

## Spectroscopic Studies on Carboxylates of Titanium in Solid State

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### ABSTRACT

*Titanium isopropoxide, boric acid, and deionized water were mixed together in the right proportions and subjected to the Sol gel process in order to make nano-sized Boron doped Titanium dioxide (B-TiO<sub>2</sub>) particles. A calcination procedure was performed on the samples, which lasted for five hours and had a temperature of 500 degrees Celsius throughout its entirety. The findings of X-ray diffraction (XRD) have demonstrated that it is possible for anatase and rutile phases of manufactured nanoparticles to coexist at the same location at the same time. The full width at half maximum measurement that was carried out utilising the XRD led to the conclusion that the size of B-TiO<sub>2</sub> nanoparticles was 20.77 nm. This led to the conclusion that the size of B-TiO<sub>2</sub> nanoparticles was 20.77 nm. The method of UV-vis spectroscopy was used in order to arrive at a value of 3.42 eV for the band gap energy of the boron-doped anatase nanoparticle. This was accomplished in order to get at the conclusion that the nanoparticle possessed a band gap. We were able to determine this by analysing the wavelength of the light that was emitted by the substance. The FTIR method was applied to analyse both the powdered form of B-TiO<sub>2</sub> as well as the form of B-TiO<sub>2</sub> that had been capped with citric acid. In the course of researching B-TiO<sub>2</sub> nanoparticles with Raman spectroscopy, it was discovered that the particles include a phase mixture that is composed of both anatase and rutile.*

**KEYWORDS:** *Titanium, Carboxylates.*

### INTRODUCTION

Titanium dioxide (also known as TiO<sub>2</sub>) is one of the promising materials that may be utilised in a broad range of contexts. These uses span from everyday things like paint and toothpaste to cutting-edge technology applications like photovoltaic cells, photocatalytic breakdown of pollutants, bio-sensing, and antibacterial activity in water. One of the components that may be discovered in dental paste is called titanium dioxide (TiO<sub>2</sub>). These applications range from simple things like paintings and cosmetics to more involved applications like toothpaste and even more advanced sorts of technology. TiO<sub>2</sub> is the n-type semiconductor that is used the most commonly because of its great photocatalytic activity and stability, in addition to its comparatively inexpensive cost and non-toxic nature. The reason for this is due to its tremendous photocatalytic activity and stability. This is as a result of the substantial photocatalytic activity that it possesses. Anatase, rutile, and brookite are the three distinct variations of crystalline forms of titanium dioxide that may frequently be found together. The phase with the greatest level of thermodynamic stability is rutile, while anatase and brookite are regarded to be metastable phases. Rutile is the phase that contains the element ruthenium. The photocatalytic activity of anatase TiO<sub>2</sub>, on the other hand, is much higher than that of rutile. This is because it has a larger surface area in contrast to both its mass and its volume. The reason for this is because it boasts a larger surface area. It has been determined that the band gap of anatase is 3.2 eV, which is comparable to an absorption wavelength of 385 nm when measured at an ultraviolet frequency. In contrast to rutile, which is an oxide of ruthenium and has a band gap of just 3.0 eV (in the visible light range of 410 nm), rutherfordite is an element that contains the element ruthenium. In spite of this, anatase is often always thought of as the phase of TiO<sub>2</sub> that contains the

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highest level of photochemical activity. This increased activity is brought about by the synergistic impact that is brought about by the larger rate of hole trapping and the higher surface adsorptive capacity that anatase possesses. Both of these factors contribute to anatase's synergistic effect. These two characteristics may be responsible, at least in part, for the increased activity of the anatase enzyme.

