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MULTI-ITERATIVE GREEN HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF CERIUM OXIDE NANO STRUCTURED MATERIALS

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ABSTRACT
Cerium oxide (CeO_2) or ceria is the most promising material due to its unique physical and chemical properties among the other materials. The CeO_2 nanostructures are significant nanomaterials used for different applications such as electrochromic devices, environmental catalysis, cosmetics, pharmaceuticals, solar cells, gas sensors, fuel cells, oxygen pumps, etc. The conventional nanomaterials synthesis methods namely solvothermal and solution combustion procedures are non-friendly and high-cost chemicals, applicable to small-scale industrial applications. Therefore, there is a need to develop a novel synthesis of nanostructured particle methods with eco friendly and cost-effective. In this paper, a novel synthesis of CeO_2 nanoparticles using the Multi-iterative Green Hydrothermal Synthesis method is developed. The designed hydrothermal synthesis methods are simple, direct, cost-effective, eco-friendly, and applicable on a large industrial scale. The Multi-iterative Green Hydrothermal Synthesis method uses the Centella Asiatica (CA) and Indigofera Tinctoria (IT) aqueous leaf extract as surfactants for different textured morphologies green synthesizes of CeO_2 Nano Superstructures (NSS) at low temperatures. The green synthesizes of CeO_2 nanoparticles are used in biomedical applications. The subcritical water extraction method is applied to extract the aqueous leaf and exploited as a 'green principle' for the synthesis of CeO_2 nanoparticles. With the use of aqueous leaf extracts, the synthesis of CeO_2 is carried out by applying the multi-iterative green hydrothermal synthesis. The cerium ammonium nitrate solution and aqueous leaf extract solution are stirred and heating the reaction system at a particular temperature to obtain the synthesized CeO_2. The green synthesizes of CeO_2 provide significant advantages including improved crystallinity, reduced particle size, and surface area of superstructures in a

minimum time. After that, the synthesized CeO_2 samples are characterized by using different techniques such as X-ray diffraction (XRD), Fourier transforms infrared (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV- Visible spectroscopy (UV-Vis) to identify the structural, optical and surface morphological properties. The observed results indicate that the hydrothermal methods attain more efficiency in the synthesis of CeO_2 nanoparticles by means of a cost-effective and preserve their catalytic quality under large-scale industrial environments than the solvothermal and solution combustion synthesis methods.

Keywords: Synthesis of CeO_2 nanoparticles**,** Multi-iterative Green Hydrothermal Synthesis method, subcritical water extraction method, aqueous leaves extract, synthesized CeO_2 characterization

1. INTRODUCTION

Metal and nanoparticles (NPs) are developing active ingredients, in the field of nanoscience and nanotechnology. The nanoparticles (NPs) have exposed great attention owing to their high surface area, high reactivity, unique size and shape, and particle morphology. Nanoparticles utilize nano-size structures in the ranges between 1 to 100 nm. These nano-scale structures have unique physiochemical properties and it applied in various fields of physics, biology, and chemistry. Therefore, the nanoparticles are utilized as promising tools in numerous sectors such as consumer products, pharmaceutics, cosmetics, and agriculture.

Various types of inorganic nanoparticles have been used due to their physicochemical properties. Among these nanoparticles, cerium oxide nanoparticle is significant consideration due to their promising applications such as microelectronics, optoelectronics, fuel cell technologies, gas sensors, medicine, etc. The nanoparticles are relatively stable and low-cost.

Synthesis refers to the process of creating the nanoparticles. This method has the ability to provide a uniform size, shape, and well-distributed nanomaterials. This method is employed to synthesize the cerium oxide nanoparticles. Green synthesis is the method of creating nanoparticles that involve the use of plant or plant parts. The process of green synthesis is more efficient, simpler, and economical, and it is simply scaled up to perform larger operations. Plants are said to have more benefits such as easy accessibility, safe handling, etc. Moreover, the nanoparticles created by green synthesis are more stable and it increases the speed of the CeO_2 nanostructure materials synthesis process than the other microorganisms. Many routes have been developed to green synthesize the CeO_2 nanostructure materials, such as sol–gel process, chemical vapor deposition, electrochemical deposition technique, microemulsion method, template method, solvothermal, solution combustion, and hydrothermal synthesis. Among these different methods, hydrothermal synthesis is a one-step low-temperature synthesis process with more efficient and economical routes.

Solvothermal synthesis method was developed in [1] for cerium oxides nanoparticles preparation and manufacturing and it used for emerging applications. But the designed targeted synthesis of novel materials difficult to include more in-depth structural characterization to provide appropriate structure property correlations. A one-pot, urea solution combustion method was developed in [2] to synthesize highly catalytic CeO_2 nanoparticles under varying thermal conditions were characterized.

The CeO_2-NPs were effectively synthesized and characterized in [3] by means of Solanum nigrum leaf extract. This leaf extract was used to control and stabilize the agents in the biosynthesis of CeO_2 nanoparticles. However, the accurate leaf extraction process was not performed to minimize the time consumption of CeO_2 synthesis. A hydrothermal method was developed in [4] for the synthesis of CeO_2 nanoparticles. But the designed method was not used for sensing and detecting hazardous chemicals.

A green precipitation scheme was introduced in [5] for the synthesis of cerium oxide nanoparticles with the use of Moringa oleifera leaf extract with a small reaction time. But the designed scheme was not efficient for biomedical applications. The synthesis and characterization of CeO_2 nanoparticles were performed in [6] using the Abelmoschus esculentus leaf extract. This medicinal plant extract operates as both a reducing and stabilizing agent. But the designed synthesis method failed to provide a fruitful platform to explore the green synthesis of nanoparticles in different biomedical therapeutics.

The cerium nanoparticles were synthesized in [7] using the green method by means of origanum Majorana leaf extract. But the designed leaf extract was promising nanoparticles but it did not apply in nanomedicine to treat cancers. A Citrate-coated nanoceria particle was created in [8] by the use of hydrothermal synthesis to remove the unreacted cerium ions and salts. The different characterization techniques were applied to find the particle size and morphology, crystalline structure, and properties. This extensive physicochemical characterization of a cleansed nanoceria that failed to apply in the selection and interpretation of results in the material, chemical, and medical applications

A pseudocapacitive electrode materials synthesis was carried out in [9] by using a safe co-precipitation method to enhance the performance of environmentally friendly cerium oxide (CeO_2) synthesis. However, the obtained results reported in this method indicate that the enhancement of the electrochemical properties of oxide materials was not achieved by simply modifying the electronic structures and introducing Frenkel defects into the materials.

Synthesis and Characterization of Cerium Oxide saturated Titanium Oxide Photoanodes was introduced in [10] for improving the efficient Dye-Sensitized Solar Cells (DSSC) application. However, CeO_2/TiO_2 based photoanode was not useful for energy-efficient DSSCs and relevant applications.

1.1 Contributions

From the analyses of existing work, a novel multi-iterative green hydrothermal synthesis methodis introduced with the following contributions.

- To improve the synthesis of CeO_2 nanoparticles, a novel multi-iterative green hydrothermal synthesis methodis developed based on aqueous leaf extraction, CeO_2 synthesis and characterization.
- First, subcritical water extraction method is applied for aqueous leaf extraction to perform hydrothermal synthesis of CeO_2 with minimum time consumption.
- Secondly, multi-iterative green hydrothermal synthesis process is carried out with the aqueous leaf extract and cerium ammonium nitrate solution to get the CeO_2 nanoparticles. The reaction process is repeated multiple times to get the optimum ratio of two solutions hence it called as multi-iterative synthesis method.
- The synthesized CeO_2 nanoparticles are characterized with different methods to identify the structural and surface morphological properties.
- Finally, results and discussions is carried out to estimate the performance of our multi-iterative green hydrothermal synthesis and other synthesis method.

1.2 Organization of the article

The article is organized into different sections as follows. Section 2 reviews the related works. Section 3 provides a brief description of the Multi-iterative Green Hydrothermal Synthesis method with a neat diagram. Section 4 describes the performance results and discussion of Hydrothermal Synthesis method and existing methods. At last, Section 5 concludes the paper.

2. RELATED WORKS

A two-shaped cerium oxide nanoparticles (NPs) were synthesized in [11] by using a hydrothermal approach by changing the reaction temperatures. However, the shape-dependent findings were not efficient to attain many optimized and controlled outcomes. A microwave-assisted hydrothermal synthesis method was developed in [12] with small reaction times. The microstructural, dilatometric, and electrical characterization of CeO_2 was obtained. But the designed synthesis technique was not an effective approach to decreasing the sintering temperature of CeO_2 ceramics with enhanced electrical properties.

