Synthesis and Characterization of Zirconium Oxide Nanoparticles via Chemical Method

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ABSTRACT

Zirconium oxide (ZrO_2), is a versatile material with exceptional properties, making it valuable in various fields. This research focuses on the synthesis of ZrO_2 nanoparticles through a chemical method and explores the influence of synthesis parameters. The Sol-Gel method was employed, and structural and morphological characterizations were performed. X-ray diffraction analysis revealed crystalline properties, with an average crystallite size of 13.21 nm. Field Emission Scanning Electron Microscopy (FE-SEM) showed spherical particles with an average size of 14 nm. FTIR spectroscopy confirmed the formation of ZrO_2. This study enhances our understanding of ZrO_2 nanoparticles and their potential applications.

Keywords: Zirconium Oxide Nanoparticles (ZrO_2), Chemical Synthesis, Sol-Gel Method Biomaterials, Nanomaterials,

1.Introduction

Zirconium oxide, often called zirconia (ZrO_2), is a versatile and promising material that has garnered significant attention in various fields, including catalysis, electronics, ceramics, and biomedicine, owing to its exceptional physical and chemical properties. Among the different methods employed for the synthesis of zirconium oxide nanoparticles, chemical methods have gained prominence due to their controllability, scalability, and ability to tailor the material's properties. [1] Zirconium oxide nanoparticles exhibit remarkable characteristics such as high thermal stability, excellent chemical resistance, low thermal conductivity, and a wide bandgap, making them suitable for applications ranging from solid oxide fuel cells and high-temperature insulation to catalyst supports and drug delivery systems. These nanoparticles have demonstrated immense potential in addressing some of the most pressing challenges in modern technology.[2] In this research, we delve into the synthesis and characterization of zirconium oxide nanoparticles through a chemical method, with a primary focus on elucidating the influence of various synthesis parameters on the final product's properties. Using chemical synthesis routes offers distinct advantages in terms of controlling particle size, morphology, and phase composition, enabling the tailoring of zirconium oxide nanoparticles for specific applications. A biomaterial is a substance created with the intention of forming a bond with biological systems in order to measure, support, or exchange any tissue, member, or bodily function. able to come into regular or irregular touch with bodily fluids [3]. The bio-functionality and bio-compatibility of

materials utilized in the medical industry are assessed. The capacity of a device to carry out a certain function over the course of its useful life is referred to as biocompatibility, which is closely connected to how biomaterials interact with the tissues they come into touch with during biological use[4]. Numerous substances, referred to as biomaterials, have been designated as being safe for use within the human body. "Any materials used to replace or restore function to the body's tissues" are considered biomaterials. They involve metals, polymers, ceramics, and composite materials. Many researchers reported on the different types of glasses and ceramics designed for Bio applications called "Bioceramics" Bioceramics contain glass-ceramic, alumina (Al_2O_3), and zirconia (ZrO_2), Hydroxyapatite (HA), and resorbable calcium phosphates[5]. They have generally been used for dental and orthopedic applications

2.Experimental procedure

2.1 Material

The name, type, and company that supplied the materials used in the current research which listed in Table 1

Table 1. Materials Used.

Name	hemical formula	Type	Company
Zirconium nitrate	Zr $(NO_3)_4$	White powder	gma-Aldrich (UK)
Sodium hydroxide	NaOH	White powder	Avonchem Ltd

2.2 Preparation method

The Sol-Gel method was used to prepare ZrO_2, including some steps. Firstly, the "sol" was prepared by mixing precursor materials with (NaOH) as a reaction fuel and adjusting the alkalinity at pH 8. Secondly, the Sol gradually forms a "gel" diphasic system containing a liquid and solid phase; Thirdly, a drying process was used to remove the remaining liquid phase, typically accompanied by a significant amount of shrinkage. Finally, a thermal treatment, or calcination process, is often necessary to obtain the final ZrO_2

