GREEN SYNTHESIS OF TiO_2 NANOPARTICLES USING CHROMOLAENA ODORATA

Dissertation submitted to

MAHATMA GANDHI UNIVERSITY, KOTTAYAM

In partial fulfillment of the requirements for the award of the

Bachelor of Science in

CHEMISTRY

BY

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(Affiliated to Mahatma Gandhi University, Kottayam)

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CERTIFICATE

This is to certify that the project report entitled **"GREEN SYNTHESIS OF TiO_2 NANOPARTICLES USING *CHROMOLAENA ODORATA*"** is an authentic record of the project work carried out by Ms. **LABEEBA T N** (Reg.no:200021026022) in partial fulfillment of the award of the degree of Bachelor of Science in Chemistry at Bharata Mata College, Thrikkakara affiliated to Mahatma Gandhi University, Kottayam under my guidance and supervision.

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DECLARATION

I, LABEEBA T N, hereby declare that the project report entitled "**GREEN SYNTHESIS OF TiO_2 NANOPARTICLES USING CHROMOLAENA ODORATA**" is an authentic record of the work submitted to mahatma Gandhi university in partial fulfillment of the requirement for the award of the degree of B.Sc. in Chemistry carried out by me under the guidance of Dr ANU K JOHN, Assistant Professor Department of Chemistry, Bharata Mata College Thrikkakara.

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GREEN SYNTHESIS OF TiO_2 NANOPARTICLES USING CHROMOLAENA ODORATA

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CHAPTER 1

INTRODUCTION

Nanotechnology

Nanotechnology alludes to research and technological development of particles within the length scale 1-100 nanometer (A nanometer is one billionth of a meter) at atomic, molecular, macromolecular range. Objects within the range are called nanomaterials, which shows a wide range of properties which differ from the bulk of the material and also possess selfassembly behaviours. For the use in Information Technology, Bioengineering and Environmental application, materials with outstanding electrical, optical, mechanical and magnetic properties are developed. Nanotechnology refers to the synthesis and characterization of materials implemented at nanoscale. It is used effectively in the field of medicine for the early detection, diagnosis and treatment of various diseases and also in the development of target specific drugs.

It was first put forward by the physicist Richard Feymann in his lecture at the American Physical Society entitled “There’s Plenty of Room at the Bottom: An Invitation to Enter a New Field of Physics”, which was mainly concerned with the idea of manufacturing things on a very small scale. It was uncovered that the selection of nanotechnology not only improved the usefulness of conventional materials but also decreased carbon emissions and reduced energy consumption.

Nanotechnology has various applications in the fields of purification water systems, energy systems, physical enhancement and better food producing methods.

Nanomaterials

Materials whose size is confined to about 100 nanometers are called nanomaterials. The chemical and physical properties of nanomaterials change at the nanoscale; their properties will be different from those of bulk materials of the same composition since the number of atoms or ions becomes a significant fraction of the total number of atoms.

Crystalline nanomaterials generally have reduced lattice structures and a low melting point as compared to bulk materials. Thermal stability is also associated with surface energy. They show unique physical and chemical features, such as stability, shape, chemical composition, etc.

Based on the number of dimensions that lie within the nanoscale, nanomaterials are classified into 0 dimensional, 1 dimensional, 2 dimensional, and 3 dimensional nanomaterials. All three dimensions are contained at the nanoscale in zero-dimenional nanomaterials. One dimension is present outside of the nanoscale in one-dimensional nanomaterials. Nanorods, nanowires, and nanotubes are all members of this class. Nanomaterials with two dimensions outside the nanoscale are referred to as two-dimensional nanomaterials. It consists of nanofilms, nanocoatings, nanolayers, graphene, etc. Materials with dimensions outside of the nanoscale are referred to as three-dimensional nanomaterials. Quantum dots, fullerenes, dendrimers, powdered metal, and metal oxide are some of these nanoparticles.

Different types of nanomaterials

Nanoparticles are generally classified into Carbon-based, Organic, and Inorganic nanomaterials.

1.Carbon based nanomaterials

Carbon based nanomaterials are completely made-up of carbon. They are further divided into Fullerenes, Graphemes, Carbon Nano Tubes, Carbon Nano fibers and Carbon black

Fullerene

Fullerene is a C_{60} carbon molecule that forms a spherical structure containing about 28 to 1500 carbon atoms held together by sp^2 hybridization. It shows different photochemical, electrochemical, and physical properties that can be utilised in biological systems. In the presence of light, fullerene can produce high quantum yields of singlet oxygen. Fullerenes can transfer electrons from their excited state, so they can be used to cleave DNA.

Graphene

Allotrope of carbon with a thickness of around 1 nanometer. On a two-dimensional planar surface, graphene forms a hexagonal network of carbon atoms. Graphene possesses high optical transparency, electrical conductivity, and a great ability to work with biological systems. In biological cells, there is a chance of graphene-induced toxicity due to the presence of contaminants

Carbon nanotubes

Made up of one or more graphene cylinders or sheets with sp^2-hybridised carbon atoms forming a hexagonal network. It consists of hollow or closed ends with lengths varying from a few micrometres to a few millimetres. It exhibits features of low-dimensional materials. At high temperatures, it provides a high yield with good-quality samples. They have unique physical and chemical properties.