Cerium oxide nanoparticles (CeO_2 -NPs) were synthesized in [13] using Acorus calamus aqueous extract and tested. The findings underline the potential of CeO_2 nanoparticles for both positive and negative bacterial agents. But the new alternative antimicrobial agent was not exploited. CeO_2 NPs are synthesized [14] through precipitation with mixed water-alcohol solutions. The synthesized CeO_2 NPs were not normalizing the oxidative stress in the biological environment

A new method was developed in [15] for the synthesis of cerium oxide nanoparticles by means of Dillenia indica aqueous extract. However, the cost-effective green synthesis method was not applied for the mass production of CeO_2 without environmental pollution. A CeO2/reduced graphene oxide (rGO) nanocomposites were synthesized in [16] by using a simple one-pot hydrothermal method. A CeO_2 nanoparticles powder was synthesized [17] by using leaf extract acquired from tea waste. A facile hydrothermal preparation method was developed in [18] for CeO_2 nanocomposites synthesis.

However, better stability was not estimated to cause potential environmental applications. A hydrothermal synthesis method on the Physio-chemical characteristics of cerium oxide was developed in [19] from salt solution using ammonia precipitation. A mesoporous CeO2 nanosheet synthesis was introduced in [20] by using cerium nitrate impregnation and calcination with tree leaves as the pattern.

3. METHODOLOGY

CeO_2 -based materials are important to fast ion-oxide conductors among the most promising nanoparticles. The size of the nanoparticle ranges from 1–100 nm and it has unique physiochemical properties and is utilized in a variety of fields of physics, biology, and chemistry. The nanoparticles are of great attention because of their high surface area, high reactivity, unique size and shape, and element morphology. Therefore, the nanoparticles are considered promising tools for their optimistic impact in improving various sectors of the economy, consumer products, pharmaceutics, cosmetics, medicine, and agriculture. The nanotechnology includes different flexible nanomaterials such as AgO_2, $Al2O_3$, MgO, ZrO_2, TiO_2, ZnO, CeO_2, and Fe_2O_3. It is mainly applied to a wide range of applications such as catalysts, sensors, solid oxide fuel cells, sunscreen cosmetics, bioimaging, biotransformation, antibacterial movement, drug delivery livelihoods, and anti-parasitic gels.

Synthesis of nanoparticles is an emerging field of nanoscience and technology due to their physical, chemical, electrical, and optical properties. The process of nanoparticle synthesis reduces the hazards to the global attempts and implantation of sustainable processes. In particular, a CeO_2 synthesis is considered technologically important due to its versatile applications. The CeO_2 synthesis is mainly included two methods, such as physical and chemical. However, these methods are toxic and unstable, making them less efficient. Therefore, a safe, less toxic method has been developed called Green Synthesis. These methods make use of different biological resources such as plants, microbes, or any other biological derivative.

Plants are identified to acquire a wide range of bioactive compounds that are used for the green synthesis of metal nanoparticles. It reduces the toxicity of nanoparticles instead of chemical agents. Conventional techniques such as solvothermal, solution combustion methods has been developed for CeO_2 nanoparticle synthesis

3.1 Solvothermal Method based CeO_2 Synthesis

The solvothermal method was applied for CeO_2 synthesis and producing chemical compounds, in which a solvent including a reagent is positioned under high pressure and temperature in an autoclave. Here the solvents were used other than the water such as ammonia, carbon dioxide, dimethylformamide, and various alcohols such as methanol, etc. Among the various synthesis methods, the solvothermal process was the most promising synthetic route due to its minimum cost, high efficiency, and good crystallization of the product.

Applying the solvothermal method, where reagents were heated at high pressure and temperature i.e. above the boiling point of the solvent to induce nanoparticles directly from the solution. This method produces nanomaterials of varying sizes and unusual compositions with nanoparticle morphology

By applying a solvothermal method for CeO_2 synthesis, the following experimental settings were utilized. First, the Cerium acetate hydrate with 20 mmol is dissolved in the solvent of 120 mL of butanediol or ethylene glycol. After that, these two solutions were mixed and set in an autoclave. The autoclave was completely cleaned with nitrogen and heated at the temperature for 2 hours. The reaction in the presence of octanoic acid provided a clear solution, and a product was obtained by the addition of 90 mL of deionized water to the solution. The products were cleaned with methanol and calcined in the air at the temperature for 30 minutes. Finally, the CeO_2 samples are obtained by the solvothermal reaction.

3.2 Solution Combustion Method Based Ceo2 Synthesis

The solution combustion method was also applied to produce simple and complex nanoparticles with a preferred morphology of size and shape. The advantages of the solution combustion method were to minimize the cost, simplicity, and energy-efficient synthesis. The combustion synthesis reaction was carried out by using fuel and the fuel-to-oxidizer as a solvent.

CeO2-nanoparticles were synthesized with cerium nitrate as an oxidizer and Ammonium nitrate was used as a fuel since it is low-priced and rich in nitrogen, hydrogen, and oxygen. During the combustion process, the Ammonium nitrate released corresponding oxides and no carbonate species were formed.

First, all glassware used in the experiments was cleaned with ethanol, washed thoroughly using distilled water, and dried before use. Double distilled water was used in all experiments. The CeO2 nanoparticles were synthesized by the combustion of aqueous solutions including cerium nitrate, glycine, and ammonium nitrate.

These three aqueous solutions were mixed in glassware and applied to a temperature of 450°C and it undergoes combustion with fire to produce the ceria. Therefore, the combustion process for CeO_2 nanoparticle synthesis as given below,

$$Ce(NO_3)_3.6\,H_2O + 2C_2H_2O_2\,N + NH_4NO_3 \rightarrow CeO_2 + 4CO_2 + 13H_20 + \frac{7}{2}N_2$$

From the above equation, the combustion reaction was done completely and obtains the formation of ceria ($Ce(NO_3)_3$) with ammonium nitrate (NH_4NO_3) and glycine ($C_2H_2O_2\,N$). The obtained nanoparticles were re-washed in distilled water. Finally, the product was dried in hot air and further cooled in a room temperature.

Thee conventionally designed methods are complex, time-consuming, expensive, and hazardous. Considering all the above issues, there is a need to develop an environmentally benign green route and search for new catalysts for the synthesis of CeO_2. Therefore, multi-iterative green hydrothermal synthesis methods aimed at the development of new methods for simple, lesser time-consumption and cost-effective.

3.3 Multi-iterative Green Hydrothermal Synthesis method based CeO_2 synthesis

The Multi-iterative Green Hydrothermal Synthesis is a novel method to crystallize the nanoparticles from high-temperature plant aqueous solutions at high vapor pressures hence it is called a green hydrothermal method. Biological ways of synthesizing CeO_2 nanoparticles using plant extracts are eco-friendly green synthesis other than the chemical and physical methods. Green synthesized nanostructured materials play a significant role in real-time applications, including biomedical and environmental.

Plants used in the synthesis process are said to include many advantages such as easy availability and safe handling and it contains a wide variety of biomolecules such as alkaloids, terpenoids, phenols, flavonoids, proteins, etc. These are used for mediating the synthesis of nanoparticles. Moreover, plant based nanoparticle synthesis is more stable and the speed of synthesis is earlier than the other microorganisms.

A green hydrothermal method is applied for CeO_2 synthesis with the use of two plants such as Centella Asiatica (CA) and Indigofera Tinctoria (IT). In the hydrothermal method, the water is used as a solvent to dissolve the Cerium Ammonium Nitrate (CAE) and the aqueous leaf extracts surfactant from the two plants CA and IT and it is used for the green synthesis of CeO_2 at low temperatures. Compared to the other two methods namely solvothermal and solution combustion, the Multi-iterative green hydrothermal synthesis method performs efficient CeO_2 nanoparticles synthesis with low temperature, cost-effective, and minimum time consumption. Here, the Multi-iterative represents the solution of the CAE and the aqueous leaf extracts solution is taken in different ratios and perform multiple iterations to get the optimal ratio for synthesizing the CeO_2 nanoparticles.

Figure 1 reveals an architecture diagram of the multi-iterative green hydrothermal synthesis method to perform three different processes namely aqueous leaf extraction, CeO_2 synthesis, and characterization. The methodology considers the input as biological resources or microorganisms such as two types of plants namely Centella Asiatica (CA) and Indigofera Tinctoria (IT). Synthesis of nanoparticle methods using different plant extracts is very cost-effective and it has several advantages such as easy availability, safe handling and handles a broad range of biomolecules. In addition, nanoparticles produced by plants are more stable and the rate of synthesis is faster than the use of other microorganisms.