Recent research has shown that the level of activity achieved when utilising anatase in conjunction with rutile or brookite is superior to that achieved when using anatase on its own. During the process of creating TiO<sub>2</sub> nanoparticles with boron doping, we were successful in accomplishing what we set out to do because we kept in mind the aforementioned facts and information. We will present a method for the manufacture of B-TiO<sub>2</sub> nanoparticles in this study that does not involve an unduly difficult process, and we will also characterise these particles. Over the course of the last several decades, a great deal of progress has been made in terms of the development of various ways of synthesis. Some of these processes include hydrothermal solvothermal sol-gel direct oxidation. In the process of producing nanostructured materials formed of TiO<sub>2</sub>, a wide variety of techniques, such as chemical vapour deposition (CVD), electro-deposition, sonochemical deposition, and microwave deposition, have been utilised. In this study, the synthesis of boron-doped TiO<sub>2</sub> nanoparticles was finished using a straightforward approach called sol-gel, which was chosen because it is simple, economically practical, and environmentally inconspicuous. The sol-gel technique is the name given to this particular approach. Boron was introduced into the body as a result of the usage of boric acid as a source. Boron is a chemical element. In order to determine the characteristics of the B-TiO<sub>2</sub> sample, several spectroscopic analyzers, including X-ray diffraction, UV-Vis spectrophotometers, Fourier transform infrared spectrometers, and Raman spectrophotometers, were employed. In addition to that, Raman spectrophotometers were used.

Reactions of metal alkoxides with bidentate ligands, such as -diketonates, carboxylates, or sulfonates, are essential because they are necessary for the moderation of the exceptionally high reactivity of metal alkoxides towards nucleophilic reagents. This reactivity can be moderated by the reactions of metal alkoxides with bidentate ligands. These reactions are significant because they are required for the moderation of the extraordinarily high reactivity of metal alkoxides towards nucleophilic reagents. As a result, these reactions are crucial for the moderation of this reactivity. As a direct consequence of this need, the use of metal alkoxides is essential.<sup>1–5</sup> Because of this, the use of metal alkoxides, specifically Ti(OR)<sub>4</sub>, as precursors in sol-gel, chemical vapour deposition (CVD), and atomic layer deposition (ALD) techniques, which are typically utilised in the fabrication of titanium oxide containing ceramics or titanium dioxide nanoparticle layers, is constrained. In particular, the use of metal alkoxides as precursors in sol-gel techniques is constrained. In example, one cannot make extensive use of metal alkoxides as precursors in sol-gel processes due to the limitations imposed by these methods. One of these methods is atomic layer deposition, which is sometimes referred to by its acronym, ALD.<sup>6–15</sup> During the process of making organic-inorganic hybrid materials, the reactions that were just gone over make it possible to construct clusters of organically modified transition metal oxides. This is performed by introducing organic molecules into the transition metal oxides in order to alter them.<sup>16,17</sup> such multinucleated complexes that are oxy titanium alkoxo carboxylate and have the general formula of oxo titanium alkoxo carboxylate. An analysis of the spectrum data has shown that the transformation of the previously acquired unstable intermediate species led to the generation of the hexanuclear systems that were mentioned earlier in this passage. This was discovered as a result of the discovery that the transformation of the spectrum data. As a result of the development of the hexanuclear systems, we were able to arrive at this conclusion.

Due to the low quality of the crystals that were separated, it was unable to determine the crystallographic structures of the intermediate species. This fact is directly responsible for the fact that the mystery has not been solved. In addition to this, Papernik et al. (2013) discovered that

