Materials and its Characterization

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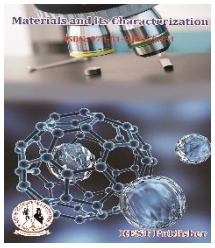

Green Synthesis of Zirconia Nanoparticles Using Moringa Oleifera and Psidium Guajava Leaves Extract and Their Characterization

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Abstract: *Zirconia nanoparticles (ZrO2 NPs) have many physical, chemical properties and applied in ceramic as a inert material. In this paper, we present the green synthesis of ZrO2 NPs using Moringa oleifera leaves and Psidium guajava leaves extracts as stabilizing agent. Zirconyl oxychloride was used as a precursor solution. The synthesized ZrO2 NPs were characterized using various spectral techniques such as UV-Visible spectroscopy (UV-Vis), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction study (XRD), Scanning electron microscope (SEM) and Energy dispersive X-ray diffraction study.*

Keywords: *Moringa oleifera, Psidium guajava, ZrO2, Characterization*

1. INTRODUCTION

Zirconia nanoparticles ceramics are widely used in manufacturing of polishers, pigments, catalysts, refractory materials, electric materials, solid oxide fuel cells, and structural materials owing to their specific chemical and physical properties, including high chemical stability, high temperature insulation capability, and ionic conductivity (Noh et al., 2003). Nanomaterials play a central role in nanotechnology, raising the demands for sustainable development (Srivastava et al. 2021). The chemical synthesis of nanomaterials chiefly does not satisfy the strict requirements for this trend, and also mismatches with the principles of green chemistry. Currently, the green approaches for the synthesis of nanomaterials are of remarkable significance due to their eco-friendliness as well as cost-effectiveness (Tran et al. 2020a). Zirconium (Zr) is classified as a transition metal element in the titanium family with the atomic number. Zr does not basically capture neutrons, offering a potential metallic cladding for the fuel rods in the nuclear reactors (Cazado et al. 2021). Zirconium dioxide is one of the highly stable oxides, created by thermalizing zirconium compounds (Hassan and Jalil 2022). Depending on the various synthesis routes, ZrO_2 can present in the crystalline phases involving monoclinic, tetragonal, and cubic (Zhang et al. 2018). Bulk ZrO_2 has wide bandgap energy, typically ranging from 5.0 to 7.0 eV. Because of the ultrahigh stability and very low toxicity, ZrO_2 has exhibited an intensive range of practical technologies for heat-resistant ceramic superalloys (Wang et al. 2021), dental restorations (Chen et al. 2021), fuel cells (Rambabu et al. 2020), and heterogeneous catalysis (Jiang et al. 2020). Such promising utilizations make ZrO_2 an ideal nanomaterial, promoting the green strategy for synthesizing ZrO_2 nanoparticles. In general, there are two major approaches to synthesize the ZrO_2 nanoparticles, involving top-down and bottom-up (Jadoun et al. 2021). The former implies the conversion of bulk material into thinner crystallites by the physical route. This means that it needs the participation of enormous mechanical energy sources such as milling, and ionic sputtering (Shrimal et al. 2020). As a result, top-down strategy brings many inevitable drawbacks of causing secondary impressions or intermediates, altering the physicochemical property and surface chemistry of as-synthesized nanoparticles (Indiarto et al. 2021). More importantly, nano-sized particles are mostly unattainable by top-down approach. Meanwhile, the latter implies the formation of particles by creating building blocks from ultra-small particles such as atoms or molecules, and then assembling them together. By this way, the nanostructured particles can be intentionally attainable under the control of fabrication conditions (Rana et al. 2020). Zirconium dioxide nanoparticles have numerous applications in various fields and got much attention from the researcher because of their valuable and unique characteristics which include catalytic, sensing, mechanical, thermal, electrical, biocompatible, and optical characteristics and due to these characteristics ZrO_2 NPs are utilizing in various bone implants (Gillani et al., 2015) solar cells, gas sensor, fuel cell, seed germination, photo-catalysis, refractory and energy (Nikam et al., 2019). Green synthesis of ZrO_2 nanoparticles and their applications in biomedical, adsorption, catalysis, and nanocomposite fabrication. The plant tissues including flowers, fruits, seeds, leaves, roots, etc. possess many phytochemicals such as polyphenols, saponins,