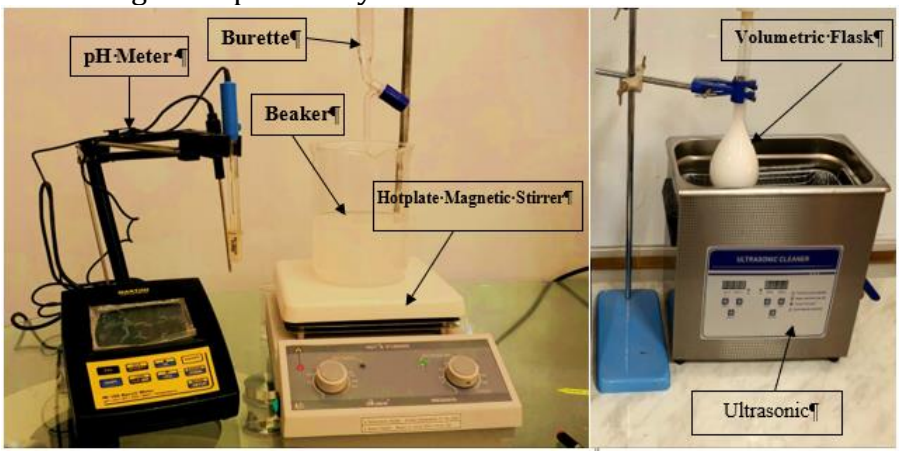

Figure 1 Setup of Chemical Sol-Gel Process System

3.Characterization

The structural characteristics of ZrO_2 were investigated utilizing "Japanese X-ray diffraction equipment (Rigaku)". The FE-SEM ("FEI NOVANANOSEM450I") was used to analyze the morphology (e.g., particle sizes and shapes), and Fourier Transform Infrared Spectroscopy FTIR

4.Results and Discussion

4.1 X-Ray Diffraction Analysis

The crystalline structure and other structural parameters, such as; (the distance between planes in crystals and the crystallite size) were measured for ceramic nanocomposite using X-ray diffraction (XRD) device. The Debye–Scherer equation (1) was used to calculate the average crystalline size

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \quad (1)$$

"D is the crystallite size (nm)", (λ)is the X-ray wavelength Cu-K radiation (0.15406 nm)، (θ)is the Bragg diffraction angle (degree)، and (β) is the diffraction peak's Full Width at Half Maximum (FWHM). The distance between the inter-planar spacings involved was measured using Equation (2). (d).

$$n\lambda = 2dsin\theta \quad (2)$$

Where:

d: denotes the distance between atomic layers in a crystal. λ represents the wavelength of incoming X-ray radiation, 2θ denotes the diffraction angle (Bragg's angle), and n is the order of the diffraction peak.[6, 7]

TABLE 2. Structural properties of ZrO_2.

Sample	**ZrO_2 at 600 ℃**		
Two θ° (Deg.)	Strongest 3 Peaks		
	0.29	0.51	0.04
hkl	111	202	311
d_{hkl} Exp. (A°)	.948	.805	.539
d_{hkl} Std. (A°)	.938	.799	.534
Crystallite size (nm)	5.07	3.29	1.26
The average Crystallite size (nm)	13.21		
Card No.	96-152-1754 cubic		

X-ray diffraction pattern analysis of the ZrO_2 calcination at 600 °C for 3 hours as shown in figure (2). The characteristic peaks for ZrO_2. Appear at 30.29,50.51and60.04 and are associated with the [111],[202], and [311] respectively, which are in good agreement Matching with (COD. No. 96-152-1754).

The average crystallite size was from the diffraction lines' full width half maximum (FWHM) Table 2).[6]

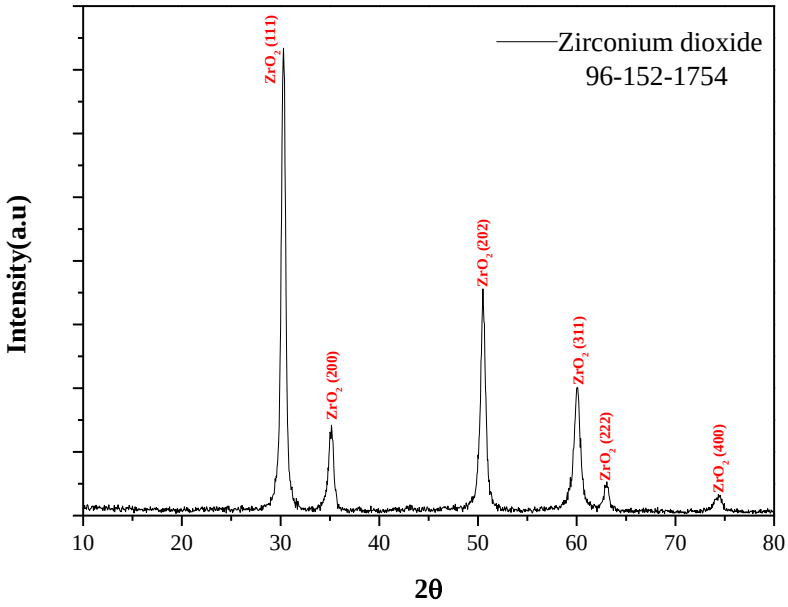

Figure 2 XRD Pattern of ZrO_2

4.2 Field Emission Scanning Electron Microscopy (FE-SEM) Results

Figures (3) show the surface morphology investigations by the FE-SEM of the ZrO_2. It is found that the average particle size is (14) nm, with a spherical shape and different agglomeration rate, the surface morphology of ZrO_2 nanoparticles has dendritic morphology, as shown in Figure (3). The particle's size and surface area were calculated by Image J software, and the average diameter was calculated by Origin pro software Table (3).[8, 9]

Table 3 The particle size and specific surface area of ZrO_2.