Carbon nanofibers (CNF)

Carbon nanofibers possess limited evidence for cytocompatibility and high conductivity properties, as well as high strength and a high Young's modulus. Concentration of CNF in concrete improves the properties required for strain checking, damage analysis, etc. and also improves the quality. 70% carbon gas to fibre conversion at low temperatures is cost-effective. In CNF, volume resistivity is reduced to 105 cm.

Carbon Black

Carbon black is an amorphous black pigment that is an electrically conducting material. Particle size ranges from 10 to 100 nm. They are spherical in shape, having a diameter of 20 to 70 nanometers. It is mainly used as a reinforcing agent in inks, paints, elastomers, and plastics. It has the ability to alter the mechanical, electrical, and physical properties of the medium in which it is dispersed. Elastomers and plastics can be made conductors by the addition of finely powdered carbon black.

2.Organic nanomaterials

The most common organic nanomaterials or polymers include dendrimers, micelles, liposomes, ferritin, etc. Organic nanoparticles are very sensitive to electromagnetic radiation and are non-toxic, biodegradable particles. Organic nanoparticles are widely used in the biomedical field. They are used in drug delivery systems due to their high stability, drug-carrying capacity, and drug delivery systems.

3.Inorganic nanomaterials

Metal and metal oxide nanoparticles generally fall under the category of inorganic nanosizedparticles. They are not made of carbon.

Metal-based nanoparticles

Metal-based nanoparticles are tiny particles made of metal that can be created in two different ways: either by building them up or by breaking down a larger piece of metal. They are very sensitive to things around them and have a lot of surface area compared to their size. They can be made from many different metals, like copper (Cu), Iron (Fe), Lead (Pb), Gold (Au), Silver (Ag), and zinc (Zn). The size of these particles is usually between 1 and 100 nanometers.

Metal oxide-based nanoparticles

Based on metal oxide nanoparticles, Metal oxide nanoparticles are created in order to alter the characteristics of metal nanoparticles.They are created chemically to boost the effectiveness and reactivity of metal nanoparticles.Aluminium oxide (Al2O3), cerium oxide (CeO2), iron oxide (Fe2O3), titanium dioxide (TiO2), and others are among the metal oxide nanoparticles that are frequently synthesised.

TiO_2 Nanoparticles

TiO_2 is a powder that is non-combustible, white, and odourless. Titania, titanium (IV) oxide, titania, titanic acid anhydride, and Ti white are only a few of the various names for titanium dioxide. Due to their distinctive textural and structural properties, nanostructured materials have attracted a lot of attention in the last ten years for use as catalysts and other applications. There has been a lot of focus on significant metal oxides like TiO_2 and others. Due to the stability of its chemical structure, biocompatibility, and physical, optical, and electrical properties, titanium is a highly well-known and extensively explored substance. Many consumer products contain TiO_2 NPs to improve their whiteness or opacity.

TiO_2 exists in three mineral shapes: anatase, Rutile, and Brookite (Fig.1). Anatase sort TiO_2 contains a crystalline structure that compares to the tetragonal framework (with di pyramidal propensity) and is utilized primarily as a photocatalyst under UV light. Rutile sort TiO_2 too contains a tetragonal precious stone structure (with prismatic propensity). This sort of Titania is basically utilized as white shade in paint. Brookite sort TiO2 has an orthorhombic crystalline structure. TiO_2 is favoured in anatase frame since of its high photocatalytic action, since it includes a more negative conduction band edge potential (higher potential vitality of photogenerated electrons), high particular region, non-toxic, photochemically steady and generally cheap. TiO_2 morphologies have basically included nanostructures such as nanotubes, nanowires, nanorods, and mesoporous structures.

