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Green Synthesis of Titanium Oxide Nanoparticles Using Potato Peel Extract and Investigation of Their Structural and Morphological Properties: Sintesis Ramah Lingkungan Partikel Nano Oksida Titanium Menggunakan Ekstrak Kulit Kentang serta Kajian Sifat Struktur dan Morfologinya

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Abstract

General Background: Nanotechnology offers advanced approaches for developing functional materials with unique physicochemical properties. **Specific Background:** Titanium dioxide nanoparticles are widely studied due to their stability, non-toxicity, and applicability in photocatalysis and environmental remediation. **Knowledge Gap:** Conventional synthesis methods often involve toxic chemicals and energy-intensive processes, creating a need for sustainable alternatives. **Aims:** This study aims to synthesize TiO_2 nanoparticles using potato peel extract through a green sol-gel method and to investigate their structural and morphological properties. **Results:** X-ray diffraction confirmed the formation of phase-pure anatase TiO_2 with a tetragonal structure and an average crystallite size of 22.687 nm. FTIR analysis verified Ti–O–Ti bonding and surface hydroxyl groups, while FE-SEM revealed near-spherical morphology with slight agglomeration. EDX analysis demonstrated high chemical purity and homogeneous elemental distribution of titanium and oxygen. **Novelty:** The study utilizes potato peel waste as a biogenic reducing and stabilizing agent, contributing to sustainable nanomaterial synthesis. **Implications:** The obtained nanoparticles exhibit properties suitable for applications in photocatalysis, environmental remediation, and biomedical fields, supporting the development of eco-friendly nanomaterial production strategies.

Keywords: Titanium Dioxide Nanoparticles, Green Synthesis, Potato Peel Extract, Structural Characterization, Nanomaterials

Key Findings Highlights

1. Phase-pure anatase structure confirmed through diffraction analysis
2. Biogenic route produced nanoscale particles with uniform composition
3. Surface functional groups support catalytic application potential

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1. Introduction

Nanotechnology is considered to be one of the fastest developing fronts in modern science and engineering that provides previously unknown opportunities to handle matter at an atomic and molecular level [1]. The metal oxide nanoparticles have taken a center stage among the various set of nanomaterials that have received a significant scientific focus because of their outstanding physicochemical features that include high surface to volume ratios, adjustable optical attributes, high chemical reactivity, and exquisite mechanical stability. Such distinctive properties make metal oxide nanoparticles to be very promising to a wide range of applications in photocatalysis, environmental remediation, biomedicine, energy conversion, sensing technologies and advanced functional material [2].

Titanium dioxide (TiO2), also called titania, is one of the most researched metal oxide nanomaterials due to its remarkable chemical stability, non toxicity, natural occurrence, and excellent photocatalytic performance [3]. There are three main crystalline forms of TiO2 nanoparticles: anatase, rutile, and brookite, each of which has the particular structural and electronic characteristics that define their functional behaviour. Anatase which is the most photocatalytically active polymorph with a bandgap of about 3.2 e V is well known and commonly used in org. pollutants degradation (ultraviolet irradiation), solar-energy conversion (dye-sensitized solar cells), antibacterial and antifungal bioactivity, and self-cleaning and anti-fogging paint [4]. This wide range of application of the TiO2 nanoparticles in all these fields has driven serious research activities aimed at coming up with efficient, cost effective and environmentally friendly production processes [5].

Traditional methods to the synthesis of TiO2 nanoparticles such as sol-gel, hydrothermal synthesis, chemical vapor deposition, solvothermal and co-precipitation methods proved to be quite effective in the fabrication of nanoparticles of controlled size and morphology [6]. Nevertheless, such methodologies often incorporate use of toxic chemical reducing as well capping agents, toxic organic solvents, energy-intensive processing conditions and multi-step processes that produce by-products that are harmful to the environment. Such restrictions have prompted an increased amount of interest in the creation of more environmentally friendly and sustainable options that will avoid the intrinsic limitations of traditional synthetic pathways without disrupting or diminishing the quality and functionality of the end products in the form of nanoparticles [7].