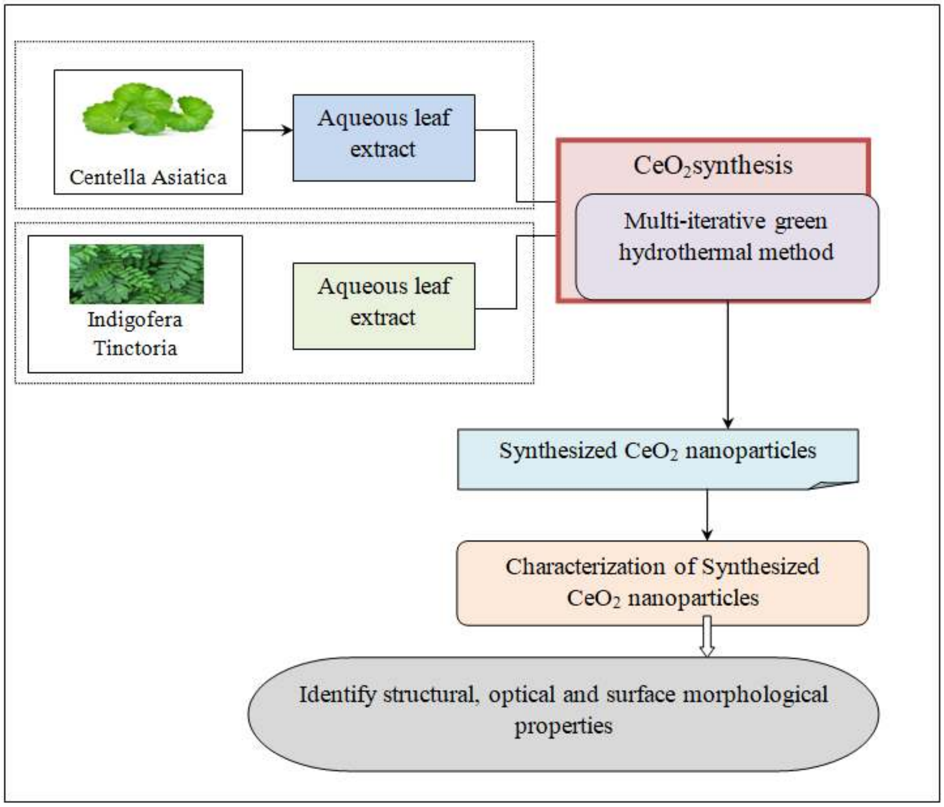

Figure 1 Architecture of the Multi-Iterative green Hydrothermal Synthesis Method

Centella Asiatica is a perpetual herb used in Ayurvedic medicine to treat various disorders. It contains numerous energetic components such as triterpenoids, saponins, volatile oils, flavonoids, tannins, phytosterols, amino acids, and sugars. Fresh leaf extracts and active compounds possessed various pharmacological effects, such as anti-oxidant effects, cardioprotective effects, antihypertensive effects, anti-cancer activities, etc. Another plant is Indigofera Tinctoria used for the synthesis of CeO_2 metal nanoparticles from their respective salt by using aqueous leaf extract. This leaf extraction plays a double function as stabilizing and minimizing agent for the construction of nanoparticles.

The aqueous leaf solution is extracted and performed by using the Subcritical water extraction method from the two types of plants such as Centella Asiatica and Indigofera Tinctoria. After the extraction, the synthesis method called h multi-iterative green hydrothermal synthesis is applied. First, the CeO2 is synthesized with a low-temperature hydrothermal process. This kind of synthesizing process uses the solvents as water under high pressure. The particle growth is performed in an apparatus called an autoclave, in which a nutrient is provided along with water. A temperature is maintained between the opposite ends of the growth chamber. At the hotter end, the nutrient solute melts, while at the cooler end, it is deposited on a seed crystal. The main advantage of the hydrothermal-based CeO2 synthesis is also particularly suitable for the development of large good-quality nanoparticles while maintaining control over their composition. The different processes of the CeO2 synthesis and the characterization processes are discussed in the following subsections.

3.3.1 Multi-iterative green hydrothermal synthesis method with Centella Asiatica (CA)

The CeO2 nano superstructures with different textured morphologies are synthesized by applying the novel Multi-iterative green hydrothermal method at low temperatures using Centella Asiatica (CA) leaves extract as surfactants. Centella Asiatica (CA) is a medicinal herb used to reduce the oxidative stress related cardiovascular diseases. The use of plant aqueous leaf extracts provides significant advantages to improving the crystallinity and minimizing the particle-size and performing synthesis of larger surface area with lesser time consumption. Therefore, this synergistic route of surfactant-mediated hydrothermal synthesis of CeO2 provides a simple and cost-effective synthesis and it applied to potential therapeutic mechanism in the treatment of oxidative stress related cardiovascular diseases. The flow process of the CeO2 Hydrothermal synthesis is shown in figure 2.

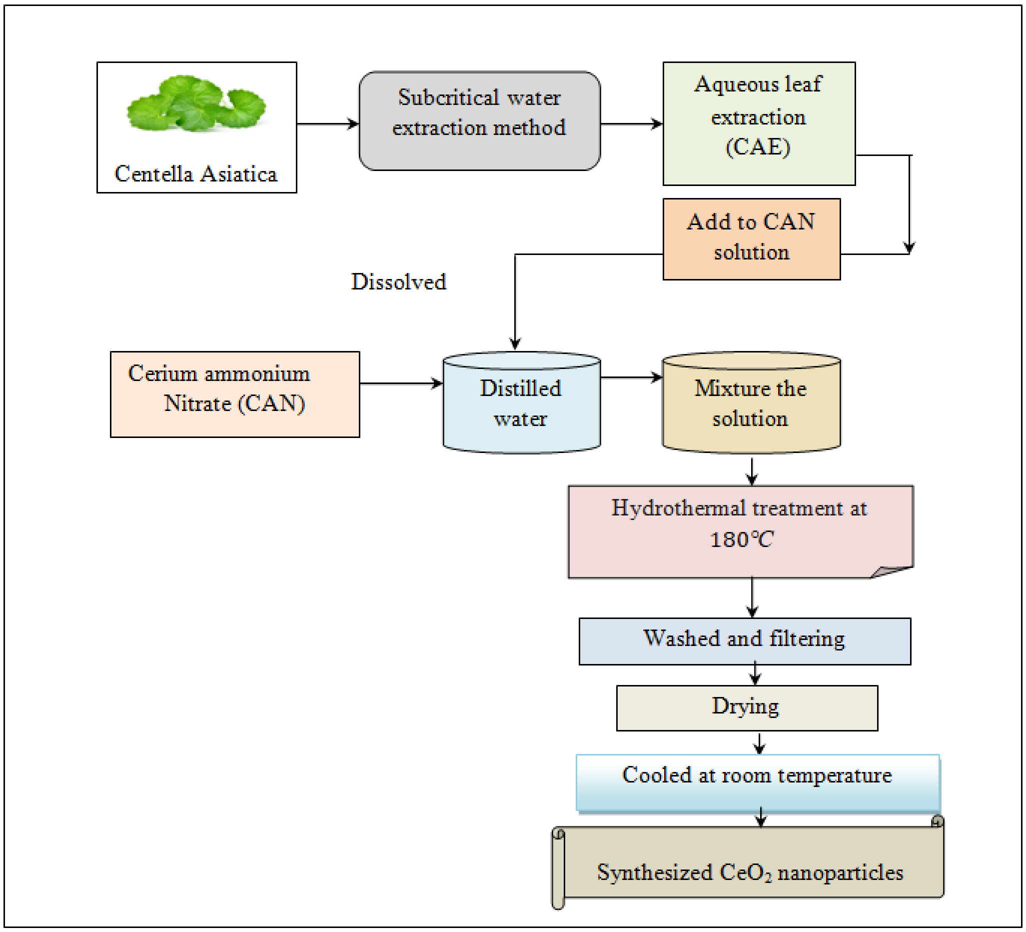

Figure 2 Synthesis of CeO_2 nanoparticles with Centella Asiaticasurfactant

As shown in above figure 2, the process of CeO_2 nanoparticle synthesis is carried out with Centella Asiatica as a surfactant. First, the Centella Asiatica (CA) leaves are used as surfactant and an aqueous leaf solution is extracted by using the subcritical water extraction method. This extraction method is an effective approach that uses water as the solvent at high temperatures as well as pressures to attain the safe, rapid, and efficient extraction of the required aqueous leaf compounds from the CA plant. In addition, these extraction processes are used to get highly purified aqueous leaf solution compounds.

Subcritical water extraction is a method that altered the physical properties of water in high pressure and increases the temperature above its boiling point (up to *374℃*) to preserve the water in its fluid state, hence improving it as an extraction solvent. Compared to other extraction methods, subcritical water extraction is an efficient, harmless, and eco-friendly method to extract the aqueous compounds from leaf samples. In addition, this method utilizes a lesser extraction time and acquires a higher quality of extraction.