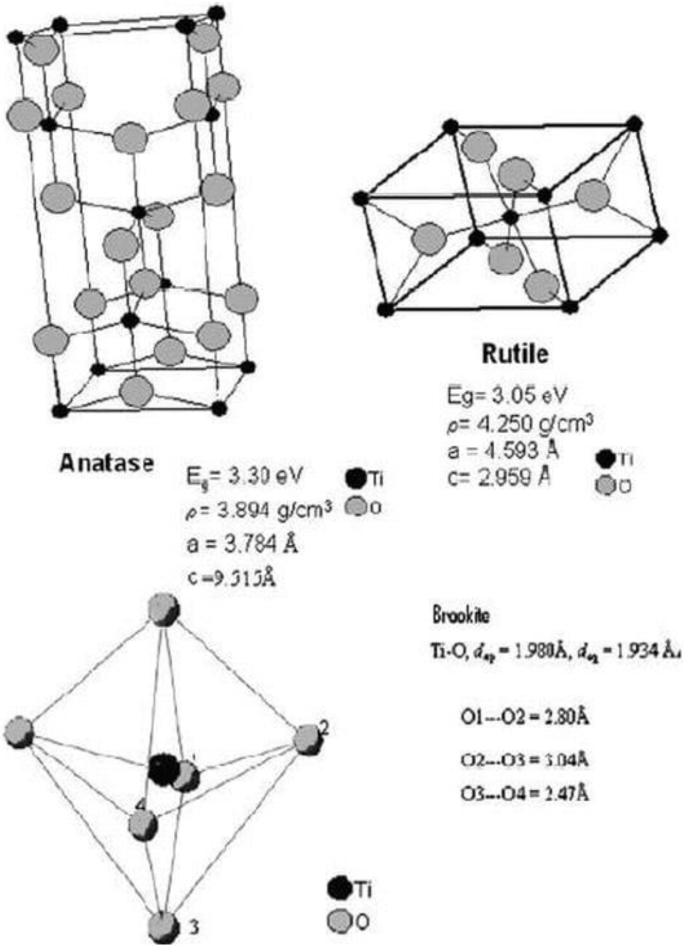

Fig.1 Different forms of TiO₂ [D.P. Macwan, P.N. Dave, J.Mater.Sci.46(2011) 36693686]

Applications of TiO_2 Nanoparticles

TiO2 may be a multifunctional fabric, broadly utilized in photocatalysis for water part and natural toxin expulsion, sun powered cells, gas sensors, biosensors, and so on. TiO2 nanobelts, as one of the foremost critical 1D TiO2 nanostructures, have extraordinary electronic and optical properties that bring almost significant improvement of their execution, and one of a kind auxiliaryfeatures that extend their potential applications.

Photocatalysis: - Fujishima and Honda said something might be possible. In 1972, scientists figured out how to split water using a TiO_2 electrode and sunlight. Scientists have tried really hard to find new ways to use TiO_2 to remove pollution and create clean hydrogen fuel using sunlight. Scientists have found that TiO_2 nanobelts work better than nanospheres in making things happen with light. This is a really important discovery. This study showed how TiO_2 nano belts work better than TiO_2 nanospheres for cleaning pollutants from the air. Our team created a special device that combines TiO_2 nanowires and graphite fibers with a wind-powered generator. It helps to use light to turn harmful substances into harmless ones. In photocatalysis, TiO_2 helps move energy from light to an important part called the charge carrier.

Solar Cells: -TiO_2 is used to make sun powered cells, especially dye sensitized and organometallic halide perovskite solar cells. TiO_2 helps transport electrons in both cases. In some solar cells called DSSCs, they use TiO_2 tiny materials that are made sensitive to light by dye. This is cheaper than

using silicon in other solar cells. When using TiO_2 nanoparticles, the distance that electrons can travel is shortened because the nanoparticles doesn’t touch well and have blemishes on their surfaces.

Gas sensors: - Gas sensors made of metal oxide semiconductor work by measuring how electricity flows through a special porous film when gas molecules stick to it. The semiconductor sensor's resistance changes depending on the type and amount of gas it detects. This is how it can tell different gases apart.

Biosensors: - TiO2 nanobelts are extremely stable and compatible with various biomaterials. They are able to enhance chemical catalytic performance concurrently.These advantages make TiO2 a fantastic contender for many biosensors. Furthermore, excellent electronic characteristics and a high surface to volume ratio provide rapid transit of charge carriers, which guarantees a high affectability. TiO2 is regarded as the ideal material for optical biosensors because of its exceptional fluid stability and expanding index of refraction.

Synthesis of TiO_2 Nanoparticles

Sol Gel Method

The sol-gel method is a way to make different types of ceramics. It is very flexible and can be used for many things. In a usual solution-A gel is made by mixing fluids that react with each other and stick together. This creates a thick substance called a colloidal suspension or sol. Usually, metal salts or metal alkoxides are used as starting materials. These react to make something else. liquid state to the solid state of the polymer material. When the polymer material finishes forming and the liquid is gone, it turns into a solid. Change liquid to solid gel. You can make thin layers on a surface by spinning or dipping it in a liquid. When you put sol in a mould, it becomes a wet gel. Then, the wet gel changes into something else. A very hard ceramic that is dried and heated more. substance is extracted from a gel and allowed to dry in a manner that prevents shrinking. This material is very lightweight and has a lot of tiny holes in it, making it good at insulating things from heat or cold.

We take out the liquid from a wet gel using a special condition called supercritical. We can make tiny and even ceramic powder using different methods like precipitation, spray pyrolysis, or emulsion techniques. The easy way to make small pieces of TiO_2 is to mix chemicals called precursors and mix them with water (hydrolysis). This process is called sol-gel method.

First, some kind of chemical called an acid helps to break apart a certain substance called titanium (IV) alkoxide. This is done through a process called hydrolysis. Then, the resulting pieces (or molecules) of titanium (IV) alkoxide link back together in a new way. This is called condensation. Ti-O-Ti chains are easier to make when there is less energy involved.

Sonochemical Method

In the blending of a variety of nanostructured materials, including high-surface area move metals, combinations, carbides, oxides, and colloids, ultrasound has proven to be extremely helpful. Ultrasound doesn't interact with atomic species in a coordinated manner to produce chemical effects. Sonochemistry, on the other hand, results from acoustic cavitation, which is the arrangement, growth, and implosive collapse of bubbles in a fluid.

