

In Situ Phase Study of $\text{La}_2\text{O}_3+\text{TiO}_2$ Composite By XRD

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ABSTRACT

With growing emphasis on environmental sustainability, green synthesis methods for nanomaterials have emerged as viable alternatives to conventional chemical techniques. These eco-friendly approaches are particularly attractive for their low toxicity, cost-effectiveness, and minimal environmental impact. This study presents a green synthesis strategy for developing a $\text{La}_2\text{O}_3:\text{TiO}_2$ nanocomposite using the leaf extract of *Dalbergia sissoo* (Indian Rosewood) as a natural reducing and stabilizing agent. The synthesis followed a simple solution method, where the phytochemicals present in the *D. sissoo* extract acted as bio-reductants to facilitate nanoparticle formation without toxic chemicals. Structural analysis via X-ray Diffraction (XRD) confirmed the formation of a composite material comprising anatase-phase TiO_2 and cubic-phase La_2O_3 . The nanocomposite showed a reduced crystallite size compared to pure TiO_2 , suggesting enhanced control over nucleation and growth due to the bioactive compounds in the extract. Fourier Transform Infrared Spectroscopy (FTIR) revealed absorption bands corresponding to hydroxyl, carbonyl, and other organic groups, indicating the involvement of plant-derived compounds in nanoparticle capping and stabilization. These surface-bound functional groups may also enhance reactivity and environmental interactions. Scanning Electron Microscopy (SEM) showed semi-spherical nanoparticles with uniform distribution and minimal agglomeration. This morphology reflects the effective role of the plant extract in directing particle shape

and preventing clustering, contributing to an increased surface area. The successful integration of La_2O_3 into the TiO_2 matrix through a green synthesis route highlights the potential of plant-based methods for engineering advanced nanocomposites. This approach enables tailoring of material properties for targeted applications such as photocatalysis, sensing, and environmental remediation.

Keywords: La_2O_3 , TiO_2 , XRD

Introduction

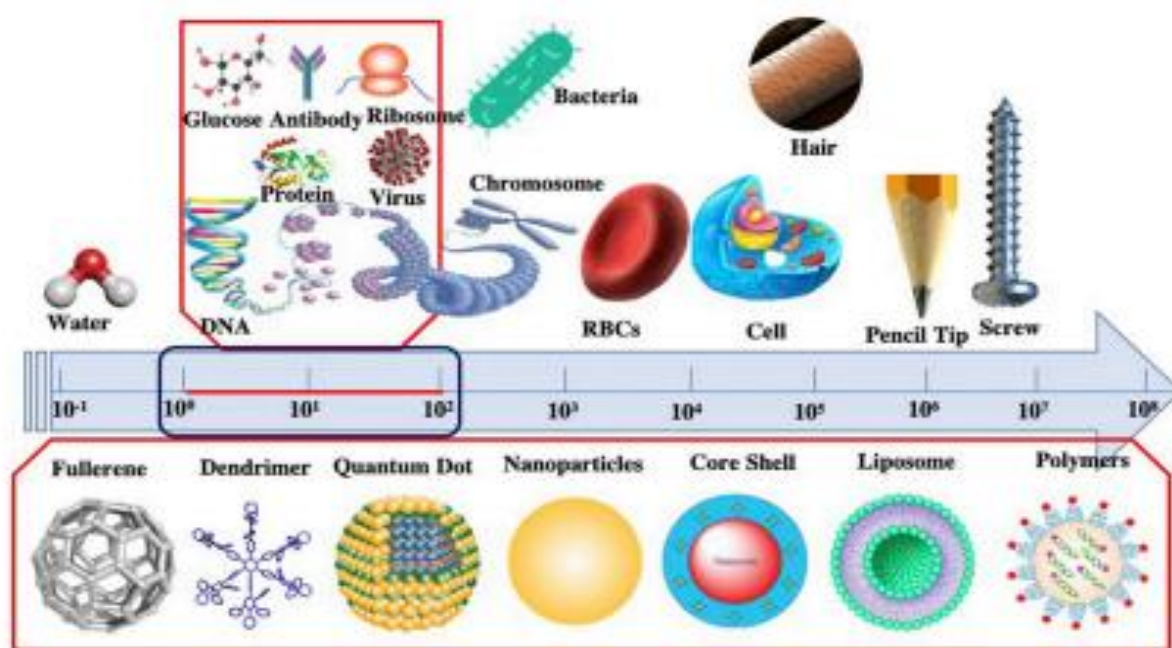
Nano Sciences

Nano science is a multidisciplinary field of science that studies the properties and behavior of materials at the nano scale level. The term "Nano science" refers to the study of phenomena that occur at the nanometer scale, which is typically defined as the range from 1 to 100 nanometers (nm) (Binns, 2021).

Nano scale level is characterized by the properties that are not found in the larger scale level of materials. For instance, Nano materials have a large surface area then their volume, thus they are highly reactive besides having unusual optical, electronic, magnetic, and mechanical behavior. Furthermore, the behaviour of nanomaterials in most instances is dictated by quantum physics a fact that makes them remotely sensitive to external influences (Nalwa, 2004).

Various techniques are used in the manipulation and identification of Nano scale materials which forms the topic of discussion in nanoscience. Some of the techniques is Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Atomic Force Microscopy (AFM), X-Ray Diffraction (XRD), and Spectroscopy Method or Methodology such as UV Diffractions Techniques, UV FTIR, Ramand Spectroscopy and etc. , with their findings. Such techniques enable the scientists to gather information on the configurational, optical, electronic, magnetic and mechanical characteristics of the nanomaterial's (Kulkarni & Kulkarni, 2015).

Nano materials have a great importance in different fields of life. It includes medicine, electronics and energy and environmental applications. Nano science results in the fabrication of new materials and devices with enhanced performance like Nano scale transistors, sensors, and memory devices. In medicine, nano science has been used to develop new drug delivery systems, imaging agents, and therapeutics for the treatment of diseases such as cancer. In energy, Nano science has led to the advancement of new materials for solar cells, fuel cells, and batteries (Wilson, Kannangara, Raguse, Simmons, & Smith, 2002). In materials science, Nano science has been used to develop new materials with enhanced properties, such as strength, durability, and conductivity. In environmental science, nanoscience has been used to develop new technologies for water purification, air filtration, and soil remediation (Wilson et al., 2002). Nanotechnology is constructed and drawn its, depiction and its impact, fabrication and its vast application of its structures,



devices and frameworks by supervise the shape and size at the nanoscale. The integration of biomolecules at the nanoscale is shown in Figure 1.1(Saallah & Lenggoro, 2018).

Figure 1.1: integration of biomolecules at nano scale range.

Over the past few decades, science has made tremendous progress. One of the

greatest inventions of science is physics, in which we study matter and laws. While science can be defined as a methodology that deals with the study of natural phenomena. The knowledge we gain from experimentation and observation is called science. If we observe everyday objects, we learn the phenomena of science. Nanotechnology plays an important role in new inventions of science (Wolf & Medikonda, 2012).

History of Nanotechnology

Nanotechnology is a relatively new field, and its history can be divided into several phases. The early phase, which began in the early 20th century, was characterized by the discovery of quantum mechanics and the realization that matter could be understood and utilized and employed at the atomic and subatomic level. The term nano-technology was not coined at this time, but, indeed, the ideas of orchestrating matter at the atomic molecular level were broached by many scientists from Feynman to more recent geologist Nano Technology.

During the 1950s and the 1960s researchers were able to directly investigate matter at the Nano scale level using tools like the electron microscope and X-ray diffractions. The scanning tunneling microscope was developed in 1981 by Gird Binnig and Heinrich Rohrer enabled scientists to scan through a surface in order to observe the atoms and molecules that are present as well as move them from one position to the other. This development caused marker of the modern era of nanotechnology in which scientists were able to work at the atomic and molecular level with a high degree of control.

The term nanotechnology was coined in 1986 by K. Eric Drexler, in his book titled “Engines of creation” that talked about the synthesis of new machines and devices at Nano scale. This vision of nanotechnology later on came to be called the “molecular nanotechnology”, which focused on the possibility of developing ultra-small systems that would be made from atomic and molecular components. Despite the fact that the molecular nanotechnology is still a subject of research interest, other forms of nanotechnology have been developed over the years some of which include the top-down technology which include lithography and etching as well as the bottom- up technology which include self-assembly and molecular beam epitaxial.

Nanotechnology as a field only came into focus in the early and mid-nineties when the governments, funding agencies and industries saw the prospect in the applications of nanomaterials and devices. The U. S. National Nanotechnology Initiative (NNI) was launched in 2001 with the mandate of co-coordinating and funding of research in the field of nanotechnology. ;The European Union also launched another corresponding project, called the European Nanotechnology Initiative (ENNI), in 2001m(Drexler, 1987) .

Since then the field has blossomed, and nanotechnology has practically influenced numerous arenas of study. For instance in the electronics field, the application of Nano material's has yielded enhanced and faster, Miniature and efficient gadgets. In medicine, applications of nanoparticles are in delivery of drug and imaging and sensing. In energy, nanotechnology is been applied to come up with a better solar cell and batteries. By applying approaches in materials science, new and advanced nanomaterials are created offering enhanced desires performance characteristics.

Overall, the history of nanotechnology is characterized by significant advances in our ability to manipulate and understand matter at the Nano scale level, as well as the development of new tools, techniques, and approaches for studying and manipulating nanomaterial's. Ongoing research in nanotechnology is expected to lead to the development of new materials and technologies with significant applications in many different fields(Binnig & Rohrer, 1987) .

It is such a small particle the width of a human hair . Here, one nanometer is equal to 1 billionth of a meter. It is widely used in various areas of nanotechnology, which are given below in Figure 1.2.



Figure 1.2: Practical Applications of Nanotechnology

Applications of Nanotechnology

The nanotechnology make it entity in different fields are discussed in table 1.1

Table 1.1 applications of nanotechnology in different fields.

Medical and drugs	Cancer Treatment, Surgery, Drug delivery, Medicine.
Nanodevices	A solar cell, Fuel cell, Batteries, and Biofuel.
Optical Electronics	Audio, Cameras, Mobile devices, and Computer hardware.
Bioengineering	Food and Beverages Advance packing materials, Sensors.
Nanofibers	Stain-proof, Waterproof, Wrinkle-free textiles. • Space: Low power, Integrable Nano.
Energy Cosmetics	Cosmetic products, Sunscreen, and Anti-aging .

