**Faculty of Natural and Applied Sciences Journal of Applied and Physical Sciences**
**Print ISSN: 3026-9857**
www.fnasjournals.com
**Volume 2; Issue 1; September 2024; Page No. 32-36.**

# Exploration and Analysis of Titanium Oxide Nanomaterials Using Scanning Electron Microscopy and Energy Dispersive Spectroscopy

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**Abstract**
This research study delves into the synthesis and comprehensive characterization of TiO2 nanomaterials using the sol-gel method. Thorough material property determination is essential before integrating substances into diverse fields such as Science, Engineering, and Technology. This study focuses on the synthesis of titanium oxide nanoparticles via the sol-gel technique employing Titanium Tetra chloride (TiCl4) and Ethanol (CH3CH2OH) as precursor materials at ambient temperature. The resultant gel was subjected to calcification at 600°C for one hour, and subsequent characterization was performed using Scanning Electron Microscopy (SEM) with a JEOL JSM-7600F model, as well as Elemental Dispersion Spectroscopy (EDS). The findings reveal the prevalence of the Anastasia phase within the TiO2 antiparticles, a highly valuable state for various applications. Electron micrographs of the titanium oxide nano particles portray delicate, aggregate, and fiber-like structures. EDS analysis attests to the composition, with Titanium and Oxygen elements constituting 70.24% and 20.30% of the total weight, respectively, indicating a composition ally pure sample comprising 90.54% of the analyzed components. Detected as impurities, carbon and silicon are present in trace amounts, contributing 6.24% and 3.23% by weight, respectively, culminating in an impurity content of 9.46%. The significance of the Anastasia variant of titanium oxide extends to diverse applications, including but not limited to lithium-ion batteries, filtration systems, anti-reflective coatings, and various environmental uses. Furthermore, this nanomaterial exhibits promise in ultraviolet (UV) screening agents like sunscreens, and sunglasses, and serves as a preservative in food packaging applications. This research offers a comprehensive understanding of the synthesis process, material characteristics, and potential utility of Anastasia titanium oxide nanomaterials in numerous practical applications.

**Keywords:** Titanium oxide, Characterization, SEM, EDS, Synthesis

## Introduction

Nanomaterials encompass a wide range of materials with sizes ranging between 1 to 100 nanometres and structures that exhibit dimensions smaller than 100 nanometre across one, two, or three dimensions. These materials can arise from diverse sources: natural phenomena, anthropocentric activities, and deliberate engineering efforts. Natural nano materials, for instance, originate from processes like photochemical reactions, volcanic eruptions, forest fires, erosion, and the byproducts of plants and animals (Buzea et al., 2007). Additionally, unwanted materials, often generated as incidental byproducts from activities such as simple combustion, food preparation, chemical manufacturing, and various industrial processes including welding, refining, smelting, and vehicle and aircraft engine combustion, contribute to this category (Nowack & Bucheli, 2007). In the anthropocentric class of nano materials, materials like metal oxides, zero-valence metals, quantum dots, and dendrites come into play (Buzea et al., 2007). Within this expansive landscape, titanium, a transition metal belonging to Group IV of the periodic table, assumes a vital role. Its oxide derivatives, collectively known as titanium oxides, form through the reaction of titanium and oxygen at elevated temperatures. Present in plants, animal tissues, and igneous rocks like futile and entailment, titanium is the ninth most abundant element, accounting for 0.63% of mass (Tian &Tatsuma, 2004), it boasts an atomic number of 22 and an atomic weight of 47.88. With a melting point of 1,660°C, boiling point of 3,287°C, and specific gravity of 4.54, titanium manifests as a lustrous white metal characterized by low density, robust strength, and resistance to corrosion and potent acids (ENSDF,2010). The realm of titanium oxides holds profound significance due to their high refractive index, UV-absorbing capabilities, and photovoltaic potential. Occurring naturally as oxide compounds of titanium, these materials come in various sizes, morphology, and crystalline structures, enabling their application across catalysis, electronics, sensing, phonic, and medical fields. The processed form of titanium mineral ores, namely TiO2, yields products such as pigments and titanium sponge metal. Distinguished by polymorphic, titanium dioxide (TiO2) exists in multiple phases, including Anastasia, brook-lime, and futile, characterized by their distinct refractive indices. Anastasia exhibits a refractive index of 2.488, while futile and brook-lime possess refractive indices of 2.609 and 2.583, respectively. These phases showcase minimal absorption and dispersion within visible and near-infrared ranges, along with robust chemical and thermal stability ( Zallannn & Moret, 2006; Wang et al., 2004). The Anastasia phase particularly finds application in diverse fields, including lithium-ion batteries, filters, anti-reflective coatings, and highly reflective coatings (Wu et al, 2011). However, maintaining the stability of Anastasia against transformation into futile presents a notable challenge. Abundantly available, titanium oxide is mined extensively in countries like the USA, Brazil, and Canada.