	(N)Total	Mean	St.D.	Sum	Min.	Med.	Max.
Diameter (nm)	63	14.883	4.50497	937.629	8.823	14.064	30.64
Aera (nm^2)	73	85.641	27.480	6166.15	29.401	82.857	160.368

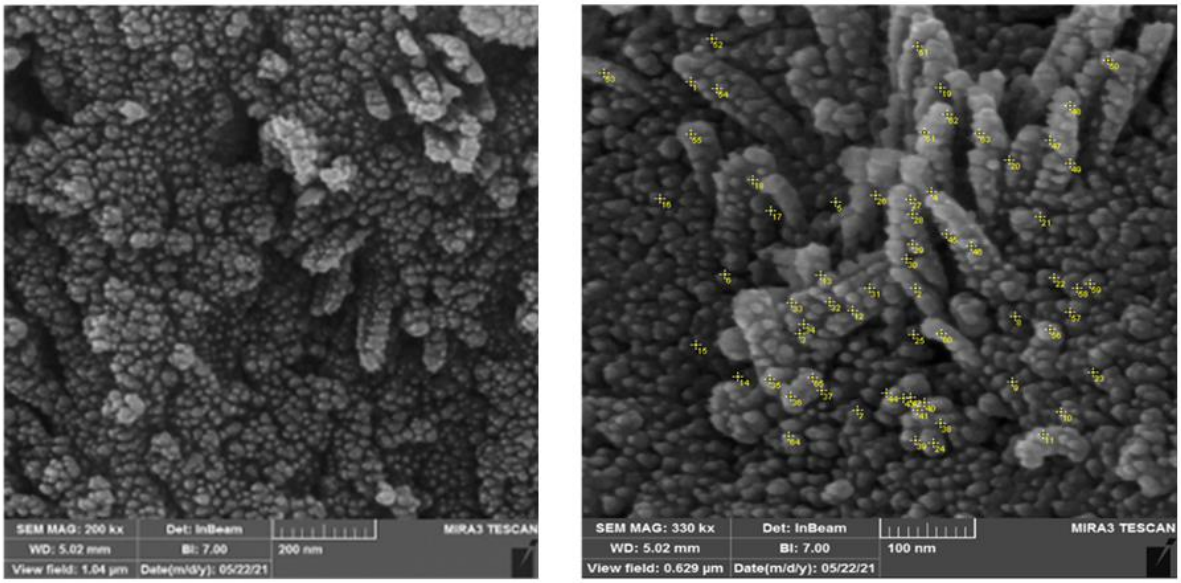

Figure 3 The SEM images of ZrO2

4.3 FTIR spectroscopy

To evaluate the formation and quality of the samples, FTIR spectroscopy is performed. The primary functional groups of ZrO_2 are shown in Figures 4. As a result of the stretching vibration of the C-H and O-H groups of water molecules adsorbed on the surface of ZrO_2 nanoparticles, a significant absorption band was found in the range of 2958 to 3720 cm^{-1}, with the peak cantered at 2036.46 cm^{-1}. This vibration represents the typical bending vibration of the Zr-OH group of hydroxyl zirconium. The C-O-H carboxylic acid bond is seen by the peaks at 1423.20 cm1. sharp peaks were observed around 620.96 to 867.81 cm^{-1}, corresponding to the stretching vibration of Zr–O of the ZrO_2 phase, and this confirms the Formation of ZrO_2 as the crystalline phase[10]