Nanomaterials

Nanomaterials are materials that have a size range of 1-100 nanometers (nm) in at least one dimension. They have unique physical, chemical, and biological properties that differ from their bulk counterparts due to their small size and high surface-to-volume ratio. The properties of nanomaterials can be tuned by controlling their size, shape, composition, and structure, making them useful in various applications (Nair & Laurencin, 2008).

Nanomaterials can be grouped depending on their compound, their arrangement and their size. Some of the most well-known major classes of nanomaterials include; nanoparticles, nanotubes, nanowires, nano films, and nanocomposites. These can be made using many techniques including; two tier top up and down approaches; biological, chemical or physical (Mirkin, Letsinger, Mucic, & Storhoff, 2020).

Nanomaterials are used in different sectors, for instance; Electronics, energy, medical uses, and environmental uses. In electronics Nano materials are applied in manufacturing transistors, memory devices and sensors because of there electrical

characteristics. In energy they are applied in solar cells, fuel cells and batteries due to their high surface area and energy transformation rate. In medicine, they are applied for the treatment of diseases, regeneration of tissues and imaging because they are biocompatible and additionally, are able to deliver their load to a particular place in the body. In the environment, they are employed in water purification, air purification and waste disposal since they unravel adsorption and catalytic characteristics (Nasrollahzadeh, Sajadi, Sajjadi, & Issaabadi, 2019). A material that has Nano scale particles or constituents, or a material produced by nanotechnology. Nanomaterials can be defined as materials having at least one outer dimension of 1-100 nm.

Classification of Nanomaterial on the Bases of Dimensions

The properties of nanomaterials strongly depend on their morphology. Based on the electron dimension constraint, nanomaterials are classified as (also shown in Figure 1.4)

3-D (Nanoparticles)

2-D (Nanowires/Nanotubes)

1-D (Thin surface coating)

0-D (Quantum dot)

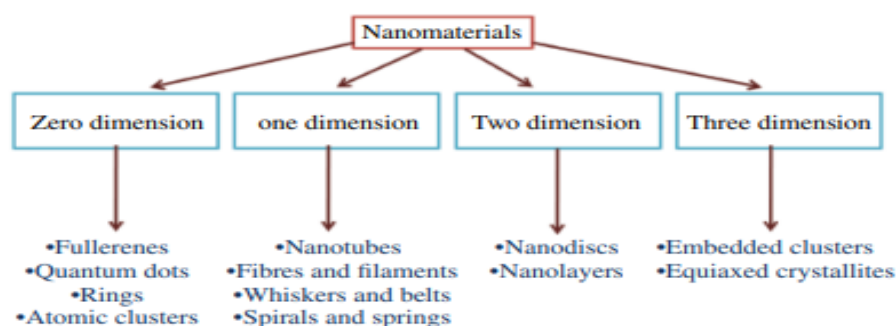


Figure 1.3: Classification of nanomaterial on the bases of Morphology (Giacovazzo, 2002)

Zero-dimensional Nanomaterials (0-D)

All dimensions are measured at the Nano scale (no dimension exceeds 100 nm). 0D nanomaterials are nanoparticles. 0D nanoparticles are long and wide in the nanometer range, e.g., quantum dots, rings and atomic clusters. 0D nanoparticles have great potential in bimolecular recognition, ion detection, and disease diagnosis.

They can also be used in printing.

Dimensional Nanomaterials (1-D)

Dimensional (1-D) nanomaterial's are materials that are in the Nano scale in two dimensions. The best examples of dimensional 1-D nanomaterial's are nanowires, Nano rods, nanotubes, and Nano ribbons from the important class of one-dimensional nanomaterial's, as compared to bulk materials. One-dimensional nanomaterial's have a larger surface area and the possibility of quantum-configured effects. In one-dimensional nanomaterial's, confined electrons are in two dimensions and can move freely in only one dimension (Nel, Xia, Madler, & Li, 2006).

Two-dimensional (2-D) Nanomaterials

In two-dimensional materials, the motion of electrons is limited to one dimension, while electrons can move freely in two dimensions. Particles with a high aspect ratio are also considered two-dimensional nanomaterials .Two-dimensional materials are not limited to Nano scale dimensions. Two-dimensional nanomaterials are shaped like a plate. Some of the Examples of two-dimensional Nano materials are Nano films, Nano layers, Nano coatings, and quantum wells(Kargarzadeh et al., 2018) .

Three-Dimensional Nano materials (3-D)

A size greater than 100 nanometers, having three dimensions, possessing a nano crystalline shape at the nanoscale, similar to a bulk material. Nanoparticles with multiple layers, nanoparticle frameworks, nanotubes and fiber frameworks are the best examples of three-dimensional materials. Three-dimensional nanomaterials, such as nanowires and Nano sheets, have attractive characteristics for use in bio imaging and other semiconductor devices. On the other hand, composite three-dimensional nanomaterial's have found applications in science.

Nanomaterial in different forms can also be shown with the help of diagrams, which are shown in Figure 1.4.

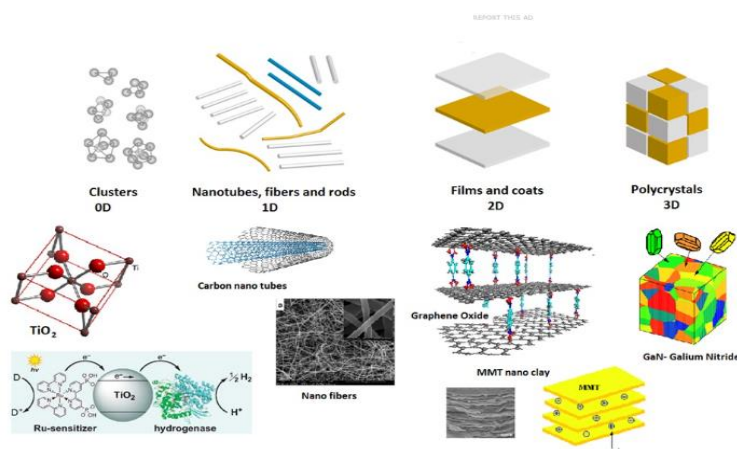


Figure 1.4: Classifications of Nanomaterial on the Bases of Dimensionality.

Methods of Synthesis of Nanoparticles

Nanomaterial's are of great importance to people engaged in scientific research. Nanomaterials have become one of the most interesting topics in various fields such as physics, chemistry, engineering, and biology. The form of nanostructures can be dimensionless, which includes nanoparticles, one-dimensional - nanowires, two-dimensional - thin films, and three-dimensional - arrays, hierarchical structures. Nano scale materials determine their physical and chemical properties. The properties depend on such factors as the size, shape, and crystal structure of nanomaterial's, so many studies have been conducted on the control synthesis of nanostructured materials(Rikta, 2019). There are two techniques by which Nano materials are formed:

- Physical method (top down)
- Chemical method (bottom up)

Top-down Approach

The most commonly used top-down approach is milling. One of the biggest problem with the top-down approach is damage to the surface structure and crystallographic damage to the machined structures. These disadvantages, in turn, lead to additional problems in device design and manufacturing. However, this approach leads to the creation of bulk Nano fabrics. Despite the drawbacks of the top-down approach, it will continue to play an important role in the creation of nanostructures.

Bottom-up Approach

The bottom-up approach is building tissue from head to toe, molecule by particle, particle by atom or cluster by cluster. Colloidal scattering can serve as an excellent illustration of the bottom-up approach in a mixture of nanoparticles. Although the bottom-up approach is often used in nanotechnology, it is not a new concept. All living things in nature observe development using this approach, in addition, it has been used in mechanics for over a century. Examples include salt and nitrate production in the chemical industry. Although the bottom-up approach is not unusual, it plays an important role in the creation and processing of nanostructures(Zhang, Uchaker, Candelaria, & Cao, 2013) .

The flow diagram for top up and top down approach for the synthesis of nanomaterials are shown in the figure 1.5.

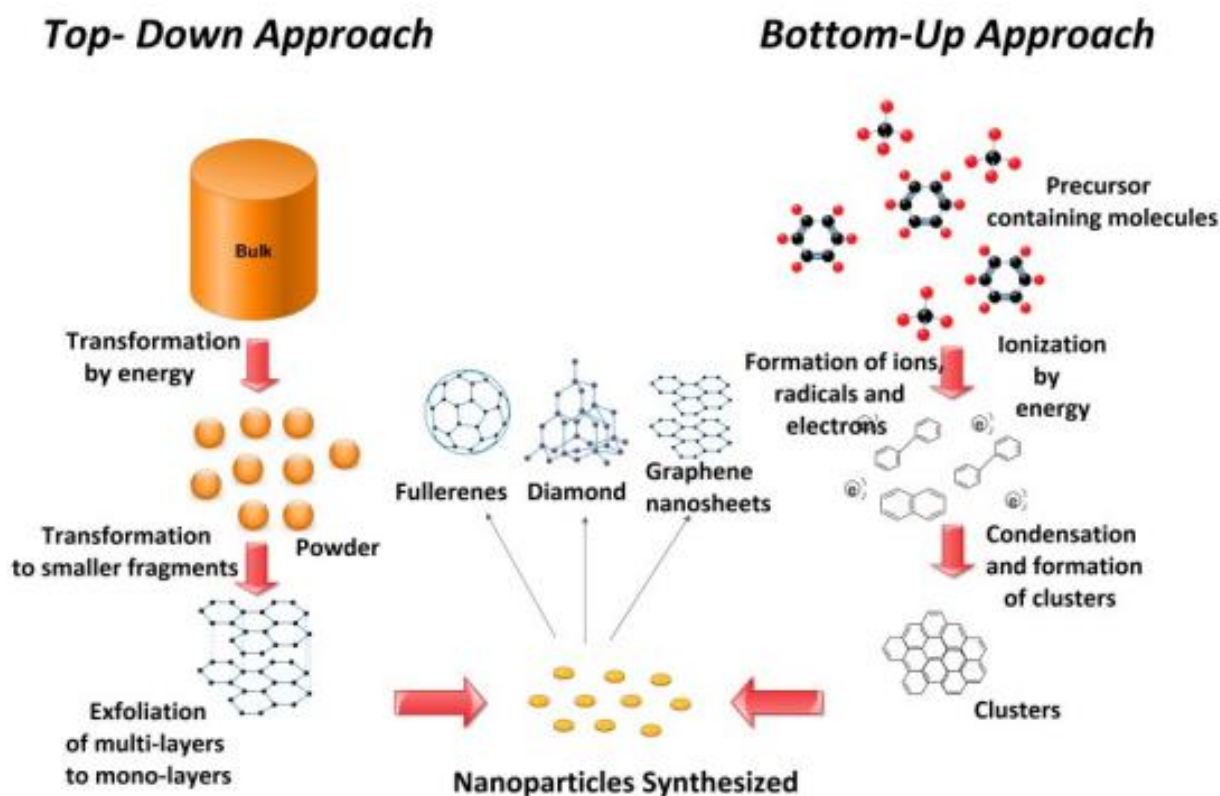


Figure 1.5 : Top Up and Top Down Approaches for the Synthesis of Nanoparticles

Introduction to Titanium Dioxide

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Titanium dioxide is a mineral that occurs naturally in nature. It has the chemical formula TiO_2 . There are many semiconductors that can be used, but titanium is a rich, biocompatible, and inexpensive semiconductor. It is very unique because of its physical and chemical properties. It has good conductivity and chemical stability. Because of its various properties and unique characteristics, we can use this oxide in a variety of commercial, industrial and residential applications.