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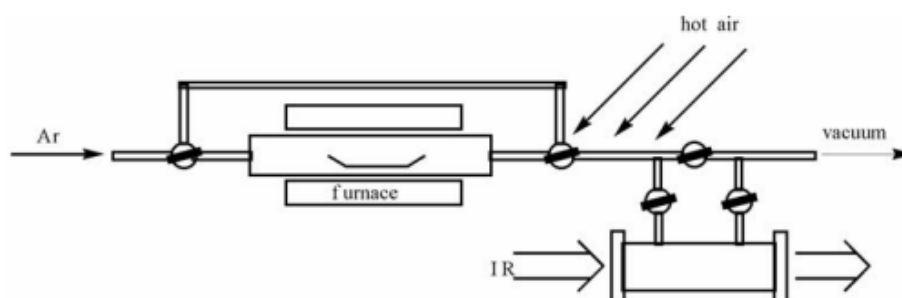
hexanuclear systems may be formed from multinuclear oxo Ti(IV) complexes. According to this study, Schubert presented, on the basis of reports from the relevant literature, the plan of the reaction pathway, starting from the tiny units and working its way up to For the synthesis of novel materials, in which the structure of  $Ti_x(i-O)_y$  clusters needs to be properly regulated, the correct identification of the reactions route is of critical relevance and has a major bearing on the process. In the following report. This study focuses on the structural and spectral characterisation of multinuclear -oxo complexes that were created in contacts of tetrasobutoxide with 2,2-dimethylpropionic acid, 2,2-dimethylbutyric acid, and 3,3-dimethylbutyric acid. These complexes were generated in contacts of tetrasobutoxide with these acids. The formation of these complexes required the presence of tetrasobutoxide throughout the process. The combination of tetrasobutoxide with the aforementioned three unique types of dimethylpropionic acid led to the development of these complexes, and it was this reaction that was responsible for their production. Titanium(IV) tetraisobutoxide, in contrast to the other titanium alkoxides, was chosen for this experiment because it had the capability to form intermediate species that are structurally more stable. This was the primary factor that led to its selection.

## OBJECTIVES

1. The Study Carboxylates of Titanium in Solid State.
2. The Study Titanium Isopropoxide, Boric Acid and Deionized Water in Appropriate Proportion.

## RESEARCH METHODOLOGY

were carried out using the use of the conventional Schlenk line and glovebox methods, as described in the publications that were published on the subject. These experiments were conducted out in an environment that included argon throughout. Microanalysis,  $^{13}C$  NMR, and  $^1H$  NMR, as well as infrared (IR) approaches were the ones that the researchers opted to utilise in order to validate their structure. In addition, infrared (IR) methods were also utilised. Both of the objects were kept in a facility that had an argon atmosphere, and the temperature of the facility where they were kept was kept at the same level during their stay there. Infrared (IR) spectra were obtained with the use of a spectrometer that was modelled after an FT-IR SPECTRUM 2000. Experiments in infrared spectroscopy were able to be performed in the solid phase owing to the employment of a SPECAC temperature variable cell, which enabled the temperatures to be altered. This made it possible for the experiments to be carried out. TGA, which stands for thermogravimetric analysis, was carried out using SDT 2960 TA Instruments in an atmosphere consisting of nitrogen at temperatures ranging from 298 to 773 degrees Celsius with a ramp rate of 3 kelvins per minute. Researchers examined the infrared spectra of vapours that were carried along with the carrier gas (Ar) by using a reactor that had been created particularly for the purpose of conducting the experiment. The results of these studies were written up and published in a document that was a part of the thermogravimetric analysis (TGA) that was carried out. In order to get mass spectra, an EI technique was utilised, and an MS AMD-604, MASPEC system was used for the data gathering procedure. Both of these pieces of equipment are referred to as "instrumentation."



**Fig. 1. A diagrammatic representation of the reactor that was utilised in the IR studies at different temperatures**

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For the studies on deposition, a horizontal 'cold-wall' CVD reactor was utilised. This reactor had a capacity of 10 dm<sup>3</sup>, a heated substrate electrode on top measuring 56 mm in size, a gas input shower that was 15 mm away, and a gas flow rate of 60 cm<sup>3</sup> min<sup>-1</sup>. In order to conduct an analysis of the morphology of the metal oxide layers, a scanning electron microscopy instrument known as a JSM 5500LV was employed in combination with an EDXS system.