The physical properties influencing the utility of titanium oxide span density, melting point, and refractive index (U.S. Geological Survey Minerals Commodity Summaries 2008; Kirk-Other Encyclopedia of Chemical Technology, Wiley: New York, 2000). Given its high refractive index, TiO2 has been employed as a pigment in both white and coloured systems for an extended period. Its applications extend to paper, printing inks, fibres, and various consumer products, such as sunscreens, cosmetics, and paints. Furthermore, TiO2 nanomaterials have proven efficacious in purifying drinking water and serve as components in food additives, paint pigments, UV-protection creams, and anti-fouling agents. Within the realm of nanomaterials, titanium oxide encounters the phenomenon of aggregation, wherein primary particles aggregate without disintegrating into their elemental constituents. These aggregates can merge to form transient agglomerates, often requiring substantial forces to break them apart. In the case of cryogenic titanium oxide, primary particles sized at approximately 10 to 20 nm persist for only 10 to 200 milliseconds before transitioning into aggregates and agglomerates. Capitalizing on these properties, metallic oxide nanoparticles like titanium oxide are harnessed across electronics, thermal catalysis, phonics, biosensors, and microelectronics. As the sizes of these materials approach the nanosecond, their properties undergo notable modifications (Kamil et al, 2016; Tian&Tatsuma,2004). Thus, the present study centres on the synthesis and characterization of TiO2 nanoparticles using Scanning Electron Microscopy (SEM) (Owolabi,2022).

**Materials and Methods**

The materials used in this study are Titanium Tetra Chloride (TiCl4) with purity of 99.99%, ethanol (C2H5OH) with purity of 99.99%) , Hydrochloride acid (HCl) with purity of 99.7% and distilled water. The instruments used for the preparation are a Heating Mantle (CORNING PC – 420D), Oven, Muffle Furnace , Fume cupboard, mortar, crucibles, weighing balance, spatula, thermometer, pH meter, funnels, stirrer and beakers. Sol-gel technique was employed in the synthesis of Titanium Oxide nanoparticles in which 20 ml of Titanium Tetra chloride (TiCl4) was slowly added drop wise into 200 ml of absolute ethanol (CH3CH2OH) at room temperature. The reaction was performed in a fume cupboard due to the large amount of Cl2 and HCl released during the mixing process. A light yellow solution was obtained and initialized to form a sol-gel precipitate. The pH of the solution was monitored at the range of (Buzea et al., 2007). The sol- gel solution was later vaporized at 80 $^{o}$C until a dry-gel was obtained. The dry-gel precursor was calcined for one hour in the Muffle Furnace at calcination temperatures of 6000C to form TiO2 Nano powders. The laboratory work was carried out in the Pure Chemistry and Industrial Chemistry Laboratory, Prince Abubakar Audu University, Anyigba, Kogi State, Nigeria.

