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Morphological and Biochemical Characterization of Synthesized TiO2 Nanoparticles for Enhanced Biotreatment of Pharmaceutical, Ayurvedic, and Culinary Wastewater

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ABSTRACT

The rapid increase in pharmaceutical, Ayurvedic, and culinary wastewater production has led to growing environmental concerns, particularly in the context of wastewater treatment. These effluents are often rich in organic and inorganic pollutants, including biological oxygen demand (BOD), chemical oxygen demand (COD), color, trace metals, and oils, posing significant challenges for wastewater treatment. This study investigates the synthesis and characterization of titanium dioxide (TiO2) nanoparticles using plant extracts from *Parthenium hysterophorus* and *Saccharum spontaneum*. The green synthesis method, facilitated through spray drying, produced TiO2 nanoparticles with distinct morphological and biochemical properties that hold promise for biotreatment applications. The nanoparticles were characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and Field Emission Scanning Electron Microscopy (FE-SEM), revealing functional groups, crystal phases (rutile and anatase), and nanoparticle morphology ranging from spherical to hexagonal with sizes between 72.9 nm and 190 nm. The study also demonstrates the potential of electrochemical coagulation (ECC) as an efficient biotreatment method for pharmaceutical, Ayurvedic, and culinary wastewater. The results suggest that TiO2 nanoparticles, synthesized through a green approach, could serve as effective agents in the treatment of wastewater from diverse sectors, offering a sustainable and eco-friendly alternative for environmental remediation.

Keywords: Saccharum spontaneum, Spray drying, FTIR (Fourier Transform Infrared Spectroscopy), XRD (X-Ray Diffraction), FE-SEM (Field Emission Scanning Electron Microscopy), Nanoparticle morphology

1. INTRODUCTION:

There are many types of wastewater that are produced by treatment rooms. These include suspended solids (SS), particle solids, chemical oxygen demand (COD), biochemical oxygen demand (BOD5), and oil and grease (O&G). The aforementioned effluents have the potential to have a detrimental effect on the rivers in the surrounding area. The Ayurvedic sector is responsible for producing effluent that is both oily and filthy, and it seems that there is no legal mechanism that is suitable for dealing with this wastewater. It is important to note that the flow of these wastewaters is completely distinct from the flow of effluent in allopathic healthcare installations. Standard procedures are followed in order to dispose of the liquid waste that is produced by Ayurvedic treatments such as Vamana, Virechana, Raktamoksha, and Basti, amongst others. There are a great number of highly serious pollutants included in ayurvedic wastewater.[1] Some of these pollutants include biological oxygen demand (BOD), color, metals, dissolved particles, trace chemicals, organic and inorganic substances (O&G), and chemical oxygen demand (COD). Other pollutants include trace levels of trace compounds. With the exception of taila, which is composed of oils, the rest of the Dhara, Avagaha, Salvana, Upanaha, and Ksalana are all composed of liquids.[2] There are natural drains that are used to dispose of these liquids. Various decoctions are used to make kashayas. The vast majority of herbal treatments are completely risk-free to use, provided that they are disposed of in the appropriate manner within the sewage system. In this scenario, liquid waste that has been permitted to remain in a stationary position would attract flies and scents that are considered to be unpleasant. With the little amount of information that was accessible, we decided to start on an innovative journey in order to acquire the knowledge necessary to cleanse raw Ayurvedic effluent. We investigate the use of electrochemical coagulation (ECC) as a treatment technique for wastewater treatment from the actual Ayurvedic industry as well as effluent from treatment rooms in order to remove main pollutants.[3]

The first testing was carried out in accordance with the requirements set out by the American Public Health Association (APHA) for the main quality criterion on the raw effluent that was generated by the Ayurvedic enterprise.[4] This was done prior to the implementation of batch electrochemical processing (ECC) using electrodes that were constructed of two-dimensional metal plates that were created of a variety of metals. It is possible that the mineralization of wastewaters, as well as a decrease in hydraulic retention time (HRT), unit operations, and treatment train procedures, might occur as a consequence of the use of contemporary technology for the treatment of wastewaters that present unique challenges.

Among the many different kinds of wastewater that ECC has successfully treated, some examples include electroplating, slaughterhouses, dairy farms, auto garages, washing facilities, plantations that harvest sunflower oil, and the chocolate industry[5] Wastewater from washing facilities is another kind of wastewater that ECC has successfully treated in many instances. In the past, we had a lot of trouble achieving the lowest HRT, great floc settle, and clear water following ECC. The use of gravity filtering, SVI control, and strong ECC byproducts were utilized in order to accomplish this. The problems have been resolved thanks to a research that was recently carried out on raw AHWW.[6]
In the context of this article, the term "waste" refers to items that individuals do not intend to use or that they do not consider to be important enough to merit the retention of. Wastes, which have the potential to damage both people and the environment, should be eliminated as soon as it is possible to do so in order to avoid some of the most serious health problems from occurring. One of the most important things to do is to find out how to transform waste into a resource that can be used. There are many different types of waste, some examples of which include garbage from households, sewage, sludge, waste from factories, packaging, scrap metal, obsolete electrical equipment, detritus from gardens, paint canisters, and abandoned automobiles. In light of this, it is quite probable that a large variety of waste kinds might be generated from a variety of sources over the course of daily operations. The following types of facilities have the potential to be sources of pollution: residential structures, commercial buildings (such as restaurants, stores, and factories), industries (such as pharmacies and garment manufacturers), agricultural (slurry), construction (including demolition), energy generation (such as mining and quarrying), among other types of businesses.[7] In light of the enormous amount of garbage that is produced, it is of the utmost importance to manage it in a way that does not do any damage to either the environment or the human population. The management of waste may be approached from a variety of angles, including, but not limited to, the recovery of energy, recycling, reuse, disposal, and reduction of waste along with other approaches. According to the findings of study carried out wasted pharmaceutical products may be classified as either hazardous or non-hazardous.

1.1. TREATMENT OF PHARMACEUTICAL EFFLUENTS USING NATURAL COAGULANTS

The use of biological treatment is the method that is both the most practicable and the most cost-effective for removing organic contaminants from wastewater from pharmaceutical companies. The reason for this is because organic matter is the most prevalent contaminant that may be discovered in wastewater from pharmaceutical companies. The objective of this investigation was to investigate seven naturally occurring coagulants that are easily accessible and with the intention of treating wastewater that is produced by pharmaceutical manufacturing systems.[8] Within the pharmaceutical business, there has been research conducted on the use of natural coagulants for the treatment of wastewater. Based on the findings of the study conducted on natural coagulants, the following are some of the most important conclusions that were drawn

2. AIM AND OBJECTIVES

1. The purpose of this work is to investigate the morphological and biochemical features of synthesised TiO2 nanoparticles, with a particular emphasis on the possible applications of these nanoparticles in the bio-treatment of wastewater from the pharmaceutical, Ayurvedic, and culinary sectors.
2. The objective of this project is to identify and implement the biotreatment approach that is the most effective and efficient.

3. METHODOLOGY

3.1. GREEN SYNTHESIS OF TIO_2-NP:

In the fields of physical, biological, chemical, mechanical, optical, medicinal, and engineering sciences, the manufacture of inorganic metal oxide nanoparticles is gaining prominence. In these fields, inorganic metal oxide nanoparticles are being produced in order to investigate and interact with molecules and individual atoms.[9] Metal oxide and nanoparticles possess antibacterial, magnetic, electrical, and catalytic capabilities. These features are a result of the high surface-to-volume ratio that they possess. As a result of their high oxidation resistance, nontoxicity, and light stability, titanium dioxide nanoparticles have a wide range of applications. Some of these applications include dye-sensitive solar cells (DSSC), water and air cleaning, and others. Metal oxide nanoparticles have a variety of uses in the pharmaceutical industry. Historically, a wide range of physical and chemical techniques have been used in order to successfully manufacture metal oxide nanoparticles.[10] Among the most common types of synthesis procedures, reduction, sol-gel, solvothermal, electrochemical, and non-sputtering techniques are among the most widely used. A significant amount of work and effort is required for the therapies that have been listed above, in addition to the fact that they are expensive and potentially harmful. Because of their biocompatibility, environmental friendliness, and the long-term economic feasibility of dependable biosynthetic and ecologically friendly processes, these characteristics have become more significant in the process of limiting adverse repercussions that occur during their usage, particularly in the medical field. This is especially noteworthy with regard to the pharmaceutical industry. The bottom-up manufacture of nanoparticles requires careful consideration of the reduction and oxidation processes that take place throughout the manufacturing process. In 2004, the production of customised nanoparticles was estimated to be 2,000 metric tonnes. By the year 2020, it is anticipated that the production of these nanoparticles would have climbed to 58,000 pounds. When it comes to the manufacture of metal

and metal oxide nanoparticles, one of the most common methods involves the utilisation of plant phytochemicals as reducing or antioxidant constituents. The production of nanoparticles that have a significantly reduced impact on the environment may be accomplished via three primary chemical processes. When selecting a nanoparticle stabilisation material, it is essential to choose a solvent medium, a reducing agent, and a non-toxic nanoparticle stabilisation substance. These are all essential components to take into consideration. The production of nanoparticles was made possible by the utilisation of plant extracts that satisfy the requirements of green chemistry. The extracts of Saccharum spontaneum and Parthenium hysterogenicus were included in this group of plant substances. Additionally, plant extract is easily available and has a large variety of beneficial metabolites, which distinguishes it from other biosynthetic processes thanks to its unique characteristics. In addition, when it is handled correctly, it does not present any dangers to one's health or safety. In the beginning, we used a flask to combine twenty millilitres of ethanolic plant extract with fifty millilitres of titanium tetraisopropoxide. Following that, we heated the mixture to a temperature of fifty degrees Celsius for a period of five hours while stirring it. After that, the mixture was spun at a speed of 5,000 revolutions per minute for fifteen minutes in order to remove the nanoparticles. In accordance with the research carried out by Karuppuswamy and colleagues in 2023, the nanoparticles were dried in a hot air oven for a period of five hours at a temperature of 500 degrees Celsius.[11]