The technique of green synthesis or biogenic synthesis has become an attractive and viable paradigm in the synthesis of metal and metal oxide nanoparticles relying on biological reducing and stabilizing entities like plant extract, fungi, bacteria, algae, and by-products in agriculture[8]. Plant-mediated synthesis has been of special interest because of the strong accessibility of plant biomass, the simplicity and scalability of the synthesis procedures and because plant-derived phytochemicals, such as polyphenols, flavonoids, alkaloids, terpenoids, and organic acids, are capable of reducing metal preceptor ions and stabilizing the formed nanoparticles against aggregation at the same time. These biomolecules do not only mediate the reduction and nucleation of metal oxide nanoparticles, but they also act as natural capping agents that provide colloidal stability and size, shape and surface chemistry control of nanoparticles[9].

Solanum tuberosum (potato peel) is a highly promising and under-explored agricultural waste stream that is produced in massive amounts across the globe as a food processing by-product and domestic waste. Bioactive phytochemicals, including chlorogenic acid, caffeic acid, ferulic acid, glycoalkaloids (solanine and chaconine), flavonoids (quercetin and kaempferol), vitamins (ascorbic acid and vitamin E) and other polysaccharides are abundant in potato peels [10]. This complicated phytochemical structure endows potato peel aqueous extracts with robust reducing and chelating features rendering them very apt in green synthesis of metal oxide nanoparticles. The idea of using potato peel waste as a functional reagent to produce nanoparticles is in line with the concepts of green chemistry and a circular economy, where a low-value waste product is converted to a high-value resource without the production of other hazardous chemical waste[10, 11].

2. Experimental

2.1 Materials

The chemical reagents were of the analytical grade in all the chemical procedures used in this study and were not further purified except when indicated. Titanium(IV) isopropoxide (Ti [OCH(CH3) 2]. 4, 97% purity was a precursor of titanium nanoparticles and was purchased at Sigma-Aldrich (St. Louis, MO, USA). A solvent of titanium precursor in the first-stage preparation was isopropyl alcohol (IPA, 99.7% purity) obtained by Merck KGaA (Darmstadt, Germany). Sodium hydroxide (NaOH, 97% purity) was purchased at Fluka Chemical Corp and was added to the medium of synthesis as needed to adjust the pH. Hydrochloric acid (HCl, 37% w/v) of BDH Chemicals was used to achieve the purposes of modifying pH. All experimental activities, such as the preparation of extract, reactions during synthesis, and washing were done using deionized water (resistivity ≥18.2 MΩ cm at 25C) supplied by a Milli-Q water purification system (Millipore, Billerica, MA, USA). Fresh potato samples (Solanum tuberosum) were purchased at a nearby agricultural market and utilized immediately or refrigerated at 4 ℃ not exceeding 48 hours before use to reduce biochemical degradation of the phytochemical compositions.

2.2 Preparation of Potato Peel Aqueous Extract

The aqueous extract of Potato Peel was prepared by first drying the peels, followed by blending the mixtures of potato peels with water and ethanol.

The potato peels were gently peeled off the potato flesh with a sterile peeling knife and very little contamination of the

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starchy potato interior was allowed. The peels obtained were rinsed with a lot of tap water to remove all dirt and agricultural residues on the surface and then three consecutive rinses with deionized water to remove any ionic contamination. The peels were washed and placed on clean absorbent paper whereby they were left to dry in ambient temperature or about 24 hours to dry the excess surface moisture. The semi-dried peels were then sliced into small pieces about 1-2 cm2 in size so as to have as many surface areas to extract as possible[12].

To prepare the extract, 20 grams of the dried pieces of potato peel were weighed accurately using an analytical balance and transferred to a 500 mL round-bottom Erlenmeyer flask which contained 200 mL of deionized water making the peel-to-water ratio 1:10 (w/v). The mixture was stirred with magnetic hotplate stirrer and heated at 80 o C with continuous stirring with 300 rpm of the flask of 30 minutes. This temperature and time were chosen on the basis of the preliminary optimization experiments targeted to achieve the maximum extraction of heat-labile bioactive compounds, in particular polyphenols and organic acids, and to avoid their excessive thermal damage. After the heating step, the extract was left to cool to a room temperature and then filtered using a set of filtration media: Coarse Whatman No. 1 filter paper was added at first to remove large particulate matter and then filtration through a fine Whatman No. 42 filter paper (2.5 µm pore size) was used to obtain a clear and light yellow-brown filtrate. The subsequent extract was kept in amber glass bottles at 4 o C and consumed within 72 hours of preparation in order to reduce phytochemical oxidation and microbial contamination [12].