**Results**

**The characterization of Tio2 using SEM-EDS**

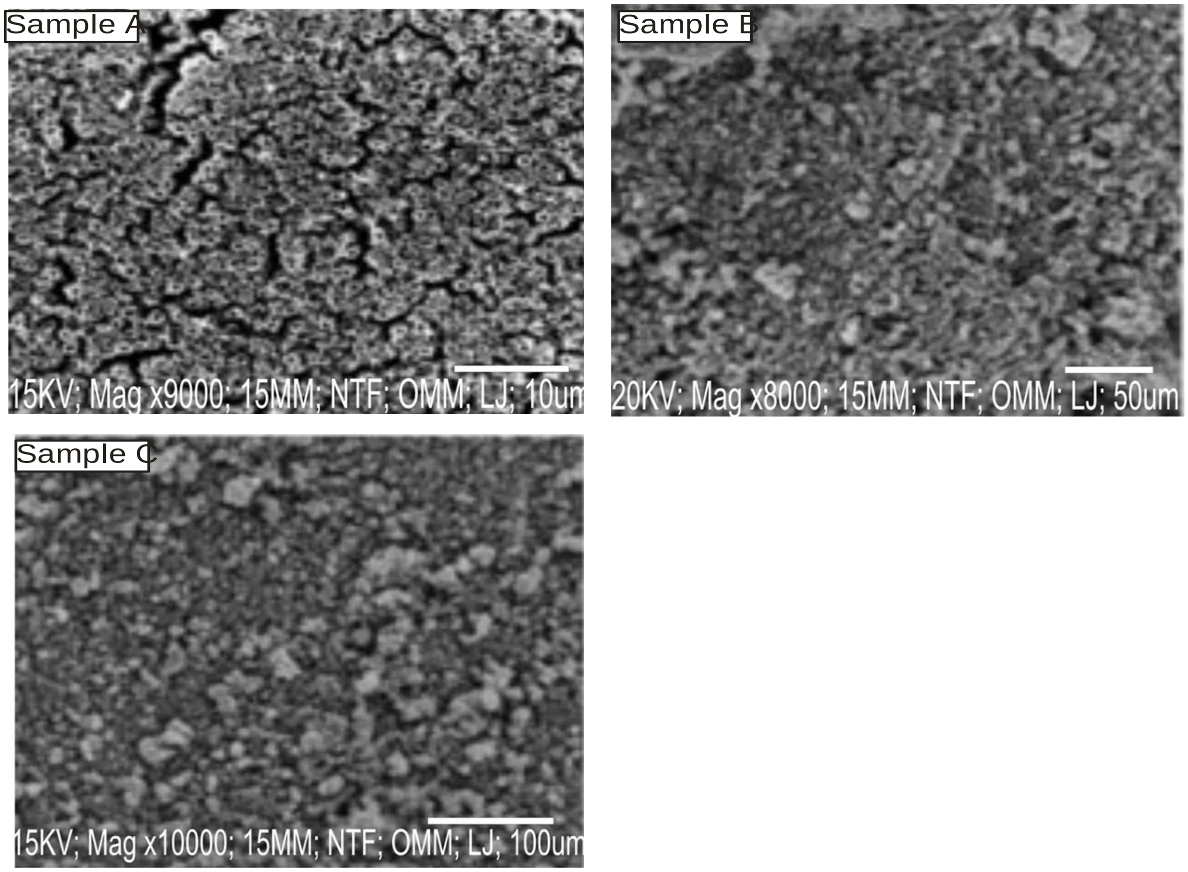


Figure 1: SEM Micro graphs of $TiO_2$ at different magnifications

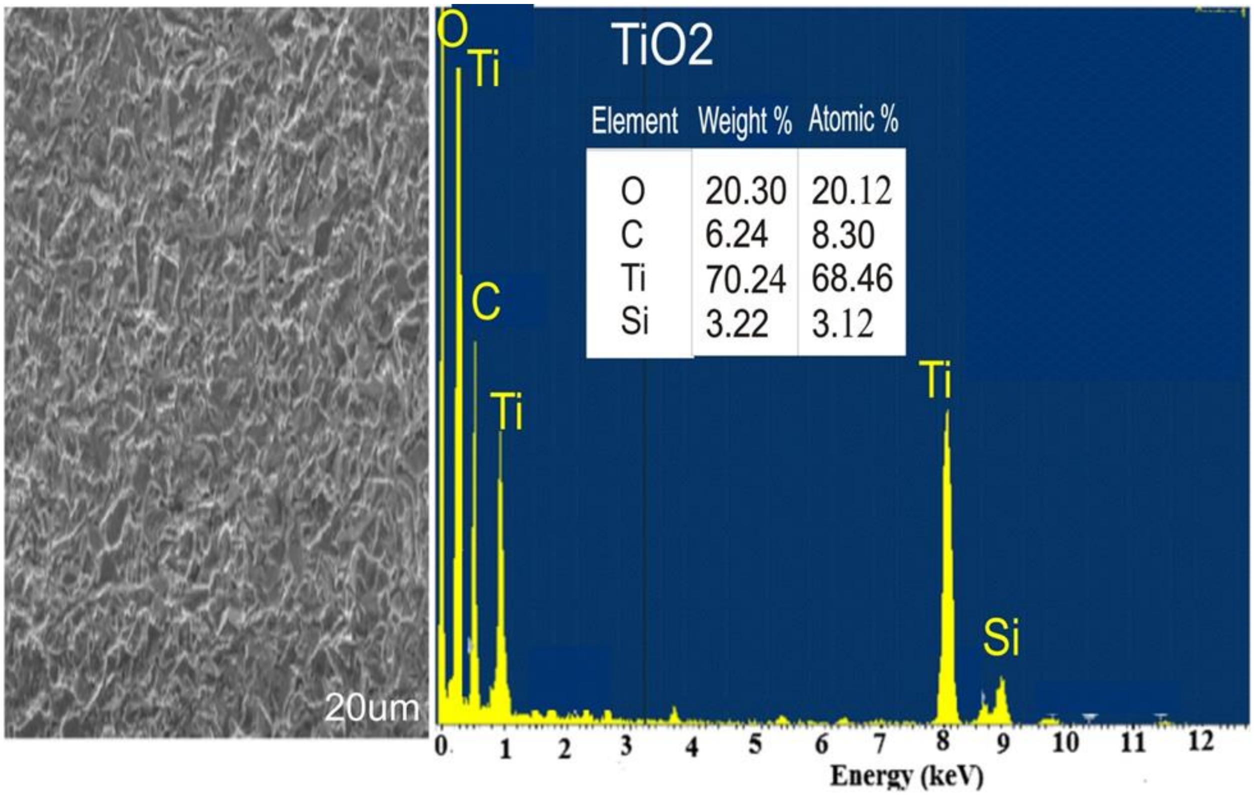


Figure 2: Elemental composition of $TiO_2$ with percentage weights

The analyses were carried out at Rolab Research and Diagnostic Laboratory Ibadan, Oyo State, Nigeria using SEM. The morphology and the structure were determined at different voltages and magnifications of Magx8000, Magx9000 and Magx10000 respectively as shown in figure 1. The structure and textures of the nano material particles calcined at temperature of 6000C are shown below. Figure 1: SEM Micro graphs of TiO2 at different magnifications The EDS was also carried out at Rolab Research and Diagnostic Laboratory Ibadan, Oyo State, Nigeria using the JEOL JSM. 7600F machine. Energy dispersal X-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. This is based on the investigation of samples through the interactions between electromagnetic radiation and that matter, analyzing x-rays emitted by the matter in response to being hit with charged particles. Its characterization are due to the fundamental principle that each element has a unique atomic structure allowing X-rays to identify the elemental composition of the samples.

**Discussion**

From Figure 1, Sample (a), at Magnification of X9000, the observed result/structure is seen to be spherical and spongy. Sample (b), at Magnification of X8000, the observed result/structure appears to be more aggregated and irregular in shape. Sample (c), at Magnification of X10000, shows individual particles with some agglomeration. The EDS analysis in Figure 2 reveals the elemental composition of the synthesized TiO2 