

Optical and Vibrational Characterization of Zirconium Oxide Nanoparticles Synthesized in Distilled Water Using The Pulsed Laser Ablation Method

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Abstract– In this study, Zirconium oxide nanoparticles were successfully synthesized in distilled water using the Pulsed Laser Ablation in Liquid method. This study aimed to determine the optical properties and functional groups of the synthesized zirconium oxide nanoparticles by Ultraviolet-Visible Spectroscopy (UV-Vis) and Fourier Transform Infrared (FTIR) analysis. The UV-Vis data indicated a maximum absorption peak at a around 298 nm, signifying the development of zirconium oxide nanoparticles with optical properties characteristic of metal oxide semiconductors. The absorption spectrum gradually decreased in the visible light region, indicating dominant absorption in the ultraviolet region. The FTIR results showed the presence of a Zr–O bond absorption band as the main characteristic of zirconium oxide formation, as well as an –OH group band originating from water molecules adsorbed on the nanoparticle surface. The research results indicate that the pulsed laser ablation method is effective for synthesizing zirconium oxide nanoparticles in distilled water with optical properties and chemical structures consistent with theory.

Keywords – Zirconium Oxide Nanoparticles; Pulsed Laser Ablation; Ultraviolet-Visible Spectroscopy; Fourier Transform Infrared Spectroscopy; Distilled Water.

I. INTRODUCTION

The application of nanoparticles has been increased in many industries because it is able to manufacture nano size materials with physical, chemical and optical properties [1]. One of the materials that is widely developed is zirconium oxide because it has high thermal stability, good corrosion resistance, high biocompatibility, and superior mechanical and optical properties. Zirconium oxide nanoparticles are widely used in several fields such as biomedicine, sensors, chemistry and catalysts [2]. The nanosize of the particles increases the surface area and hence increases the reactivity and performance of the material in numerous applications [3]. Various methods have been used for the synthesis of zirconium oxide nanoparticles, such as sol-gel, coprecipitation, hydrothermal, and other chemical methods [4]. However, these methods generally require additional chemicals, high temperatures, and complex purification processes, potentially resulting in contamination of the final product [5]. The pulsed laser ablation approach is an intriguing option, since it is capable of manufacturing nanoparticles with high purity, simple procedure and is environmental friendly without the requirement of extra chemical precursors [6]. The liquid medium used in the laser ablation process has a major influence on the characteristics of the resulting nanoparticles [7]. Distilled water is frequently utilized as an ablation medium because it is inert, easily obtained, inexpensive, and capable of producing nanoparticles with a high degree of purity. In addition, the use of distilled water

supports the concept of environmentally friendly synthesis because it does not produce harmful chemical residues. The distilled water medium also helps disperse nanoparticles so that they form a relatively stable colloid [8].

Analysis methods are needed to determine the properties of the synthesized material. The methods used include UV-Vis analysis to determine the optical properties of nanoparticles and FTIR is used to identify functional groups and chemical bond vibrations based on infrared radiation absorption patterns. In zirconium oxide nanoparticles, FTIR can be used to identify Zr–O bond vibrations and other functional groups present on the nanoparticle surface [9]. UV-Vis and FTIR analyses are very important to ensure the successful formation of zirconium oxide nanoparticles based on the synthesized results.

This study was conducted to synthesize zirconium oxide nanoparticles in distilled water using the pulsed laser ablation method and to characterize the optical properties and analyze the characteristics of the functional groups of the resulting nanoparticles. This study is expected to provide information on the effectiveness of the pulsed laser ablation method in producing high-quality zirconium oxide nanoparticles and support the development of zirconium oxide based nanomaterials for various advanced applications.

II. EXPERIMENTAL PROCEDURE

2.1. Materials

The materials used in this study were zirconium (Zr) plates with a purity level of 99.99%, distilled water, and glass beakers.

The equipment used included an Nd:YAG laser with a wavelength of 1064 nm.

Figure 1 The zirconium plate used was 1.5 x 1.5cm in size and was sterilized with 70% alcohol to remove any bacteria present on its surface. Then the metal plate was washed with distilled water to eliminate remaining alcohol. The beaker for the synthesis of the sample was washed with alcohol and rinsed with distilled water. The cleaned zirconium plate was then transferred into a beaker containing 10 mL of distilled water.