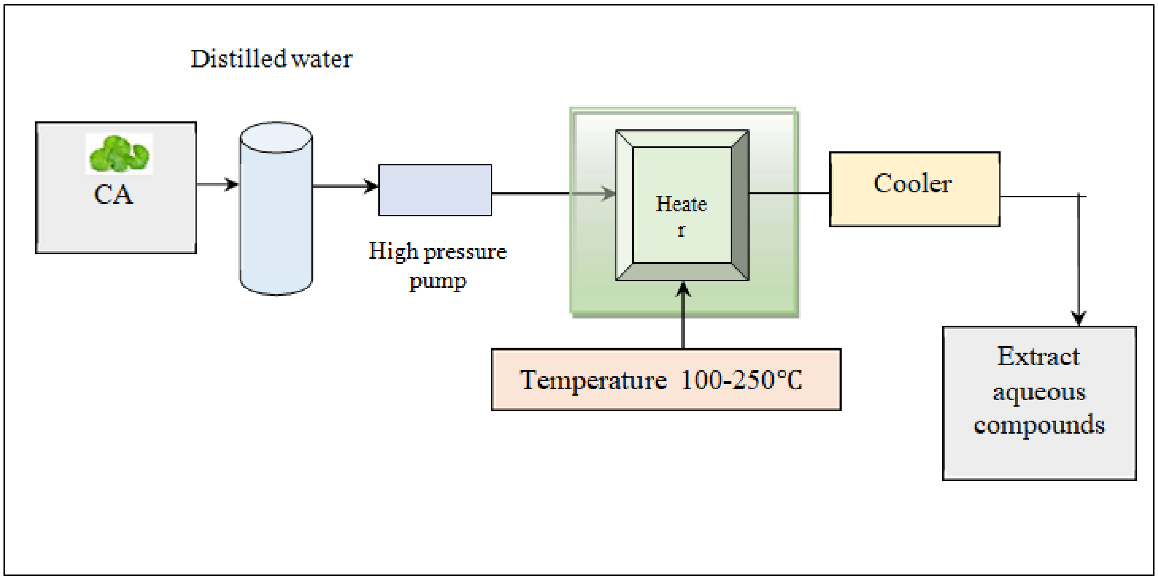

Figure 3. Basic diagrams for the subcritical water extraction process

The basic diagram for an extraction process used for the plant is presented in Figure 3. Then, the solute compound is transferred into the solvent (i.e. distilled water). This type of extraction process manages three important parameters such as pressure, time, and temperature. The extraction setup consists of a solvent reservoir with a high-pressure pump to push the water into the system. The pump is used to pump the water into the heater. The optimum extraction conditions of bioactive components are at 100-250 and pressure 10-40MPa. The extraction yields of bioactive components get increased while increasing the temperature and pressure. Under these conditions, 5 mg/g of aqueous compounds from leaf samples are extracted.

For the synthesis, 2.5 mg/g of Cerium Ammonium Nitrate (CAN) is dissolved in the distilled water. The 5 mg/g of aqueous leaf extract solution is added drop wise to the cerium ammonium nitrate solution. In order to optimize the construction of CeO_2nanoparticles, different ratios of CAE and CAN (1: 1 to 1: 9) are iteratively tested with multiple times. The main aim of the multi-iterative refers to a process where the design of a product is improved by repeated testing. After finding the optimal ratio, these two solutions are stirred for 15 minutes. Then the stirred solutions are transferred into hydrothermal treatment with external temperature at 180℃ and it maintained for 12 hours. The final obtained particles are re-washed at 2-3 minutes with the distilled water and filtered. The product is dried in hot air oven for 2 hours and further cooled to room temperature. Finally, the uniformly sized CeO2 particles are obtained to determine physicochemical and biocompatible properties. Therefore, green synthesis of cerium nanoparticles with the Centella Asiatica (CA) leaves extract solution, are efficiency against cardio myoblast hypertrophy treatment in the biomedical applications.

3.3.2.CeO_2Hydrothermal Synthesis with Indigofera Tinctoria (IT)

The synthesis of CeO_2 nanoparticles is carried out by the Multi-iterative green hydrothermal synthesis method with the Indigofera Tinctoria (IT) aqueous leaf extracts surfactant. This process is similar to the process of CeO_2 nanoparticle synthesis with the Centella Asiatica method. The fresh leaves are collected from the Indigofera Tinctoria plants. It is cleaned thoroughly with water and then with double distilled water to remove any soil and other impurities on it. The leaves are partitioned into small pieces and applied to a subcritical water extraction method.

The obtained aqueous compounds leaf extracts is then cooled and filtered. In order to perform the synthesis, Cerium Ammonium Nitrate (CAN) with the concentration of 2.5 mg/g of is dissolved in the distilled water. Then the extracted aqueous from the Indigofera Tinctoria (IT) plant and the CAN solution are stirred for 15 minutes and it applied to a Hydrothermal treatment with the temperature of 180°C for 12 hours. Finally, the obtained particles are filtered for 2-3 minutes in distilled water and get the superstructure CeO_2 nanoparticles.

3.4 Characterization of Synthesized CeO_2 nanoparticles

After the synthesis of the CeO_2 nanoparticles with the Centella Asiatica and Indigofera Tinctoria plants, the characterization process is said to be performed to identify the structural and morphological properties of the cerium oxide nanoparticles. There are different characterizations techniques are used to understand the structural, optical, and surface morphological properties of all synthesized samples from the hydrothermal route method. The different techniques are X-ray diffraction (XRD), Fourier transforms infrared (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and UV-Visible spectroscopy (UV-Vis).

The characterization of the synthesis of the CeO_2 nanoparticles with the Centella Asiatica leaf extract is used as potential tool against cardiomyoblast hypertrophy treatment. Cardiomyoblast hypertrophy is a thickening of the heart muscle that occurred when cardiac muscle mass increases due to extended and enlarged stress on the heart. Therefore, the cardiac hypertrophy is a major cause of mortality by the means of heart failure and sudden death. Hence, prevention of hypertrophy is a significant therapeutic target. Oxidative stress is an important cause of cardiac hypertrophy. The characterization of synthesized CeO_2 nanoparticles with the Centella Asiatica leaf extract is used for reducing the oxidative stress.

The X-ray diffraction (XRD), Fourier transforms infrared (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and UV-Visible spectroscopy (UV-Vis) characterization of the green synthesis of the CeO_2 nanoparticles with the Indigofera Tinctoria leaf extract is examined to treat the lung cancer. The abnormal cancerous cell is removed due to the reactive oxygen species induced by CeO_2 nanoparticles, it causes damages to the cellular component and leads to the cell death. Therefore, the CeO_2 nanoparticles with the Indigofera Tinctoria leaf provide the good anticancer properties against lung cancer. Due to these properties, synthesized CeO_2 nanoparticles have remarkable applications in biomedical field and it effectively used as a powerful tool against cancer.

3.5 Aqueous gamma-Radiation Dosimeter for synthesized CeO_2 nanoparticles

The CeO_2 nanoparticles synthesized by a Multi-iterative Green Hydrothermal Synthesis method are investigated for the purpose of developing a new gamma-Radiation Dosimeter. This dosimeter is used as an extremely sensitive and cost-effective in ultra-low-dose environment. This dosimeter is used to identify the radiation properties of nanoparticles. The CeO_2 thin films are exposed to gamma rays with an average energy of 1.25 MeV, radiation activity rate 7.328 kGy/h in a closed chamber. The thin films placed vertically at a distance of 6.5 cm from the source active area. The thin films were exposed for 207, 415 and 828 minutes to obtain the required doses.

4. RESULTS AND DISCUSSIONS

In this section, performance of the CeO_2nanoparticles synthesis is analyzed with three different methods namely Multi-iterative Green Hydrothermal Synthesis method, solvothermal and solution combustion method. The results of different synthesis methods are analyzed with different metrics such as extraction yield and the different CeO_2 characterization are X-ray diffraction (XRD), Fourier transform infrared (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV-Visible spectroscopy (UV-Vis) and Aqueous gamma-Radiation Dosimeter. The performance are analyzed with the help of a table and graphical representation.

4.1 Performance analysis of extraction yield

Extraction yields of Centella Asiatica (CA) and Indigofera Tinctoria (IT) plant aqueous leaf surfactantare analyzed with the varying temperature using Multi-iterative Green Hydrothermal Synthesis method. The extraction yield is measured in terms of milligram per gram (mg/g) by varying the temperature in degree Celsius (℃).