Occurrence

There are many common metals on Earth, and titanium is one of them. Because titanium is an oxide, it is found as a mineral in the Earth's crust. We can also find it together with calcium and iron. In addition, at very high pressures, other forms of titanium exist, viz.

Monoclinic baddeleyite.

The other form is orthorhombic, known as brukite.

Both forms can be found in Germany. Ilmenite and rutile are two forms in which titanium is abundant, but the highest percentage is found in rutile, which is 98%(Cullity & Smoluchowski, 1957) .

Structure

Titanium (TiO_2), which is white in color, has a very unique structure and shape, namely

- Rutile, which has a tetragonal structure.
- Anatase, which also has a tetragonal structure.
- Brukite differs from the above forms and has an orthorhombic structure.
- Pure titanium dioxide can be a fine white powder that gives a brilliant and white hue.

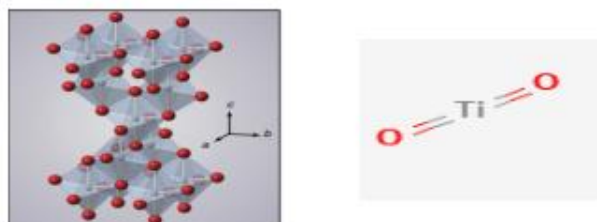


Figure 1.6: Structure of TiO_2

Properties

Titanium dioxide being different from other minerals and oxides and has various

properties. It has physical as well as chemical properties due to which it is widely used.

Physical Properties

If we look at the properties of titanium dioxide we will see and observe some special things about this material first if we look at its melting point so we will observe that titanium dioxide has a melting point of 1843°C . After this if we discuss its boiling point we will see that it shows that due to its non-polarity it is non-polar in nature and it is not soluble in water as it occurs in solid form. Nature-wise due to this its boiling point is 2972°C . If we discuss its brilliant, it looks dark in color TiO_2 as like at other materials and substances it shows a good haziness and brilliance in nature. It is also used as a shield factor to make it a good resistor from heat and light.

It has defensive capacity since it can shield skin from UV radiations which makes it a significant fixer for sunscreen, securing the skin from harmful cancer-causing UV beams.

TiO_2 is used as a photocatalyst in solar panels as well as reducing pollutants in the air (Giacovazzo, 2002). These are its properties as discussed in detail.

Types of Titanium Dioxide

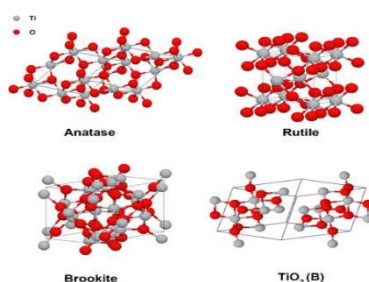


Figure 1.7: Structures of Types of TiO_2

It has basically three types which are as follows:

- i. Rutile titanium dioxide nanoparticles
- ii. Anatase titanium dioxide nanoparticle
- iii. Brookite

Rutile Titanium Dioxide Nanoparticles

It has high stability and appears in dark red color. At high temperature and high pressure we obtain this form of titanium dioxide. In rutile 98% of titanium is present so it is very abundant form of titanium dioxide. As in above figure rutile's have a

good crystalline structure encompasses a tetragonal unit cell having oxygen anions and titanium cation. The oxidation number of titanium cation (Ti^{+4}) is 6 whereas the coordination number of oxygen anion (O_2^-) is 3.

Properties

Rutile has very unique properties due to which makes it ideally different from other forms. Following are its properties

- It has greater dispersion.
- It can display double refraction of light i.e., high birefringence.
- Greater refractive index (RI) at visible wavelengths (Qi, Stein, & Friščić, 2014).

Applications

- It is used in manufacturing of metallic titanium and titanium dioxide pigments.
- It is used in plastics, paints and papers (Mingos, 2020).

Anatase Titanium Dioxide Nanoparticles

The nanoparticles of anatase titanium dioxide are found colorless or yellow in color. The characteristic frame of anatase titanium dioxide contains a parcel of pollutions which makes it dark. Anatase titanium dioxide is very distinctive from rutile and is tetragonal in crystalline structure. It is metallic in appearance. It can be synthesized to be utilized within the fabricating of semiconductor.

Brookite Nanoparticles

Brookite is the orthorhombic variation of titanium dioxide and happens in numerous common polymorphic shapes. Brookite is very rare as compared to the other forms of titanium dioxide i.e. rutile and anatase. It has bigger cell volume than either anatase or rutile. It has 8 TiO_2 bunches per unit cell compared with 4 for anatase and 2 for rutile. Due to its frame, it shows photocatalytic action (Cullity & Smoluchowski, 1957).

Applications of Titanium Dioxide

- It has wide range of applications in many fields which are as follow
- Paints and Coatings
- Plastics, Adhesives and Rubber
- cosmetics
- Food
- Food Contact Materials and Ingredients

Introduction to La_2O_3

La_2O_3 , Y_2O_3 , Lu_2O_3 , and other rare-earth (RE) sesquioxides are well known as excellent optical materials for active lanthanide ions. They are crystal clear and more gleaming to visible and infrared light, and luminescence of lanthanides can be effectively harmonized by optical pumping (Cullity & Smoluchowski, 1957). With the rapid development of nano science and nanotechnology, research on nano scale luminescent materials composite with REE is also developing.

Nanostructured REE sesquioxides composite with active lanthanide ions enable the development of nanophosphors for various applications, including solid-state lasers, fluorescent lamps, planar displays, fiber-optic communication systems, and other photonic devices. However, the size of these materials affects some of their physical characteristics, including concentration quenching, luminescence quantum efficiency, and emission lifetime. Lanthanum oxide (La_2O_3) with a bandwidth of 4.3 eV is the sesquioxides RE with the largest bandwidth among semiconductors. La_2O_3 forms crystals with the hexagonal system structure of the $P3m1$ space group.

These compounds and materials has large verity of uses and applications they acts a catalyst and a part of them they also found applications in ceramics and used to unhealed the structure of materials and their strength, they played a guide in optical waveguide they are also used as filters and are maximum of the parts of the auto mobiles and their parts and they are used as a exhaust gas convertors. The trivalent europium (Eu^{3+}) is activated and used in the activated form as phosphors and has been used in a very large scale in the gas exhaust convectors. Different techniques has been discussed and previously it has been discussed in detailed that number of material are researched and take in to interest they have contains La_2O_3

using different methods to amalgamate the more accurate methods such as the method of ciliation the method of combustion amalgamate and other hydrothermal methods

The La_2O_3 Structure

The notion of the valence as it was developed and taught in the 20th century led to assign to the oxygen in ionic compounds a charge of two electrons that is O^{2-} . The study of the structure type of oxide La_2O_3 , (figure 1.9), studied initially in “Crystal Structures and Bonds” 1983, led to a different approach. Indeed it is characterized by close packing hcp of the lanthanum atoms. In this packing one oxygen atom occupies the octahedral site and two others the tetrahedral site. The oxygen atom of the octahedral site is easily replaced by S, Se, Te, SO_4 etc., the tetrahedral site is stable and strongly covalent characterized by absorption spectroscopy of rare earths by a strong shift towards red in La_2O_3 and $\text{La}_2\text{O}_2\text{S}$. Also the importance of the group (LnO) or (Ln $_2\text{O}_2$), where Ln- O distance is stable nearby 2,30Å, has been underlined by Caro indicating a strong La—O link. The structure La_2O_3 is also the thermodynamically stable structure for sesquioxides of Ce, Pr and Nd. In this structure the oxygen occupies two very different crystallographic sites, which initially led me to the hypothesis that the deep electron of lanthanum did not leave its atom. This assumption leads to two different types of oxygen ions, one O_2^- and the two other O^{1-} , allowing thus a simple interpretation of the structure in question. In this interpretation there is a fundamental point, the space exists only through the objects composing it, as a result if two zones of the studied space exhibit different properties, the objects that occupy these areas exhibit different properties .

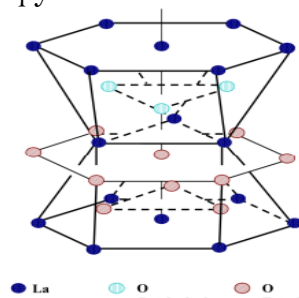


Figure 1.8: Structure of La_2O_3 .

Applications of La_2O_3

La_2O_3 (lanthanum oxide) exhibits a large number of applications. Some of them are given here.

1. **Catalysis:** La_2O_3 is used as a catalyst in several reactions because of its effect of

improving efficacy and selectivity of the catalysts. For instance in refining high-octane gasoline, the material known as La_2O_3 is employed as catalysts in an attempt to transform low-octane naphtha to high-octane gasoline. In the preparation of hydrogen peroxide La_2O_3 is applied to enhance the rate of reaction because it is a catalyst (Bunaciu, Udriş Tioiu, & Aboul-Enein, 2015).

2. Optics: It finds applications in production of optical glasses and lenses because of high refractive indices and low dispersion of La_2O_3 . Incorporation of La_2O_3 into the optical glasses has enhanced features such as high transmission and low absorption besides having a high index of refraction. La_2O_3 is also employed in the fabrication of infrared absorbing glasses for thermal imaging uses as well.

3. Electronics: La_2O_3 has numerous applications in electricity mainly as a dielectric material in capacitors and other digital commodities inclusive of memory devices and transistors. Dielectric constant of La_2O_3 is high; thus, this material is often used in capacitance applications such as energy storage. It is also used as gate dielectric in MOSFETs because of high dielectric strength coupled with low leakage current.