Figure 1. Figure 1 shows the stages of preparing the plant extract

2.3 Green Synthesis of TiO_2 Nanoparticles

TiO 2 nanoparticles were prepared by a biogenic sol-gel method. In a classic synthesis, 5 mL of titanium(IV) isopropoxide was initially dissolved in 20 mL of isopropyl alcohol under constant magnetic stirring in a 100 mL beaker to get a homogeneous solution of titanium precursor. 50 mL of the newly made potato peel aqueous extract was then dropwise added to this precursor solution at a constant rate of about 1 mL/min by adding a calibrated drop of the precursor solution to the neatly stirred at 400 rpm and room temperature. Introduction of titanium precursor into the aqueous extract activates instant hydrolysis and condensation reactions where the phytochemical constituents of the extract are involved in facilitating the development and stabilization of the titanium oxide colloidal network. The pH of the reaction mixture was kept at 9 10 range by dropwise addition of 0.1 M NaOH solution to the reaction mixture and this was adjusted to 9 10 level to ensure that the titanium precursor was fully hydrolyzed and that the titanate species were formed.

The obtained gel-like precipitate was kept under constant stirring during a further 2 hours at room temperature to allow the reaction to take place fully and to evenly cover the particles of the precursor with the phytochemical capping solutions. Centrifugation was then used to separate the precipitate and the supernatant by centrifugation at 5000 rpm in 15 minutes on refrigerated centrifuge (Sigma 3-18K, Germany). Precipitate gathered was washed three times with deionized water and once with absolute ethanol to get rid of leftover organic matter and unreacted precursor species, and centrifugation was done between washing. The precipitate was washed and soaked in a clean ceramic crucible and dried in the laboratory furnace at 100 o C over a 2-hour period to eliminate any water molecules attached to it. The dried powder was then calcified in a muffle furnace (Nabertherm L 3/11, Germany) at a temperature of 500 C over 2 hours, but with a regulated heating

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rate of 5 C/min, to cause crystallization and conversion of the amorphous titanium oxide starting material to the crystalline TiO 2 anatase phase. The resulting white powder was gently ground in agate mortar and mortar using agate pestle, sieved with 200-mesh sieve and stored in sealed glass vials at ambient temperature until further characterization[13].

.3. Results and Discussion

3.1 X-Ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) was used to determine the crystalline structure and phase composition of synthesized TiO2 NPs and the obtained diffractogram is depicted in Figure 2. The three clear, sharp peaks of the XRD pattern were of 25.16, 37.36, and 47.635 o at the (011), (004), and (020) crystal planes, respectively. These feature reflection peaks are well in accordance with the tetragonal anatase form of TiO2 as verified by comparison with the Crystallography Open Database (COD) reference entry No. 96-900-8217. The fact that no other diffraction peaks appeared as a result of rutile or brookite or other polymorphic phase is a clear indication that phase-pure anatase TiO2 nanoparticles have been formed successfully [14].