Table 1 Performance of extraction yields of Centella Asiatica (CA) and Indigofera Tinctoria (IT)

Temperature (℃)	Extraction yields (mg/g) using (CA)	Extraction yields (mg/g) using (IT)
100	0.2	0.3
150	1.8	2
200	3	3.2
250	5	5.1

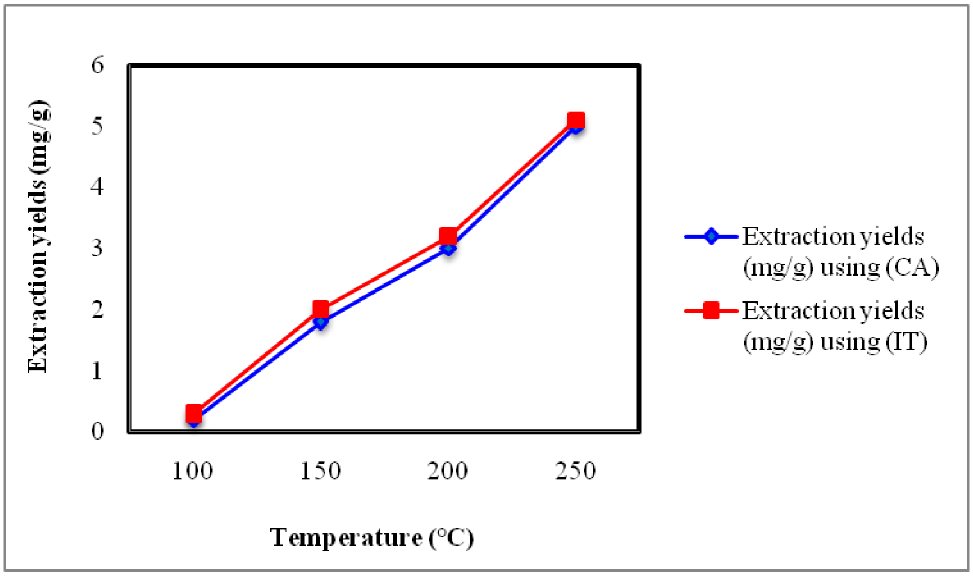

Figure 4 The Effect of Temperature on the Extraction Yields of Aqueous leaf surfactant

The performance analysis of extraction yields of hydrothermal method with two plants namely Centella Asiatica (CA) and Indigofera Tinctoria (IT)as in table 1 and figure 4. As shown in figure 4, the horizontal axis represents a temperature in °C while the vertical axis represents the extraction yields in mg/g. From the results, the extraction yield of aqueous leaf surfactant increased significantly from 0.2 to 5 mg/g when temperature increased from 100 to 250 °C using CA plant. Similarly, the extraction yield of aqueous leaf surfactant increased significantly from 0.3 to 51 mg/g when temperature increased from 100 to 250 °C using IT plant. From the analysis, extraction yield of aqueous leaf surfactant gets increased while increasing the temperature. The optimum extraction condition is obtained at 250 °C. Under these conditions, 5mg/g of aqueous leaf surfactant were extracted. Therefore, the higher extraction temperature is valuable to extract larger amount of aqueous leaf surfactant.

4.2 X-ray diffraction (XRD)

X-ray diffraction (XRD) pattern of CeO_2 nanoparticles characterization is performed by using the Multi-iterative Green Hydrothermal Synthesis method. X-ray diffraction analysis (XRD) is a method used in nanotechnology to determine the crystallographic structure of CeO_2 nanoparticles.

The CeO_2 nanoparticles crystallite size in XRD pattern is calculated by using given formula,

$$crystallitesize(\mathrm{S}) = \frac{k*\lambda_{Xray}}{b*\cos\theta} \quad (1)$$

Where, k denotes a constant depending on the crystallite size, λ_{Kray} denotes a wavelength of $X-ray$, b denotes a full width half maximum in radians, θdenotes a peak position in degree.

Sample calculation for Multi-iterative Green Hydrothermal Synthesis method

X-ray diffraction pattern of CeO2-NPs using CA

- Let us consider$k = 0.94$, $\lambda_{\mathrm{Kray}} = 1.5406\,\dot{A}$, $b = 0.26mm$, $2\theta = 29degree$ with fist peak position (111)

$$crystallitesize(\mathrm{S}) = \frac{0.94 * 1.5406}{0.26 * \cos\ (29)} = 329.51nm$$

X-ray diffraction pattern of CeO2-NPs using I

- Let us consider $k = 0.94$, $\lambda_{\mathrm{Kray}} = 1.5406\,\dot{A}$, $b = 0.32mm$, $2\theta = 28.5degree$ with fist peak position (111)

$$crystallitesize(\mathrm{S}) = \frac{0.94 * 1.5406}{0.32 * \cos\ (28.5)} = 267.42nm$$

Table 2 Intensity versus angle with CA

$2\theta\ (degree)$	**Intensity (a.u)**		
	Multi-iterative Green Hydrothermal Synthesis method	**solvothermal method**	**Solution combustion methods.**
10	220	125	25
20	240	140	40
30	250	150	50
40	220	120	26
50	220	120	26
60	220	120	26
70	210	110	25
80	200	100	10

Table 2 indicates that the X-ray diffraction characteristics of CeO_2-NPs using CA using three methods using As shown in the table, the intensity value of the multi-iterative green hydrothermal synthesis method is better than the solvothermal method and solution combustion methods.

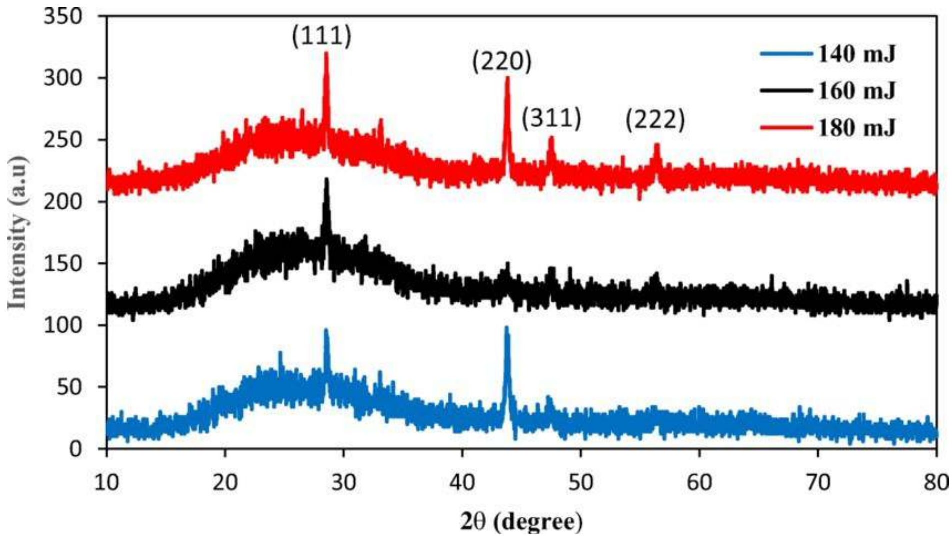

Figure 5 (a) X-ray diffraction pattern of CeO2-NPs using CA

Figure 5 (a) reveals the performance of X-ray diffraction patterns using different plants namely Centella Asiatica (CA) for CeO_2 nanoparticles synthesis. As shown in the above graphical plot, the vertical axis represents intensity in absorption unit (A.U) and the horizontal axis represents the diffraction angle in degrees. As shown in the graph, the intensity value of the multi-iterative green hydrothermal synthesis method in red color and solvothermal method in black color. Similarly, the blue color indicates solution combustion methods. Therefore, green synthesis of cerium nanoparticles with the Centella Asiatica (CA) leaves with X-ray diffraction patterns used for cardiomyoblast hypertrophy treatment in the biomedical applications.

Table 3 intensity versus angle with IT

$2\theta\ (degree)$	**Intensity (a.u)**		
	Multi-iterative Green Hydrothermal Synthesis method	**solvothermal method**	**Solution combustion methods.**
10	150	100	5
20	150	100	50
30	150	100	50
40	150	100	50
50	150	100	50
60	150	102	50
70	152	102	51
80	151	101	49

Table 2 indicates that the X-ray diffraction characteristics of CeO_2-NPs using IT using three methods. As shown in the table, the intensity value of the multi-iterative green hydrothermal synthesis method is better than the solvothermal method and solution combustion methods.