4. Fuel cells: The reasons for the use of La_2O_3 in electrolyte material of solid oxide fuel cells are because of high ionic conductance. In fuel cells La_2O_3 play an important role of the transport of ions in between the anode and the cathode and is important in generating electrical power.

5. Ceramics: La_2O_3 is employed as a dopant to ceramics in order to modify their mechanical properties, high-temperature stabilities, and electrical characteristics. For instance, La_2O_3 -composite ZrO_2 ceramics characterized by enhanced mechanical properties is utilized for fabrication of cutting tools, bearings, and fuel cells.

6. Coatings: La_2O_3 is used as a coated material to enhance metals and its alloy formation towards corrosion. La_2O_3 coatings act as a shielding layer between the metal surface and surrounding atmosphere thus protecting the metal from corrosion and prolonging the metal 'durability'.

7. Medical applications: La_2O_3 can be used in medical fields like orthopedic applications like bone implants because of its good bio-compatibility and that it can improve on the mechanical properties of any implant that is to be used in the bones. La_2O_3 can enhance the mechanical properties of the bone implant such as increase in the strength and toughness of the implant hence minimizing implant failure. La_2O_3

is also used as a coating material in dental applications such as, implants and prosthetics.

Problem of the Statement

Despite the growing interest in $\text{La}_2\text{O}_3-\text{TiO}_2$ composites for advanced functional materials, their phase evolution during high-temperature synthesis remains insufficiently understood. In situ X-ray diffraction (XRD) offers a powerful tool to monitor real-time phase transformations. However, limited data exist on the thermal behavior and interaction mechanisms within this composite system. This study aims to bridge that gap by providing a detailed in situ phase analysis of $\text{La}_2\text{O}_3-\text{TiO}_2$ composites using XRD.

Nanoparticles Synthesis by Plant Extracts

Nanoparticles synthesis employing plant extracts is an emerging field currently experiencing remarkable growth in the field of nanotechnology, where plant abstracts are employed in reducing and capping of nanoparticles. As a green synthesis method, this approach has the benefits of being effective, cost friendly, environmentally friendly and more capable of synthesizing nanoparticles of various sizes and shapes as compared to chemical methods.

Green synthesis of nanoparticles using plant extracts is relatively a new approach that can be considered superior to many chemical methods. It is environment friendly technique being a part of reducing and stabilizing reagents containing in plant phytochemicals. Such phytochemicals are flavonoid s, terpenoids, alkaloids, polyphenols that have various biological activities and curing potentialities.

There are certain steps that are involved like use of metal salt solution, plant extract and then use a reducing agent. The presence of plant extract lead to reduction of metal ions which forms nanoparticles. In the plant extracts, there are reducing agents that partake in the process of reduction of metal ions into metal nanoparticles. The capping agents in the plant extracts as the stabilizers for the nanoparticles to prohibit them from the aggregation. Plant extract concentration, pH and temperature and reaction time affect the size, shape, and properties of the formed nanoparticles.

There are many papers available that discusses about the preparation of

nanoparticle using plant extracts and there are many plant extracts which are used for the preparation of different types of nanoparticles. Some of the nanoparticles that have been synthesized through plant extracts include; silver, gold, platinum, iron oxide and zinc oxide. The synthesized nanoparticles have been characterized using some techniques including UV-Vis spectroscopy, Transmission Electron Microscopy (TEM), Dynamic Light Scattering (DLS) and Fourier Transform Infrared Spectroscopy (FTIR) (Rikta, 2019).

Another benefit of using plant extract in synthesizing nanoparticles is that no toxic materials are involved, and the environmental harm the process causes is minimal making them beneficial and good. Plant extracted may be used in reduction process and the process is cheaper than using the normal chemicals in the market. Furthermore, these plant extracts contain phytochemicals which have other therapeutic advantages and as such will be useful in biomedical areas including drug delivery and cancer therapy.

However, some difficulties are also found related to the synthesis of nanoparticles using plant extracts. Among the issues, there is the problem of batch-to-batch variability of the phytochemical composition, making the size and properties of nanoparticles vary as well. Another difficulty is the lack of standardized protocols for extraction of different plant extracts so as to achieve reproducible means.

Aim and Objectives

- To synthesize the La_2O_3 and TiO_2 composite by green method
- Characterization of La_2O_3 and TiO_2 composite

Literature Review

Composite materials have received considerable interest in research with a focus on structural and functional characteristics. Among them, $\text{La}_2\text{Zr}_2\text{O}_7$ (LZO) with TiO_2 composite shows significant features (Mezouar et al., 2024). XRD is primarily used for the identification of the phase, composition of materials and the appearance of new phases. In situ study, making it a valuable tool for studying changes in phase composition during thermal treatments or under mechanical load (Wang et al., 2020). The synthesis of LZO- TiO_2 composites for various industrial applications has been driven great attention due to temperature stability and mechanical

properties (Cazan et al., 2021).

In particular, the integration of TiO_2 in LZO composites improves stability at high temperatures and restricts undesirable phase changes, making them suitable for high-temperature applications like thermal barrier coatings (Zhao et al., 2024). X-ray diffraction imaging (XRI) investigations are beneficial in studying phase transformations in composite materials, an essential factor in material science. Advanced techniques such as in situ synchrotron X-ray diffraction provide insights into phase formation, crystallinity, and thermal expansion of LZO-based composites, which are essential for their use in high-temperature conditions (Singh et al., 2024).

Microstructural characteristics of LZO- TiO_2 composites are highly influential on their property profile. Depending on whether the morphology of these composites is well-defined or ill-defined, as determined by XRD analysis, their mechanical and thermal characteristics can be significantly altered (Hemath et al., 2020). LZO- TiO_2 composites exhibit enhanced sintering stability, contributing to better structural performance at elevated temperatures, especially in applications like thermal barrier coatings where minimizing thermal conductivity while preventing material destruction (Mezouar et al., 2024).

Phase stability of LZO with TiO_2 is maintained even under conditions where LZO's pyrochlore phase, known for high thermal stability, might otherwise degrade at high temperatures (Singh et al., 2024). Incorporation of TiO_2 may lead to the development of other phases, such as perovskite, which could affect the composite's behavior which can be determined by XRD studies (Zhao et al., 2024). In situ XRI allows real-time monitoring of these reactions, providing researchers with the ability to adjust processing conditions to ensure optimal phase composition (Mezouar et al., 2024).

The synergy between LZO and TiO_2 significantly improves the mechanical properties of the composites. High hardness of TiO_2 and wear resistance balance the thermal characteristics of LZO, enhancing the overall mechanical behavior (Cazan et al., 2021). In situ XRD analysis has shown that the dispersion of TiO_2 can reduce residual stresses, thus improving mechanical properties such as fracture toughness, making these composites suitable for applications that require both heat and wear resistance (Osman et al., 2022).

XRD is a valuable tool for evaluating crystallographic texture, which is influential in determining the material properties of LZO-TiO₂ composites. In situ XRI allows for real-time monitoring of texture changes during material processing, which directly impacts properties like thermal conductivity and mechanical strength (Mezouar et al., 2024). Advanced X-ray imaging technologies have expanded the potential for non-destructive investigation of phase evolution and defect formation under operating conditions, providing critical insights into material behavior and failure modes during thermal or mechanical cycling tests (Wang et al., 2020).

Studies using in situ XRD have provided valuable insights into the phase transformation characteristics of LZO-TiO₂ composites under varying environmental conditions. For instance, LZO samples incorporating oxygen might destabilize the pyrochlore phase, while TiO₂ stabilizes the structure, information that is crucial for practical applications in severe conditions such as aerospace or energy industries (Zhao et al., 2024). The well-defined interface between LZO and TiO₂ phases promotes load transfer, enhancing mechanical properties (Mezouar et al., 2024).

The application of advanced characterization techniques like XRD and XRI has significantly improved the understanding of LZO-TiO₂ composites structural and microstructural properties. These methods provide insights into phase composition, crystalline structure, and overall material performance, aiding in the development of superior processing techniques (Singh et al., 2024). In particular, spark plasma sintering (SPS) is highlighted for its ability to achieve high densification at relatively low temperatures and in short times, with in situ XRD providing real-time data on phase transformations and microstructural changes during sintering (Mezouar et al., 2024). The structural and functional properties of La₂ Zr₂ O₇ (LZO)-TiO₂ composites make them promising candidates for various industrial applications, particularly where high-temperature stability and enhanced mechanical properties are essential (Zhao et al., 2024). The addition of TiO₂ enhances the overall performance of the composite by introducing secondary phases that may contribute beneficially to the structural integrity under severe conditions (Mezouar et al., 2024). The combination of these materials in a composite form leverages the strengths of each, creating a synergistic effect that enhances properties

such as thermal conductivity, phase stability, and mechanical toughness.

Phase composition plays a vital role in defining the performance of LZO-TiO₂ composites. X-ray diffraction (XRD) is an invaluable tool in the characterization of these composites, allowing researchers to identify phase compositions and detect any phase transformations that might occur during processing or in-service conditions (Wang et al., 2020). TiO₂ incorporation may also lead to the formation of unwanted phases such as perovskite, which can alter the thermal and mechanical properties of the composite. XRD studies, particularly those conducted in situ, provide critical data that can be used to adjust processing conditions to favor the formation of desired phases (Osman et al., 2022).

The presence of well-defined crystalline phases, as revealed through XRD analysis, contributes to enhanced mechanical stability at elevated temperatures (Singh et al., 2024). The fine-tuning of microstructures through controlled processing conditions can optimize the thermal and mechanical properties of the composites, making them suitable for use in harsh environments (Hemath et al., 2020).

Residual stresses within LZO-TiO₂ composites are another critical factor affecting their performance. XRD analysis has been used to assess these stresses, providing insights into how the distribution of TiO₂ affects the overall stress state of the material (Cazan et al., 2021). In addition to structural and mechanical characteristics, the crystallographic texture of LZO-TiO₂ composites plays a crucial role in determining their thermal properties. The preferred orientation of crystalline phases can significantly impact the thermal conductivity and mechanical strength of the material (Mezouar et al., 2024). In situ X-ray diffraction imaging (XRI) allows researchers to monitor changes in crystallographic texture during material processing, providing real-time feedback that can be used to adjust processing parameters. This capability is particularly valuable in optimizing the microstructure of LZO-TiO₂ composites to achieve the best possible performance in high-temperature environments (Zhao et al., 2024).

Innovations in X-ray imaging technologies, such as in situ XRI, have broadened the scope of research on composite materials by enabling non-destructive analysis of phase evolution and defect formation (Wang et al., 2020). The ability to

observe these changes in real time provides valuable insights into material behavior and failure modes, allowing for more precise control over the material's properties during processing (Mezouar et al., 2024).