3.2. SPRAY DRYING TECHNIQUE:

Spray drying is a well-established technique with a long history of use in a variety of fields (e.g., foods and chemicals). The use of spray drying for the pharmaceutical industry dates to the early 20th century when it was used for the drying of blood. Since then, it has been employed for various pharmaceutical applications including formation of amorphous solid dispersions, encapsulation of drugs and essential oils in excipient matrices, and spray drying of biopharmaceuticals (e.g., proteins, vaccines, Deoxyribonucleic Acid (DNA), antibodies). Spray drying is widely applied to produce pharmaceutical powders with particle size ranging from the nanometer to the micrometer scale. It has been extensively used for the production of inhalation particles as it allows manipulation and control of properties such as particle size distribution, shape, density, flow ability, moisture content, crystallinity and dispersibility of the powders. Additionally, spray drying can be used both as a particle-engineering and as a crystal-engineering platform Spray drying has been used to a significant degree by the chemical and food sectors for a considerable amount of time.[12] During the early 1900s, the pharmaceutical business was the first to use spray drying as a method for drying blood. Following that, this method has been used in a variety of applications, including the encapsulation of drugs and essential oils in excipient matrices, the production of amorphous solid dispersions, and the spray drying of biopharmaceuticals, amongst others. The production of pharmaceutical powders with diameters ranging from nanometres to micrometres is often accomplished via the use of the spray drying technique. Because of its capacity to regulate and manage attributes such as form, density, flow ability, crystallinity, shape distribution, moisture content, and dispersibility, powder is suitable for use in the production of inhalation particles. This is because powder can be used to effectively create inhalation particles.Spray drying has the potential to serve as a basis for the development of concepts that may be used in the field of particle and crystal engineering.

3.2.1. PROCEDURE:

i. Twenty millilitres of ethanol and 0.1 percent titanium tetra isopropoxide were combined with a base and stirred together for a period of thirty minutes. Adding a few drops of distilled water to the mixture is the next stage in the process of making the dispersion medium.
ii. In relation to the product, an ultrasonication session of twenty minutes was carried out. Following the sonication of the solution, it was heated to a temperature of 150 degrees Celsius and then autoclaved for a period of three hours.
iii. It was centrifuged and then washed with deionised water after the solution had been allowed to reach room temperature.
iv. This was done in order to remove any possible pollutants that may have been present. It is possible for you to pass through it by using Whatman No. 1 filter paper.
v. After being dried at 110 degrees Celsius for five hours, the sample that had been filtered was then annealed for two more hours at a temperature of 500 degrees Celsius.
vi. TiO_2 nanoparticles were characterised after they were collected after they had been collected.

Table no. 1: Composition of factorial batches of TiO_2-NP

Formulation Code	Titanium tetra isopropoxide	Carbapol 934 : Glycerol	Stirring Speed
F1	5 mg	1:1	500
F2	5 mg	1:2	500
F3	5 mg	1:3	500
F4	5 mg	1:1	1000
F5	5 mg	1:2	1000
F6	5 mg	1:3	1000
F7	5 mg	1:1	1500
F8	5 mg	1:2	1500
F9	5 mg	1:3	1500

4. CHARACTERIZATION OF FORMULATION:

The JEM-2100F investigates the process by which photocatalytic nanomaterials induce fibres to aggregate, whilst the transmission electron microscope (TEM) operating at 200 kilovolts investigates the morphology of the sample. In order to determine the dimensions and contours of the nanofibers, scanning electron microscopy (SEM) was used. ICP, which stands for inductively coupled plasma, was used in an earlier experiment for the purpose of determining the various metal concentrations that were being investigated (iCAP 6500, Thermo Fisher). Through the use of ultraviolet-visible spectroscopy, the quantities of components, particularly organic molecules, were examined and determined.[13] In addition, the quantities of metal were discovered by the use of both of these methods. In order to capture pictures of TiO2 and other elemental conformations, the X-ray detector is responsible for collecting X-ray measurements and determining their contents. Instruments such as the TA and the TGA-Q5000 are used in the process of thermogravimetric analysis, which is abbreviated as TGA for short. Nanofiber and nanoparticle spectra were obtained by the use of Fourier transform infrared (FTIR) spectroscopy, which was carried out with the assistance of a Thermo Nicolet iS10 equipment.

4.1. ELECTRON MICROSCOPY

Electrons are the principal particles that are used in the process of removing material in electron microscopy. The development of a steady and monochromatic electron beam is required in order to guarantee the successful completion of the microstructural characterisation technique. The scanning transmission electron microscope, often known as a STEM, is an example of an electromagnetic device that may be used to display the characteristics of electron waves. The SEM and TEM techniques are considered to be part of the STEM field

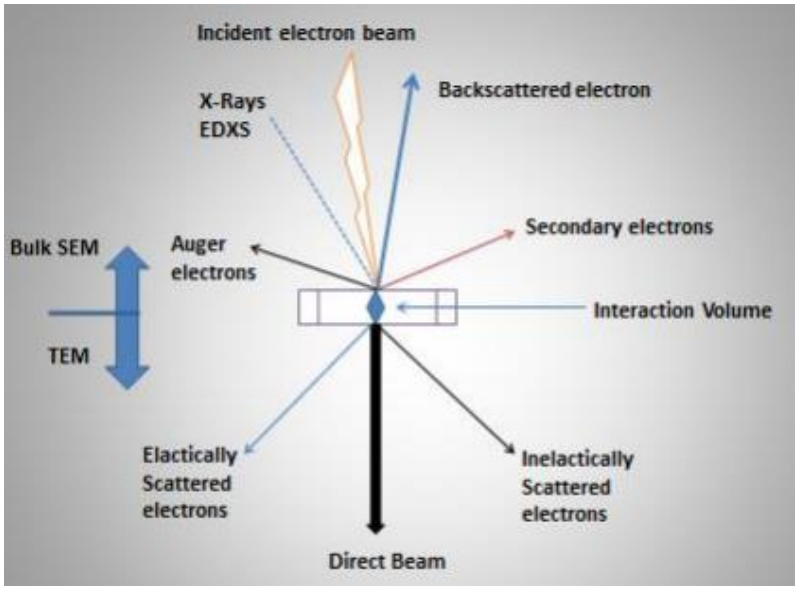


Figure no. 1: Electron Microscopically Beam consequence after interaction with incident beam with specimen

4.2. SCANNING ELECTRON MICROSCOPE (SEM)

This technique, known as scanning electron microscopy (SEM), involves scanning the surface of an item in order to produce images. Initially, a high-energy electron beam was directed onto the surface of the sample. Subsequently, the

beam signals were reflected back onto the material, which enabled atoms and electrons to interact with one another and create the images. By using the image morphology system, it is also able to examine the surface domain, as well as the composition and properties of the surface. The scanning electron microscope (SEM) first made use of scattered electrons. Considering the enormous depth of focus that scanning electron microscopes possess, they are able to selectively display a greater number of sample images. The diffusion of electrons from an electron beam is the process that results in the production of various images. It is possible to refer to these electrons as backscattered electrons or secondary electrons (SE), depending on the characteristics that they manifest.[14] In contrast to backscattered electron images, secondary pictures are produced by electrons with lower energy that are expelled from the initial energy beam. Backscattered electron images are produced by particles with greater energy that are lifted. Although a greater number of electrons are produced during BSE, the number of electrons produced during SE image generation is far lower, with less than fifty electron volts being produced. The SE picture displays inelastic scattering in a deep synergistic range, in contrast to the BSE image, which displays elastic scattering in the same range as the SE image. For the purpose of maximising the resolution and analysis of surface morphology, it is essential to keep the surface clean and to manage the conductivity between the sample and the electron beam in an appropriate manner.

Figure no. 2: Scanning electron microscope (SEM)

4.3. TRANSMISSION ELECTRON MICROSCOPY (TEM)

With the help of an electron beam, the transmission electron microscope is a piece of apparatus that magnifies an image of an item that is very thin. The acronym "TEM" is used to refer to this piece of apparatus. The picture is captured by a CCD camera sensor after it has been magnified and focused on a photographic film imaging device during the photographing process. In comparison to earlier mass spectrometers, it demonstrates superior performance in terms of specimen resolution and sensitivity to minute features.[15] Through the use of transmission electron microscopy (TEM), it is possible to demonstrate that a space that is two-dimensional has a structure that is three-dimensional. The brightness of an image captured by a transmission electron microscope (TEM) may be affected by a variety of contrasts. Controlling the mass-thickness contrast or the picture absorption may be accomplished with the help of this technique. In addition to this, contrast is applied to the whole sample once crystallographic plane diffraction has been applied to it

Figure no. Error! No text of specified style in document.**: Transmission Electron Microscopy (Ref. Google)**

4.4. ULTRAVIOLET-VISIBLE (UV-Vis)

The technique of absorption spectroscopy may be used in clinical laboratories for the purpose of determining the identity of a broad variety of molecular biological products, elements, compounds, and inorganic substances. These integrated analysers make use of a wide variety of technologies, such as turbidimetry, ion-selective electrodes, and UV-Vis absorption, amongst others. The method of spectroscopy that is being discussed here is used to investigate the reaction of light. The transmission rules let the remaining light to travel through the sample container, despite the fact that the sample container may only permit a certain amount of light to pass through it. On the other hand, the symbol (Io) represents the proportion of the entering light that may be used for spectroscopy, while the sign (I1) denotes the intensity of the excitation light[11] Consequently, the transmittance % is made up of the following:

$$\%\ \text{of transmittance}\ (\%T) = \left(\frac{Io}{I1}\right) \times 100$$

By using this equation absorbance (A) can be write down in terms of transmittance,
A = (-log T)

Despite the fact that the majority of the UV-Vis equipment that is now available functions in the wavelength range of 190 to 700 nm, this is not always the case. Within the confines of our laboratory, we have ultra-modern UV-Vis equipment that is capable of detecting wavelengths ranging from 190 to 800 nanometres. The Beer-Lambert Law, which is widely considered to be among the most important absorption ideas, may be evaluated with the use of our technique. Under perfect circumstances, the relationship between concentration and absorbance is linear, provided that the length of the route does not change continuously.