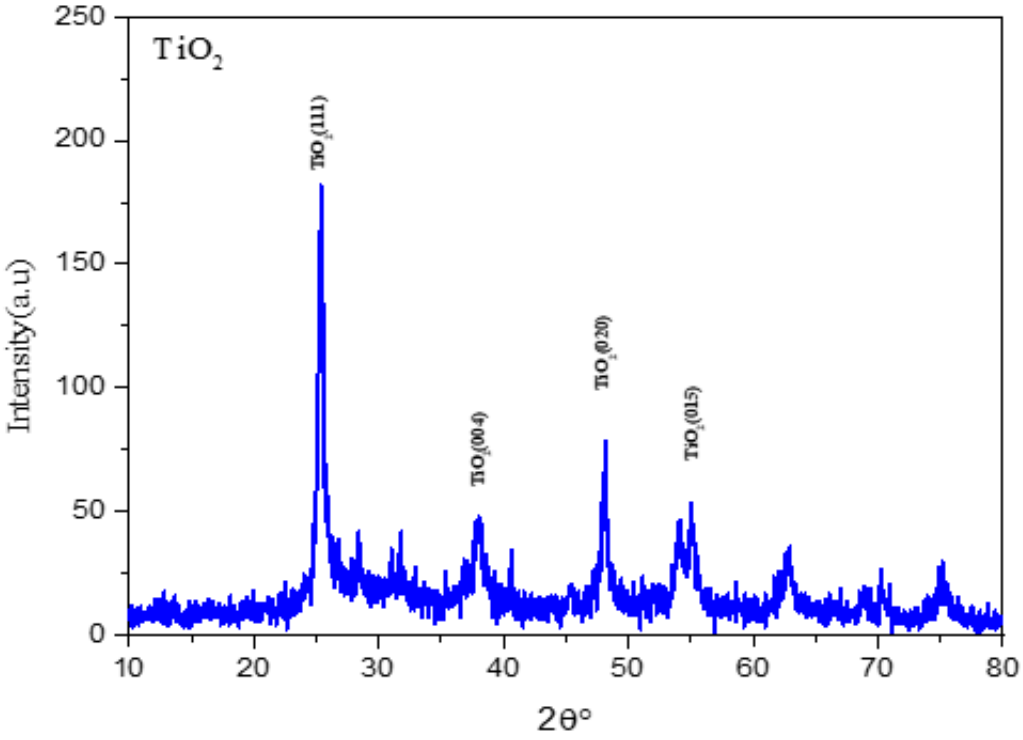

Figure 2. Figure 2 XRD pattern of TiO2 NPs

The steepness and high-quality of the diffraction peaks points at high level of crystallinity in the synthesized nanoparticles. The values of full width at half maximum (FWHM) of the peaks at the 25.16 = 2 theta, 37.36 = 3 theta, and 47.635 = 4 theta were obtained to be 0.2826, 0.4065 and 0.552, respectively. The gradual expansion of the peaks with increasing 2theta is typical of materials of nanoscale, in which the broadening of the lines is inversely related to the crystallite dimension based on the Scherrer equation:

D = Kλ / (β cos θ)

where D is the mean crystallite size, K is the Scherrer constant(0.94), with 2 being the X-ray wavelength (CuKalpha, 15406 nm), B being the FWHM in radians, and the Bragg diffraction angle, 0. The crystallite sizes determined with the three major diffraction peaks were 30.083 nm, 21.547 nm, and 16.431 nm giving the average size of the crystallites of 22.687 nm. The difference in the size of crystallites on various planes of diffraction can be explained by the anisotropic growth behavior exhibited along the various crystallographic orientations, which is common with tetragonal anatase nanostructures.

These results complement the previously obtained values of anatase TiO2 nanoparticles made through chemical and sol-gel manufacturing methods and in which the crystallites size is generally between 10 and 35 nm. The size and more importantly the nanoscale dimensions of the crystallites are also important since the dimensions and specifically their specific surface area are anticipated to increase the photocatalytic activity due to quantum effects of confinement and the overall reactivity of the material to be used in future applications..

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Parameter	Peak 1	Peak 2	Peak 3	Average / COD Entry
2θ (°)	25.16	37.36	47.635	22.687 nm (avg)
(hkl) Plane	(011)	(004)	(020)	
FWHM (°)	0.2826	0.4065	0.552	
Crystallite Size (nm)	30.083	21.547	16.431	
Crystal Phase	Anatase	Anatase	Anatase	Tetragonal TiO_2
COD Reference			96-900-8217	

Table 1. Table 1. Structural Parameters of TiO2 NPs Derived from XRD Analysis

1.2 Fourier-Transform Infrared Spectroscopy (FTIR) Analysis

To determine the typical functional groups and vibrational frequencies within the nanoparticles, the FTIR spectrum of the prepared TiO2 NPs Figure 3 was obtained in the wavenumber range of 400 -4000 cm -1. The wide and strong absorption band that is present in the low-wavenumber region (400900 cm -1) can be said to be due to TiO stretching and TiO Ti bridging modes which is the characteristic fingerprint of TiO2 lattice framework. This finding is closely correlated with the crystal structure which was identified using XRD analysis and is also in line with spectral data reported before on anatase-phase TiO2 nanoparticles.

The O-H stretching vibration of physisorbed water molecules and surface hydroxyl groups (Ti -OH) on nanoparticle surface gives the broad absorption band at around 3200 -3500 cm -1. The corresponding water adsorbed bending mode is usually found to be at 1620-1640 cm -1. The existence of surface hydroxyl groups is of special concern, as they are active sites of the creation of radicals under photocatalysis, and of vital importance in the process of photodegradation of organic pollutants. The FTIR data unanimously prove purity of chemical integrity of the synthesized TiO2 NPs and no contamination of organic residues of the precursors, so validating the purity of the final product [15].