In situ XRD during SPS provides real-time data on phase transformations and microstructural changes, allowing researchers to adjust sintering parameters such as temperature, pressure, and heating rate to optimize the material's density, mechanical strength, and thermal stability (Mezouar et al., 2024).

The ability to tailor the phase composition and microstructure of LZO-TiO₂ composites in real time through in situ XRD and XRI techniques significantly enhances the control over the materials properties. This capability is crucial in applications that demand specific thermal or mechanical characteristics, such as reducing the formation of detrimental phases that could compromise the composite's performance (Zhao et al., 2024). For instance, in situ analysis during sintering processes has shown that fine-tuning the temperature can stabilize specific crystalline structures while preventing the formation of unwanted secondary phases, directly impacting material properties such as electrical conductivity and catalytic activity (Singh et al., 2024).

Advanced characterization methods like XRD and XRI have not only improved the processing techniques for LZO-TiO₂ composites but have also paved the way for new approaches to synthesis. Hybrid sintering methods, where SPS is combined with other techniques, are being explored to further enhance the structural and functional characteristics of these composites (Wang et al., 2020). The integration of in situ XRD and XRI into the development cycle of LZO-TiO₂ composites ensures that each processing step is optimized, resulting in measurable improvements in the final material properties, such as hardness, porosity, and dielectric constant (Osman et al., 2022).

The continued advancement of X-ray diffraction and imaging techniques promises to drive further innovations in the study and application of LZO-TiO₂ composites. By enabling real-time monitoring and precise control of processing conditions, these methods bridge the gap between theoretical modeling and practical application, facilitating the development of high-performance materials tailored to meet the demands of specific industrial sectors (Cazan et al., 2021). As the

understanding of these composites phase transformations, crystallographic textures, and interface structures continues to deepen, new opportunities will emerge for their use in cutting-edge technologies across aerospace, energy, and electronics industries (Mezouar et al., 2024).

The integration of XRD and XRI in the analysis of LZO-TiO₂ composites exemplifies the importance of advanced characterization in material science, enabling real-time monitoring and control of processing conditions that bridge the gap between theoretical modeling and practical application. The continued development of these techniques promises further advancements in the synthesis and application of such composites across various technological sectors (Wang et al., 2020).

Research Methodology

This section discusses the green synthesis and its detailed mechanism for the conglomerates of La₂O₃-TiO₂ nanocomposites. After the synthesis process, the prepared sample was characterized by using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) techniques.

Green Synthesis

Green synthesis process involves extracting biological compounds from leaves, stems, and barks of various plant species. This environmentally friendly method is not only cost-effective but also sustainable. The green synthesis approach leverages a diverse range of plants for the production of nanomaterials. These steps are shown in figure 3.1

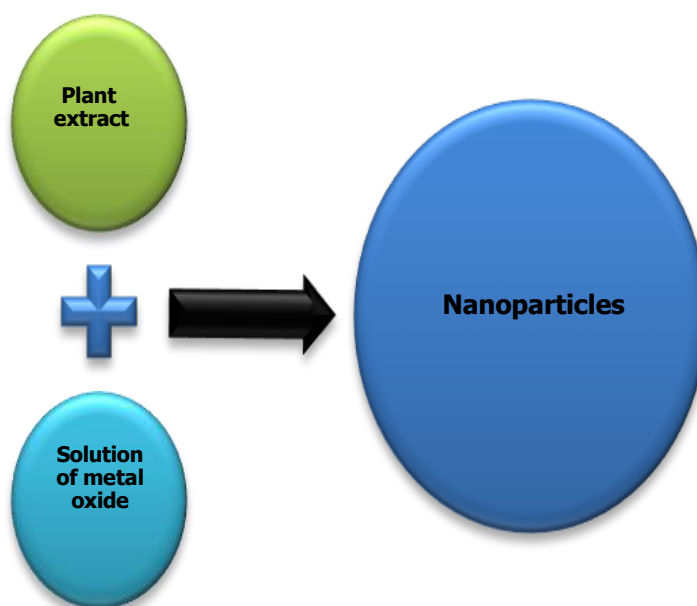


Figure 3.1 Graphical Representation of Green Synthesis

Solvent -Based Green Synthesis

The solvent process is the fundamental constituent in the green synthesis process, The water is always recognized an ideal fluid and very much a good and suitable solvent system for the green synthesis process (Kecheng, Boshui, & Yong, 2013). Water is the most economical and more frequently available solvent on this planet as far as per concern of the advancement in the Nano sciences and the Nano technology. The use of water for the solvent process in the green synthesis methods is extensively used. These techniques as used in the aqueous solution of water in the per chapter literature review we emphasize on the two major steps named as follows:

- Water is used as a solvent in this process
- Extract from leaves of plant is dissolved in water.

Mechanism of Green Synthesis for Metals and their Oxide Nanoparticles

Green synthesis of nanoparticles has gained significant attention due to its eco-friendly and cost-effective approach, utilizing natural resources such as plant extracts, microorganisms, or biopolymers as reducing and stabilizing agents. This method eliminates the need for toxic chemicals typically used in conventional synthesis processes, reducing environmental impact and ensuring biocompatibility. Plant extracts, rich in bioactive compounds like flavonoids, tannins, and phenolics,

are frequently used in the green synthesis.

Plant, leaf abstraction performs a dual role by acting like reducing and stabilizing agent to facilities Nnno particles abstraction. Different plants have changing concentration level of phyto-chemicals. Major moieties that are present in plants are frons, terpenoids, sugar , ketones and aldehyde's, carboxylic acid and amides, which are responsible for reduction process Flavorioids have an increased capacity to reduce metal ions.

Preparation of Dalbergia Sissoo Extract

Dilbergia Sissoo leaves were obtained from Sahiwal, Pakistan. To remove dust and other debris from the leaves, they were rinsed with fresh tap water. Dilbergia Sissoo Plant leaves were dried in dark and then converted into powder form. Afterwards, 10 grams of powder was boiled in 1000 ml distilled water. After an hour, a dark green extract was obtained. The obtained extract was filtered to remove impurities. The filtered extract was placed in refrigerator for further use. The process is shown in figure 3.2.

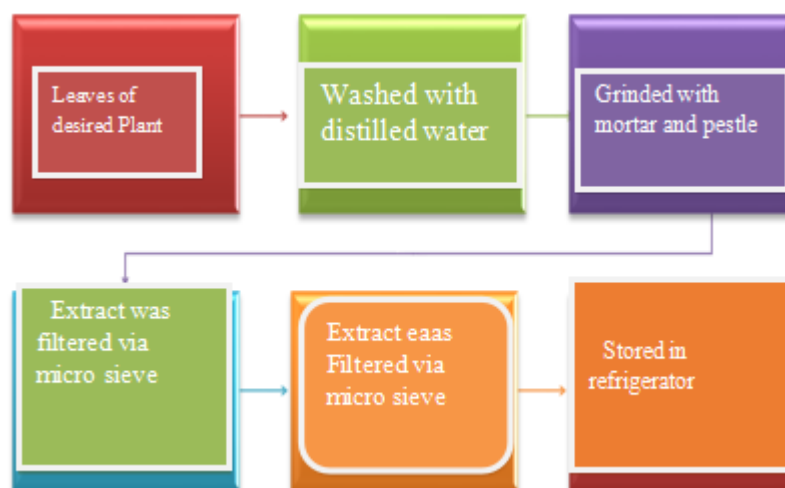


Figure 3. 2- Green Extract of Dalbergia Sissoo Leaves

Size and Morphology of the Synthesized Nano Particle Controlled in Green Synthesis

In green synthesis, the size and morphology of nanoparticles are controlled through various factors such as the concentration of plant extracts, the type of reducing agents, and the pH of the reaction medium. The plant-based compounds, including polyphenols and flavoring, act as both reducing agents and stabilizers, influencing

the growth rate and shape of the nanoparticles.

Concentration of Plant Extract: A higher concentration of plant extract can lead to faster reduction of metal ions, resulting in smaller nanoparticles. A lower concentration tends to produce larger particles.

pH: The pH of the solution plays a crucial role in controlling particle size and shape. A more acidic environment typically favors the formation of smaller nanoparticles, while basic conditions can result in larger or more irregularly shaped particles.

Temperature: Higher temperatures often speed up the reduction process, leading to smaller nanoparticles with different morphological.

Reaction Time: Longer reaction times may result in the growth of larger nanoparticles as the reduction process continues.

please explain short that how can the green synthesis processed further optimized to controlled the size shape and composition of Nano composite

To optimize the green synthesis process for controlling the size, shape, and composition of nanocomposites, several strategies can be applied:

Green Synthesis of TiO₂ Composite

Titanium salt($\text{Ti}(\text{C}_4\text{H}_9\text{O})_4$) is used to prepare TiO_2 . The precursor was dissolved in 200ml of leaf extract. After this the mixture was heated at 60 °C for 6 hours. After this the solution was centrifuged to remove the impurities. After each centrifuge the water at the surface of the solution is removed, and some of the fresh distilled water has been added to repeat the process of centrifuge again to remove more impurities at its surface. This process is repeated 5 times and at the last we get the white precipitates at the bottom of centrifuge tube. The paste of the prepared sample was shifted to China dish and dried at 80 °C in oven. After drying, the prepared sample was further annealed at 400 °C to burn all the organic impurities and improve the crystallinity of prepared sample. The sample was cooled down to room temperature. It was further ground with mortar and pestle and shifted to sample veils for further characterization.

Green Synthesis of La₂O₃-TiO₂ Composite

Lanthanum ($\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) and titanium ($\text{Ti}(\text{C}_4\text{H}_9\text{O})_4$) salts were used as precursors to prepare La_2O_3 - TiO_2 composite. These salts were dissolved in 200ml

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extract in 30:70 wt. % ratios. The prepared solution was hot stirred at 60 °C for 5hrs. After that, the solution was centrifuged to separate the prepared sample from water and impurities.

After each centrifuge, the surface water was discarded, and fresh distilled water was added and centrifuge again. This process was repeated five times. In the last we get thick slurry at the bottom of the centrifuge tube.

The thick paste of the prepared sample was shifted to China dish and dried at 80 °C in oven. After drying, the prepared sample was further annealed at 400 °C to burn all the organic impurities and improve the crystallinity of prepared sample. The sample was cooled down to room temperature, these were further ground in alumina crucible and shifted to sample veils for further characterization. After doing above all the steps the anatase phase of TiO_2 was obtained. The above discussed process is done for the sample at 600°C which gives clear images as discussed in chapter 4. This process is shown in figure 3.3.