nanoparticles. The major elements detected are titanium (Ti) and oxygen (O), which are the main constituents of TiO2. The percentage weights of these elements are shown in Figure 2 indicating the relative abundance of each element in the sample. The use of SEM and EDS is valuable for characterizing the morphology, structure, and elemental composition of materials, particularly nanomaterials like TiO2 nanoparticles. The differences in morphology observed at different magnifications suggest variations in particle size, aggregation, and distribution. The EDS analysis provides quantitative information about the elemental composition, confirming the presence of the desired titanium and oxygen elements in the synthesized TiO2 nanoparticles.

## Conclusion

A titanium oxide nano particle has been synthesized through the sol-gel route and characterized using Scanning Electron Microscope (SEM) and Energy Disperse Spectroscope (EDS). The higher calcification temperature helps in increasing the nanoparticle crystalline. The result of EDX confirms the presence of titanium and oxygen with percentages of 70.4% and 20.30% while impurities like carbon and silicon in trace forms have percentage compositions of 6.24% and 3.23% weights respectively totalling 9.46%. The Anastasia form synthesized is suitable for applications such as Lithium-ion batteries, filters, anti-reflective and high-reflective coatings, and a host of other environmental applications.

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*Cite this article as*:
Owolabi, J., & Ojadi, P. (2024). Exploration and analysis of titanium oxide nanomaterials using scanning electron microscopy and energy dispersive spectroscopy. *FNAS Journal of Applied and Physical Sciences,* 2(1), 32-36.