### EPR Studies

In this experiment, electron paramagnetic resonance spectroscopy, which is commonly referred to by its abbreviated form, EPR, was used so that the generation of reactive oxygen species on the surface of the composite materials that were under examination could be confirmed. Because electron paramagnetic resonance is a longer term, this concept is often referred to using the abbreviation EPR. The observations made use of an X-band EPR SE/X-2541M spectrometer that was given by Radiopen in Poznan, Poland. This spectrometer was modulated at 100 kHz. The automated NMR-type JTM-147 magnetometer that was employed in the field measurement of the magnetic field was manufactured by Radiopan in Poznan, which is located in Poland. Radiopan is responsible for the fabrication of the magnetometer. The microwave frequency was monitored with the use of a frequency metre at all times. During the measurements, the following parameters were taken into account: a microwave frequency of approximately 9.33 GHz; a range of microwave power from 6 to 58 mW; a modulation amplitude between 0.2 and 1 mT; a sweep with a range from 10 to 50 mT; a sweep time that varied from 1 to 8 minutes; a time constant that varied from 0.03 to 0.3 seconds; and a receiver gain that varied from 3.2 to 5 10<sup>5</sup>. Only extremely weak signals were picked up by the recording equipment when it was set to the accumulation mode. While the temperature of the surroundings around the experiments stayed the same, the measurements were taken using foil samples that were concurrently generated for each of the experiments. The section of the material that had been exposed to sunlight was analysed using EPR in order to get information about its properties. The EPR and catalytic measurements were both conducted in an independent manner, despite the fact that they were carried out quite close to one another in terms of timing.

### Antimicrobial Activity of PMMA + TOCs Composite Films

Gram-negative bacteria (*Escherichia coli* ATCC 25922, *Escherichia coli* ATCC 8739), Gram-positive bacteria (*Staphylococcus aureus* ATCC 25923, *Staphylococcus aureus* ATCC 6538), and *Candida albicans* ATCC 10231 were employed in order to assess the antibacterial activity of the samples. After being cleaned with UVC for 15 minutes on both sides of the foils that were 20 mm x 20 mm, the PMMA + TOCs foils that were being assessed were initially exposed to visible indoor light before the antimicrobial investigation could commence. Following this, the foils were evaluated for quality. Following the completion of this technique, the foils were positioned inside of 12-well plates, each of which contained one millilitre of a microbial inoculum that had been suspended in sterile deionized water. After that, the plates were sterilised by deionization. The plates were kept within an incubator for a period of twenty-four hours at a temperature of 37 degrees Celsius and a rotating speed of 80 revolutions per minute while the air inside the incubator remained humid. These conditions were maintained in order to ensure that the plates would not become damaged.

Utilisation of a densitometer that was produced in Latvia by Biosan helped bring the level of microbial density down to 0.5 on the McFarland scale. This was made possible owing to the fact that the scale was used. First, the inoculum was diluted one hundred times with sterile deionized water before it was used in the experiment. This was done so that the appropriate concentration level could be achieved. Colony counts were used to determine the final concentration after 100 litres of the inoculum had been spread across the surface of Sabouraud Dextrose Agar (SDA; Becton Dickinson for *C. albicans*) or Tryptic Soy Agar (TSA; Becton Dickinson for *C. albicans*) in Petri plates. The control for this specific experiment consisted of floating the test microorganisms in a well that was empty and did not include the item that was being tested. After incubation, the inoculum was

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recovered from the wells by first collecting it, and then later, diluting it serially by a factor of ten. This was done after the inoculum had been exposed to the temperature of 37 degrees Celsius. Following the completion of the incubation procedure, this further step was carried out. After that, one hundred microliters of each of the dilutions were evenly distributed across the appropriate medium on Petri plates. This process was repeated three times. After a further 24 hours of incubation at 37 degrees Celsius, the number of colony-forming units (c.f.u.) that were present on plates that had been infected was measured. The reduction (R) index was computed so that it could be used to represent the antibacterial activity of the goods that were tested. These products were put to the test in order to determine their effectiveness. In order to assess whether or not these items were useful, we put them through their paces and conducted some tests. The following formula was applied in order to carry out the task of computing the R index: R is equal to  $U_t$  plus  $A_t$ , where  $U_t$  is the common logarithm of the number of microorganisms present in the microbial inoculum and  $A_t$  is the common logarithm of the number of bacteria present in the treated microbial inoculum. In other words, R is the ratio of the two logarithms. R represents the relationship between the two. The biocidal activity of a sample may be evaluated using a number of different criteria, one of which is the R-value of the sample.