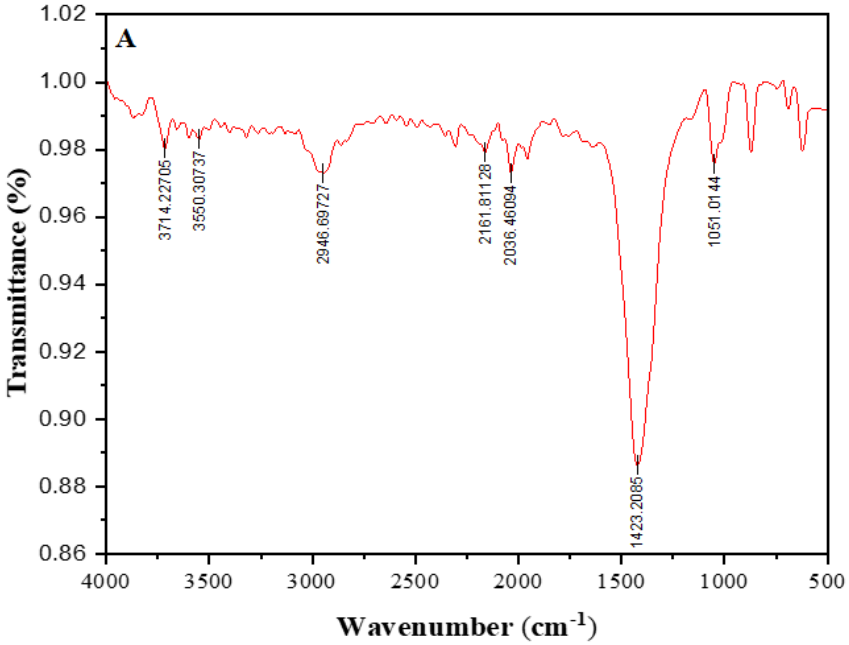

Figure 4 FTIR of ZrO2

Conclusion:

In this study, we successfully synthesized and characterized zirconium oxide (ZrO_2) nanoparticles using a chemical method, specifically the Sol-Gel process. The structural analysis through X-ray diffraction demonstrated the crystalline nature of the nanoparticles, with an average crystallite size of 13.21 nm. Field Emission Scanning Electron Microscopy (FE-SEM) revealed spherical particles with an average size of 14 nm and dendritic morphology. FTIR spectroscopy confirmed the presence of functional groups and the formation of ZrO_2. These findings underscore the controllability and scalability of chemical synthesis methods for tailoring ZrO_2 nanoparticles to specific applications. The properties observed in this study, such as small particle size and crystalline structure, make these nanoparticles promising for various fields, including catalysis, electronics, ceramics, and biomedicine. Further research can explore the utilization of these ZrO_2 nanoparticles in practical applications, contributing to advancements in technology and materials science.

References

[1] S. Jayakumar, P. Ananthapadmanabhan, K. Perumal, T. Thiyagarajan, S. Mishra, L. Su, A. Tok, J. Guo, Characterization of nano-crystalline ZrO2 synthesized via reactive plasma processing, Materials Science and Engineering: B 176(12) (2011) 894-899.

[2] N.A. Patil, B. Kandasubramanian, Biological and mechanical enhancement of zirconium dioxide for medical applications, Ceramics International 46(4) (2020) 4041-4057.

[3] M. Berner, V. Zarko, M. Talawar, Nanoparticles of energetic materials: synthesis and properties, Combustion, Explosion, and Shock Waves 49 (2013) 625-647.

[4] M. Euler, Nanotechnology in a Nutshell. From Simple to Complex Systems. Von Christian Ngô und Marcel Van de Voorde, Physik in unserer Zeit 46(1) (2015) 48-48.

[5] R.A. McCauley, Corrosion of ceramic materials, Crc Press2013.

[6] P. Parida, A. Behera, S.C. Mishra, Classification of Biomaterials used in Medicine, (2012).

[7] H.H. Al-Kayiem, S.C. Lin, A. Lukmon, Review on nanomaterials for thermal energy storage technologies, Nanoscience & Nanotechnology-Asia 3(1) (2013) 60-71.

[8] G. Cerrato, S. Bordiga, S. Barbera, C. Morterra, Surface characterization of monoclinic ZrO2: I. Morphology, FTIR spectral features, and computer modelling, Applied surface science 115(1) (1997) 53-65.

[9] A.A. Thamir, N.J. Jubier, J.F. Odah, Antimicrobial activity of zirconium oxide nanoparticles prepared by the sol-gel method, Journal of Physics: Conference Series, IOP Publishing, 2021, p. 012058.

[10] K. Kaviyarasu, L. Kotsedi, A. Simo, X. Fuku, G.T. Mola, J. Kennedy, M. Maaza, Photocatalytic activity of ZrO2 doped lead dioxide nanocomposites: Investigation of structural and optical microscopy of RhB organic dye, Applied surface science 421 (2017) 234-239.