymmetrical C–H vibrations. Band at 457.6cm^{-1} corresponds to the Ti - O - Ti. Vibrational bands appearing around 723 cm^{-1} correspond to TiO_2 Modes in the anatase phase(Kandavelu et al., 2004; Khan et al., 2019).

FTIR spectrum of onion extract is characterized by different function groups such as the band at 3409.8 cm^{-1} is corresponding to N-H stretching of proteins and O-H stretching of carbohydrate and water. Band at2934.1 cm^{-1} is representing the CH_2 antisymmetric stretch of methyl groups mainly: lipids. Band at1624.6 cm^{-1} is representing amid I and presence of water. Band at 1416 cm^{-1} is represents CH_3 asymmetric. Band at 1265 cm^{-1} is represents amide III for protein. Bands at 1123.2 cm^{-1} and 1053 cm^{-1} are due to C-OH stretching band of oligosaccharide residue. Bands at927.8 cm^{-1} and 873.5cm^{-1} is described left-handed helix DNA. Band at 810.2 cm^{-1} is due to ring CH and reflect structure information of polyphenols(León et al., 2017; Mugundan et al., 2014).

The green synthesis of TiO_2 NPs using onion extract was confirmed by the presence of function groups of both TiO_2 and onion.

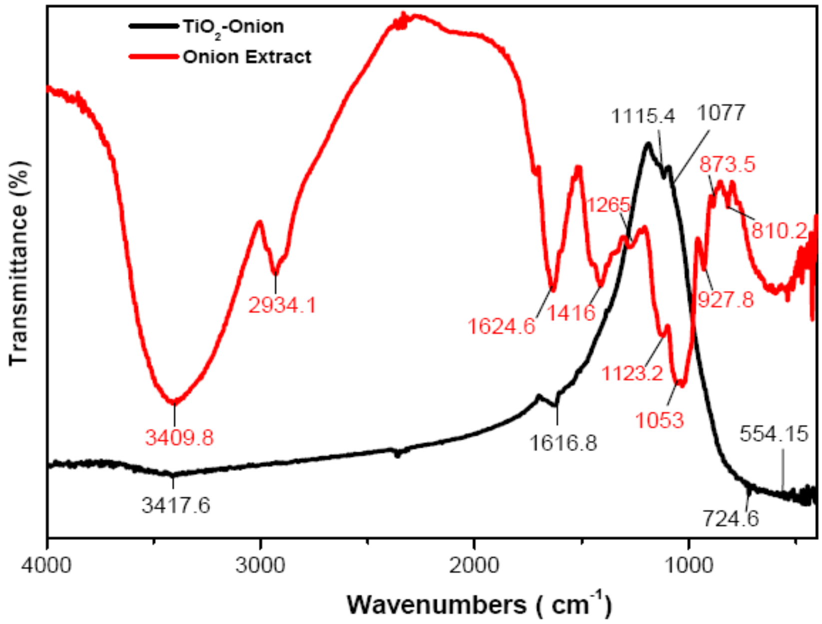

Fig. 2: FTIR spectra of onion extract and green synthesized TiO_2 NPs using onion extract.

3.3 High Resolution Transmission Electron Microscope (HRTEM):

HRTEM image of green synthesized TiO_2 NPs reflect a sort of agglomeration with a spherical or irregular spherical shape, **Fig. 3**.

