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Investigations of Structural, Optical, and Stability Analysis of ZrO_2 Nanoparticles

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Abstract- In this research article, ZrO_2 metal oxide nanoparticles were prepared using microwave assisted solution method. The prepared sample were characterized by X ray diffraction (XRD), High Resolution Transmission electron microscopy (HRTEM), Particle size analyzer, Photoluminesence (PL), and Zeta potential. XRD pattern shows the prepared ZrO_2 nanoparticles having monoclinic structure which shows the average crystalline size around 25 nm. This was well agreed with TEM image also. PL spectroscopy is used to study the electronic structure of the prepared material and surface defects can also be analyzed. Particle size analyzer shows the particles are mostly aggregates. From the Zeta potential analysis the electric charge on the surface and the stability of the particles are concluded.

Index Terms- ZrO_2; Microwave; XRD; Zeta potential; Particle size analyzer.

1. INTRODUCTION

Metal oxide nanoparticles are of huge importance for the reason that of their high melting point. ZrO_2 is used as cutting tools and used to make engine components and furnaces. This gives the precious information about the absorbent behavior of various purposeful properties present in living entities and material dyes. Nanoparticles are great importance because of their high surface to volume ratio. There are many synthesis routes have been employed to obtain nano-sized tetragonal zirconia particles likes co-precipitation [1], Glycothermal Processing [2] Solid-State Reaction [3], Pechini Method [4], microwave-assisted solution method [5], bio-phase protocol [6], hydrothermal method [7] and sol-gel [8] processing. Among this Sol–gel is one of the best methods for synthesizing the nanoceramics e. g. zirconia, titania, hafnia, etc. Crystallite phase, crystallite size and other properties of zirconia nanoparticles are dependent on diverse parameters such as the type of precursor, pH during hydrolysis and thermal treatment [9]. Different methods are used to synthesize metal oxide nanoparticles. But microwave assisted solution method have environmental approach. Microwaves are the electromagnetic radiations with wavelength ranging from 1mm to 1m in free space and frequencies between 300GHz to 300 MHz respectively. The most common microwave frequency used for carrying out the research work is 2.45 GHz, similar to the frequency of domestic microwave oven. By the use of microwaves the synthesis of inorganic compounds has gained great importance, because this method offers several advantages compared to conventional heating method. Such as rapid heating, energy saving, fine micro structure, better product quality, environment friendliness etc. Microwave assisted synthesis is cleaner, faster and economical than the conventional method. So ZrO_2 nanoparticle has been synthesized via microwave assisted solution method at short time (10 min). The crystalline nature, optical studies and stability of the prepared particle were discussed in this paper.

2. MATERIALS AND METHODS

2.1. Preparation ZrO_2 nanoparticles

NaOH and Cl_2H_2OZr were taken as the precursor material for the preparation of zirconium oxide nanoparticle. In this work 8.6 g of zirconium oxy chloride was added with the 100 ml of distilled water and stirred one hour at room temperature for obtaining uniform mixture, during the stirring of above solution, NaOH solution was added drop by drop till the pH value of the solution becomes 12. After that white sol has been formed. This prepared solution kept at microwave oven with 60^oC at 5 minutes. Then the powder samples were filtered by deionized water and it's dried at room temperature for one hour. The resultant product was white colour ZrO_2 nanoparticles.

2.2 Characterizations

Powder XRD of the prepared sample was investigated by X-ray diffractometer (X PERT PRO PANALYTICAL, Netherland) to analysis the crystallite behavior. Cu Kα of wavelength λ =1.5 Å

was used and from the diffraction range 10 - 80°. The morphological analysis of the prepared samples was analysised by High Resolution Transmission electron microscope (model JOEL – J2000) with the operating voltage at 200 kV. ZrO_2 nanoparticle size has been analysed with the help of Shimadzu SALD – 2300 (wing SALD II: version 3.1.1) particle size analyser. The optical studies of the prepared sample were recorded using PL spectrometer. The prepared nanoparticle has also been studied the zeta potential analysis using Malvern Zetasizer 3000.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