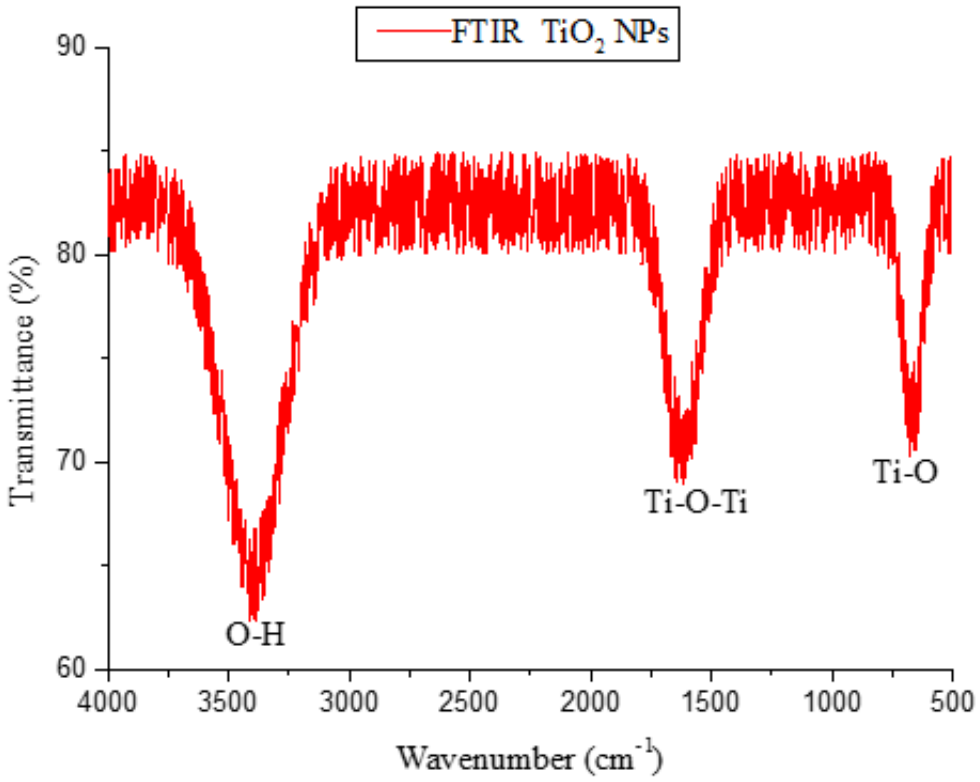

Figure 3. Figure 3 FTIR spectroscopy of TiO2 NPs

1.3 Field-Emission Scanning Electron Microscopy (FE-SEM) Analysis

The morphology and microstructure of the synthesized TiO2 NPs were studied through the field-emission scanning electron microscopy (FE-SEM), and the representative micrographs at various magnifications are shown in Figure 4 The image presented by FE-SEM has indicated that the nanoparticles assumed a relatively homogenous morphology, which is in agreement with the nanoscale sizes of crystallites obtained through XRD analysis. The particle particles tend to stick together and this is a well-known phenomenon in nanoparticle systems because the surface energy is high and van der Waals forces exist on individual particles between particles on the nanoscale.

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According to the morphological analysis, the nanoparticles are mainly of near-spherical to slightly irregular shape. The size distribution observed in the FE-SEM micrographs is generally in compliance with the average size of crystallites calculated by XRD-Scherrer analysis as 22.687 nm, but the size of the particles could be a little bit bigger because of agglomeration effects and because of the resolution limits of electron microscopy at this range. The small dimensions in the nanoscale are likely to dramatically increase the functional properties of the nanoparticles in surface-mediated processes (photocatalysis, sensing and drug delivery) because of the high surface-area-volume ratio [16].