In a standard procedure for preparing TiO2 nanoparticles, 0.5 g of TiO2 pellets were dissolved in 30 ml of 10 M NaOH solution and mixed vigorously at room temperature for 2 hours. Then, for two hours in ambient temperature, the yellowish arrangement was lighted by an ultrasonic shower (Control Sonic 405, 40 kHz, and 350 W). At that point, the resulting accelerates were centrifuged, cleaned, and tapped with deionised water a few times and dreid at 60°C for 24 hours.

Electrodeposition Method

Electrodeposition is a way to add a metal coating to a surface. Making something smaller at the cathode. The material that needs to be coated is used as the negative electrode in a process. Put into a mixture that has metal salt to create a coating. The metal pieces break into small pieces. Drawn to negative electrode and changed into metal. We can make TiO_2 nanowires by putting a special layer called Anodic alumina membrane (AAA) on a surface and then adding electricity.

Solvothermal Method

The solvothermal method is almost the same as the hydrothermal method, but it uses a different liquid called a solvent. The thing being used here is not water-based. The temperature can be made much hotter than in the hydrothermal process because you can use different types of strong-smelling liquids that are hard to boil. The solvothermal method is usually better at controlling the size, shape, and crystal quality of TiO_2 than hydrothermal methods. Tiny particles. We found a way to make many types of tiny particles in a consistent and even way using a method called solvothermal. The solvothermal method is a way to make materials by using a liquid (usually a solvent) and heating it up. This can help create materials that have special properties, like being very small or having a specific shape. It's a complicated process that requires special equipment and knowledge. We used a way to make very small TiO_2 particles.

Direct Oxidation Method

At high temperature conditions. Essentially, this means that tiny particles of TiO_2 can be made by heating up titanium and adding certain chemicals. Anodization is the process of adding a protective coating to metal by making it undergo a chemical reaction with an electric current. Tiny rod-shaped crystals made of TiO_2 were made by putting titanium metal in a process called direct oxidation. A plate that has a substance called hydrogen peroxide with a percentage of 28 to 30. Usually, nano rods made of TiO_2 grow on a Ti plate during a process.

Green synthesis of TiO2 nanoparticles from leaf extract

Due to the little usage of chemicals and other agents during biological synthesis, green production of nanoparticles employing various biological metabolites can aid in overcoming chemical and physical approaches. The most expert, often adaptable, physiologically sound, and practical method for creating nanoparticles is known as "green synthesis". Because green nanoparticle synthesis is simple, inexpensive, and generally reproducible, it offers an advantage over existing procedures.

In the Green Synthesis experiment conducted by Waseem Ahmad and co-workers in 2020, they examined different bacterial and fungal inhibiting properties of greenly produced nano sized titanium dioxide (TiO_2) particles employing Mentha arvensis leaf extract as the precursor and reduction agent. Diffraction by x-ray, spectroscopy such as UV-Visible , Fourier transform infrared, and scanning electron microscopy techniques were used to analyse the synthesised nanoparticles. The produced nanoparticles' crystalline structure can be shown in the X-ray diffraction analysis, and the incidence of peaks at 25.27° corresponds to 110 anatase form. The synthesised nanoparticle was analysed to be spherical in shape and range in size from 20 to 70 nm, according to a study with the help of scanning electron microscope. Interesting antibacterial and antifungal activity was displayed by green TiO_2 nanoparticles that were produced.

Similarly in another paper published in 2022 November, they described a novel biogenic source, a capping and reducing agent, and a creative sustainable method for producing titania (TiO_2) from Acorus calamus (A. calamus) leaf extract. Using the techniques UV, FTIR, SEM, DLS, BET, and TGA-DSC analysis, were used to study the optical, structural or morphological and surface and thermal properties of biosynthesized nanoparticles. The XRD pattern demonstrated the phase development and the existence of nanocrystalline

TiO_2. Conjugation and the presence of the Ti-O and O-H bands of vibrations were discovered by FTIR research. The morphological study revealed that the nanoparticles

were globular, which have an average size of 15–40 nm. The effect of size assessment was also examined using DLS. The photocatalytic nature of plain, synthetic P-25, and Rhodamine B (Rh B) dye was dissolved in aqueous solution, and the photocatalytic action of bare, commercial P-25, and nano sized biosynthesized TiO_2 (G-TiO_2) particles was examined under visible light irradiation at various time intervals. By destroying 96.59% of the Rh B dye, the biosynthesized TiO_2 nanoparticles demonstrated potent photocatalytic activity. A variety of kinetic representations were used to examine equilibrium information. With equilibrium rate constant (K1) and regression coefficient (R2) values of 3.72 104 and 0.99, respectively, the pseudo-first-order reaction was best matched. The disc diffusion technique was used to examine the produced nanoparticles' antibacterial effectiveness. Moreover, compared to bare TiO_2, biosynthesized TiO_2 displayed superior antibacterial action against the chosen gram-positive staining pathogenic bacteria (B. subtilis, S. aureus) over gram-negative (P. aeruginosa, E. coli).