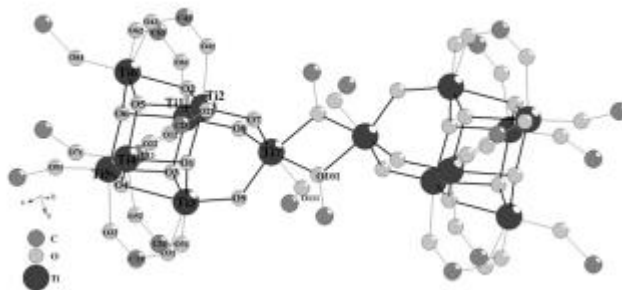
## DATA ANALYSIS

Crystalline compounds were produced by reacting titanium(IV) tetraisobutoxide with branched organic acids ( $\text{HOOCR}'$ ;  $R' = t \text{ Bu}, \text{CH}_2 t \text{ Bu}, \text{CMe}_2\text{Et}$ ) at ambient temperature in an argon environment while applying a molar ratio of ligand to titanium. The reaction took place at a tetrabutyl group of the titanium(IV) tetraisobutoxide. The reaction was carried out in an atmosphere consisting of argon. The development of a solitary crystal that is colourless and odourless will take place over the course of a few days as a result of the gradual evaporation of the reaction mixture that takes place in an atmosphere that is composed of argon. This will happen in the future. After isolating crystals of a quality that was adequate for the X-ray single-crystal diffraction measurements, those crystals were then submitted to crystallographic and spectral (IR, NMR) examinations. The X-ray single-crystal diffraction measurements were successful. Additional studies of the building were carried out somewhere between ten and fourteen and twenty-five and thirty days after the initial ones. explains the process of gathering X-ray data for complexes and highlights the many components that go into making up the complex. In addition, the particular bond lengths and angles of the compounds that were researched are provided in ESI Tables S1 and S2 respectively. These tables may be found in the ESI document. You should be able to find these tables in the ESI document. It was not possible to solve the crystallographic structure since the problematic crystals were of poor quality; as a result, the structure was created on the basis of an examination of the spectrum data.

## Multinuclear Ti(IV) complexes synthesized in the 1 : 1 reaction of $\text{Ti}(\text{O}i \text{ Bu})_4$ with $\text{HOOC}(\text{CH}_3)_2\text{Et}$

After a period of five days during which the reaction mixture was allowed to gradually evaporate at room temperature in the presence of gas, the colourless crystals of complex were efficiently separated. This was accomplished by separating the crystals of complex. Studies using X-ray diffraction have showed that the structure of is made up of dimeric units that have the general formula  $[\text{Ti}_7\text{O}_9(\text{O}i$  and are generated around the inversion centre. These units make up the structure of the inversion centre. To generate the dimeric structures, the hexanuclear cores of 3-oxo titanium are coupled with three different alkoxide ligands at the terminals of the dimeric skeletons.

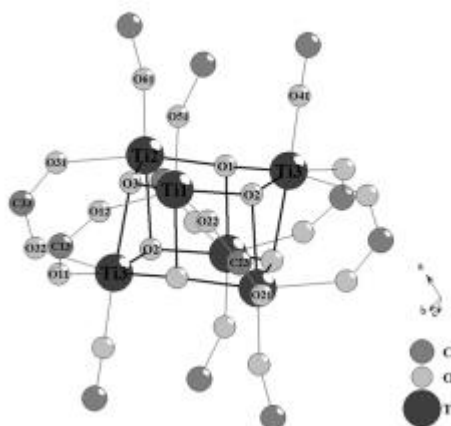
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**Fig. 2 structure of (1) showing some of the crystallographic labeling scheme; hydrogen and carbon atoms have been omitted for clarity.**