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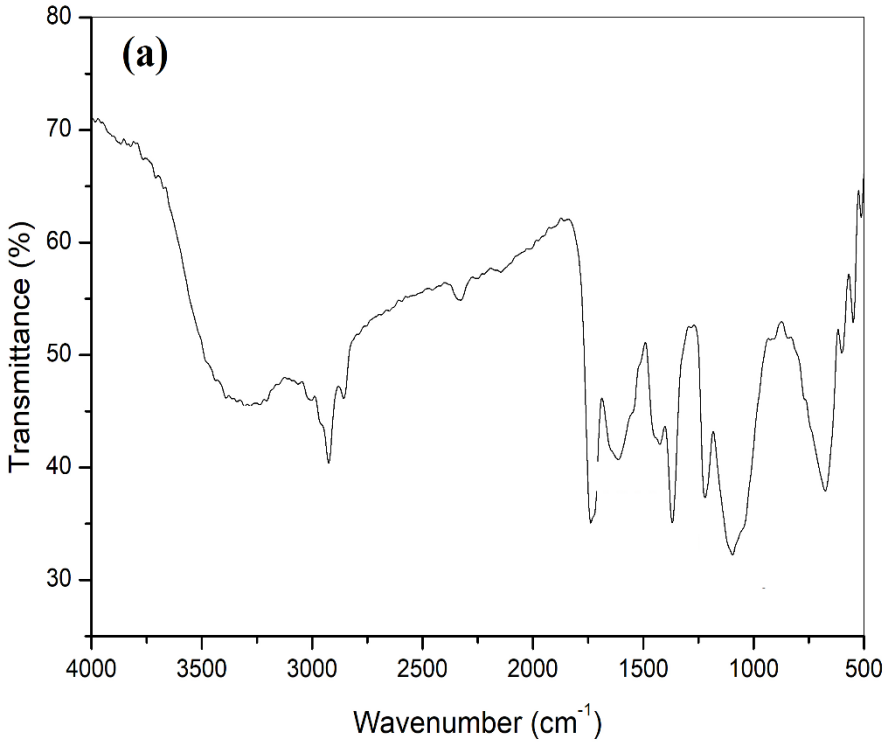
quercetin, and gallic acid. Microorganisms including bacteria, fungi, algae, etc. can secrete biomolecules, metabolites, enzymes, and proteins. These phytochemicals and biomolecules participate in the green synthesis of ZrO_2 NPs.

2. MATERIALS AND METHODS

Zirconyl oxychloride and Sodium hydroxide (NaOH) were purchased from Merck, India. All chemicals were used without further purification. Deionised water was employed as a solvent for the synthesis of ZrO_2 NPs. Psidium Gujava and Moringa oleifera leaves were collected from Tiruvannamalai, Tamil Nadu. The leaves were washed twice with distilled water to remove dust particles and other contaminants. About 50 g of the leaves were added to 100 ml of distilled water and boiled for 3 h at 80ºC. The extract was filtered using Whatman filter paper. To synthesize ZrO_2 NPs, 0.1 M solution of zirconyl oxychloride was prepared in 100 ml of distilled water and extract was added drop wise under constant stirring. The mixture was subjected to continuous stirring and the hydroxide formed during the process was separated using Whatman filter paper. The hydroxide obtained was repeatedly washed with absolute ethanol followed by distilled water and dried at 100ºC for 3. This paste was then collected in a ceramic crucible and heated in an air heated furnace at 400ºC for 3 h. A light white coloured powder was obtained and this powder was carefully collected and sent for characterization. Crystalline nature of ZrO_2 nanoparticles was analysed using X-ray diffraction studies. Functional groups present in the ZrO_2 nanoparticles were characterized by Fourier Transform Infrared spectra the 400–4000 cm^{-1}. The morphology and size distribution were characterized using FE-SEM (JEOL JSM 6701-F) and coupled with EDAX analysis.

3. RESULTS AND DISCUSSION

Green synthesis ZrO_2 NPs using Psidium Guajava and Moringa oleifera leaves extract are rich in biomolecules, while adding this extract with zirconyl oxychloride solution and subjected to heating at 80-90ºC, we get the off-white precipitate. Here, sodium hydroxide was used to maintain the pH values of the reaction mixture. The structures of the synthesized nanoparticles were confirmed FTIR, XRD and SEM-EDAX spectrum. FTIR analysis was carried out to assess the possible bonds in the ZrO_2 NPs. The Figure 1 illustrates the FTIR spectra of ZrO_2 NPs synthesized using Psidium Guajava leaves extract. The ZrO_2 NPs show broad, sharp and weak absorption bands at 3280, 2910, 1600, 1410, 1090 and 679 cm^{-1}. A broad band noticed in the range between 3280 cm^{-1} reveals the presence of moisture content during sample preparation. The absorption band obtained between 680 cm^{-1} is characteristic of Zr–O–Zr vibrations. In the region between 1410 and 1090 cm^{-1} indicates the presence of Zr-O bond supported the tetragonal form of ZrO_2 NPs. A small band at 2900 cm^{-1} reveals the unknown impurity in the sample.