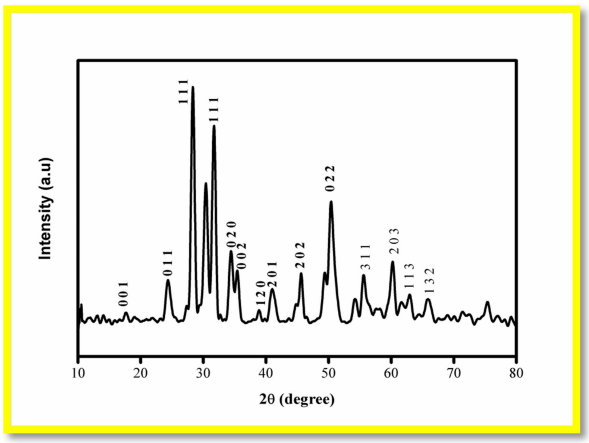

Figure 1. XRD spectrum of ZrO_2 nanoparticles

A zirconium dioxide nanoparticle synthesized by microwave assisted method using zirconium oxychloride as an oxidizer and was analyzed using powder XRD to confirm the structural information. The powder XRD pattern of fully dried powder of ZrO_2 shows clear diffraction peaks at angle in between 10 to 80°. Powder diffraction pattern of the prepared nanoparticles are shown in Figure 1. which clearly shows a main strong diffraction peaks along 28.28°, 24.16°, 30.32°, 34.2°, 35.4°, 50.19°, 40.9°, 45.6°, 60.3°, 65.8° with h k l values (011), (111), (020), (120), (201), (202), (022), (311), (203), (113), (132). These all diffraction peaks were clearly good consisted with standard diffraction data of ZrO_2 (JCPDS file no. 37-1484). Very less other peaks was shown in figure 1 and it was related to impurity which confirm to the purity of the prepared samples [10]. The broad diffraction peaks shows the prepared samples are nanosized. The crystallite size was calculated as around 25 nm from Scherrer equation. XRD studies are very important to know the behavior of the ZrO_2 nanoparticles formed and was revealed that the nanoparticles contain monoclinic structure [11].

3.2 HRTEM Analysis

Figure 2 shows the HRTEM micrograph of the freshly prepared ZrO_2 nanoparticle. Some nanoparticles were agglomerated due to electrostatic and magnetic attraction forces [12]. From the image is measured that the formed black spherical shape was evidence of formation of the nanoparticles. Figure 2 shows that every individual particle appears as black color. This shows the ZrO_2 nanoparticles having nearly spherical shape. The nanoparticle thickness clearly obtained from HRTEM micrograph and can be found as around 25 nm which is well agreed with XRD result.

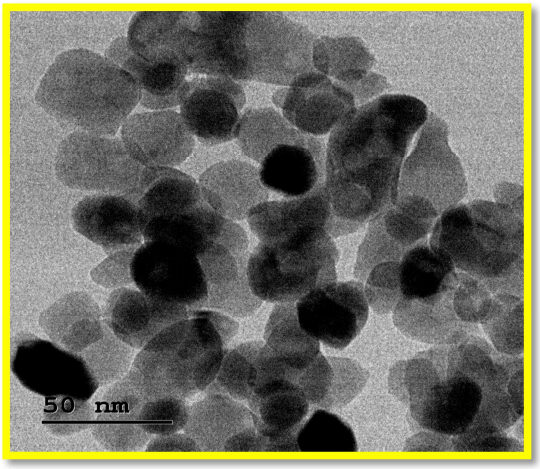

Figure 2. HRTEM image of ZrO_2 nanoparticle.

3.3 Particle Size Analysis

Figure 3 shows the particle size analysis of the prepared ZrO_2 nanoparticle the results are plotted as cumulative percentage frequency curve which shows the particle sizes are in micrometer range. According to XRD result the particle size are in nm range. So the prepared ZrO_2 nanoparticles are aggregated, from the graph we conclude the particle diameter is evenly distributed so the prepared nanoaprticles are spherical. This was also support to confirm the result by HRTEM image of the prepared sample. Thus, the particle size results are also in nm range.