A=εlc
Where, ε is the absorptivity of Substance, l is the Path Length and C is the Concentration. Here,

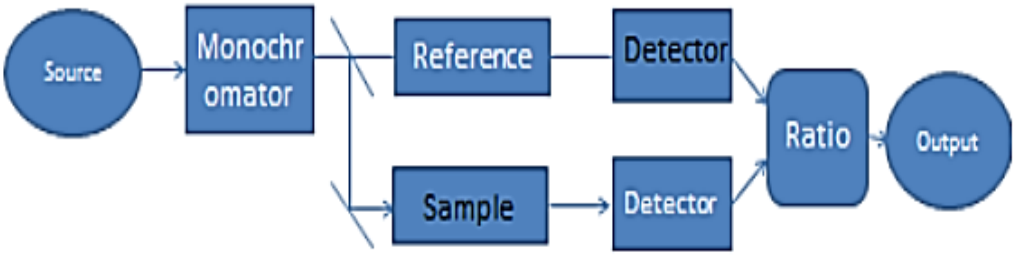


Figure 4 Schematic diagram of UV-Vis instrument pathway and construction overview

For the purpose of holding samples, cuvettes are containers that are see-through. Retail outlets are the places where the cuvettes that are manufactured of quartz and optical glass may be purchased. When light is allowed to flow through optical glass, the quantity of light that can do so is restricted. In contrast to quartz cuvettes, which have the ability to transmit light between 190 and 320 nanometres, their light transmission is restricted to 320 nanometres.[16] For the purpose of measuring the absorbance of organic substances (ibuprofen and cetirizine), this experiment calls for the use of quartz cuvettes that have a low transmitting light absorbance that is more than 300 different nanometres. It was reported by that a VARIAN Cary® 300 UV-Vis spectrometer was used in order to assist with the inquiry regarding the fluctuation of medicinal medication concentrations.

4.5. DIFFERENTIAL SCANNING CALORIMETER (DSC)

You may use the thermoanalytical differential scanning calorimetry (DSC) approach to determine how much heat is required to get a sample up to a reference temperature and how that varies with temperature. This can be done by comparing the sample to the reference temperature. Over the course of the experiment, both the reference and the sample are maintained at temperatures that are, for the most part, comparable to one another. DSC investigations often make use of temperature regimens that increase the temperature of the sample holder in a linear fashion over the course of time. For the reference sample to be considered acceptable, its heat capacity must be properly specified over the whole temperature range that was scanned. Only then can the sample be considered acceptable. Reference sample must not display any substantial changes or be contaminated in any manner during the duration of the temperature scan.[17]

Figure no. 5: Differential scanning calorimetry (DSC)

Over the course of human history, substances such as lead, bismuth, tin, and indium have been utilised in the process of establishing reference standards. In the other direction, fatty acids and polyethylene have been suggested as potential replacements for polymers and organic molecules, respectively. I.e. A. E. the plan of action was developed. At the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy in 1963, the product was first introduced to the market for advertising purposes. A. S. was the person who first conceived of the offering. In addition to M. In 1962, and in conjunction with J. It is Neill. A helpful instrument in the field of biological sciences, the adiabatic differential scanning calorimeter was first developed by P., who was the inventor of the device. 1964 was the year that L. Jackson was born. A cooperative effort was made by Dr. Privalov and Dr. Tbilisi Institute of Physics to include R. We are the monaselidze. the term "direct spectrometer" (DSC) originates from the fact that this technological apparatus is capable of accurately measuring the amount of energy and heat that it is capable of producing[18]

4.6. POWDER X-RAY DIFFRACTION (PXRD)

X-rays are an example of the particular kind of electromagnetic radiation that might be generated. X-ray wavelengths have been discovered to be present in an area that is around 1 Å (10-10 m) in size. When used to substances that have a crystal structure, X-ray diffractometers have the capability of revealing not only the structure of the crystal but also the qualities of the crystal. Primary use of this approach in the field of solid state physics and the study of materials is the process of characterising the material[19]

Figure no. 6: Powder X-ray diffraction (Ref. Google)

In order to determine the effects that the hydrothermal processing stages had on the nanofibers, X-ray diffraction (XRD) research was performed on nitrogen butoxide-polyanol (PAN) both before and after the hydrothermal processing stages. For the purpose of determining the crystalline structure and size of titanium, the designs were developed specifically for this purpose. It is possible for atoms to liberate electrons when they absorb or deflect X-rays. This is because atoms have the ability to do so. The word that describes this phenomenon is called electron ejection. The miler index is shortened as hkl, whereas the symbols that indicate the distance between two planes are denoted by the letters d or dhkl. Figure 7 displays these statistics for your perusal. The solution to the equation of Bragg's Laws may be achieved by using the following factors: the length of the path wave, the angle of incidence of the beam with the plane (θ), and the distance between the two planes (d). This is accomplished by the use of a technique known as X-ray diffraction (XRD).

$$2dsin\theta = n\lambda$$

Powders and nanofibers both have the potential to have their atomic crystal structures analysed with very little difficulty. The phase composition as well as the size of the crystal are both under its control. Check out this article for more information on residual strain. An X-pert pro super diffractometer manufactured by Phillips was used in order to carry out the material analysis. In order to carry out the test, the samples were transferred onto a plate using tape [20]

4.7. FOURIER TRANSFORM INFRARED (FT-IR) SPECTROSCOPY

By using techniques that are based on Fourier transformed infrared, it is possible to differentiate between unknown materials without causing any damage to the surrounding environment. Using qualitative approaches that are based on infrared absorption, emission, or Raman scattering, surfaces may be categorised as either solids, liquids, or gases. This process of classification may be finished if one gives it enough time. There were Michelson interferometers and Fourier transform infrared spectroscopy used in the laboratory for the purpose of conducting the analysis of the samples. Infrared sources that are able to travel through molecules that include atomic bonds may be used to provide a description of the sample's atomic vibration frequencies in the form of wavenumbers. Within certain wavenumber ranges, the material's transmittance and absorbance will exhibit peaks. These peaks will be rather noticeable. Imaging using FT-IR spectroscopy was carried out with the Thermo Nicolet iS10, which was set up in the ATR mode. Dehydrated titanium (IV) butoxide was used in the research that was carried out. When the nanofiber samples were layered on top of the crystal window, they were able to transmit light with a wavelength range that extended from 650 cm-1 to 4500 cm-1. Assert that each and every kind of nanofiber was evaluated in relation to the nanofiber that initially served as the parent.[21]

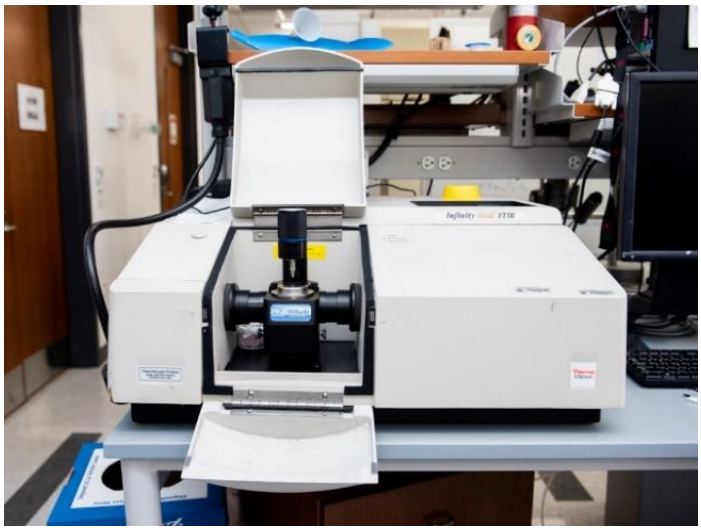


Figure no. 7: Fourier Transformed Infrared Techniques

4.8. PARTICLE SIZE DISTRIBUTION

Particle size analysis, often known as PSA, is a technique that allows for precise measurements of particle sizes down to the region of nanometres. Due to the one-of-a-kind optics that it has, the Malvern Zetasizer Nano Series is able to properly determine the size of particles in samples that include both tiny amounts of particles and significant quantities of particles. The conventional DLS apparatus, on the other hand, has a detection angle of ninety degrees, which means that backscatter optics may evaluate materials at far higher substance concentrations than the normal DLS device. There is a considerable relationship between the size of the particles and the degree to which they can be described.[22] The use of dynamic light scattering is the method that guarantees the highest level of precision when it comes to estimating the diameter of

nanoparticle dispersions. Without the use of strong lasers, it was difficult to measure samples that were excessively diluted or particles that were exceedingly minute and sparsely dispersed.