(O71, O81, O91). The hexanuclear cores are able to interact with one another and continue to be connected to one another because to the presence of three oxygen atoms that are a component of the oxo bridges that connect the sections  $[\text{Ti}_2(-\text{O})_6(\text{HOi Bu})_2(-\text{Oi Bu})_2]$ . (Fig. 2). There is a possibility of finding octahedral structures that have a little distortion and are housing titanium atoms in three distinct configurations. These situations are entirely apart from one another in every way. Ti4, Ti5, and Ti6 atoms each have one terminal Oi Bu ligand, three 3-oxo bridges, and two oxygen atoms that are a component of carboxylic groups that coordinate them. On the other hand, each of the atoms that make up Ti1, Ti2, and Ti3 are surrounded by two oxygen atoms that are a part of carboxylic groups. In addition, there are three 3-oxo bridges and one -oxo (O7–O9) bridge connecting each of these atoms. Each of the two Ti7 atoms that are a member of the  $[\text{Ti}_2(-\text{O})_6(\text{HOi Bu})_2(-\text{Oi Bu})_2]$  fragment are surrounded by oxygen atoms that are a part of one of five distinct bridges. These bridges include three -oxo bridges (O7–O9), two -Oi Bu bridges (O101), and one adducted HOi Bu bridge (O111). The TiTi distances (Ti1–Ti6) can be anywhere between 3.0892(12) and 3.5259(13), although the Ti7Ti7 distances are commonly about 3.2104(17).

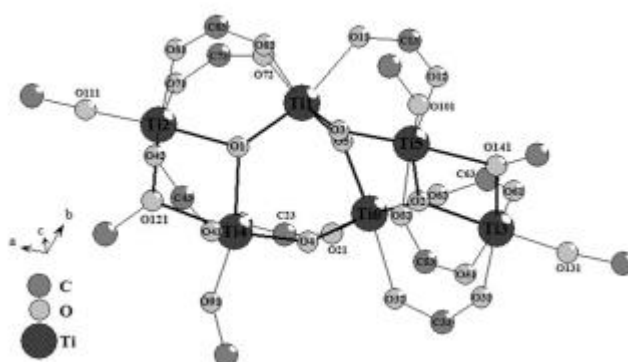
During this time, the distances that divide Ti7 and Ti1–Ti3 atoms will shift to a new value that will be somewhere in the range of 3.3243(13)–3.3418(13). This will occur as the process continues. Ti–O bond lengths may range anywhere from 1.866(5) to 1.927(5) angstroms, Ti–(3-O) bridge lengths can range anywhere from 2.079(3) to 2.222(4) angstroms, and Ti–(–O) bridge lengths can range anywhere from 1.777(3) to 1.887(3) angstroms. Six asymmetric carboxylate syn–syn bridges, sometimes referred to as -OOCR, are in charge of maintaining the stable configuration of the hexanuclear Ti–O half units. The theoretical range of length changes for shorter bonds is 2.025(4)–2.053(4), whereas the range for longer bonds is 2.059(4)–2.099(4). When it comes to shorter bonds, the range is 2.025(4)–2.053(4). The lengths of Ti–Oi Bu bonds can vary anywhere from 1.753(4)–1.770(4) angstroms for terminal ligands of Ti6–(3-O)6 cores all the way up to 2.015(3) and 2.029(3) angstroms for Ti–(–Oi Bu) bridges of  $[\text{Ti}_2(-\text{O})_6(\text{HOi bu})_2(-\text{Oi Bu})_2]$  species. These lengths are measured in angstroms. The length of the Ti–O bonds in adducted alcohol molecules was measured by the researchers and found to be 2.202(4) angstroms in length. Ti–(3-O)–Ti angles shift into two distinct ranges: the first range extends from 98.22(15) degrees to 103.77(15) degrees for three atoms that correspond to varying amounts of titanium–oxide, while the second range starts at 128.99(17) degrees. Both of these ranges are inclusive of three atoms. Both of these ranges correspond to different concentrations of titanium–oxide.