Figure 3.3: Green Synthesis of La_2O_3 - TiO_2 Composite

Characterization Techniques

The following characterization was done to study the properties of La_2O_3 - TiO_2 composite.

XRD (X-ray diffraction analysis)

XRD is a very important technique because it gives information about the phases of the nanoparticles and can make differences between crystalline and amorphous

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materials. The information about the phases of nano particles the technique is recognized by XRD which is a very important aspect and can make a difference between different type of solids

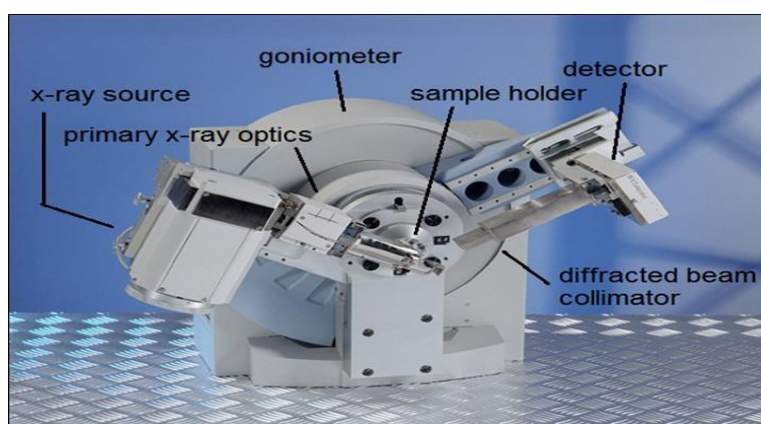


Figure 3.4: X-ray Diffraction Setup

XRD is the most accurate technique for solid structural studies because it is non-destructive and does not require complex methods to prepare samples. XRD is a technique and most accurate for solid and structure because it is nondestructive and does not require or need complex samples for further investigating the spectroscopy pattern acquired by these methods and comparison with standard results.

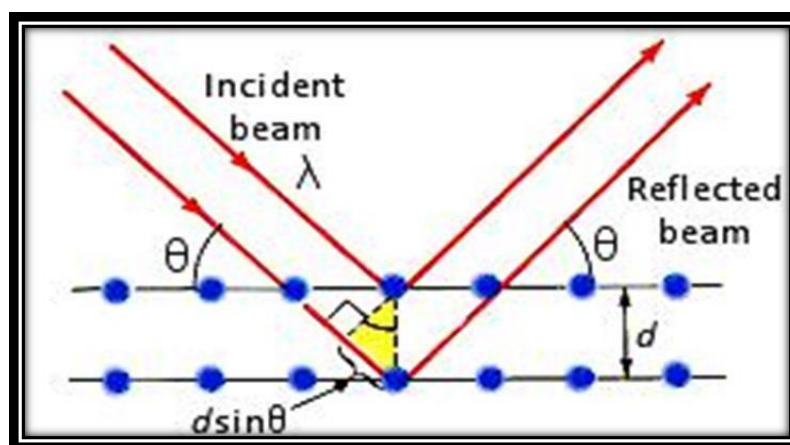


Figure 3.5- Pictorial form of Bragg's Law

The technique of X-ray diffraction (XRD) is a characterization technique that is commonly used in materials science to investigate the crystal structure of a material. The technique involves shining X-rays onto a crystalline material, which causes the X-rays to diffract, or bend, as they interact with the atoms in the crystal lattice. By analyzing the pattern of diffraction, information about the crystal structure, such as

the unit cell dimensions, crystal symmetry, and crystal orientation, can be determined. The X-ray diffraction process relies on the Bragg law, which states that when X-rays of a certain wavelength interact with a crystal, the diffracted X-rays will be in phase if the path difference between them is an integer multiple of the X-ray wavelength. The diffracted X-rays are detected and measured using a detector, such as a photographic plate, an image plate, or a detector that records electronic signals—it's the non-problematic investigating technique, which makes it a valuable tool in the investigation of materials. It is widely used in various areas of Nanomaterial's science, including chemistry, physics, geology, and engineering. The technique is useful for the analysis of crystalline solids, powders, thin films, and single crystals.

Working Principle of XRD

This process involves the shining of X-Rays into the other materials and the pattern of diffraction produced by the interjection with the atoms in the lattice. The difference in the pattern provides some information about the structure of the crystal structure and orientation of crystal. The basic working principle of X-ray diffraction (XRD) presupposes shining a beam of X-rays into a crystalline (Cullity & Smoluchowski, 1957).

Bragg equation shows the relationship between wave length of the X-rays and the angle of incident between the different planes of the crystal structure and crystal lattice (Giacovazzo, 2002):

$$n\lambda = 2d \sin(\theta)$$

where n is an integer, λ is the wavelength of the X-rays, d is the interplanar spacing of the crystal lattice, θ is the angle of incidence of the X-rays, and $\sin(\theta)$ is used to find the angle of incident between the different inter planner angle and spacing. The given fig 3.6 shows a ray diagram of Braggs law

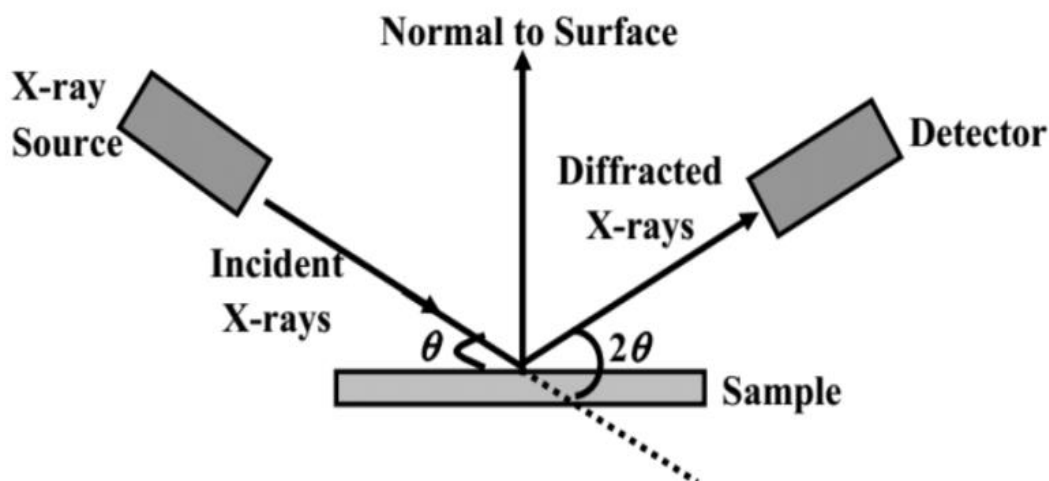


Figure.3.6: Bragg's law

When these intense rays interact with the different atoms they are diffracted in many directions producing a good and many directed pattern by measuring the angles and their intensities the required structured and shape of the Nano particles can be regulate very easily (d'Agostino et al., 2013). And identifying the phases as well as determine the crystallographic orientation and shape texture and size. This information is very much important in Nano materials sciences having the metallurgy ceramic and the solid state Physics (Cullity & Smoluchowski, 1957).

Geometry of X-Ray Diffractometer

An X-ray diffractometer is an instrument used for X-ray diffraction analysis, which involves shining a beam of X-rays onto a crystalline material and measuring the diffraction pattern produced by the interaction of the X-rays with the atoms in the crystal lattice(Kecheng et al., 2013). The geometry of an X-ray diffractometer plays an important role in the accuracy and precision of the XRD analysis.

The basic geometry of an X-ray diffractometer involves the following components:

X-Ray Source

This produces the X-ray beam that is directed towards the sample. The most common X-ray source used in XRD is a copper $K\alpha$ radiation source with a wavelength of $1.54 \text{ \AA}$ (Lavrynenko et al., 2022)

Sample Holder

The sample holder used is in such a way that; the sample is positioned in a fixed position during the XRD measurement. The sample should be arranged in a way that an X-ray beam is allowed to interact with the sample at a predetermined angle of incidence.

Goniometer

The goniometer is used for positioning of the sample holder and in the variation of the angle at which the X-ray beam is directed. If developed to an arm and a half in length, the goniometer includes a graduated circle or a digital display system for measuring the angle of incidence (Karthikeyan, Selvapandiyan, Shanavas, Anbarasan, & Acevedo, 2020).

X-Ray Detector

The X-ray pointer or locator determines the intensity/scale of the X-rays which get diffracted at the angles. The most common detector in XRD is a scintillation detector/counter/indicator or a solid state detector (Maheshwaran et al., 2021).

Incident Beam Optics

This component comprises a collimator and a monochromatic which are used to generate a narrow and monochromatic X-ray beam to be used in the experiment.

Diffracted Beam Optics

The extraction of diffracted X-rays from the sample and the directing of those towards the X-ray detector occur from this component which consists of collimator and receiving slit.

Applications of XRD

X-ray diffraction is an analytical tool whose significance can be well appreciated amongst the numerous analytical tools available in the field of material science. Here are some of the applications of XRD: Here are some of the applications of XRD:

1. Determination of crystal structure: XRD can be employed to ascertain crystal structure of material both organic and inorganic nature. From the diffraction pattern that has been generated by a sample, it can be identified that the crystal structure, and the lattice parameters (Štengl, Bakardjieva, & Murafa, 2009).
2. Phase identification: The use of XRD is the method of determining the various phases of a given sample. The diffraction pattern generated by a specific sample can

inform about crystal structure and lattice parameters that are relevant to phase identification in the given sample (Long, Zhang, Yang, Zhang, & Yu, 2014).

3. Quantitative analysis: Phase analysis can also be determined using XRD and indeed the proportion of a particular phase in the sample can be quantified. Thus, using the integrated intensity of the diffraction peak with a less intense peak from the standard reference, it is possible to evaluate the proportion of different phase in the sample(Dai, Peng, Chen, Liu, & Zan, 2009) .

4. Texture analysis: XRD is a useful technique for determining the crystallographic texture of materials which has significance in showing mechanical and physical properties of materials (Popov et al., 2015).

5. Amorphous phase analysis: XRD can be employed to check the existence of amorphous phase in given sample that is crucial in characterizing the structure and application of materials.

6.Stress/strain analysis: More specifically, XRD can be utilized for determination of the lattice strain and stress and this is useful in the study of mechanical properties of various materials.

7.Thin film analysis: XRD is used to determine the thin film crystals structures and its textures which are essential in determining the properties of thin film materials.