Figure no. 8: Malvern Zetasizer

4.9. ZETA POTENTIAL

Zeta potential is one method that may be used to evaluate the effectiveness of charge stability in colloidal nanoparticles. For the purpose of doing this, the "effective" electric charge that is present on the surface of the nanoparticles is measured. The net surface charge of the nanoparticle is "screened" by higher concentrations of ions with opposing charges that are located in close proximity to the surface of the nanoparticle. An electrical double layer is produced when the surface charge of a nanoparticle and ions with opposing charges unite to follow the nanoparticle across space. This results in the formation of the nanoparticle. Negative potential, often known as zero potential, refers to the difference in potential that exists between the secondary fluid that surrounds a nanoparticle and the main fluid in which it disseminates. The charges of the nanoparticles are offset by the charges of the ions that are present in this secondary fluid. It is also possible for positively charged particles to attach themselves to negatively charged sites, and it is also possible for negatively charged sites to attach themselves to positively charged particles. Higher Zeta Potentials, which indicate an increase in electrostatic repulsion, are not only an indicator of the stability of the particles, but they also indicate that the particles are stable[23]

5. RESULT AND DISCUSSION:

5.1. FORMULATION OF NANOPARTICLES

5.1.1. PREPARATION OF PLANT EXTRACT

Parthenium hysterophorous and Saccharum spontaneum were investigated by researchers in Baddi, Himachal Pradesh, India. These regions had been polluted by pharmaceutical effluent in the past, and the researchers were interested in looking for their presence. A portion of the extraction process included the use of ethanol. For the purpose of producing the plant powder, a mortar and pestle were used. It is necessary to make use of the Soxhlet apparatus in order to succeed in producing an ethanolic plant extract

(a) (b)

Figure no. 9: Ethanolic Plant Extracts of Parthenium hysterophorous (a) and Saccharum spontaneum (b)

5.1.2. GREEN SYNTHESIS OF TIO_2 NANOPARTICLES BY SPRAY DRYING

For the purpose of spray-granulating a titania solution that included 35% weight percent, a spray dryer that was equipped with bag filters was utilised, and Dispex N40 was utilised as the dispersant. In the event that a spray dryer with this straightforward configuration is used, there is no generation of potentially hazardous fine fractions. Granules are generally thirty-three micrometres in size, spherical in shape, non-agglomerating, and capable of flowing freely. Spray granulation, when carried out at low temperatures, does not result in the formation of grain growth or phase transitions when working with titanium powder.[24] Granules are produced as a final consequence of spray drying a nano titania solution that has been disseminated with Dispex N40. This occurs after the solution has been distributed. Following the process of dispersing these granules in water, they may be ultrasonically sonicated in order to re-establish the particle size distribution characteristic of the initial nano powder. Last but not least, spray drying nanoparticle granules that have the appropriate size and flow might be a way to make the handling of nanoparticles safer. In this way, the formation of stubborn clumps inside the material would be prevented, and the amount of dangerous particles that are breathed would be reduced. Ethanolic plant extracts of Saccharum spontaneum and Parthenium hysterophorous were used in the production of oxide nanoparticles of titanium dioxide. For the purpose of producing nanoparticles, twenty millilitres of ethanolic plant extract and fifty millilitres of titanium tetraisopropoxide were combined.[25]

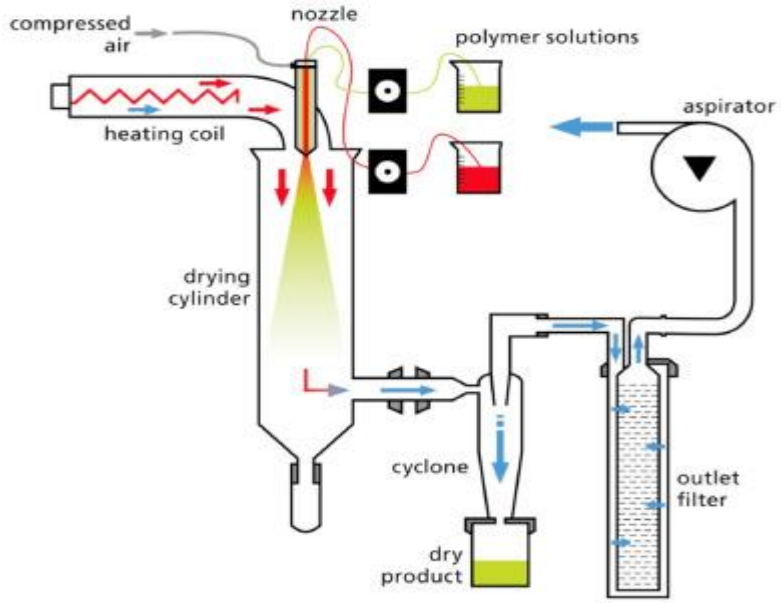


Figure no. 10: Green Synthesis of TiO_2 Nanoparticles by Spray drying

A shaker incubator was then used to place the sample, and it was allowed to incubate at a temperature of fifty degrees Celsius for a period of five hours. Afterwards, the substance was subjected to centrifugation at a speed of 5,000 revolutions per minute for a period of fifteen minutes. Following the removal of the supernatant, the particle was washed three times with a composition of ethanol that was 70% (ethanol). Immediately after the removal of the pellet from the Petri plate, it is essential to immediately add parafilm. In the subsequent step, the nanoparticles were dried in a hot air furnace at a temperature of 500 degrees Celsius for a period of five hours (for more information, refer to Figure 11).

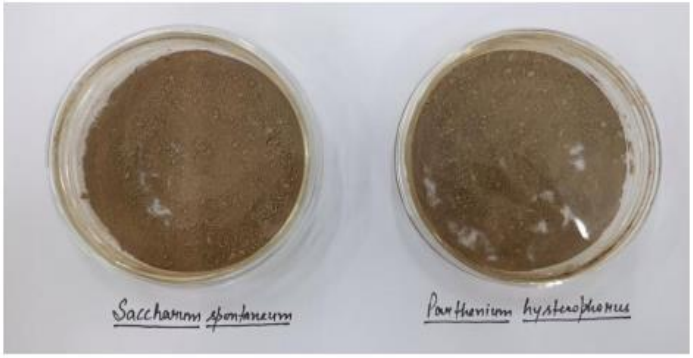


Figure 11 Synthesized TiO_2 nanoparticles by using the ethanolic *plant extract of Parthenium hysterophorous and Saccharum* spontaneum.6. Characterization of Synthesized TiO_2-NPs

6.1. FTIR SPECTROSCOPY ANALYSIS

It is possible to determine the functional groups that are present in TiO2 nanoparticles by using a technique known as Fourier transform infrared spectroscopy. The samples of titanium dioxide (TiO2) were isolated from Parthenium hysterus and Saccharum spontaneum in order to carry out Fourier transform infrared spectroscopy (FTIR) in the range of 500–4000 cm-1. Figure 4.23 is a representation of the absorbance peaks that are associated with the parthenium hysterophorus nanoparticles.[26] The frequencies at which these peaks were seen were 436.16, 1629.02, and 2919.1 cm-1. As can be seen from the spectra, stretching vibrations of Ti-O and Ti-O-Ti can be observed at 436.16 cm-1 and 1629.02 cm-1, respectively. It is possible to see the O-H stretching vibration if one focusses their attention on the peak that is located at 2919.1 cm-1. Comparing this statistics with those of is something that can be done effectively.

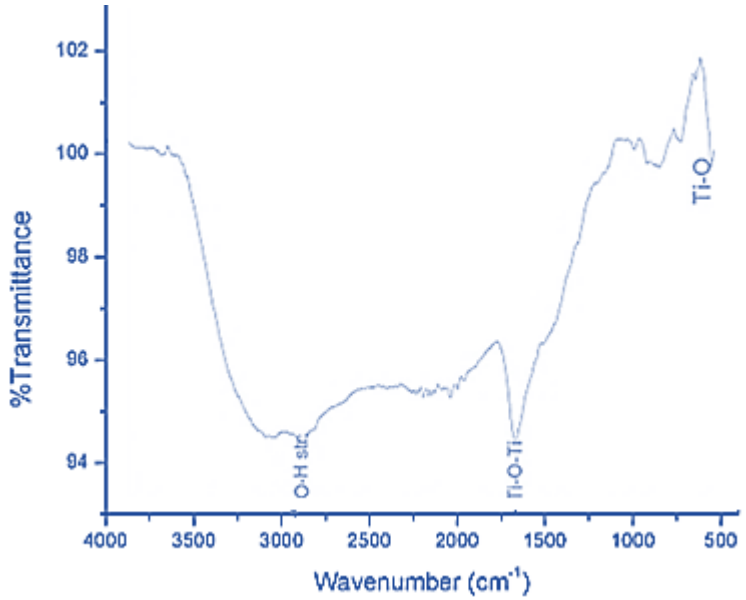


Figure no.12: FTIR spectra of TiO_2-NPs synthesized from ethanolic leaf extracts of *Parthenium hysterophorus*

Figure 13 demonstrates that the absorbance peaks for Saccharum spontaneum are particularly evident at the frequencies of 443.89 cm-1, 1544.12 cm-1, and 3023.38 cm-1. These frequencies are depicted in the figure. There is a frequency of 443.89 cm-1 at which the Ti-O bonds are stretched, and there is a frequency of 1544.12 cm-1 at which the Ti-O-Ti bonds are stretched. Specifically, the stretching vibration of the alcohol-phenol functional groups is indicated by a second signal that is located at 3023.38 cm-1. Because of these findings, it is possible that we will be able to get a better understanding of the chemical composition and structure of TiO2 nanoparticles.