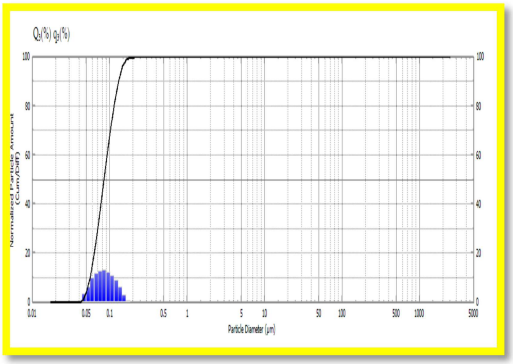

Figure 3. Particle size analysis of ZrO_2 nanoaprticle

3.4 PL Spectroscopy Analysis and Zeta Potential

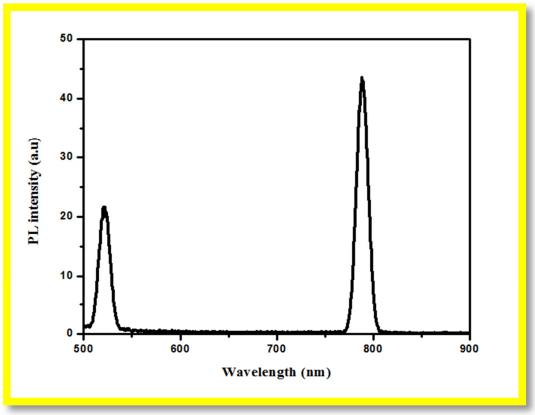

Figure 4. Photoluminesence spectrum of ZrO_2 nanoparticle

The PL methods are suitable to find the crystallite quality of the prepared nanoparticles and it is used to study the electronic structure of the prepared material. The prepared nanoparticles absorb the incident photons and reemit the photons. This analysis is used to identify the surface defects, impurity levels and disorders in the prepared nanoparticles. Figure 4 shows the PL spectrum of ZrO_2 nanoaprticle. From the figure 4 shows the room temperature photoluminescence pattern of ZrO_2 nanoparticles excited at two different wavelengths. The emission on the visible region at 550 nm shows the recombination of an excited electron in the conduction band with the hole in the valence band [13]. The emission at 563 nm should be the involvement of mid gap trap states such as surface defects and oxygen vacancies. Large amount of surface defects exist on the nanosized ZrO_2 nanoparticles because of their high surface area.

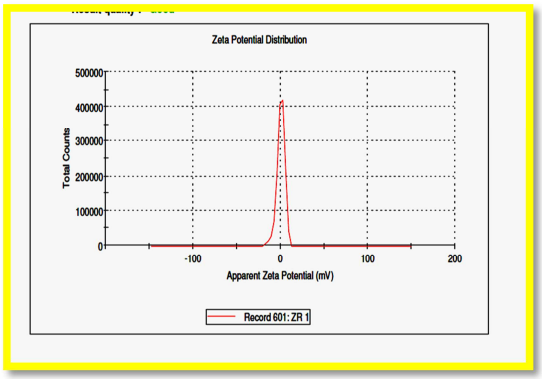

Figure 5. Zeta potential of ZrO_2 nanoparticles.

Zeta potential measures the electric charge on the surface of the prepared ZrO_2 nanoparticle. From the value of Zeta potential the stability of the particle has been concluded. A low value of Zeta potential suggest the repulsive forces are greater then the attractive force. Similarly high value of zetapotentential suggest the attractive forces are greater then the repulsive forces. Here the prepared ZrO_2 nanoparticle zeta potential value has been 0.3 mV as seen in figure 5.