Likewise, another study was conducted on green synthesis in 2020 in which an aqueous solution of Syzygium cumini leaf extract was used as a capping agent to create titanium dioxide nanoparticles (TiO_2 NPs). These environmentally friendly TiO_2 NPs were also tested for their ability to remove lead from industrial effluent by photocatalysis. Utilizing different microscopy techniques such as high-resolution scanning electron microscopy (HRSEM) and another high-resolution transmission electron microscopy (HRTEM) and also uses X-ray energy dispersive spectroscopy (EDS), Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD), dynamic light scattering (DLS), and Brunauer-Emmett-Teller analysis, analysed nanoparticles were characterised (BET). The obtained results showed that the synthetic TiO_2 NPs have a large BET surface area of 105 m2/g and a spherical morphology with anatase phase. In a self-designed reactor, photo catalytic research of TiO_2 NPs for the removal of lead from explosive wastewater were carried out. The concentration of lead was assessed using a technique called inductive coupled plasma spectroscopy (ICP). The demand of Chemical oxygen COD) was removed by 75.5% and lead (Pb2+) by 82.53% according to the results. For the first time, this use of green TiO_2 NPs is being investigated.

An article published in 2019 demonstrated the first green and efficient synthesis of TiO_2 NPs utilising leaf extract of Carica papaya, which has a potential use to function as a photocatalyst. For NPs of TiO_2, XRD analysis confirms they are tetragonal in crystal structure, crystalline in nature, and have anatase phase development. With the help of TEM pictures, the diameter of the sphere shaped TiO_2-NPs can be precisely calculated to be less than 20 nm using images of FE-SEM , which further clarify the cage-like morphology. Calculations reveal that the average particle size is 15.6 nm. The fingerprint region of the FTIR spectrum, between 750 cm1 and 650 cm1, exhibits the absorption band of Ti-O stretching mode. The mesoporous nature of TiO_2 particles is revealed by BET measurements, which have surface areas and mean pore sizes of 81.653 m2g-1 and 8.0615 nm, respectively. The UV-visible spectrum has a significant absorption peak at 303 nm, and a calculation was made to determine the band gap energy value, which was discovered to be 3.85 eV. TiO_2 NPs, when used at the recommended dosage of 25 mg, demonstrate remarkable photocatalytic efficiency (91.19%) towards the photodegradation of RO-4 dye during 180 minutes of incubation under UV illumination.

Taking into account a similar study which was held in 2019, the titanium dioxide nanoparticles were created utilising Azadiracta indica leaf extract. While XRD tests demonstrated the crystalline character of titanium dioxide nanoparticles, FTIR analyses revealed the presence of terpenoids, flavonoids, and proteins thought to be responsible for the production and stabilisation of titanium dioxide nanoparticles. The spherical form and size ranged from 15 to 50 nm, according to photographs taken using transmission electron microscopy. According to a SEM analysis, TiO_2 nanoparticles were spherical in form and ranged in size from 25 to 87 nm. Escherichia coli, Bacillus subtilis, Salmonella typhi, and Klebsiella pneumoniae were used as test organisms for the antibacterial activity of produced TiO_2 nanoparticles and Ti mpound. The results of the present study showed that TiO_2 nanoparticles inhibited the growth of all the tested microorganisms. The antibacterial effect is more pronounced in case of TiO_2 nanoparticles as compared with TiO_2 compound. The lowest MIC (minimum inhibitory concentration) value i.e. 10.42 µg/mL of nanoparticles was observed against *Salmonella typhi* and *Escherichia coli* whereas lowest MBC value i.e 83.3 µg/mL was observed against *Klebsiella pneumoniae.*

Objective and scope of the work

Objective of the current work is to synthesize titanium dioxide nano materials by a green method using the aqueous leaf extract of Chromolaena ordata. Hydrothermal method was adopted for the synthesis. The effect of calcination in the synthesised TiO_2 nanomaterials was also studied. The resulting nano materials were characterized using X-ray diffraction studies, FTIR spectroscopy and UV spectroscopy.

Titanium Dioxide nano materials have a variety of applications in the fields of cosmetics, electronics, and energy (including solar cells), food technology, metallurgy and materials, paints, pesticides, security, textiles, etc. The present work provides a simple, economic and eco-friendly way of synthesizing TiO_2 nano particles. And these particles can be used for any range of applications in the future.

CHAPTER -2

MATERIALS AND METHODS

Materials

- Titanium isopropoxide
- Chromolaena odorata leaf
- Distilled water

Experimental methods

Preparation of plant extract

The plant source, Chromolaena odorata, was collected from the premises of Ernakulam. The leaves were washed thoroughly to remove dust and other particles and kept to dry in shade. It was then made into a fine powder using a mixer grinder. For preparing the extract, 40 g of the powdered sample was dissolved in 225 mL of distilled water and heated at 50 °C for one hour. After heating, the extract was filtered with Whatman No.1 filter paper and kept in the refrigerator for further use.

Synthesis of TiO_2 nanoparticles using leaf extract

5 ml of titanium isopropoxide (TIP) was added to 40 ml of the aqueous leaf extract, and the mixture was kept for magnetic stirring for one hour. It was then transferred into a 100 mL autoclave and heated for 24 hours at 180 °C in a hot-air oven. After cooling, the product was dried at 120 °C for 2 hours. The dried sample was then grinded into fine powder using a mortar and pestle. It was then divided into two portions; one portion was kept in a muffle furnace for 2 hours at 300 °C for annealing. The annealed sample was named as TiO_2-2 and the unannealed was named as TiO_2-1.