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is one of the high-resolution techniques used in microscopy with the primary aim of providing information on the surface morphology and chemistry of samples. It operates by aiming an electron beam at a sample and generating amazingly precise pictures which can help in examination of the sample's characteristics such as microstructure, texture or composition. Investigations have conducted applying SEM in various fields including in the field of materials science.

Its inherent high resolution and imaging makes it ideal for characterization of a large variety of materials such as metals, ceramics, polymers and even biological samples. Electron Microscopy is competent and capable method to investigate the image of the sample and boundary of it. Fig 3. 5 is a scanning electron microscope previously we had defined an optical microscope, a transmission electron microscope and a scanning tunneling microscope.

Working Principle of SEM

SEM function in a way that a beam of high-energy electrons which are focused on a small area of the sample surface. The beam is scanned in a raster form on the surface and simultaneously interacting with the sample, it produces different signals that can be sensed by detectors. These signals are then used to build an image of the sample surface or to receive information about it's composition. The technique entails creation of an electron beam, focusing the developed beam to the surface of the sample, moving the beam across the surface of the sample, detecting signals resulting from the interaction of the electron beam and the sample and using the signals to build an image or gain information on the composition of sample.

Components of SEM

The Scanning Electron Microscope (SEM) is an instrumental method which can be used to investigate the structure and surface topography of solids (Hu, Kienle, Guo, & Maier, 2006).

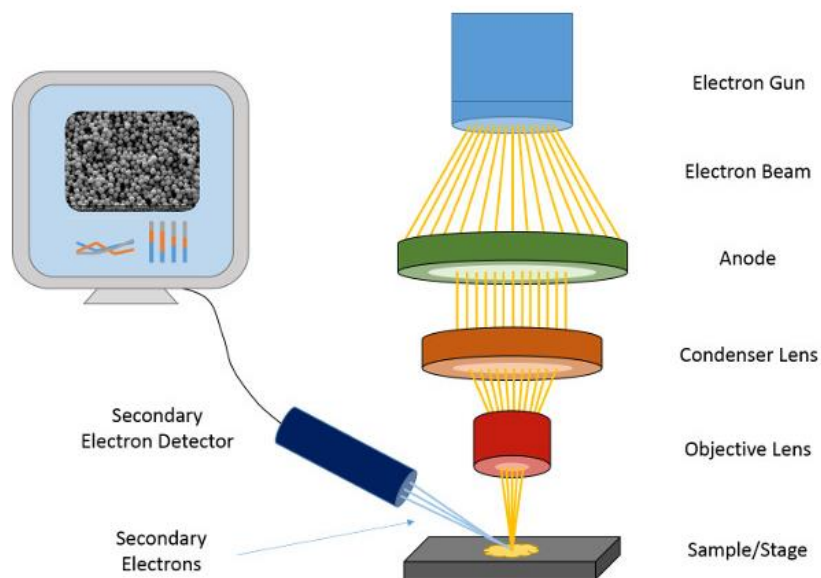


Figure 3.7 Structure and working of scanning electron microscopy

There are several major parts of the SEM model, which are explained below.

1. Electron Source

The electron source generates a beam of electrons which is focussed into a high energy accelerator and then directed to interact with the sample. Several type of

electron sources are available such as thermionic filament , field emission guns and cold field emission guns.

2. Electron Lenses

The electron lenses otherwise known as projectors help in the focusing and deflection of the electrons towards the sample. Lenses are basically made up of a series of electromagnetic coils which generate the required magnetic field to direct the electron beam(Zhang et al., 2013) .

3. Sample Stage

The stage of the holder has a sample holding capacity to position it in such a way that it can be shifted and inclined in various angles. Typically the stage is on wheels and can be programmed through a computer.

4. Detectors

The detectors record the signals which are produced by the dispersed electron beam with the sample. In SEM, there are several detectors that can be used and they include the secondary electron detector, backscattered electron detector and energy dispersion X-ray spectrometer (EDX).

5. Computer and Software

The SEM is controlled by the computer and the software, and the latter enables the user to gather and analyses data. SEM software also enables the user to control the imaging parameters such as magnification and contrast together with analysis and processing of images(B. M. Reddy, Chowdhury, & Smirniotis, 2001a).

The operation of the SEM is analyzed on the basis of the interaction between the electron beam and the sample's surface. When the electron beam strikes the sample, it can generate several types of signals, including: When the electron beam strikes the sample, it can generate several types of signals, including:

1. Secondary Electrons

These are secondary electrons which are produced from the surface of the sample when exposed to the primary electron beam.

2. Backscattered Electrons:

These are high energy electrons that are diffused backwards toward the detector after they collide with the nuclei of the atoms of the material under analysis.

3. X-Rays:

These are emitted when the small electron probe interacts with the sample atoms which in turn emit their characteristic X-rays.

Applications of SEM

Scanning Electron Microscopy (SEM) has a wide range of applications in various fields, including: Scanning Electron Microscopy (SEM) has a wide range of applications in various fields, including:

1. Material Science: Application of SEM includes examination of microstructure and elemental characterization of material including metals, ceramics, polymers and composite materials. It can also be used in the examination of characteristics of material which include hardness, fractural strength, and fatigue limit.
2. Biology and Biomedical Research: The use of SEM is in examining samples of biological nature for example the cells, tissues, and organs. It can give use to look at the structure and mode of operation of biological sample and get surface as well as internal details of such sample.
3. Forensic Science: By identifying the use of SEM in forensic analysis the following applications can be cited; Analysis of the surface of materials such as fibers, hair and gunshot residues. It can also be used for example to enhance the fingerprint and any other trace evidence found in the crime scene.
4. Environmental Science: SEM is used to study the morphology and composition of environmental samples, such as soils, sediments, and particulate matter. It can be used to identify and quantify the presence of pollutants, such as heavy metals and nanoparticles.
5. Semiconductor Industry: SEM is used to analyze the microstructure and defects of semiconductor materials, such as silicon wafers. It can also be used to study the performance of semiconductor devices, such as transistors and solar cells.

Results and Discussion

XRD Structural Studies

The pure TiO_2 sample XRD pattern is shown in the figure 4.1. The diffraction peaks were seen at $2\theta = 25.24, 37.92, 47.93, 55.52, 62.59, 69.04$ and 70.15 which corresponds to (101), (004), (200) (211), (204), (116) and (202) planes respectively for pure TiO_2 . All the peaks are indexed to be tetragonal phase of TiO_2 anatase

structure. (JCPDS card No. 21-1272).

Scorcher formula was used to estimate the crystallite size D of the nano composite. Smaller crystallite sizes and higher lattice strain are often associated with poor crystallinity of material.

$$D = K\lambda / \beta \cos\theta$$

where $\lambda = 1.54\text{\AA}$ wave length of X ray, θ = Bragg's angle, β = FWHM i.e. Full width at half maximum and proportionality constant $K = 0.94$

The crystalline size calculated at (101) plane which corresponds to highest peak at $2\theta = 25.24$ is 28.17 nm.

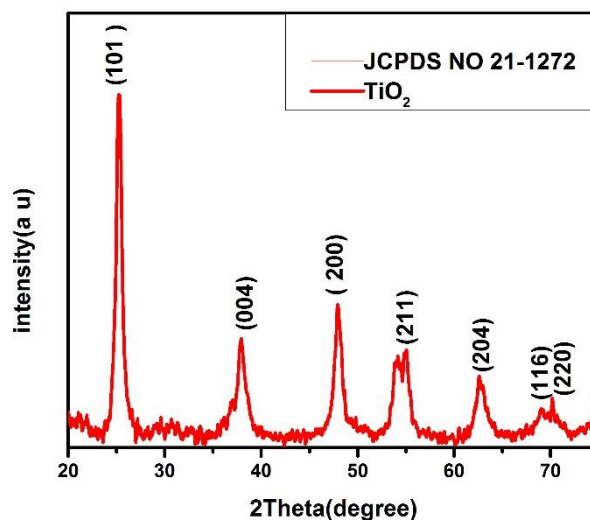


Figure 4.1 XRD Pattern of TiO₂ nanoparticles

In the given figure 4.2 shows the XRD graph of the La₂O₃-TiO₂ nano composite that is annealed at 400°C XRD scan shows few peaks, which are related to prepared nano structure of according to JCPDS-73-1764 for TiO₂ and JCODS-040856 for La₂O₃ respectively.

Due to green synthesis method, some organic impurities were present along the desired nano structures, therefore, samples were further proceeded for heat treatment and the results are discussed in the text given below

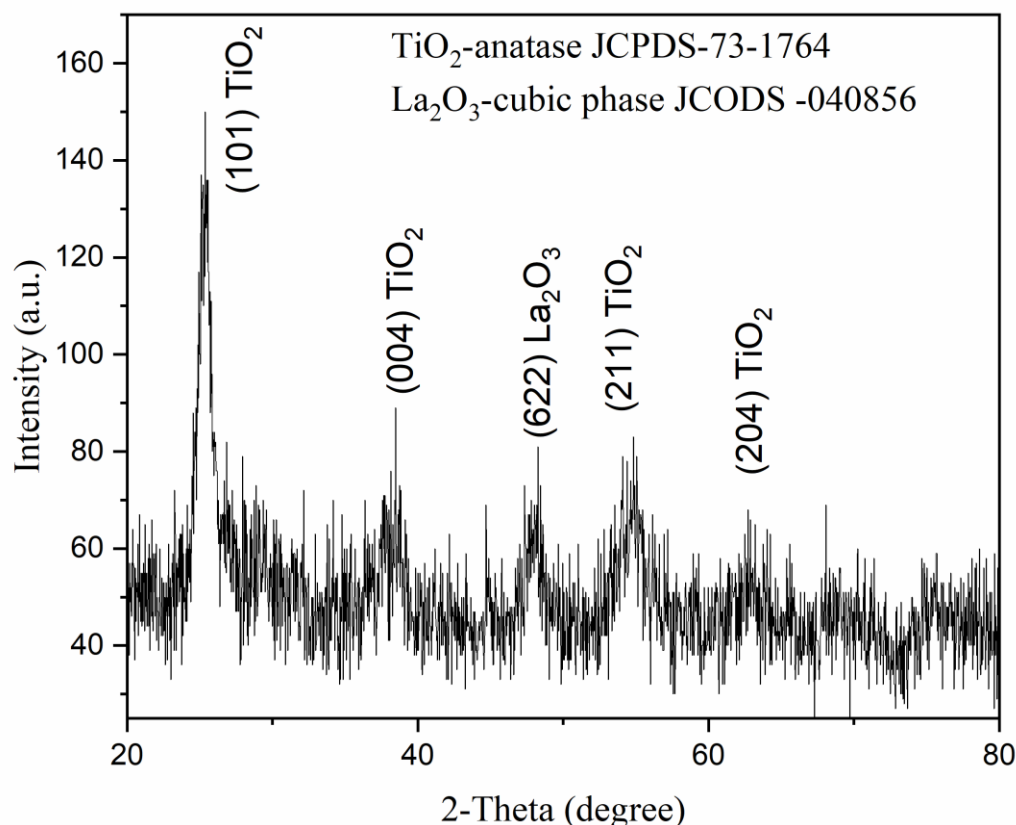


Fig 3.2 XRD Patterns of $\text{La}_2\text{O}_3:\text{TiO}_2$ Nano composite annealed at 400°C