4. CONCLUSION

Zirconium dioxide nanoparticles were synthesized by microwave assisted sol gel method. The prepared nanoparticle having monoclinic structure which is confirmed by XRD analysis and the particle size can be calculated as 25 nm using Debye scherrer formula, which was also confirmed by TEM image. Particle size analysis of the prepared ZrO_2 shows high degree of agglomeration due to the small size and intermolecular attraction. PL spectroscopy shows the prepared ZrO_2 nanoparticles having large amount of surface defect due to their high surface area. From the zeta potential analysis the high stability of the prepared particles can be confimed.

REFERENCES

[1] M.C. Chin, W.L. Li, C.W. Cheng, K.M. Cheng, Y.F. Chen, and C.S. His. “The phase transformation and crystallization kinetics of (1-x) Li_2O–x Na_2O–Al_2O_3–$4SiO_2$ glasses”. Mater. Chem. Phys., 123: 203-209, 2010.

[2] X. Liu, G. Lu, and Z. Yan. “Preliminary synthesis and characterization of mesoporous nanocrystalline zirconia”. J. Nat. Gas Chem., 12: 161-166, 2003.

[3] C. Jose, V.R. Mastelaro, V. Nascente, J.B. Zotin, E. Longo, and E.R. Leite. “Oxide surface modification: synthesis and characterization of zirconia-coated alumina”. J. Colloid Interface Sci., 343: 256–262, 2010.

[4] R. Dwivedi, A. Maurya, A. Verma, R. Prasad, and K.S. Bartwal, “Microwave assisted sol–gel synthesis of tetragonal zirconia nanoparticles”. J. Alloys Compd., 509: 6848-6851, 2011.

[5] S.C. Vella Durai, L. Guru Prasad, E. Kumar, D. Muthuraj, and V. Bena Jothy. “Investigation on Structural and Optical Properties of Manganese dioxide nanoparticles by Microwave Assisted Solution Method”. International Journal of Rearch in Advent Technology, 6: 1531-1535, 2018.

[6] F. Massodiyeh, J. Karimi, A.R. Khanchi, and M.R. Mozdianfard. “Zirconia nanoparticle synthesis in sub and supercritical water-particle morphology and chemical equilibria”. Powder Technol. 269: 461-469, 2015.

[7] E.A. Trusova, A.A. Khrushcheva, and K.K. Vokhmintcev. “Sol–gel synthesis and phase composition of ultrafine ceria-doped zirconia powders for functional ceramics”. J. Eur. Ceram. Soc., 32: 1977-1981, 2012.

[8] B. Tyagi, K. Sidhpuria, K.B. Shaik, and R.V. Jasra. “Synthesis of nanocrystalline zirconia using sol–gel and precipitation technique”. Ind. Eng. Chem. Res., 45: 8643– 8650, 2006.

[9] B. Bagchi, and R.N. Basu. "A simple sol-gel approach to synthesize nanocrystalline 8 mol% yttria stabilized zirconia from metal-chelate precursors: microstructural evolution and conductivity studies". J. Alloys Compd., 647: 620- 626, 2015.
[10] Piqi Zhao, Lingchao Lu, Xianping Liu, G.D.T. Angeles, and Xin Cheng. "Error Analysis and Correction for Quantitative Phase Analysis Based on Rietveld-Internal Standard Method: Whether the Minor Phases Can Be Ignored?". Crystals, 8: 1-11, 2018.
[11] S.K. Aysar, E. Saion. A. Zakaria. and N. Soltani. "Structural and Optical Properties of Zirconia Nanoparticles by Thermal Treatment Synthesis". Journal of Nanomaterials, 2016: 1-6, 2016.
[12] N.A. Madhan, Kun Guo. and Y. Zhixin. "A State-of-the-Art Review of Nanoparticles Application in Petroleum with a Focus on Enhanced Oil Recovery". Applied Sciences, 8: 1-29, 2018.
[13] A.K. Singh, and T.N. Umesh. "Microwave Synthesis, Characterization, and Photoluminescence Properties of Nanocrystalline Zirconia". The Scientific World Journal, 2014: 1-7, 2014.