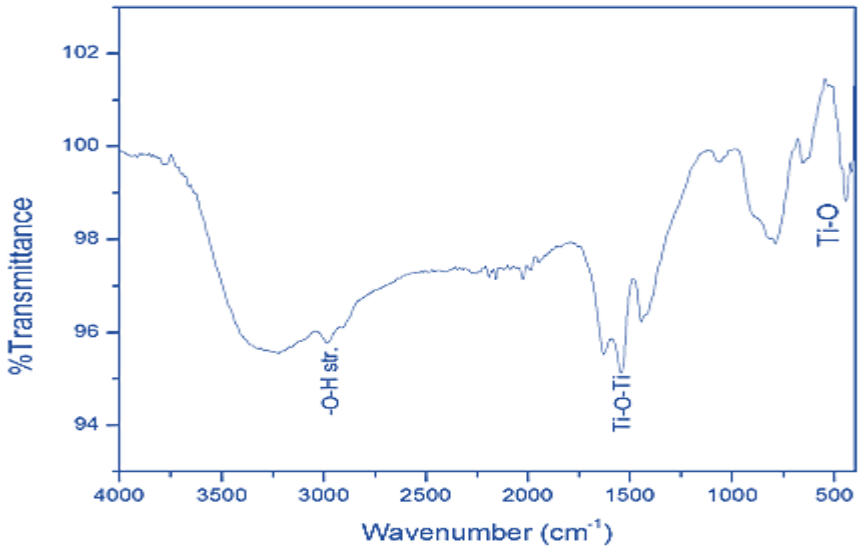
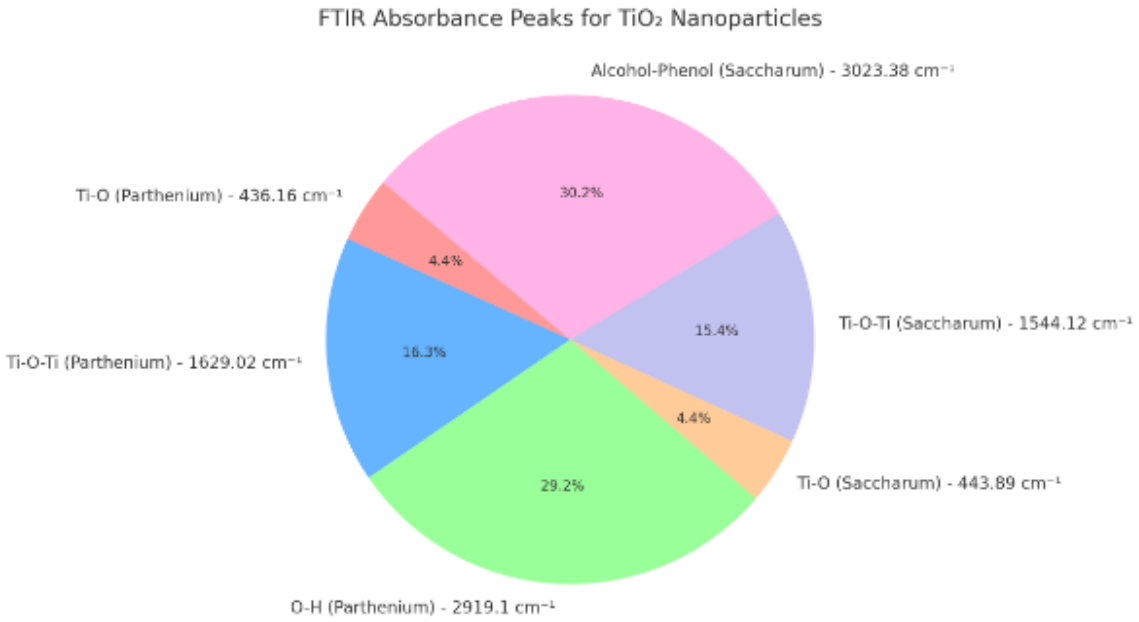


Figure no. 13: FTIR spectra of TiO_2-NPs synthesized from ethanolic leaf extracts of *Saccharum spontaneum*.



6.2. X-RAY DIFFRACTION (XRD) STUDIES

The symbol 2θ is used to denote the position of the detector, and the counts associated with the number of X-rays detected at each angle are referred to as the counts. It is common practice to represent the counts as an intensity term. The data shown in this graph was used to determine that there were 562 counts of titanium dioxide nanoparticles. These counts were identified by using X-rays at an angle of 42 degrees. Despite the fact that there was some variation between 42 and 26.59 degrees, the peak counts of Parthenium hysterophorus were consistently 400 in the range of 20–40 degrees in Figure 14. On the other hand, these results are in agreement with the JCPDS card number 21-1272 as well as the ranges of investigations conducted by every other researcher. When we compare the XRD pattern of our sample to some of the more typical patterns that have been reported by previous research, we are able to be assured that our sample includes the rutile phase of titanium dioxide nanoparticles.[27] When peaks with 2θ values of 26.59° and 42° are seen in X-ray diffraction (XRD), it indicates the presence of rutile phase.

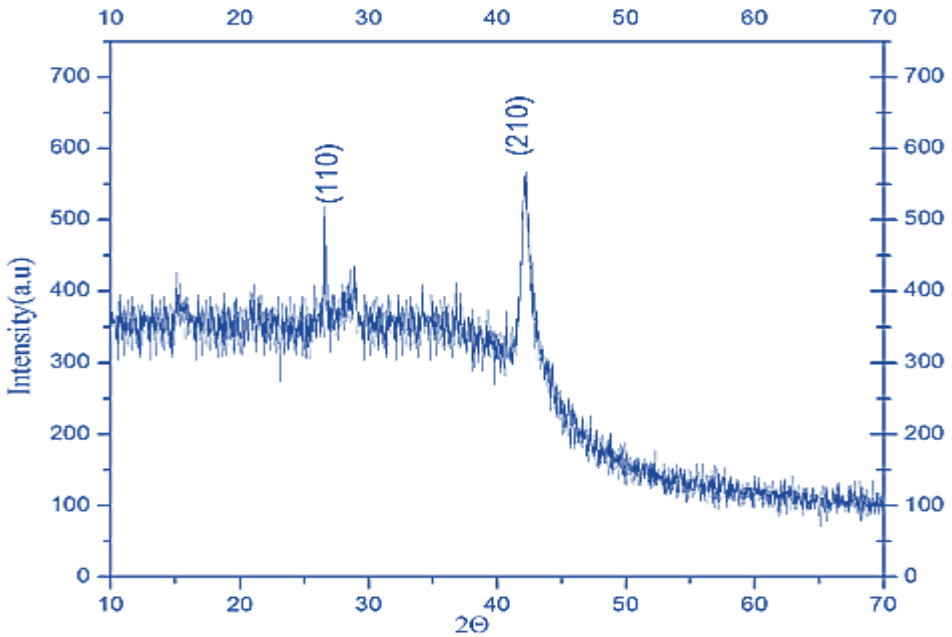


Figure no. 14: X-ray diffraction patterns of TiO_2-NPs synthesized from ethanolic leaf extracts of *Parthenium hysterophorus*

On the other hand, when X-rays were focused towards the synthesised titanium dioxide nanoparticles, the Saccharum spontaneum graph showed that there were 588 counts at 48 degrees. Highest values were found to be between 48.01 degrees and 25.4 degrees, with very little variation between the two extremes. At a temperature of 300 degrees, the counts remained stationary between 35 and 45 degrees.[28] On the other hand, these results are in agreement with the JCPDS card number 21-1272 as well as the ranges of investigations conducted by every other researcher. We will be able to tell with complete certainty whether or not titanium dioxide nanoparticles are present in the anatase phase if we compare the XRD pattern of our sample to patterns that are often reported by researchers. As can be observed in Figure 15, the X-ray diffraction pattern of the anatase phase is correlated with the X-ray diffraction peaks that occur at 25.4° and 48.01°.