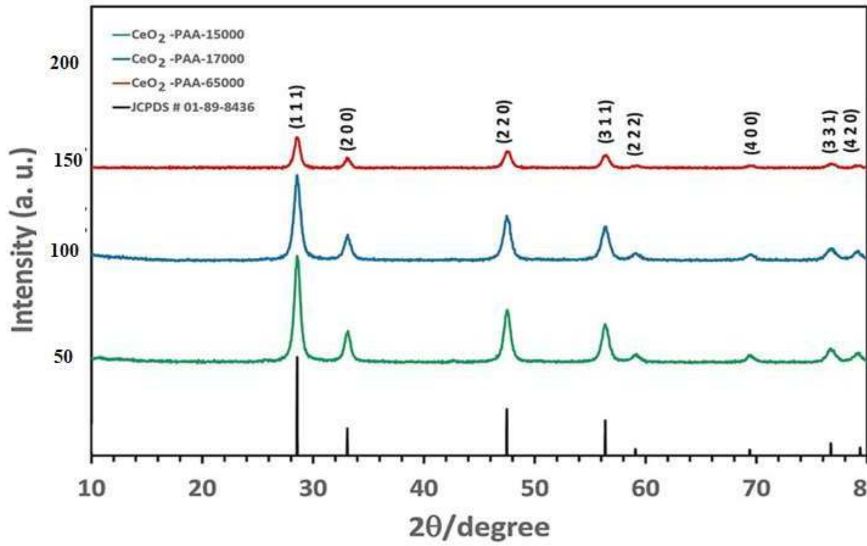

Figure 5 (b) X-ray diffraction patterns of CeO_2-NPs using IT

Figure 5 (a) (b) reveals the performance of X-ray diffraction patterns using Indigofera Tinctoria (IT) for CeO_2 nanoparticles synthesis. As shown in the above graphical plot, the vertical axis represents intensity in absorption unit (A.U) and the horizontal axis represents the diffraction angle in degrees.

As shown in the graph, the intensity value of the multi-iterative green hydrothermal synthesis method in red color and solvothermal method in blue color. Similarly, the green color indicates solution combustion methods.

4.2 Fourier transform infrared (FTIR)

FTIR is a technique used to obtain an infrared spectrum of absorption of synthesized CeO_2 nanoparticles. FTIR spectrometer collects the high-resolution spectral data and computes the intensity over a range of wavelengths at a time.

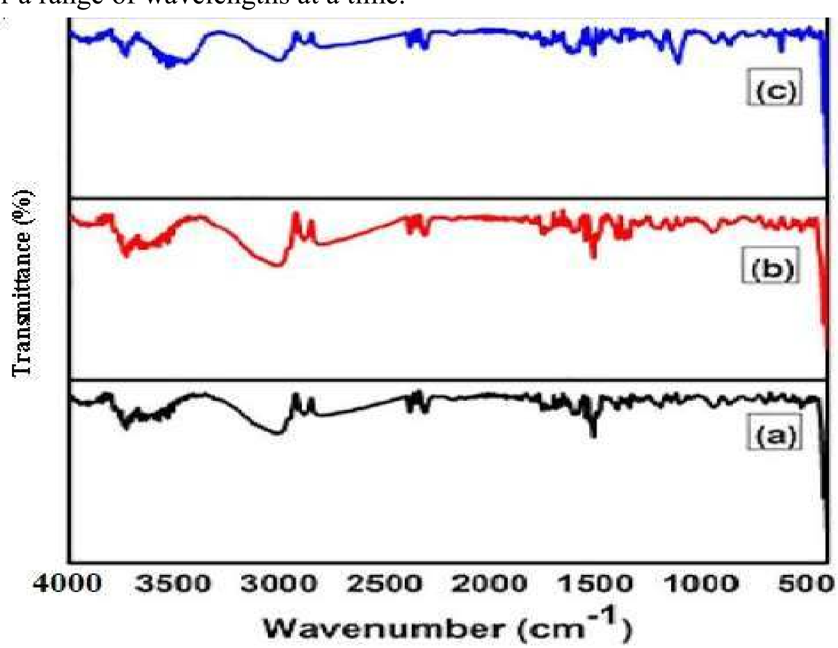

Figure 6 (a) FTIR pattern of CeO2-NPs using CA

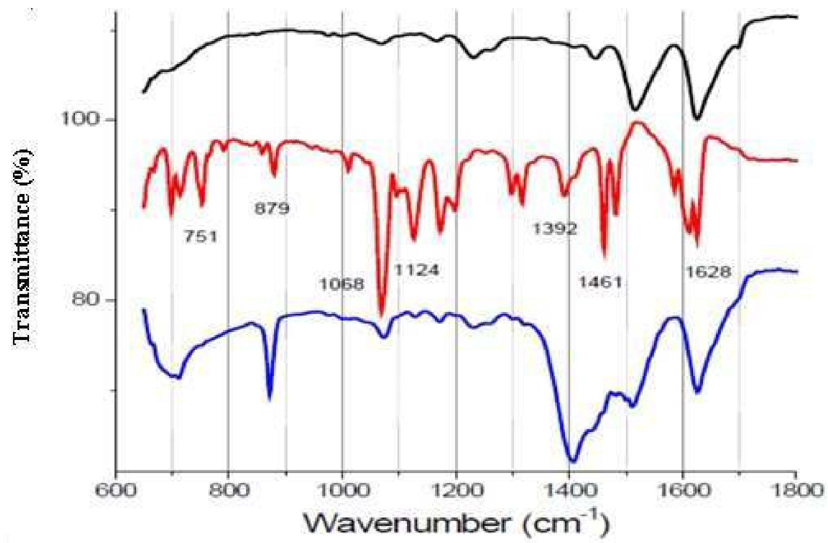

Figure 6 (b) FTIR patterns of CeO2-NPs using IT

Figure 6 (a) (b) illustrates the characterization of FTIR patterns using two different plants CA and IT for CeO_2 nanoparticles synthesis. As revealed in the above graphical chart, the percentage of transmittance is represented in the vertical axis and the horizontal axis represents the wave number in cm-1. As revealed in the graph, the percentage of transmittance of the multi-iterative green hydrothermal synthesis method is considerably improved than the existing solvothermal method and solution combustion methods. The FTIR results confirm the high purity of CeO_2 nanoparticles.

4.3 Scanning Electron Microscopy (SEM)

To analyze the effect of CeO2-NPs on the biofilm structural design of test bacteria, a Scanning Electron Microscopic (SEM) assessment of bacterial bio-films is performed. Therefore, SEM analysis shows that CeO_2-NPs minimized the bio-film development and changed the bio-film architecture of test bacteria. In order to calculate the CeO_2-NPs particle with spherical structure using SEM assessment, first the diameters of eachsphere are calculated based on the surface area of the particles.

$$D = 2 * \sqrt{\frac{Area}{\pi}} \quad (2)$$

Where, D denotes a diameter of the particle in nanometer. After calculating the diameter, the mean and standard deviation of the all the particles is computed.

$$M = \frac{\sum D}{n} \quad (3)$$

Where, M denotes a mean, $\sum D$ denotes a sum of the entire sphere diameter, n denotes a number of spheres in the given SEM image.

$$Sd = \sqrt{\frac{\sum (D-M)^2}{\pi}} \quad (4)$$

Where $Sd$is the geometric deviation, Ddiameter and, M is the mean of the diameter. Finally, mean of all the entire spheres is a size of the CeO_2-NPs particle.

Table 4 Example Calculation of CeO_2 Nanoparticles Crystallite Size in SEM Image UsingCA

Number of spheres in SEM image	Diameter of spheres in SEM image (nm)
1	98
2	45
3	79
4	67
5	82
6	69
7	96
8	85
9	65
10	96
11	98
12	88
13	78
14	73
15	85
Average mean	80.2666667

Table 4 shows the example calculation of CeO_2 nanoparticles crystallite size in SEM image using CA. Let us consider 15 spheres in the SEM image. For each image diameter is computed. Finally the average of 15 spheres is obtained as 80.26nm. This is the final CeO_2 nanoparticles crystallite size in SEM image. Similarly, the CeO_2 nanoparticles crystallite size in SEM image using IT also calculated.

Table 5 example calculation of CeO_2 nanoparticles crystallite size in SEM image using IT

Number of spheres in SEM image	Diameter of sphere in SEM image (nm)
1	93
2	42
3	76
4	65
5	80
6	68
7	92
8	84
9	54
10	90
11	82
12	84
13	65
14	74
15	89
Average mean	75.86666667

Table 5 shows the example calculation of CeO_2 nanoparticles crystallite size in SEM image using IT. Let us consider 15 spheres in the SEM image. For each image diameter is computed. Finally the average diameter of 15 spheres is obtained as 75.866nm. This is the final CeO_2 nanoparticles crystallite size in SEM image.