**Fig. 3 Structure of (2) showing some of the crystallographic labeling scheme; hydrogen and carbon atoms have been omitted for clarity.**

### Multinuclear Ti(IV) complexes obtained in the 1 : 1 reaction of Ti(Oi Bu)<sub>4</sub> with HOOCt Bu

"When left for five days in an environment free of oxygen, titanium tetrabutoxide and 2,2-dimethylpropionic acid dissolved in toluene would, as a result of being subjected to gradual evaporation, result in the separation of colourless single crystals. This process would take place after being treated to an environment devoid of oxygen. By analyzing the X-ray diffraction data that was collected at 293 K, researchers were able to demonstrate that complex 3, which has the general formula [Ti<sub>6</sub>O<sub>5</sub>(Oi Bu)<sub>6</sub>(OOCt Bu)<sub>8</sub>], was formed. This conclusion was reached after the data was analysed. The structure of (3) is made up of two titanium(IV) triangle units, each of which is composed of three atoms of titanium (Ti1, Ti2, Ti4, and Ti3, Ti5, Ti6) that are connected to one another by 3-oxo bridges. The structure of (3) is a titanium(IV) tetrahedron. The formula for three is denoted by the symbol Ti<sub>3</sub>–(3-O), and its value is three. Together, the -alkoxo bridge (O121 and O141), three bidentately coordinated OOCt Bu groups (O41, O71, O81 and O31, O51, O61), and two terminal Oi Bu ligands (O91, O111 and O101, O131) are responsible for keeping these basic units in place. The linking of the Ti<sub>3</sub>–(3-O) units is accomplished by the cooperation of three oxo bridges (O3, O4, and O5) and two syn–syn carboxylate bridges (O11 and O21)".



**Fig. 4 Structure of (3) displaying a portion of the crystallographic labelling system; for the sake of clarity, hydrogen and carbon atoms have been deleted..**

### CONCLUSION

The production of titanium dioxide was accomplished by the utilisation of a manufacturing process known as Sol gel, which is renowned for its exceptional efficiency and low cost. A calcination took place for a total of five hours with the samples being heated to a temperature of five hundred degrees

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Celsius. The use of X-ray diffraction made it possible for the observation to be made that the man-made nanoparticles had both the anatase and rutile phases. This is the realisation that was pursued and pursued successfully. When conducting an XRD analysis and making use of the equation proposed by Debye and Scherer, it was discovered that the B-TiO<sub>2</sub> nanoparticles that were generated had an average particle size of 20.77 nanometers. This was discovered when the nanoparticles were subjected to an XRD examination. It was possible to acquire this information. By making use of absorbance spectra, we were able to accomplish our goal of accurately calculating the energy of the optical band gap. The energy of the band gap in B-TiO<sub>2</sub> nanoparticles was estimated to be 3.42 eV by the use of mathematical computations. This value was arrived at. The FTIR method was applied to analyse both the powdered form of B-TiO<sub>2</sub> as well as the form of B-TiO<sub>2</sub> that had been capped with citric acid. The three primary absorption peaks that were found are located at respective wavelengths of 3950 cm<sup>-1</sup>, 1800–2200 cm<sup>-1</sup>, and 991 cm<sup>-1</sup>. It has been discovered that the bending vibrations of Ti-O display a diverse spectrum of peaks that are within the region of 1800 to 2200 cm<sup>-1</sup>. There is a chance that boron doping on TiO<sub>2</sub> is to blame for the appearance of the new peak at 3950 cm<sup>-1</sup>, as this is one of the possible explanations. A possible interaction between the carboxylic acid group and the surface of B-TiO<sub>2</sub> may have taken place, as demonstrated by the peak's migration from 1800–2200 cm<sup>-1</sup> to 1000–1200 cm<sup>-1</sup>. This is supported by the peak's movement from 1800–2200 cm<sup>-1</sup> to 1000–1200 cm<sup>-1</sup>. This strongly suggests that the material in question is compatible with living organisms.

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