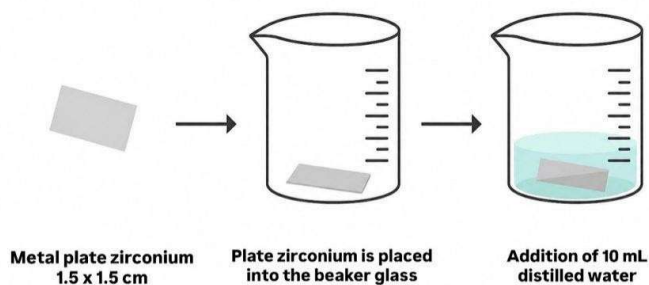


Fig. 1. Preparation of zirconium metal

Nanoparticle synthesis was carried out using the pulsed laser ablation method with a 1064 nm Nd:YAG laser. The laser parameters used included 80 mJ energy, 10 Hz repetition rate, 7 ns pulse width, and 10 cm focal length. The laser beam was focussed onto the surface of a zirconium plate in an aqueous solution for 3 hours. During the ablation process the beaker was moved gently to obtain a more homogenous distribution of the generated nanoparticles. The interaction between the laser and the target produced a plasma and it was rapidly cooled in the liquid media to make zirconium oxide nanoparticles. Figure 2

presents the schematic of the synthesis of zirconium nanoparticle colloids in distilled water.

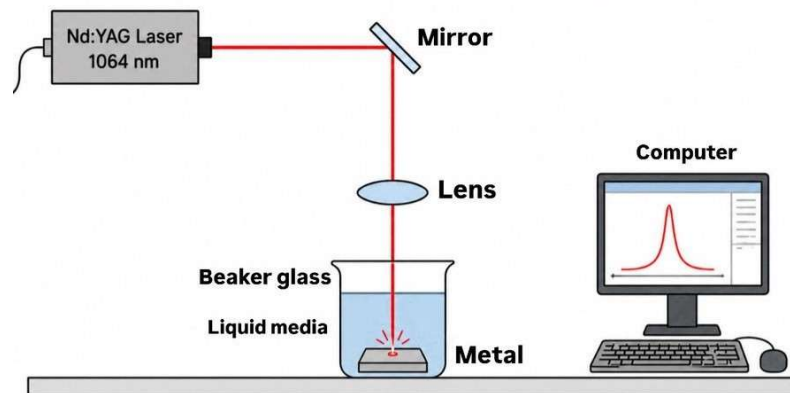


Fig. 2. Setup for Zirconium Nanoparticles Synthesis Experiment

2.2 Characterization of zirconium oxide nanoparticles

UV-Vis analysis was conducted to ascertain the properties of the nanoparticles through their absorption spectra in the ultraviolet-visible wavelength range. The nanoparticle colloid samples were diluted and placed in a cuvette, then analyzed using UV-Vis spectroscopy. FTIR analysis was performed to identify the functional groups and chemical bonds of the synthesized zirconium oxide nanoparticles. The synthesized colloid was deposited onto a silicon wafer and dried on a hot plate to obtain a solid film. The dried sample was subsequently characterized using FTIR spectroscopy in the wavenumber range of $500\text{--}4000\text{ cm}^{-1}$. Data analysis of the characterization results was performed using Origin software.

III. RESULT AND DISCUSSION

Figure 3 Zirconium oxide nanoparticles in distilled water



Fig. 3. Zirconium oxide nanoparticles in distilled water.

Figures 3 The synthesis using the pulsed laser ablation method produced a colloid of whitish-opaque zirconium oxide nanoparticles in distilled water. The color change in the medium indicates that ablation occurred on the surface of the zirconium target and that nanoparticles were formed and dispersed in the liquid.

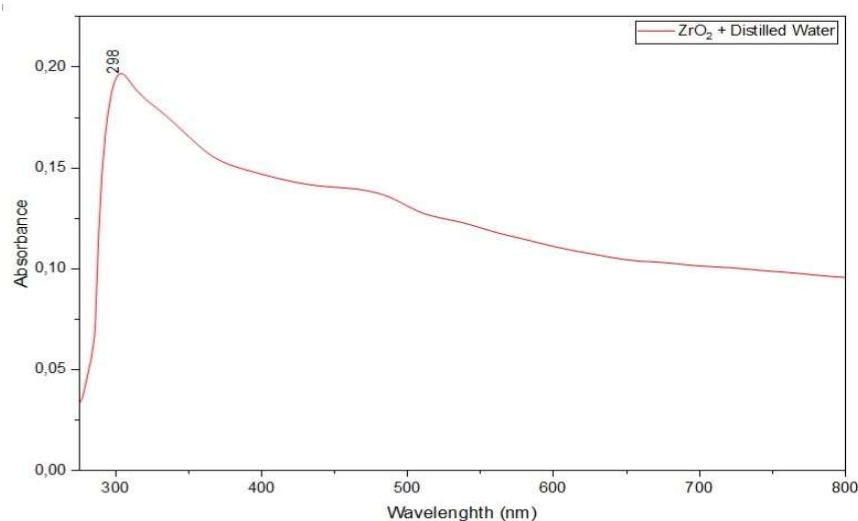


Fig. 4. UV-Vis of zirconium oxide nanoparticles

Figure 4 UV-Vis analysis of zirconium oxide nanoparticles in distilled water showed a maximum absorption peak at a wavelength of around 298 nm with an absorbance value of about 0.20. The appearance of this absorption peak in the ultraviolet range suggests the creation of zirconium oxide nanoparticles due to the pulsed laser ablation process. The curve steadily falls to the wavelength of 800 nm after the absorption peak, which shows that zirconium oxide nanoparticles have greater light absorption in the