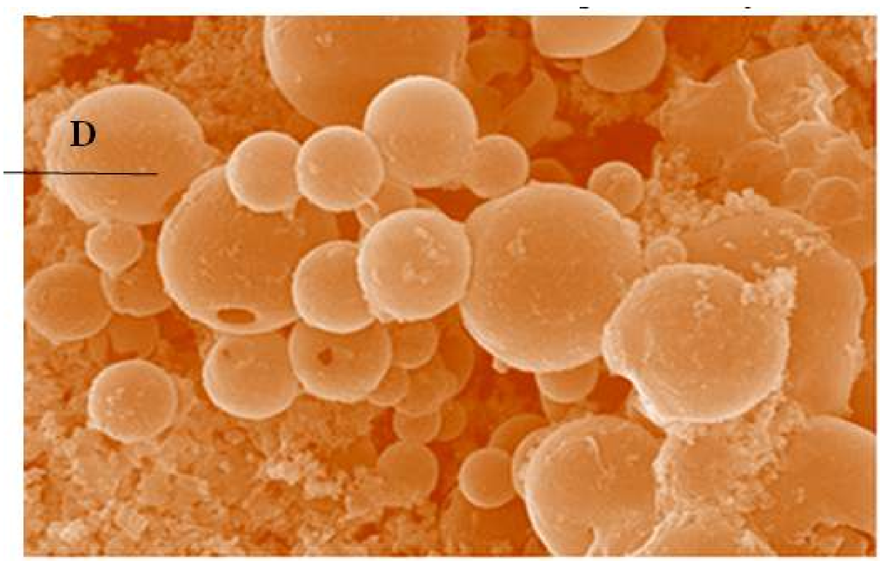

Figure 7 (a) SEM patterns of CeO2-NPs using CA

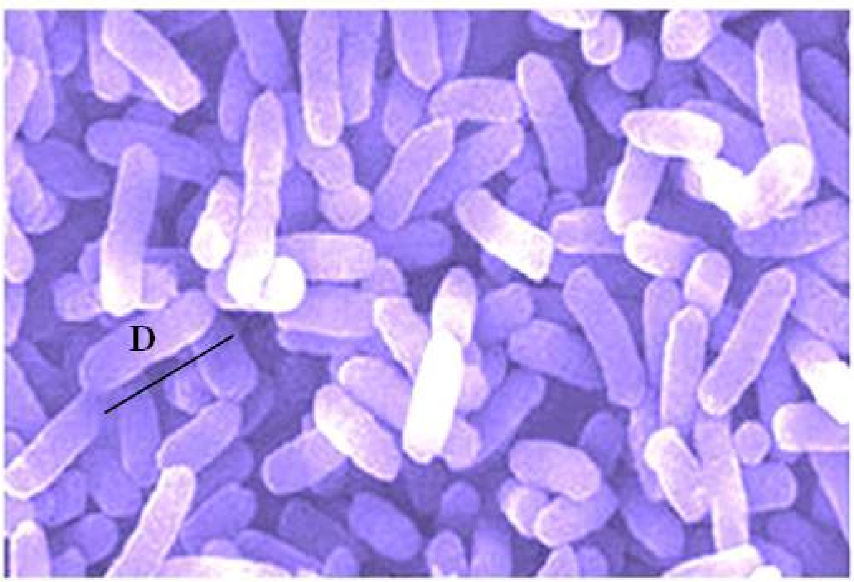

Figure 7 (b) SEM patterns of CeO2-NPs using IT

Figure 7 (a) (b) illustrates the SEM images of the pure CeO_2 nanoparticles using two plants CA and IT. A cube shaped surface morphology was observed from pure nanoparticles.

4.4 Transmission Electron Microscopy (TEM)

The TEM characterization is used to analyze the shape, size, and morphology of CeO2-NPs. As shown in the figure, the nanoparticles are observed as spherical and pseudo-spherical in shape. In some places, the nanoparticles are also observed in clusters due to the aggregation of CeO2-NPs. The CeO_2 nanoparticles crystallite size in TEM image is analyzed with hexagonal structure. For each structure in the TEM image, the area is computed as given below,

$$A = \frac{3\sqrt{3}}{2} r^2 \quad (5)$$

Where, A denotes a area of the hexagonal structure, r denotes a side of the structure.

Table 6 example calculation of CeO_2 nanoparticles crystallite size in TEM image using CA

Number of hexagons in TEM image	Area of hexagons in TEM image (nm)
1	54.5
2	57.3
3	51.3
4	45.3
5	40.2
6	45.6
7	52.3
8	45.2
9	52.1
10	65.2
11	45.2
12	51.4
13	50.2
14	44.2
15	50.1
Average mean	50

Table 6 shows the example calculation of CeO_2 nanoparticles crystallite size in TEM image using CA. Let us consider 15 hexagons in the TEM image. For each image area of the length is computed. Finally the average of 15 estimated areas is 50nm. This is the final CeO_2 nanoparticles crystallite size in TEM image.

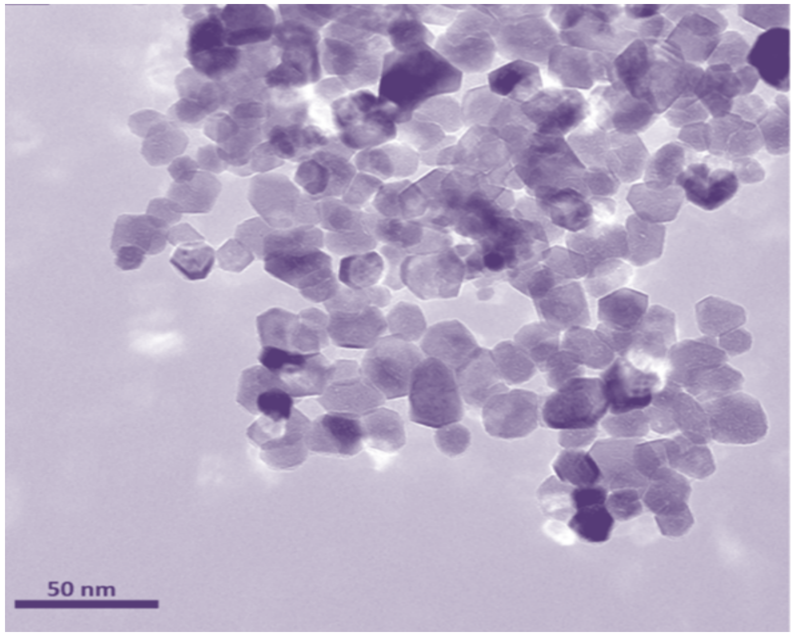

Figure 8 (a) TEM Patterns of CeO2-NPs using CA3

Figure 8 (a) illustrates the TEM images of the pure CeO_2 nanoparticles using CA. The nanoparticles of different sizes exist with the results of TEM analysis.

Table 6 example calculation of CeO_2 nanoparticles crystallite size in TEM imageusing IT

Number of hexagons in TEM image	Area of hexagons in TEM image (nm)
1	52.1
2	56.2
3	50.2
4	42.3
5	39.6
6	44.5
7	53.6
8	48.6
9	55.5
10	69.6
11	48.5
12	56.5
13	52.3
14	42.3
15	39.2
Average mean	50.067

Table 6 shows the example calculation of CeO_2 nanoparticles crystallite size in TEM image using IT. Let us consider 15 hexagons in the TEM image. For each image area of the length is computed. Finally the average of 15 estimated areas is 50.06nm. This is the final CeO_2 nanoparticles crystallite size in TEM image.

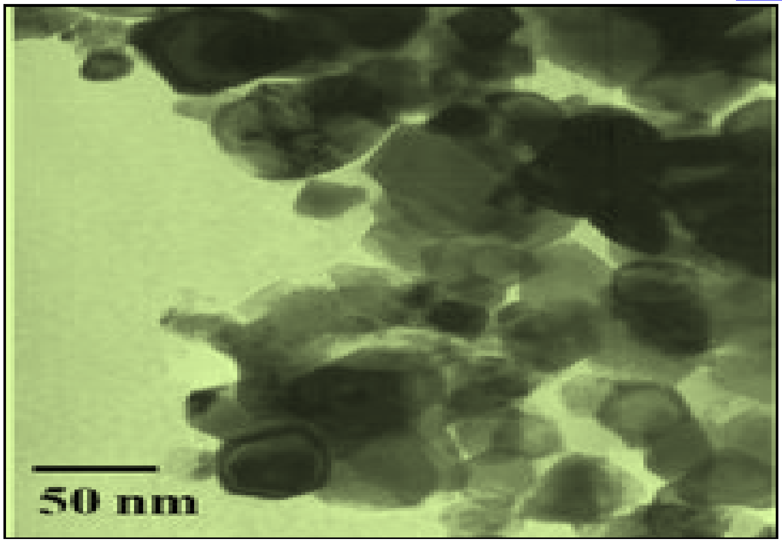

Figure 8 (b) TEM patterns of CeO2-NPs using IT

Figure 8 (b) illustrates the TEM images of the pure CeO_2 nanoparticles using IT. The nanoparticles of sizes of 50 nm exist with the results of TEM analysis.