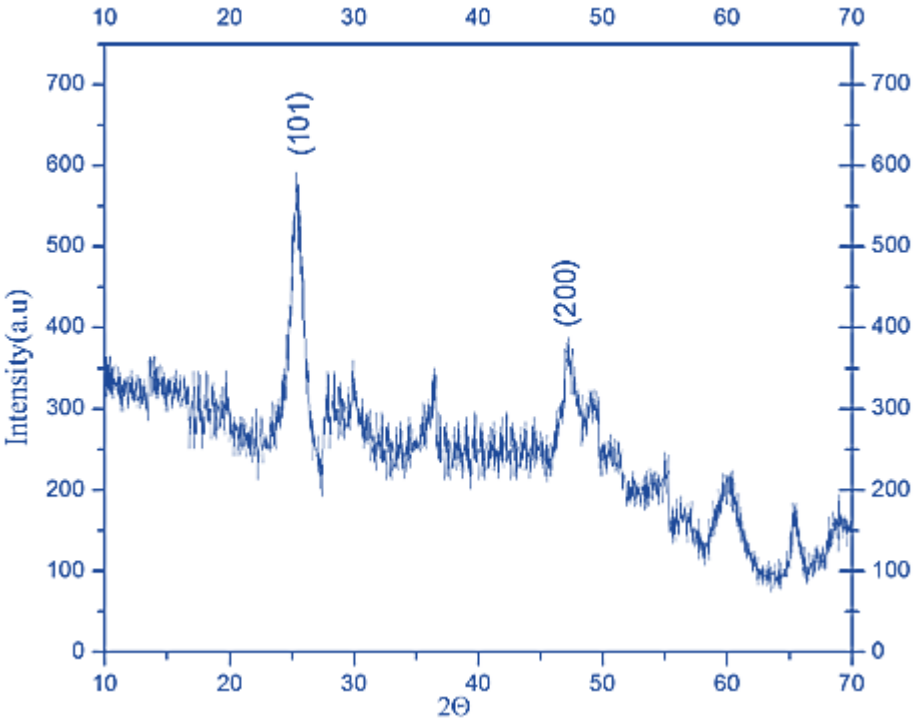
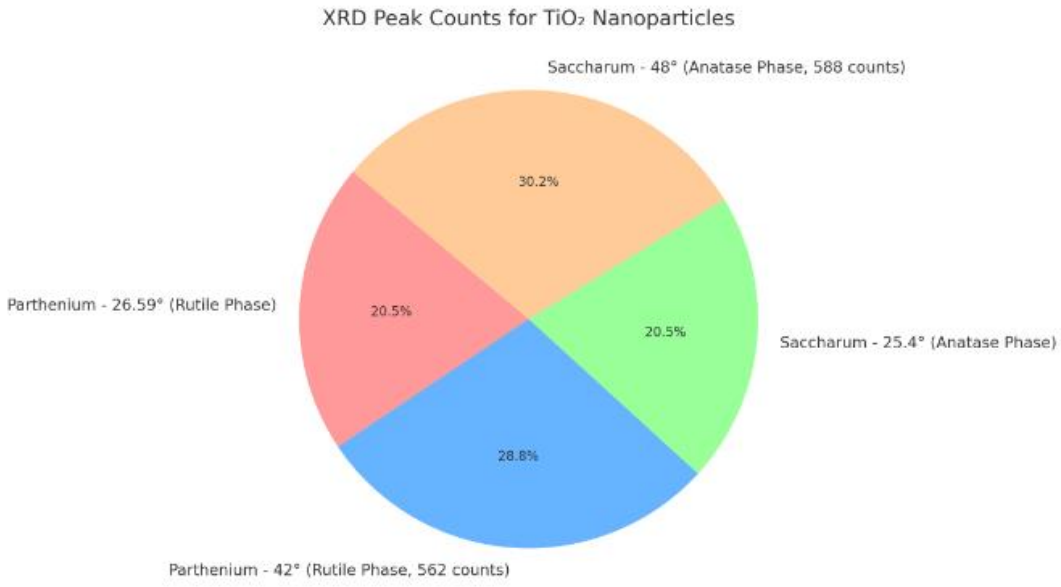


Figure 15 X-ray diffraction patterns of TiO_2-NPs synthesized from ethanolic leaf extracts of *Saccharum spontaneum*.



6.3. FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FE-SEM) STUDIES

The field-emission scanning electron microscope (FE-SEM) was used in order to attain magnifications of 10,000, 15,000, 20,000, and 25,000 in order to investigate the structure of titanium dioxide nanoparticles that were created from Parthenium hysterophorus. Depending on their shape, the nanoparticles were either spherical or hexagonal, and they seemed to have an uneven and rough topography. With an average size of 180 nanometres, the particles were very small. According to the findings of other studies, the thickness of the TiO2 was tested and determined to be 200 nanometres.

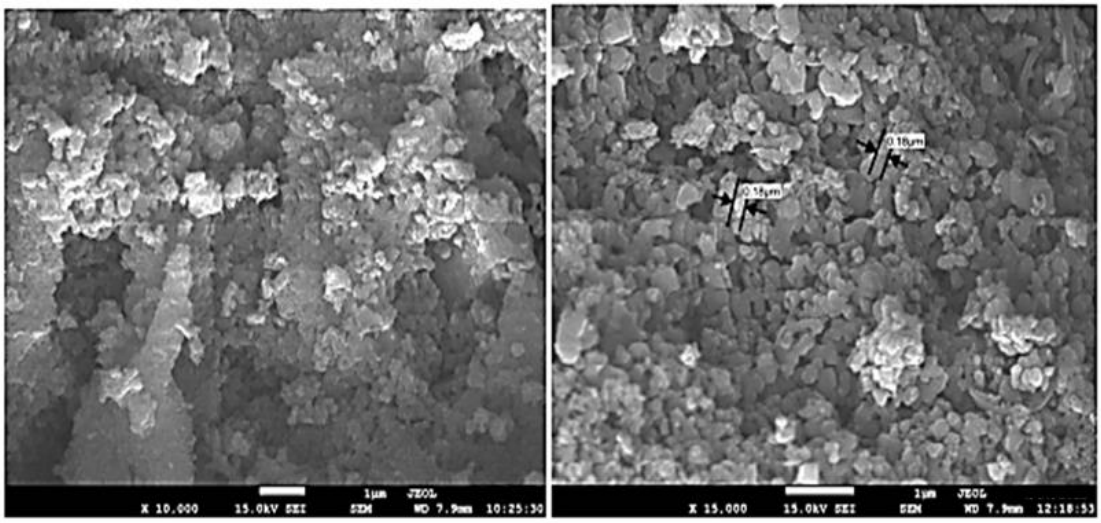


Figure no. 16: FESEM images of *Parthenium hysterophorus* derived TiO2 NPs at 10000X and 15000X

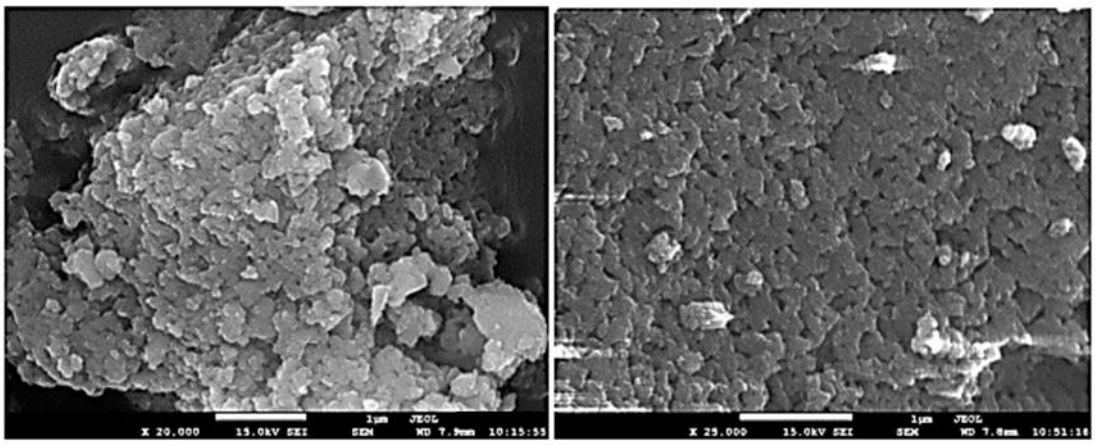


Figure no. 17: FESEM images of *Parthenium hysterophorus* derived TiO2 NPs at 20000X and 25000X

The creation of these nanoparticles, which have an average size of 180 nanometres and may have spherical or hexagonal geometries, is accomplished by the use of a synthesis procedure. An examination was performed using field-emission scanning electron microscopy (FE-SEM) at magnifications of 10,000X, 15,000X, 30,000X, and 45,000X on titanium dioxide nanoparticles that were synthesised from Saccharum spontaneum. Nanoparticles that were synthesised had a wide range of morphologies, ranging from almost spherical to rough and irregular in appearance. There was a wide variation of sizes among the particles, from very small (72.9 nm) to extremely large (190 nm). It has been found by other groups that TiO2-NPs of comparable sizes have been discovered.

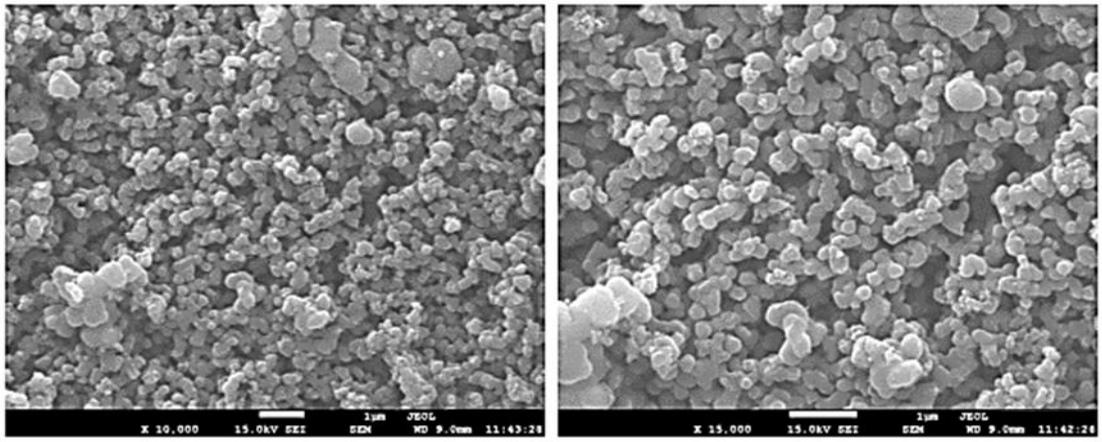


Figure no. 18: FESEM images of *Saccharum spontaneum* derived TiO_2 NPs at 10000X and 15000X

The shape of the synthesized nanoparticles was found to be nearly spherical, where smallest and largest particle size was determined to be 72.9 and 190 nm respectively.