The XRD pattern of $\text{La}_2\text{O}_3:\text{TiO}_2$ nano composite after heat treatment at 600°C is shown in figure 4.3. The diffractogram of $\text{La}_2\text{O}_3:\text{TiO}_2$ nano composite exhibited peaks corresponding to crystallographic planes (101), (004), (200), (105), (211), and (204) at 2θ : 25.23° , 37.71° , 47.72° , 54.16° , 55.32° , and 62.54° respectively. These peaks are indicative of the presence of the anatase phase of TiO_2 . In the same diffractogram, peaks corresponding to crystallographic planes (004), and (622) at 2θ : 39° and 48° respectively are the indicative of the presence of the cubic-phase of La_2O_3 . These results are in good agreement with reported literature (Zhang et al., 2013).

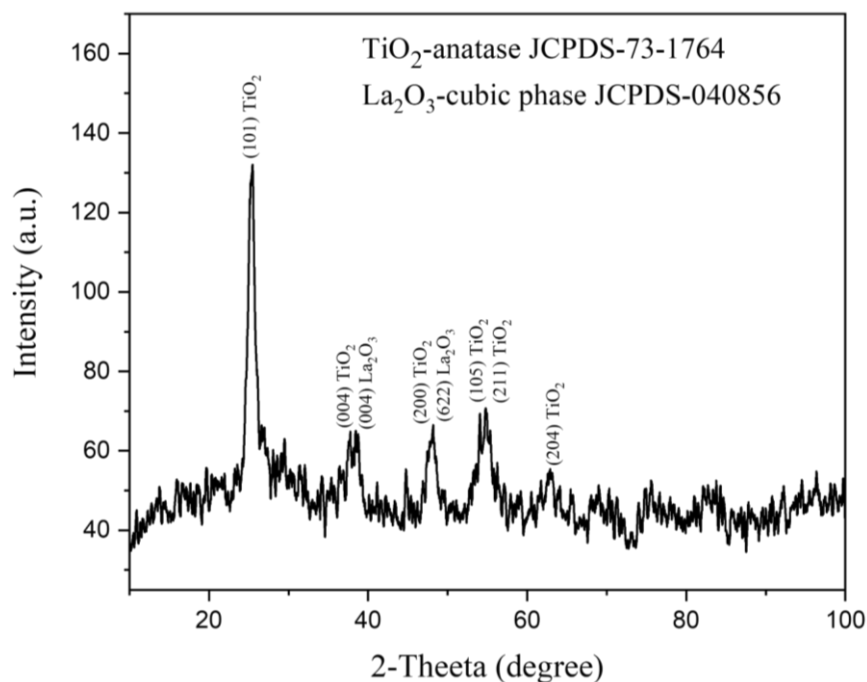


Fig 4.3 XRD Patterns of $\text{La}_2\text{O}_3:\text{TiO}_2$ Nano composite annealed at 600°C

In a nano composite, the peaks corresponding to individual components may broaden compared to pure materials. This broadening can be due to the presence of smaller crystallites or the disruption of the crystal lattice caused by the interaction between the two materials. The degree of peak broadening can provide insights into the degree of mixing or the size of the nanoparticles of individual components.

Particle size of $\text{TiO}_2\text{-La}_2\text{O}_3$ composite is calculated in the range of 10-12 nm.

Table 4. 1: Structural parameters calculated for $\text{TiO}_2\text{-La}_2\text{O}_3$ Nano composite

2theta	FWHM hkl	$\beta = \text{FWHM}$ (Radians)	Particle Size	
			(nm) at 400°C	(nm) at 600°C
25.23	0.68	101	0.011862222	11.6 $\pm$ 3
				23.4 $\pm$ 3

Morphological Study

The scanning electron microscopy images of the prepared samples are depicted in figure 4.4. Nanostructures of the $\text{La}_2\text{O}_3:\text{TiO}_2$ nanocomposite heat treatment of 600°C In micrograph, two types of grains are found. Big sponge like grains on which oval shaped small grains are attached, confirming that nano composite of two different materials have been formed.

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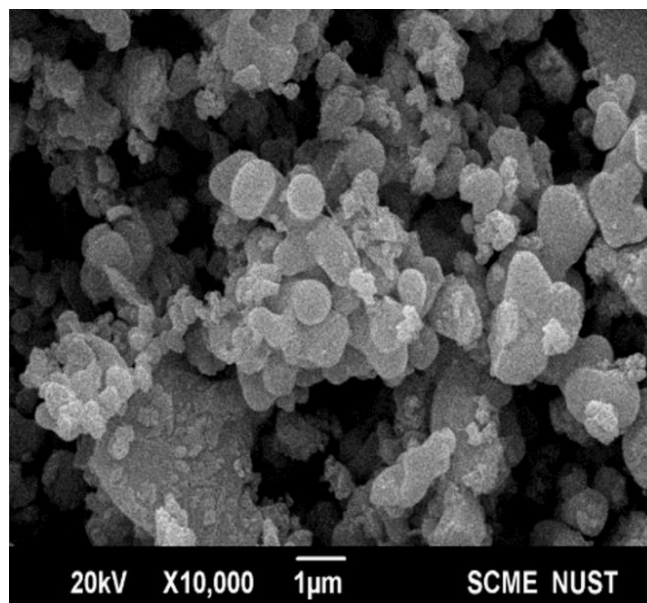


Figure 4.4 SEM images of La₂O₃:TiO₂ nanocomposite

FTIR Analysis

FTIR is a powerful analytical technique that provides information about the chemical bonds and functional groups present in a sample. In Figure 4.5, the FT-IR spectrum of La₂O₃:TiO₂ nanocomposite after heat treatment of 600°C is shown. The peaks formed at 1060 cm⁻¹ shows stretching of C-O-C bond. The peak formed at 1634 cm⁻¹ shows bending vibration of H-O-H bond. Peak at 2335cm⁻¹ is caused by absorption of atmospheric CO₂. The peaks for inorganic materials usually appeared before 800 cm⁻¹ and are not very visible. Therefore, peak of La-O or Ti-O bond is not observable in this spectrum. But any impurity peak has not been found in this spectrum.

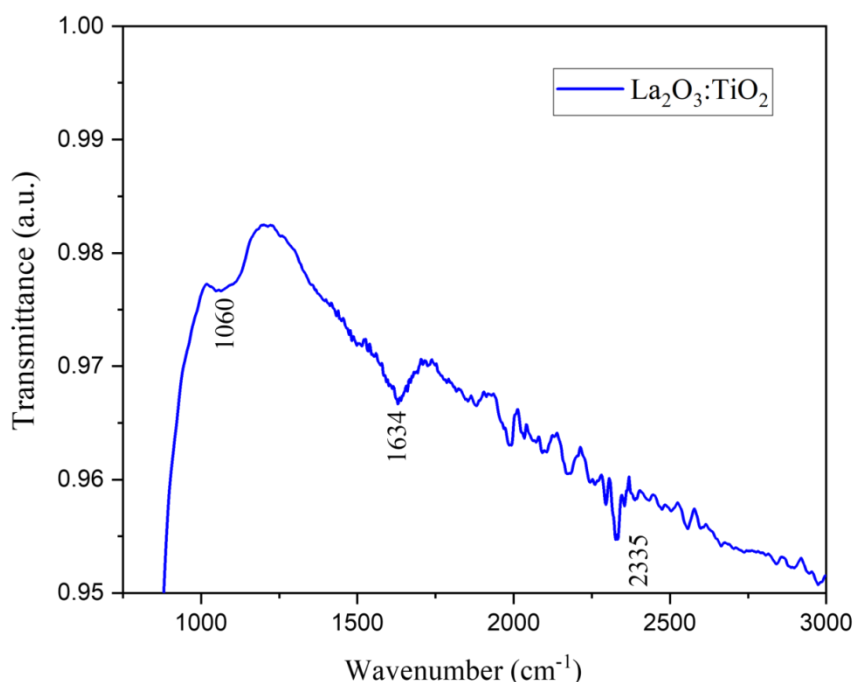


Fig 4.5-FTIR of $\text{La}_2\text{O}_3:\text{TiO}_2$ Nano composite

Conclusion & Future Prospects

Conclusion

The successful synthesis of TiO_2 and $\text{La}_2\text{O}_3:\text{TiO}_2$ nano composite is achieved through the green synthesis method. The average crystalline size of the particles of TiO_2 and $\text{La}_2\text{O}_3:\text{TiO}_2$ nano composite, as determined by X-ray Diffraction (XRD), was 28.17nm and 11.69 nm, respectively. XRD studies revealed the presence of the anatase phase of TiO_2 and cubic phase of La_2O_3 in the composite.

Scanning Electron Microscopy (SEM) is used to examine the morphology of the nano composite. Oval shaped grains of $\sim 1\text{-}2\ \mu\text{m}$ are observed in the composite.

Fourier-Transform Infrared Spectroscopy (FTIR) studies showed no impurity peak in the sample.

In conclusion, the use of *Dalbergia sissoo* extract provides a sustainable and efficient platform for synthesizing multifunctional metal oxide nanocomposites. The findings support broader adoption of green chemistry principles in nanotechnology and encourage further research into plant-mediated synthesis techniques.

Future Prospects

The green synthesis of metal and their other oxides nano particles has been attracted researchers from last few decades. Many types of natural bio components have been employed like plants, bacteria, fungi, yeast, etc. They all act as an efficient reducing agents for fabrication of nanomaterials. Among them the plants extract have very high efficiency as reducing agent. Nevertheless green nano particles synthesis based on bio-components are likely to be applied in environmental remediation and other areas likely pharmaceutical, food and cosmetic industry. Biosynthesis of metal oxide nano particles using marine algae and marine plants will be explored.

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