4.5 UV-Visible spectroscopy (UV-Vis)

The basic characterization of cerium oxide nanoparticles is performed using UV–visible spectroscopy. The powdered sample of CeO2-NPs is balanced in double distilled water and then solicited to create a homogenous suspension. From the UV–visible spectroscope analysis, the absorbance peak of the synthesized CeNPs was near 290 nm.

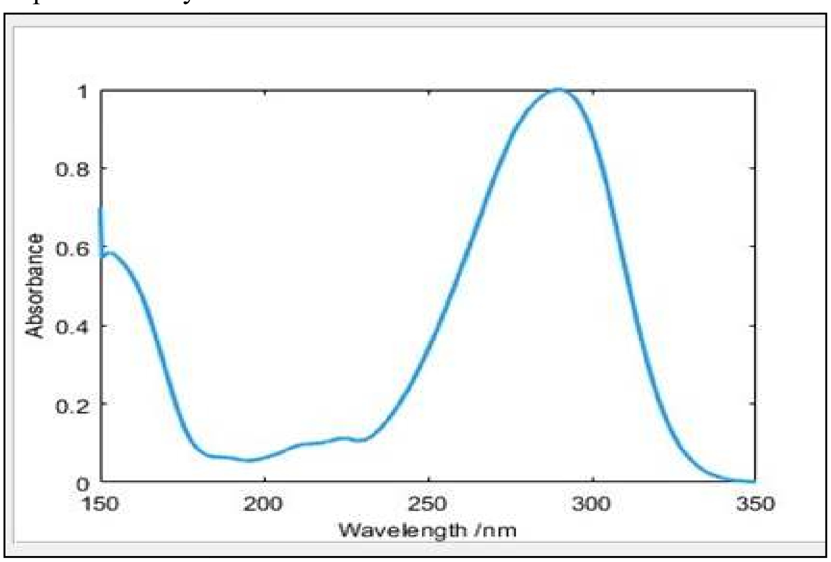

Figure 9 (a) UV-Visible spectroscopy patterns of CeO2-NPs using IT

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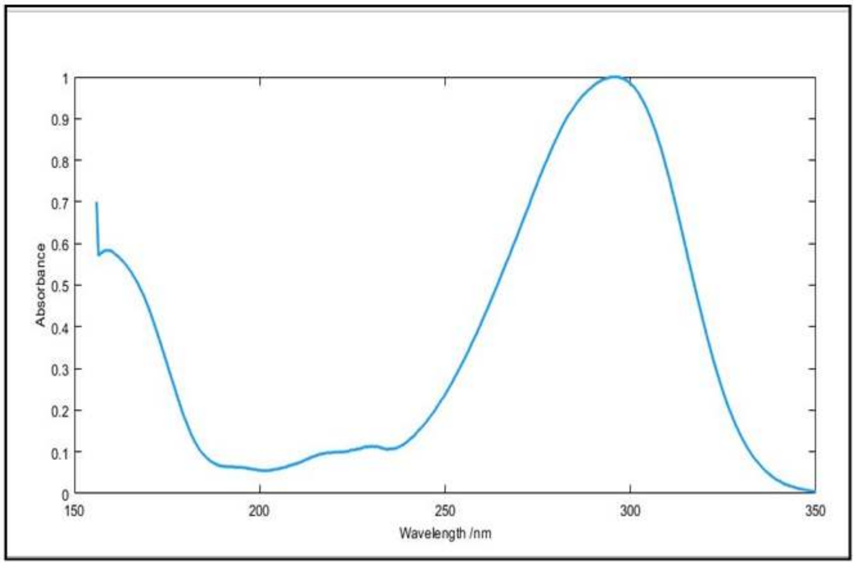

Figure 9 (b) UV-Visible spectroscopy patterns of CeO2-NPs using IT

Figure 9 (a) (b) illustrates the UV-Visible spectroscopy patterns of the pure CeO2 nanoparticles using two plants CA and IT by applying the Multi-iterative Green Hydrothermal Synthesis method. Figure 9 (a) shows the absorbance peak of the synthesized CeNPs near 290 nm. Figure 9 (b) shows the absorbance peak of the synthesized CeNPs near 295 nm.

4.6 Performance of Transmittance of gamma radiated CeO_2 nanoparticles

The pure CeO_2 nanoparticles is exposed to gamma radiation, the increase in the transmittance is observed. The relative increase of the transmittance, for each sample is calculated as given below,

$$\eta = [\frac{(t_a - t_b)}{t_b}] * 100$$

Where, η denotes a transmittance, t_a,t_b denotes a transmittance of CeO_2 nanoparticles after and before gamma-ray radiation.

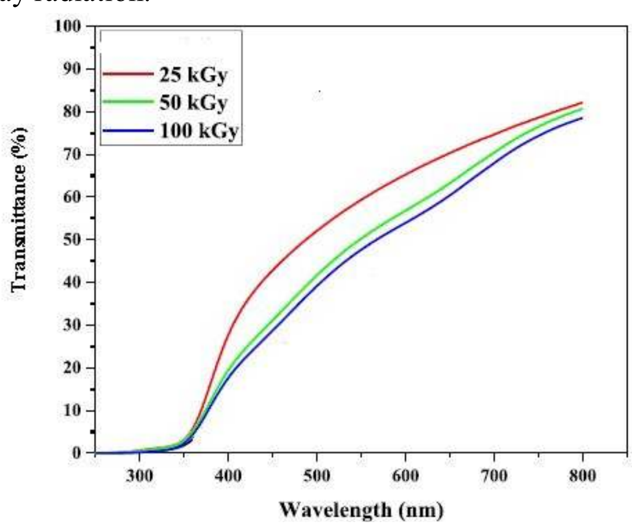

Figure 10 (a) Transmittance of gamma radiated CeO_2 nanoparticles with CA

Figure 10 (a) illustrates the Transmittance of gamma radiated CeO_2 nanoparticles by applyingthe aqueous gamma-radiation dosimetry. As shown in figure, the maximum spectra of gammaradiated CeO_2 thin films is 82% by 25 kGy of gamma ray emitted. With further increase ingamma radiation doses, the transmittance decreases and the lowest value was found to be 75%for the case of 100 kGy. This improvement is achieved by the gamma radiation sensingproperties of cerium oxide nano particles.

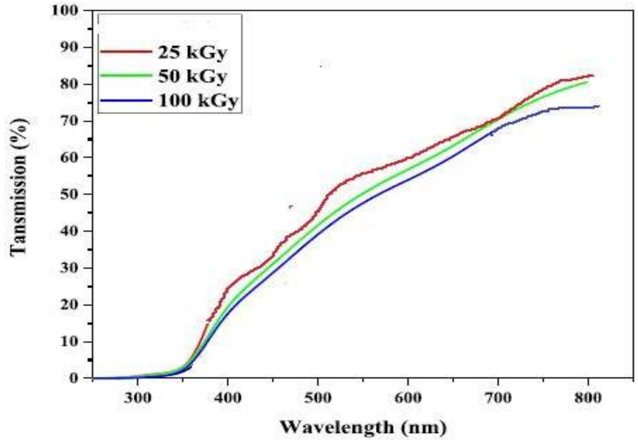

Figure 10 (b) Transmittance of gamma radiated CeO_2 nanoparticles with IT

Figure 10 (b) illustrates the Transmittance of gamma radiated CeO_2 nanoparticles by applying the aqueous gamma-radiation dosimetry. As shown in figure, the maximum spectra of gamma radiated CeO_2 thin films is 80% by 25 kGy of gamma ray emitted. While increasing the gamma radiation doses, the transmittance decreases and the lowest value was found to be 73% for the case of 100 kGy. This is achieved by applying a gamma radiation sensing properties of cerium oxide nano particles.

5. CONCLUSION

In this paper, a novel Multi-iterative Green Hydrothermal Synthesis method of cerium oxide nanoparticles and their characterization is developed for different biomedical applications. The Green Hydrothermal method uses the plant leaf for effective synthesis of CeO_2 particles. First, the aqueous leaf extract is performed by applying a subcritical water extraction method. After that, the green hydrothermal synthesis process is performed with the aqueous plant leaf extract with the cerium ammonium nitrate solution. As a result, the synthesized CeO_2 particles are obtained with minimum time consumption. The green synthesis method is an, efficient, inexpensive, and environmentally safe method for producing CeO_2 particles. Finally, the different characterization is analyzed to identify the structure of the CeO_2 nanoparticles. The results and discussed results confirmed that the Multi-iterative Green Hydrothermal Synthesis method has better performance in terms of effective synthesis of CeO_2 particles with minimum time when compared to other synthesis methods. Therefore, green synthesis of CeO_2 with CA is used for potential therapeutic tools in the treatment of oxidative stress- related cardiovascular diseases. Similarly, green synthesis of CeO_2 with IT leaf extract is applied for a anticancer effect on lung cancer cell.

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