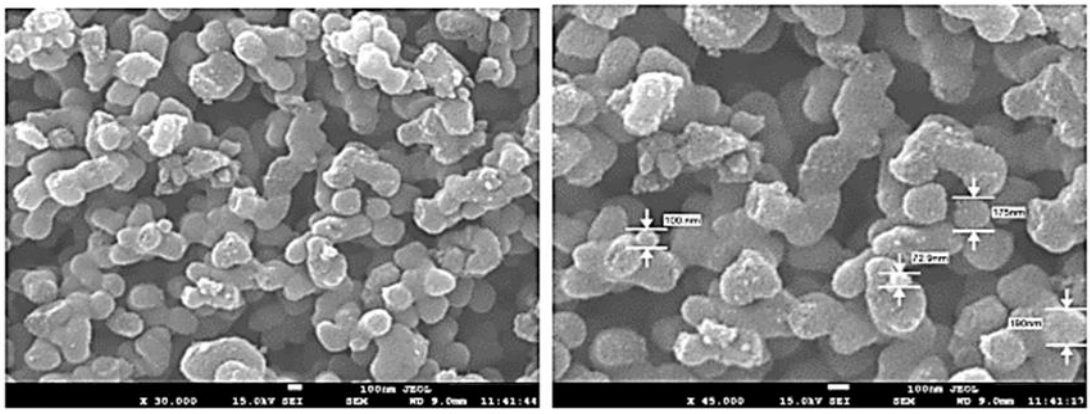
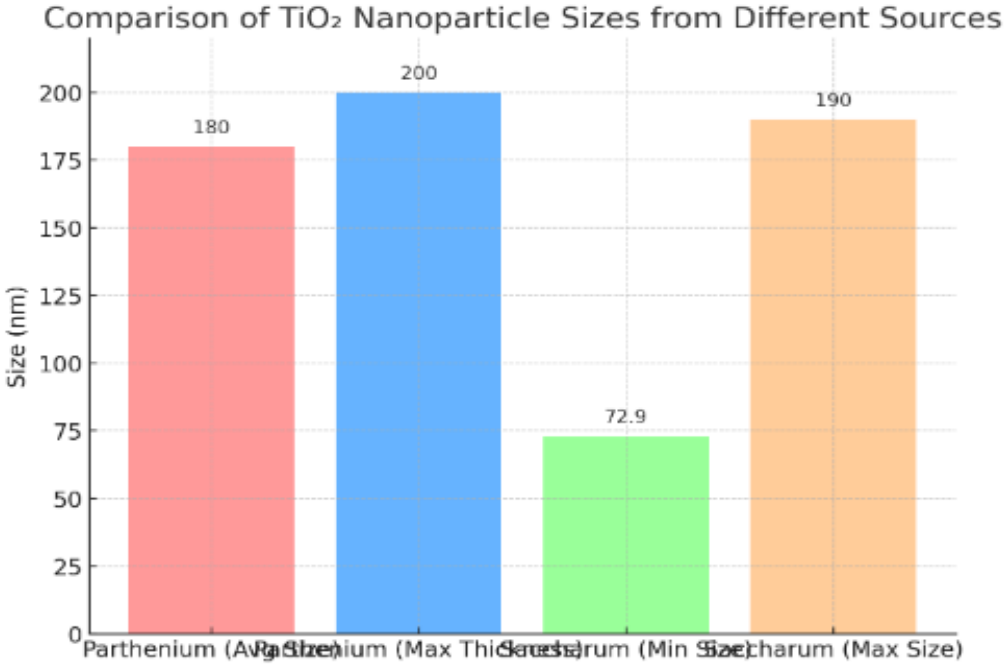


Figure no. 19: FESEM images of *Saccharum spontaneum* derived TiO_2 NPs at 30000X and 45000X



6.4. TEM and SAED ANALYSIS

Now, have a look at the following diagram. An illustration of titanium dioxide nanoparticles that have been treated with a surfactant may be seen in Figure 20. For the purpose of creating these pictures, transfer electron microscopy was used. These nanoparticles have a radius of 0.25 diameters, which is larger than the average. The size of the nanoparticles of surfactant and titanium dioxide generally varied from 25 to 30 nanometres in size. The structure of the sol-gel nanoparticles displayed an unequal distribution of charges across their whole. The results of the SAED analysis of pure TiO2 and surfactant-modified TiO2 nanoparticles indicated planes (101), (112), (200), and (204), which provide credence to the conclusions that were generated by the XRD study. An experiment was carried out by Sreethawong and colleagues (2006), which included the creation of nanoparticles of titanium dioxide using a sol-gel method. When they used transmission electron microscopy, they discovered nanoparticles that were irregular in shape.

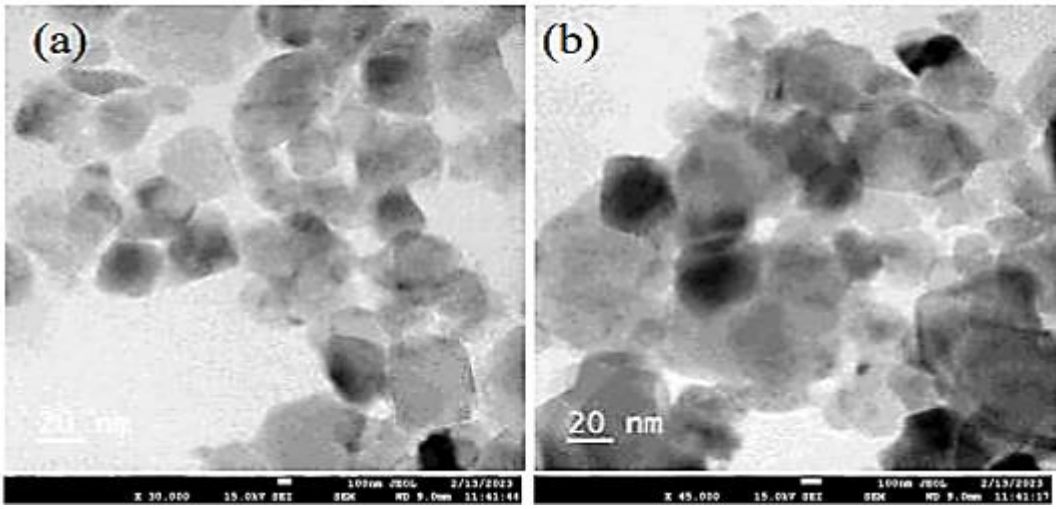
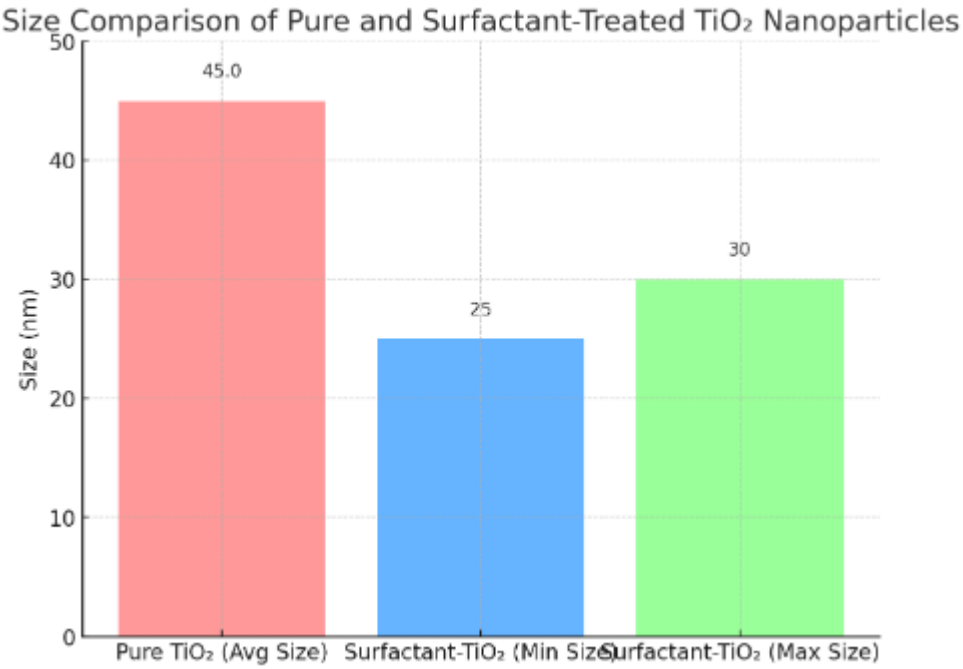


Figure no. 20: TEM images of TiO_2



7. DISCUSSION:

7.1. POTENTIAL APPLICATIONS OF NANOMATERIALS IN WATER AND WASTEWATER TREATMENT

7.1.1. ZEROVALENT METAL NANOPARTICLES

In the context of this discussion, the phrase "zerovalent metal nanoparticles" refers to iron atoms that are electrically neutral. This is a characteristic that Metallic Iron has by its very nature. It has the potential to be hazardous due to the fact that it alters oxidised materials or diminishes bulk dimensions. In order to remove pollutants from contaminated sediments and wastewater, zerovalent metal nanoparticles are an excellent choice because of their visual, mechanical, magnetic, and electrical properties, as well as their catalytic capabilities. The elements iron, zinc, and silver are all examples of nanoparticles that are made of zerovalent metals and have zerovalent values. Take a look. As an example, this demonstrates the types of nanoparticles that do not carry any charges.