Characterization techniques

UV- Visible Spectroscopy

The absorption of visible or ultraviolet light by chemical compounds, which results in the production of distinct spectra, is the basic principle of UV-Visible spectroscopy. The interaction of light and matter is the foundation of spectroscopy. A spectrum is produced as a result of the matter's excitation and de-excitation as it absorbs light. The electrons in matter are excited when they absorb ultraviolet radiation. This makes them to jump fromresult of the matter's excitation and de-excitation as it absorbs light. The electrons in matter are excited when they absorb ultraviolet radiation. This makes them to jump from a ground state to an excited state. The energy difference between the ground state and the excited state gives the amount of ultraviolet radiation or visible radiation absorbed by it.

The statement of the Beer-Lambert law can be written as follows: The rate at which the intensity of a beam of monochromatic light diminishes along the thickness of a solution that contains an absorbent of monochromatic light is directly proportional to the concentration of the absorbent in the solution and is also directly proportional to the intensity of the incident monochromatic radiation. Beer-Lambert's law states that the more absorbing molecules there are—molecules that can take in light of a specific wavelength—the more radiation is taken in.

The UV-visible spectrum of the TiO_2 sample was obtained using UV Vis NIR Spectrophotometer: Perkin Elmer Lambda 365 200nm to 1000 nm.

X-ray diffraction studies (XRD)

When a periodic array with a distant configuration scatters light, it causes constructive interference at specific angles, which is known as diffraction. The periodic arrangement of the crystal's atoms results in light diffraction. Powder X-ray diffraction (PXRD) techniques utilise this concept to identify the crystalline nature of materials since X-ray wavelengths are comparable to the distance between atoms. Atoms' X-ray scattering results in a diffraction pattern that reveals details about the crystal's atomic configuration. A sophisticated and portable tool is the Powder XRD. It has a number of

Its applications can be made even more inventive with the help of crucial components like a variable temperature assembly and a humidity chamber that show how temperature and humidity alter the nature of the material. A crystal's structural characteristics determine the way in which an X-ray scatters when it passes over it. In powder X-ray diffraction, the material's powder is used to create the diffraction pattern rather than a single crystal. Powder diffraction is typically simpler and more convenient than single crystal diffraction since it does not require individual crystals. The pinnacle position in a diffraction design depends on the frequency. When the circumstances are right, interactions between the sample and the incident X-ray beam cause intense reflected radiation. X-rays through constructive interference. The general relationship between the distance between the atoms' crystal lattice planes, the beam's incident angle, and the wavelength of the incident X-rays is outlined in this law.

Powder XRD of the sample was obtained by Bruker D8 Advance.

FTIR spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy analyse the vibration of molecules. Discrete vibrational energies are associated with each functional group. Thus, a molecule can be identified from the combination of its functional groups. FTIR has been widely used to measure the IR spectra of most materials. The property which makes FTIR more advantageous than other spectroscopic methods is that all compounds practically show characteristic absorption or emission spectra in the IR region. FTIR is concerned with the change in dipole moments of a molecule by IR light corresponding to a specific vibrational energy. The peaks in FTIR can be used for identification of molecules since the vibrational energy bands associated with each functional group is unique to every molecule.

The FTIR spectrum of the sample was obtained by Thermo scientific Nicolet IS5 FTIR spectrometer.

CHAPTER 3

RESULT AND DISCUSSION

This chapter involves the characterization results of synthesized TiO_2.

CHARACTERIZATION OF TIO_2

X-Ray Diffraction Studies

Electromagnetic waves with very small wavelengths, on the order of a fraction of a metre, are X-rays.Powder X-ray diffraction is a crucial experimental method that has been used to identify unknown phases, flaws, and grains as well as to determine the crystal structure, including lattice constants and geometry. The X-ray diffraction peaks' broadening provides information on grain size. This peak broadening is typically brought on by crystallites' finite size impact. Using the Debye-Scherrer formula, the grain size can be determined from the broadening of the diffracted beam. $t = \frac{K\lambda}{\beta cos\theta}$

Where t is the size of crystallite or grain, K is a constant with no dimension (here taken to be 0.94), is the X-ray wave length (1.5406 for Cu K radiation), is the full width at half maximum of the X-ray peak, and is the Bragg angle, which is calculated from the 2 value corresponding to the peak with the highest intensity in the XRD pattern.