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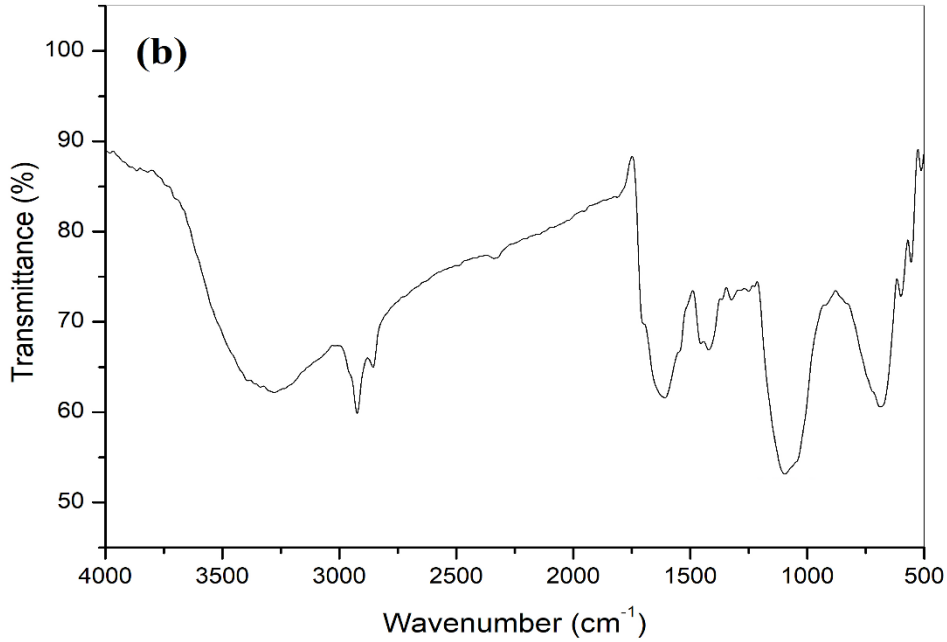
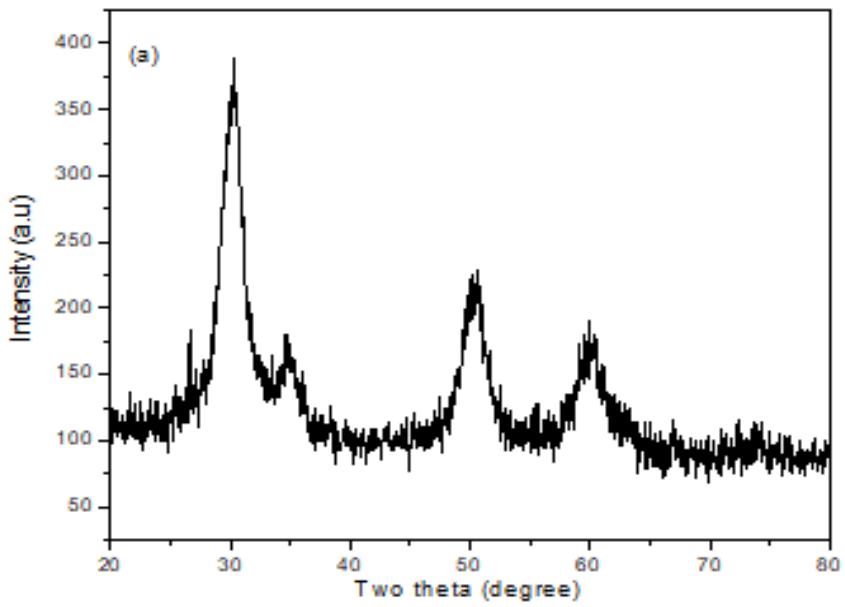


FIGURE 3. FTIR spectra of ZrO_2 NPs synthesized using (a) Moringa oleifera and (b) *Psidium Guajava* leaves extract

The consistent peaks at 1600 and 1400 cm^{-1} are due to bending vibrations of –(H–O–H)– and –(O–H–O)– bonds. The prominent peak at 1090 cm^{-1} corresponds to C–O stretching vibrations. The characteristic bands at 679 cm−1 indicate the presence of Zr–O–Zr bending vibration which confirms the formation of ZrO_2 structure [Bansal et al. 2004; Singh et al. 2014; Mallik et al. 2006]. X-ray diffraction (XRD) pattern of the green synthesized ZrO_2 NPs is shown in Figure 3 (a and b). The data shows the crystalline phase of tetragonal for ZrO_2 NPs. The distinguishing characteristic peaks for tetragonal ZrO_2 occur at 2θ=35.10, 49.50 and 61.10 corresponding to the (101), (110), (200) and (211) reflections [JCPDS No. 79-1 6]. It can be seen from the figure that the (101) plane has the highest intensity revealing the orderly growth crystallites along the (101) plane. The average crystallite size "D" has been estimated to be 15 nm using the Scherer formula.