UV region than the visible light region [10]. The absorption peak at ~298 nm is consistent with the theory and earlier research that reported zirconium oxide nanoparticles absorption in the UV region at roughly 250-350 nm [11,12]. This absorption behavior results from the transition of electrons from the $O^{2-} 2p$ valence band to the $Zr^{4+} 4d$ conduction band. The presence of this absorption peak indicates the formation of zirconium oxide species during the pulsed laser ablation process in distilled water. Generally, the change in wavelength is indicative of a considerably bigger particle size distribution or interparticle interactions in the solution,

which can be affected by the energy and time of laser ablation and the liquid medium employed in the synthesis process [13]. The characterization result indicate the formation of zirconium oxide containing particles in distilled water [14].

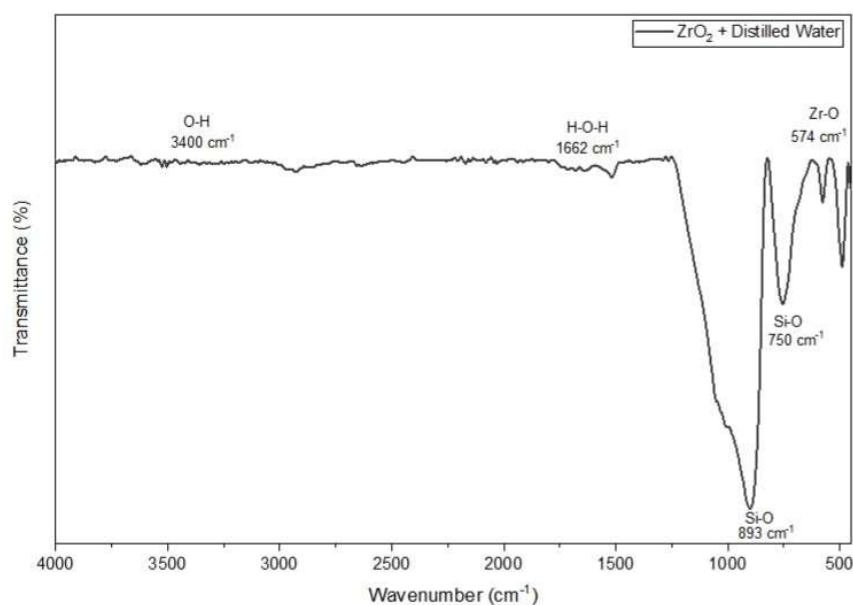


Fig. 5. FTIR of zirconium oxide nanoparticles

Figure 5 FTIR analysis was performed to identify the functional groups present in the synthesized nanoparticles. The FTIR spectrum of the zirconium oxide nanoparticles showed several characteristic absorption bands at specific wavenumber regions. The absorption band in the region around 3400 cm^{-1} indicates the presence of hydroxyl (O-H) groups originating from water molecules or water vapor adsorption on the nanoparticle surface [15]. Another peak around 1662 cm^{-1} is associated with the H-O-H molecular bending vibration of distilled water [16,17]. The absorption band in the region around $800\text{--}900\text{ cm}^{-1}$ in the spectrum of zirconium dioxide in distilled water indicates Si-O bonds caused by oxygen from the silicon present in the glass structure [18]. The Zr-O peak at wavenumbers of $400\text{--}800\text{ cm}^{-1}$ is characteristic of zirconium oxide [19]. The presence of hydroxyl groups on the nanoparticle surface can enhance the stability of the nanoparticle dispersion in the liquid medium due to the formation of interparticle hydrogen

bonding. The use of distilled water as the liquid medium offers an advantage as it minimizes chemical contamination on the nanoparticle surface. The rapid cooling process during laser ablation promotes the nucleation and formation of zirconium oxide-containing particles in the liquid medium. These attributes have a considerable effect on the optical properties and surface structure of produced nanoparticles. The FTIR results indicate the formation of zirconium oxide-containing particles synthesized in distilled water by the pulsed laser ablation method. The characteristic Zr–O bond vibration indicates that the oxidation process during ablation was successful [20].

IV. CONCLUSIONS

The pulsed laser ablation approach was used to manufacture zirconium oxide nanoparticles in distilled water successfully. UV-Vis characterization results indicated an absorption peak of around 298 nm which indicates the development of zirconium oxide nanoparticles with optical characteristics characteristic of metal oxide semiconductors. The FTIR study showed the bands of absorption corresponding to Zr-O bonds which are specific to zirconium oxide and -OH groups from the distilled water medium. The UV-Vis and FTIR results show that the pulsed laser ablation method is suitable for the synthesis of zirconium oxide nanoparticles in distilled water, the obtained optical and vibrational characteristics are consistent with the reported behavior of zirconium oxide materials.

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