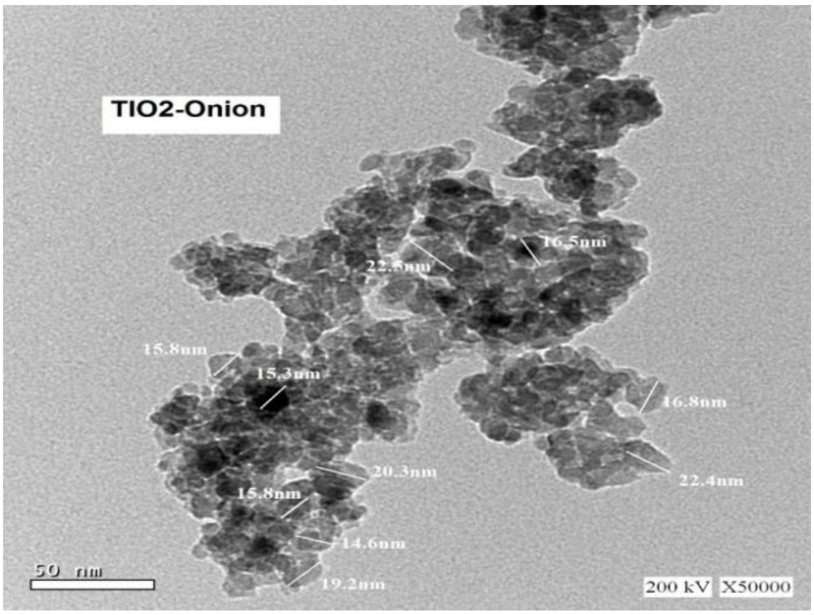

Fig. 3: HRTEM image of green synthesized TiO_2 NPs

Particle size distribution of green synthesized TiO_2 NPs as estimated from HRTEM image was presented in Fig. 4. The average size was 14.91± 1.97 nm which is smaller than the calculated values from XRD results (37.18 nm). This variation in the size may be due to the agglomeration of TiO_2 NPs. The crystallite size calculated form Scherer's equation is the apparent size, which doesn't equal the particle size especially in the case of polydisperse NPs with aggregation such as TiO_2 NPs. The green synthesis of NPs may be inducing the agglomeration due to the presence of biomolecules on the surface of NPs. The biomolecules on the surface of TiO_2 NPs attracted with other molecules due to the electrostatic force on their surface(Jadoun et al., 2021a, 2021b).

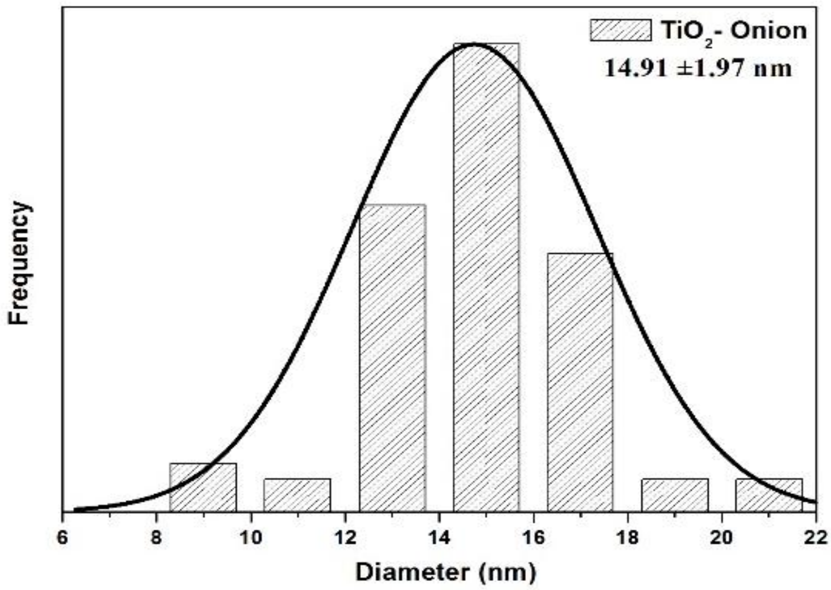

Fig. 4: Particle size distribution of green synthesized TiO_2 NPs

4.Conclusion

onion was successfully used as a reducing agent to synthesize TiO_2 NPs and the green synthesis of TiO_2 NPs doesn't alter the structure of TiO_2 NPs, which is in a good agreement with the reported results. The anatase phase formation showed the complete reduction of titanium isopropoxide using onion plants extract. The formed NPs has a spherical or irregular spherical shape with a sort of agglomeration due to the presence of biomolecules.

Acknowledgment

The authors would like to acknowledge Prof. Mohammed M. Wahman, for helpful discussions on this topic.

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