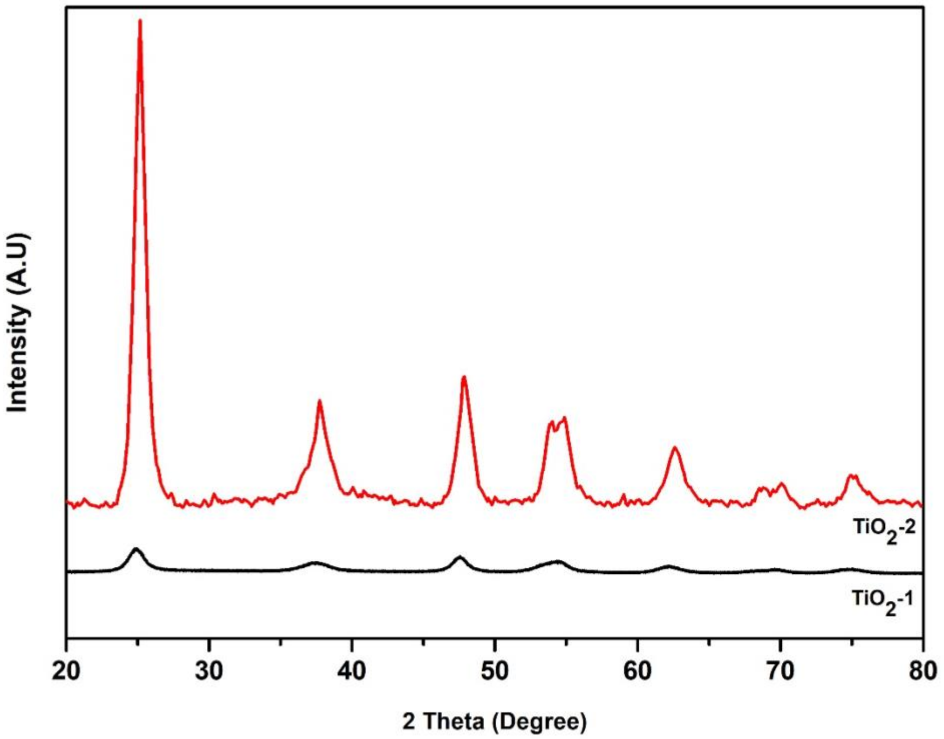

Fig. 3.1 XRD pattern of TiO_2 nano particles

Figure 3.1 shows the XRD patterns of unannealed and annealed TiO_2 samples. The peaks observed in the spectra correspond to the reflection from different planes at various 2θ values. By the comparison of the XRD peaks of synthesized TiO_2 with the standard XRD pattern of TiO_2 for anatase, rutile and brookite phases, it is observed that all the obtained peaks of TiO_2-1 and TiO_2-2 are due to anatase phase and no additional peaks due to rutile or brookite phases. Thus the TiO_2 prepared from the plant leaf extract is in a pure phase. The crystallite size was estimated from the Debye- Scherrer formula. From the calculations it is clear that the size of the particles obtained is less than 100nm. The calculated particle size for TiO_2-1 is 6.7 nm and TiO_2-2 is 10.2 nm respectively. Annealing at a high temperature causes agglomeration of the particles which leads to an increase in the particle size for the annealed sample when compared to the unannealed one. Also, by comparing the XRD peaks, it is clear that the peaks become more sharper after annealing which indicates an increase in the crystallinity of the annealed samples.

Fourier Transform Infrared Spectroscopy (FTIR)

The sample is exposed to IR radiation during infrared spectroscopy. The sample absorbs part of the infrared light, but some of it also passes through (transmits). The resulting spectrum serves as a molecular fingerprint of the material by depicting molecule absorption and transmission. No two different molecule configurations create the same infrared spectrum, similar to a fingerprint. As a result, infrared spectroscopy can be used for a variety of analyses. The FTIR spectra of the synthesised samples is given in figure 3.2.

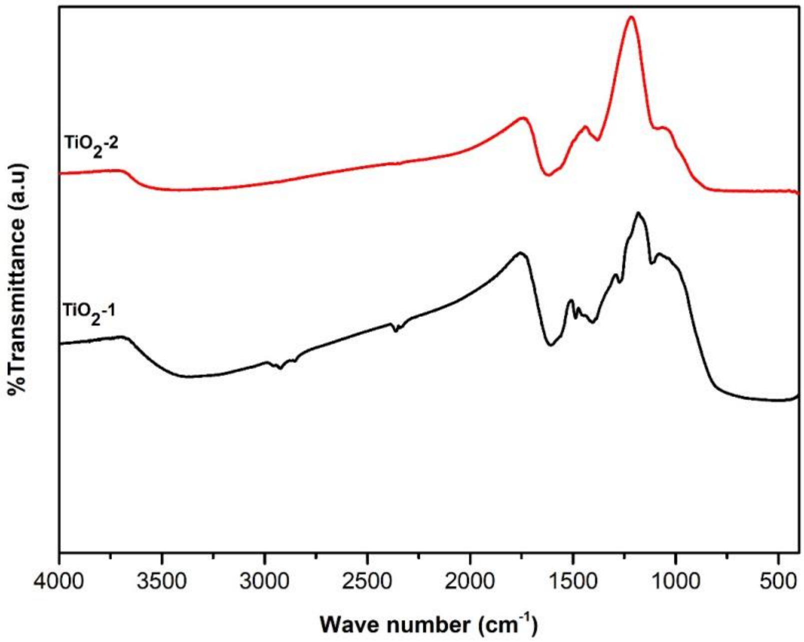

Fig. 3.2 FTIR spectrum of TiO_2 nano particles

In TiO_2-1, a broad band is observed between 3800 to 3000 cm^{-1} and it is related to stretching of hydroxyl group, indicating the presence of water as moisture. The peak at 2630 cm^{-1} is due to the -CH stretching in methylene group. Other peak at 1609 cm^{-1} is referred to the C-O stretching vibration of alcoholic group. The peaks at 1486 and 1404 indicates the existence of $-COO^-$. All these peaks are due to the presence of the phytochemicals from the plant extact and precursors. The broad band between 800 and

450 cm^{-1} is assigned to the Ti-O stretching or Ti-O-Ti bending of TiO_2. The intensities of all the bands other than that related to TiO_2 are very low indicating that the amount of these types of impurities is very less.