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where λ = 1546 Å wavelength, θ is the Bragg's diffraction angle, and β is the full width at half maximum FWHM of the diffraction peak [Kumar et al. 2015; Shuai et al. 2015].



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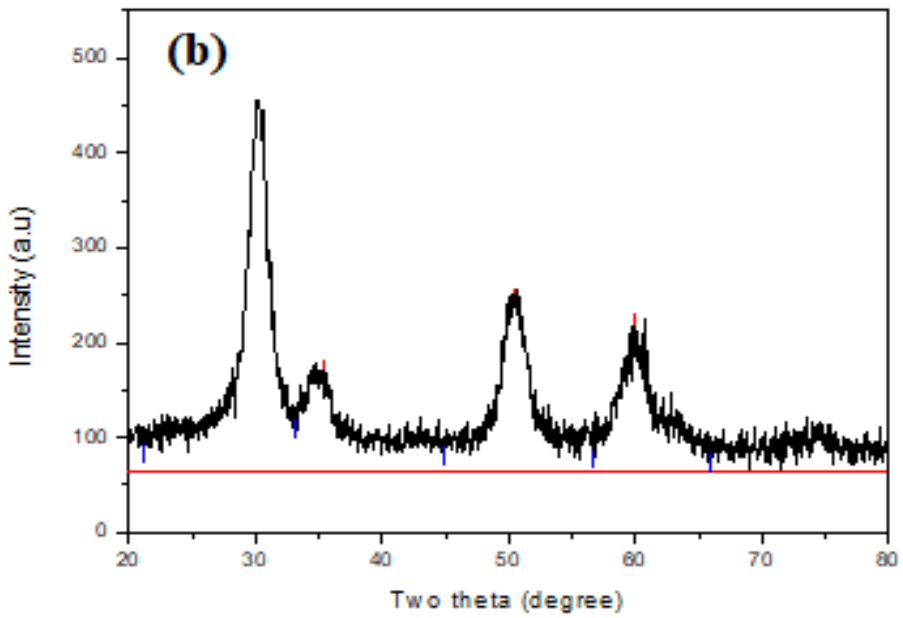
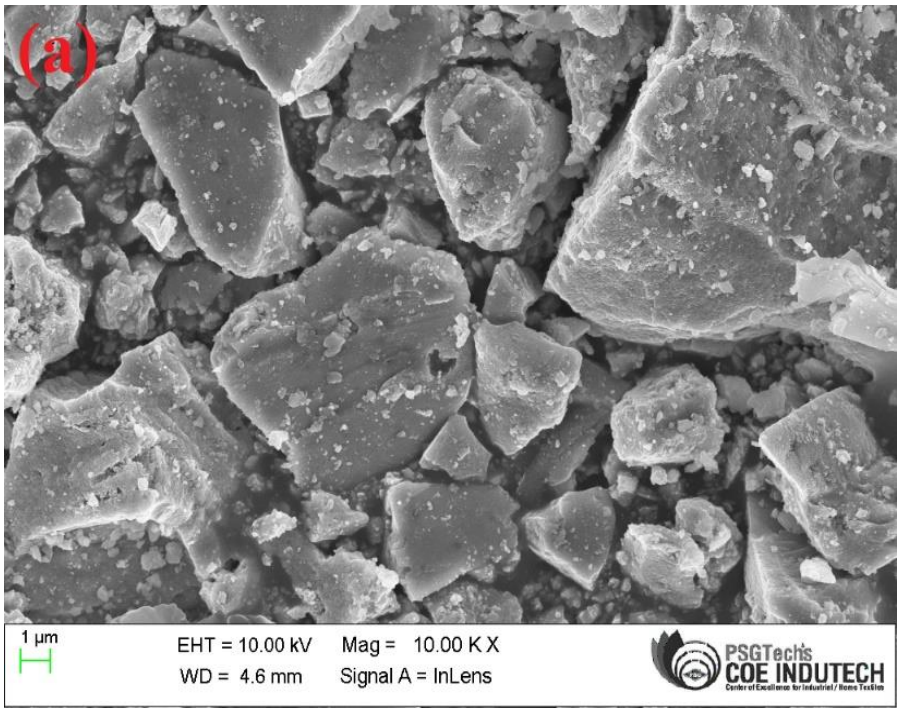