Figure no. 21: Zerovalent nanoparticles used in wastewater management. ROS reactive oxygen species

7.1.2. ZINC NANOPARTICLES

Paints, plastics, sunscreens, cosmetics, and nutritional supplements all include zinc nanoparticles, which are also referred to as Zn NPs. These nanoparticles are made up of very tiny particles. Despite this, it is not always simple to understand how these particles are released into ecosystems by the environment. Recent technological improvements have resulted in a rise in the production of zinc nanoparticles in the European Union, which has reached 1.6 metric tonnes. The extraordinary ability of zinc nanoparticles to reduce compounds more effectively than iron nanoparticles, their considerable capacity for negative reduction, and their extensive use in wastewater treatment are all reasons why zinc nanoparticles are an alternative to iron nanoparticles. This means that the pace at which pollutants are broken down by nanoparticles of zerovalent iron (ZVI) is slower than the rate at which zinc particles alone are broken down. In the majority of the research investigations that have been conducted on the treatment of Zn NP wastewater, dehalogenation procedures have been used. A number of studies have been conducted to investigate the effect that zinc nanoparticles (Zn NPs) have on the functioning of conventional activated sludge (CAS) wastewater treatment systems. The growth of ammonium-oxidizing bacteria was shown to be suppressed by zinc nanoparticles at a concentration of 5.0 mg/L, according to the findings of another laboratory investigation. Zinc nanoparticles at a concentration of 13.1 mg/L were found to inhibit the development of nitrifying bacteria in a sequencing batch reactor (SBR), which resulted in a decrease in the removal of NH4 +-N. Zinc nanoparticles have been used to break down octachlorodibenzo-p-dioxin into its chlorinated congeners in an atmospheric environment. Over the course of several research, it has been shown that zinc nanoparticles, also known as Zn NPs, have beneficial impacts on a variety of chemical compounds. Nevertheless, these benefits are only apparent in compounds that possess halogen or chlorinated groups. When taken as a whole, the notion that zinc nanoparticles are capable of removing other contaminants is not supported by a significant amount of research.

Figure no. 22: Zinc nanoparticles (Zn NPs)

7.1.3. IRON NANOPARTICLES

ZVI nanoparticles have been proven to be capable of detoxifying a variety of substances, including vinyl chloride, carbon monoxide, lindane, carbon tetrachloride, and trichloroethane, according to research. On-site wastewater treatment is thus made possible by their availability as a feasible option. Reductive dehalogenation has been used in the vast majority of wastewater testing due to the fact that it is not only very successful but also an extremely economical method. In addition, iron nanoparticles are very effective electron donors for other particles than other particles. When Fe(II) and H2 are oxidised by protons or water molecules in anaerobic conditions, the result is the production of both of these elements. As a consequence of this process, these components are discovered in wastewater in amounts that are lower than normally seen. In the event that they come into contact with pollutants, the oxidation of iron(II) to iron(III) that takes place results in the production of iron(OH)3. Organic and inorganic contaminants are removed from ecosystems by the flocculation activity of Fe(OH)3, which involves the presence of Fe(OH)3. In accordance with ZVI nanoparticles are responsible for the release of two electrons into an oxygen molecule. This, in turn, leads to the formation of hydrogen peroxide, which is subsequently reduced to water.

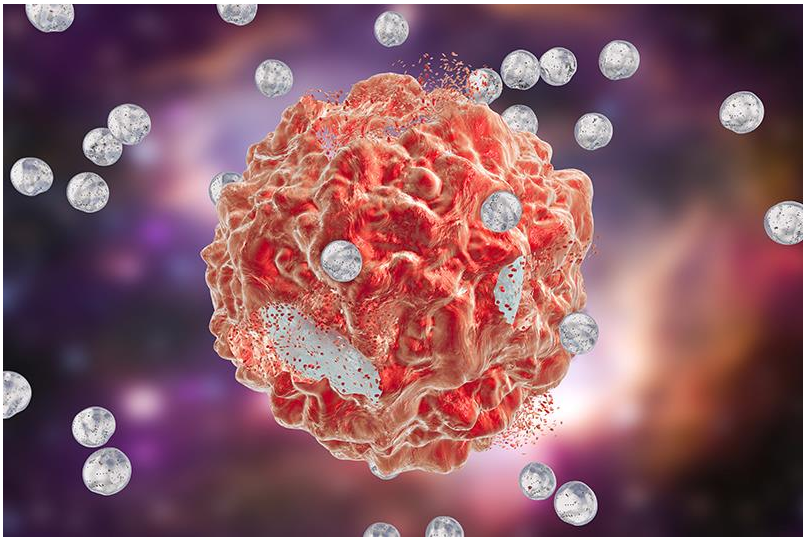


Figure no. 23: Iron Nanoparticles

There are many different organic compounds that go through the process of oxidation, which ultimately leads to the generation of hydroxyl radicals as a consequence of the interaction between hydrogen peroxide and iron (II). It is possible to remove a wide variety of pollutants from nanomaterials by employing the processes of oxidation, precipitation, reduction, and adsorption. These pollutants include nitroaromatic compounds, inorganic anions, phosphates, radio elements, nitrates, phenols, organic dyes, chlorinated and halogenated organic compounds, and nitrates. These operations are able to be carried out anywhere in the globe. Zinc oxide (ZVI) nanoparticles have a tendency to oxidise, cluster, and separate poorly from the pathway that breaks down pollutants, despite the fact that they offer a number of advantages.

With the goal of improving the effectiveness of ZVI nanoparticle molecules, a great number of innovative ways have been developed. Some of these approaches include doping with significant metal ions, conjugation with supports, surface coating, and emulsification. Emulsification is another technique. Innovative techniques ought to enhance the reactivity, aggregation, and dispersibility of ZVI while simultaneously preventing its dissociation from the wounded system.

7.1.4. SILVER NANOPARTICLES

To protect against a broad variety of illnesses, viruses, fungi, and bacteria, silver nanoparticles, also known as Ag NPs, are used in the process of disinfecting water. This is due to the fact that silver nanoparticles possess antibacterial capabilities. The permeability of bacterial cell membranes is improved when free radicals bind themselves to the membranes. The apoptosis process begins when the cell membrane breaks down and allows the cell to die. Both phosphate and sulphur are broken down by silica nanoparticles when they are present in DNA. Mahesh and his colleagues conducted a research in 2022, and the findings of that study show that the antimicrobial Ag+ ions that are produced when Ag NP is dissolved have the potential to cause the thiol group of essential enzymes to go dormant, which in turn disrupts the normal processes that occur in living organisms.

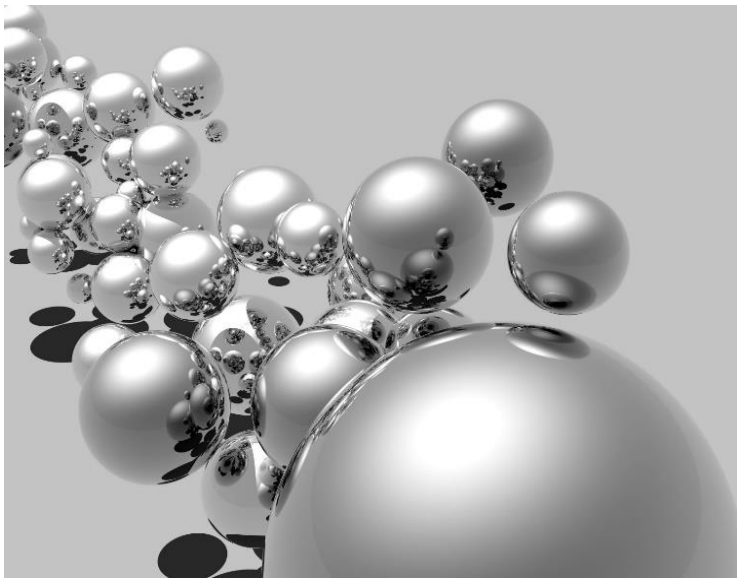


Figure no. 24: Silver nanoparticles

Because they have a tendency to gather in liquid media, their use for extended periods of time is restricted. effectiveness of antimicrobial agents intended against E. The presence of coli was discovered via the use of cellulose fibres that had been immobilised with silver nanoparticles and so utilised in in situ remediation procedures. I.e. E. coli, E. bacteria that have been rendered inactive by a filtering system, including faecalis bacteria of course. Through the process of polyether sulfone (PES) microfiltration (MF), silver nanoparticles that have been chemically reduced are introduced into the membranes that are used. The quantity of germs that were present on the membranes was found to have significantly decreased. In the process of treating wastewater, it has been shown that PES-Ag NP membranes possess powerful antibacterial properties and are highly effectively effective. During this process, nanoparticles are responsible for the destruction of a crucial cell component or the membrane structure of the cell. This occurs in an attempt to eliminate germs that are not desired.[29]

8. CONCLUSION AND SUMMARY

Nanotechnology has the potential to be used in the development of a novel structure, device, or system that incorporates better electrical, optical, magnetic, conductive, and mechanical capabilities. This is accomplished by manipulating matter at the molecular and atomic levels. There are several applications that have shown the immense potential that nanotechnology has, including the treatment of wastewater. The use of nanostructures as redox active media and catalysts for wastewater purification has a great deal of potential due to their diminutive size, large surface area, and ease of functionalization. The use of nanomaterials has the potential to eliminate heavy metals, organic and inorganic solvents, biological and colour impurities, and bacteria that are responsible for cholera and typhoid infections from wastewater.

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