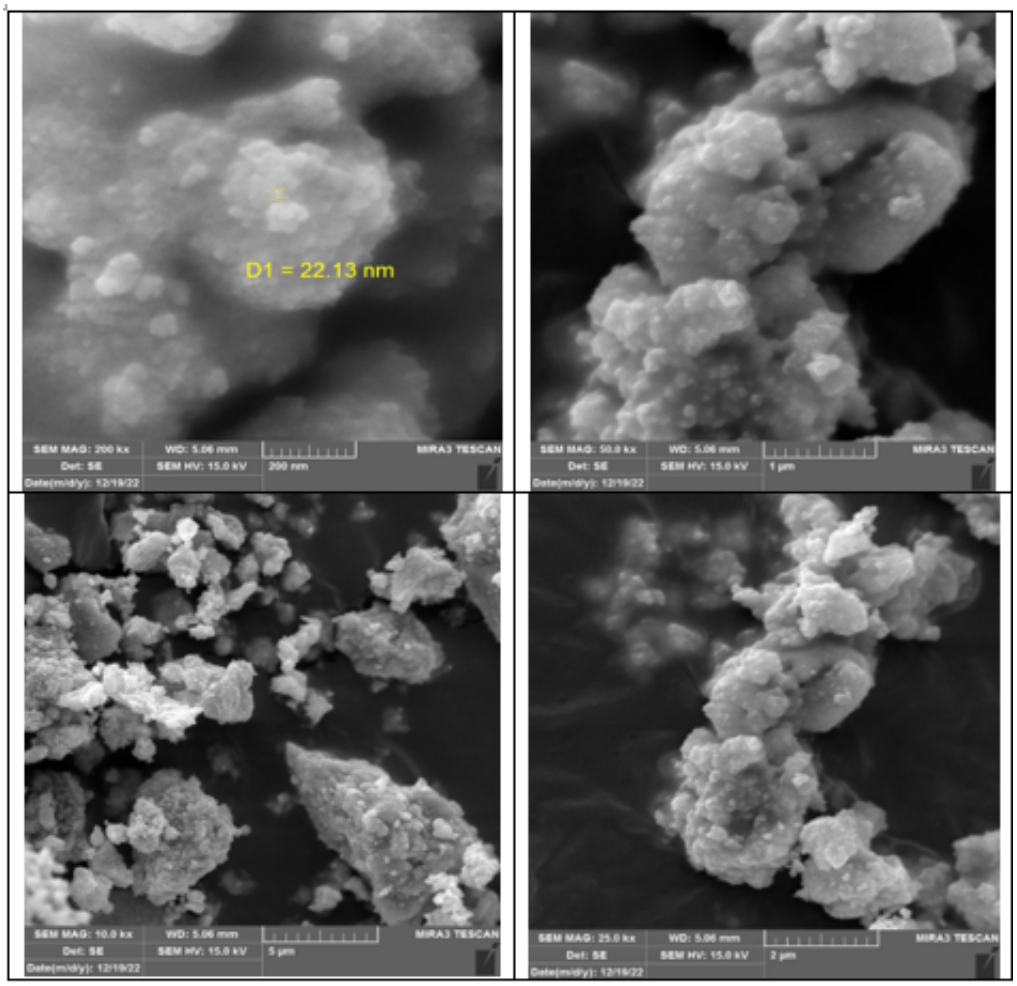

Figure 4. Figure 4 FE-SEM image for TiO2 NPs

1.4 Energy-Dispersive X-Ray Spectroscopy (EDX) and Elemental Mapping Analysis

The energy-dispersive X-ray spectroscopy (EDX) was used to determine the elemental composition of TiO2 NPs that were synthesized and the spectrum is shown in Figure 5. This was clearly indicated by the EDX analysis, which revealed two elements only, i.e., titanium (Ti) and oxygen (O), as the constitutive elements of the synthesized nanoparticles with no indications of foreign elements and impurities, thus, validating the high chemical purity of the product.

The quantitative analysis of the EDX spectrum indicated that the percentage of oxygen and titanium was 85.76 wt% and 14.24 wt%, respectively, with 94.74 at% and 5.26 at% of the percent atomic compositions, respectively. The observed oxygen-enriched surface composition, compared with the theoretical bulk stoichiometry of TiO2 (Ti:/O:apparently = 59.9/40.1) can be explained by the high surface density of hydroxyl groups (Ti–OH) and physically adsorbed water molecules on the nanoparticle surface which has been widely reported on oxide nanoparticles with high surface to volume ratios. The FTIR data also supports this enrichment of surface oxygen, having obvious OH stretching bands that are indicative of widespread surface hydroxylation.