FIGURE 3. XRD spectra of ZrO_2 NPs synthesized using (a) Moringa oleifera and (b) *Psidium Guajava* leaves extract

Where, D is the size of the particle, λ the wavelength (0.1542 nm) with Cu-Kα radiation), β the full width at half maximum and θ the Bragg angle. Small peaks at 30.00 show the establishment of phase transition from tetragonal phase. The average crystallite of (a) and (b) found to be 10 and 15 nm, respectively. The morphology, shape and formation of ZrO_2 NPs was analysed by scanning electron microscope. The SEM morphographs of the particles under two different magnifications and images are displayed in Figure 4. The images show that the particles are irregular, quasi-spherical particles and agglomerated [Selvi 2014]. Irregular shape and agglomerated particles were noticed in A, B and C. However, the SEM images of ZrO_2 NPs prepared using 0.02 M solution shows that the particles are spherical and homogeneously distributed without agglomeration.



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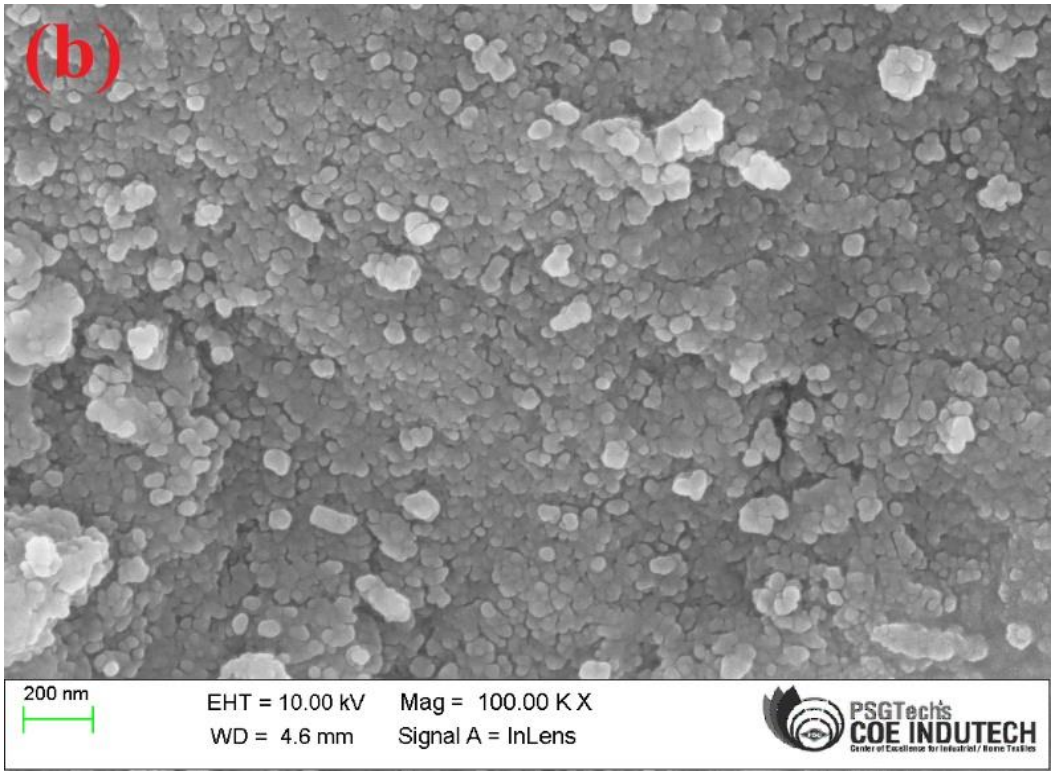

FIGURE 4. SEM image of ZrO_2 NPs synthesized using (a) Moringa oleifera leaves extract (b) ZrO_2 NPs synthesized using Psidium guajava leaves extract

The phytochemicals present in the extract having many water-soluble organic molecules, which having reducing properties are usually responsible for the synthesis of metal and metal oxide nanoparticles.

4. CONCLUSIONS

Psidium Guajava leaves extract was used as reducing agent in the synthesis of ZrO_2 NPs. The structure of pherical ZrO_2 NPs were established by UV-Vis, FTIR, XRD and SEM-EDAX analysis. The synthesis of ZrO_2 NPs using Psidium Guajava leaves extract led to the formation of spherical particles with sizes ranging between 50-80 nm. The water-soluble biomolecules present in the Psidium Guajava leaves extract might be responsible for the formation of spherical particles. Thus, Psidium Guajava proven as a suitable biomaterial for synthesis of ZrO_2 NPs.

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