In TiO_2-2, the peaks due to the phytochemicals and precursors are comparatively less and with low intensity. This indicates that the phytochemcals and precursors are expelled by the high temperature treatment and the sample purity increases.

UV-Visible Spectroscopy

The light photon absorption properties of the synthesized materials were studied by UV-Visible absorption spectroscopy. In a semiconductor, absorption of light leads to the excitation of electrons from valence band to conduction band. The energy difference between the top of the valence band to the bottom of the conduction band is called optical band gap. The UV-Visible absorption spectra of unannealed and annealed TiO_2 nano particles are given in figure 3.3.

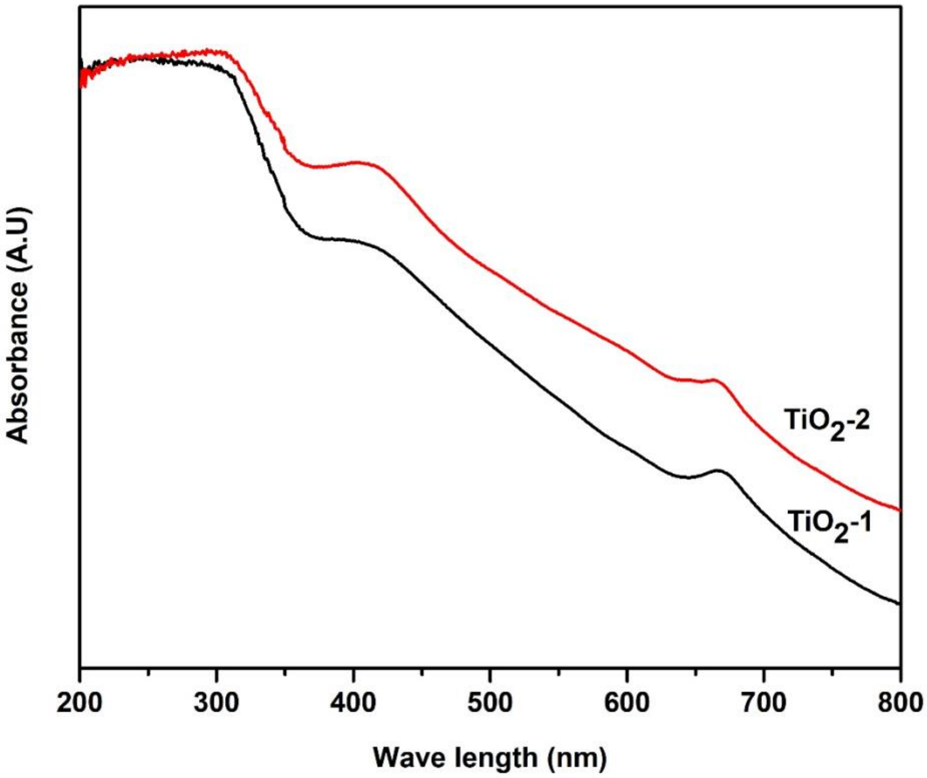

Fig.3.3 UV- Visible absorption spectra ofTiO$_2$ nano particles

The absorption maximum (λmax) value of TiO$_2$-1 is 293 nm and that of TiO$_2$-2 is 303 nm . The λmax of bulk TiO$_2$ is in the range of 400nm. The blue shift in the absorption maxima is evidence of the reduction of size to nano scale.

From the different characterization techniques, it is obvious that TiO$_2$ nanoparticles were synthesized by the phytochemical assisted green method. The proposed method eliminates the usage of chemicals as reducing agent, capping agent and solvent and hence can be considered as green method. Annealing of the hydrothermally synthesised samples cause an increase in crystallinity and purity. At the same time, particle size was increased by annealing.

CHAPTER 4

CONCLUSION

TiO_2 nano materials were synthesized from the aqueous leaf extract of the plant Chromolaena odorata using hydrothermal method. The effect of temperature on the properties of the synthsised sample has also been studied. The Characterization of the synthesised materials was done using X-ray diffraction, FTIR spectroscopy and UV-Visible spectroscopy. The XRD patterns revealed that the prepared nano particles were crystalline in nature. It also gave information about the crystalline purity of the synthesized materials. Particle size was roughly estimated from XRD patterns with the help of Debye- Scherrer formula. The calculated particle size was below 100nm. Annealing cause an increase in crystallinity and particle size. FTIR spectra gave an idea about the types of bonds present in the samples which helps to found out the presence of impurities. Annealed sample shows more purity when compared to unannealed one. UV-Visible absorption spectra of the samples showed a blue shift in absorption maximum compared to their bulk counterparts. This may be due to the increase in the band gap. From the results, it is apparent that the method used for synthesizing TiO_2 nano particles in the current work is an excellent method for preparing nano materials. This method offers a sustainable, simple and cost-effective way of producing TiO_2 nano materials which can be used for a variety of applications.

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