The EDX elemental mapping (Figure 6) was used to give spatially resolved compositional information on the assembly of the nanoparticles. The maps show that both Ti and O are distributed homogenously and uniformly across the examined region and no elemental segregation, phase separation, or clusters of impurities can be observed. Such a homogeneity of the space is an excellent sign of the chemical homogeneity of the produced TiO2 NPs and also confirms the conclusion of the success of phase-pure nanoparticle synthesis..

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Element	Series	Weight % (wt%)	Atomic % (at%)
Oxygen (O)	K-series	85.76	94.74
Titanium (Ti)	K-series	14.24	5.26
Total	—	100.00	100.00

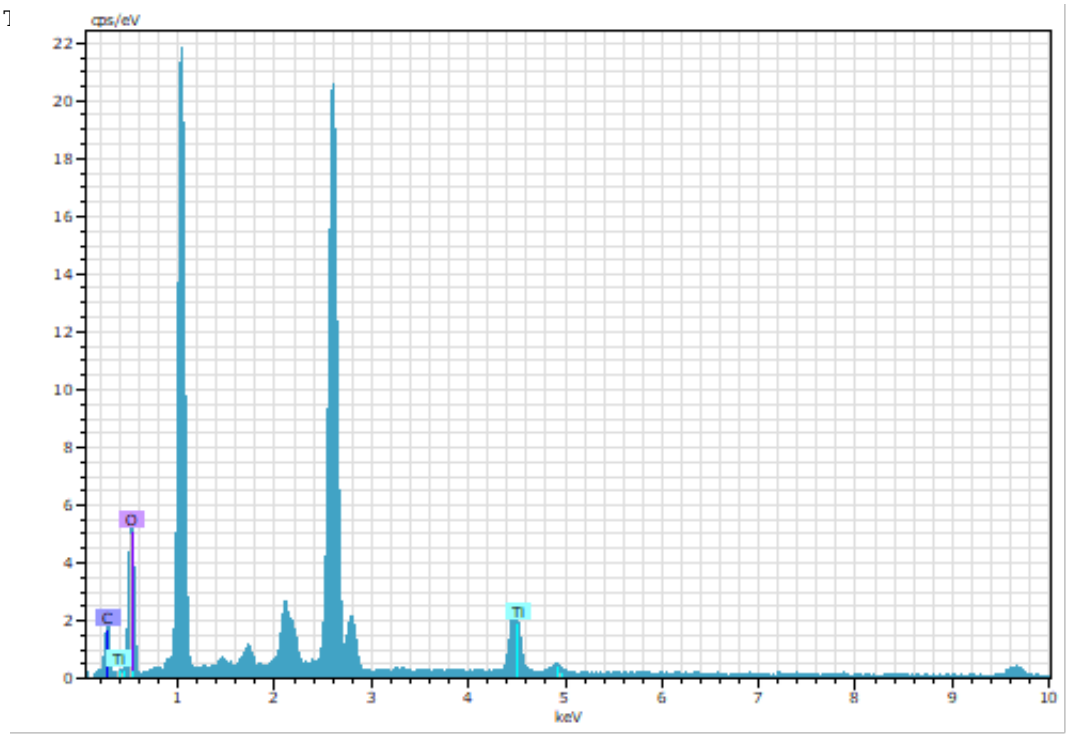

Figure 5. **Figure 5 EDX Spectroscopy TiO2 NPs**

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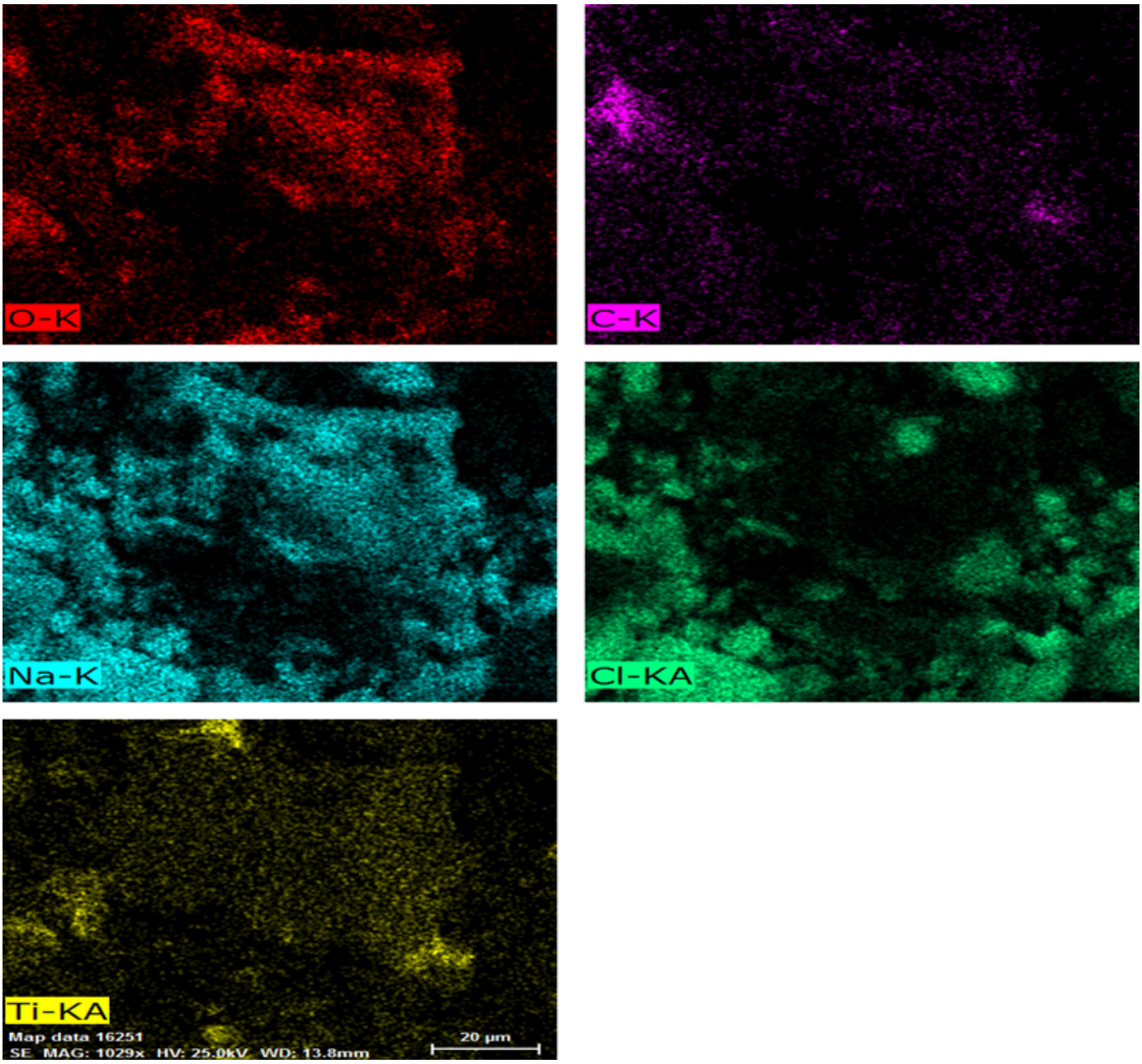

Figure 6. **Figure 6 EDX elemental mapping**

3. Conclusions

The following principal conclusions can be drawn from the comprehensive characterization of the synthesized TiO2 nanoparticles:

1. Tetragonal crystal anatase TiO2 nanoparticles with phase-pure nanoparticles were synthesized successfully as the result of the XRD analysis and as per the COD reference entry No. 96-900-8217.

2. The synthesized nanoparticles were found to have an average crystallite size of 22.687 nm through the Scherrer equation which proved that the product was of a nanoscale and was capable of use in applications concerning surface-area dependence.

3. FTIR spectroscopy was used to identify typical TiO Ti lattice vibrations and the presence of surface hydroxyls (TiOH) which form an active sites and functionalize the surface required in photocatalytic reactions.

4. FE-SEM showed that nanoparticles had near-spherical morphology with particle dimensions in agreement with those of

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crystallites obtained using XRD, which confirmed the existence of structural aspects of nanoscale in the synthesized material.

5. EDX spectroscopy and elemental mapping was used to verify that the nanoparticles were of high chemical purity, that no elements impurities were present, and that the distribution of Ti and O was homogeneous across the entire nanoparticle assembly.

6. All the data on the characterization of the successful preparation of well-crystallized, phase-pure anatase TiO2 NPs collectively confirm the achievement of the structural and physicochemical properties of the nanoparticle that is highly successful in the advancement of its applications in photocatalysis, environmental remediation, and biomedical engineering..

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