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ROLE OF PRECIPITATION AGENT ON ZRO_2 NANO PARTICLE SYNTHESIS USING CO-PRECIPITATION TECHNIQUE

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ABSTRACT- In this attempt, Zirconia (ZrO_2) was successfully synthesized using the optimized wt. % of Zirconium nitrate as a source material and the appropriate amount of sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium carbonate (Na_2CO_3) and ammonia (NH_3) as precipitation agent using co-precipitation method in the absence of surfactant. The monoclinic structure was confirmed using XRD analysis (JCPDS card no.89-9066). The crystallite size was calculated as 20-58nm using Debye-Scherer formula. Zr-O stretching vibration and Zr-O_2-Zr asymmetric vibrations were confirmed through FT-IR analysis. The different morphology was obtained through SEM analysis. The as prepared ZrO_2 could be used as filler in the lithium polymer battery electrolyte application.

Keywords – [ZrO_2, co-precipitation method]

1. INTRODUCTION

Zirconia (ZrO_2) has been extensively used in many fields enclosing such as catalysts, catalytic supports, oxygen sensors, fuel cells dielectrics, super capacitor etc., Enormous studies motivated the development of Zirconia nanoparticles with enhanced properties, which exposed that physical and chemical properties of the materials not only depended on their chemical composition and phase structure but were also associated to their morphology, size etc., [1-3]. Numerous methods including hydrothermal, sol-gel, chemical vapor deposition, sputtering, microwave assisted and co-precipitation method have been used to prepare Zirconia nano particles with different morphologies and size such as nano spheres, nano fibers, nano belts and flower-like structures.

Among these, co-precipitation method has been widely practiced owing to the production of homogenous, high-purity and crystalline oxide powders at low cost and also this simple method allows scaling up for mass production [4]. In this attempt, Zirconia (ZrO_2) was synthesized using Zirconium nitrate as a source material and different precipitation agents like NaOH, KOH, Na_2CO_3 and NH_3 using co-precipitation method in the absence of any surfactants and templates. The as prepared ZrO_2 were characterized for

structural, vibrational and morphology studies by XRD, FTIR and SEM analysis.

2. MATERIALS AND METHODS

Samples were obtained by co - precipitation method with sodium hydroxide, potassium hydroxide, sodium carbonate and ammonia as precipitation agent and zirconium nitrate as raw material. The morphology of the powder was analyzed by scanning electron microscopy (SEM, Hitachi S-4700 Type II) operated at an accelerating voltage of 25Kv. The phase and crystalline size were estimated through X-ray Diffraction measurements (XPERT-PRO with Cukα radiation). FTIR study was made using Thermo Nicolet 380 Instrument Corporation with KBr in the 4000-400 cm^{-1} region.

3. RESULTS AND DISCUSSION

A. XRD ANALYSIS

The XRD pattern of the prepared ZrO_2 nanoparticles (Fig.1) with various precipitation agents such as NaOH, KOH, $Na_2CO_3$and NH_3 were labeled as A1, B1, C1 and D1 respectively. A monoclinic structure of the ZrO_2 with lattice parameter of a= 5.146; b= 5.205; c=5.313Å is observed, and the peaks at 2θ = 23.98, 27.97, 31.43, 34.89, 50.32 and 60.15° were assigned to (011), (1̄11),(111),(102),(1̄22) and (310) reflection planes of monoclinic ZrO_2 respectively, and agreed well with that in the JCPDS card no.89-9066. The absence of impurity peaks and high intensity peaks in the pattern revealed that the prepared ZrO_2 particles were pure and in good crystalline nature. The crystalline size of the particles was estimated by Debye-Scherer formula. The values are 51, 58, 31 and 20nm for A1, B1, C1 and D1 respectively. The precipitation agent had significant role on the ZrO_2 crystalline size. This was in very close agreement with the previous literatures [5-6].

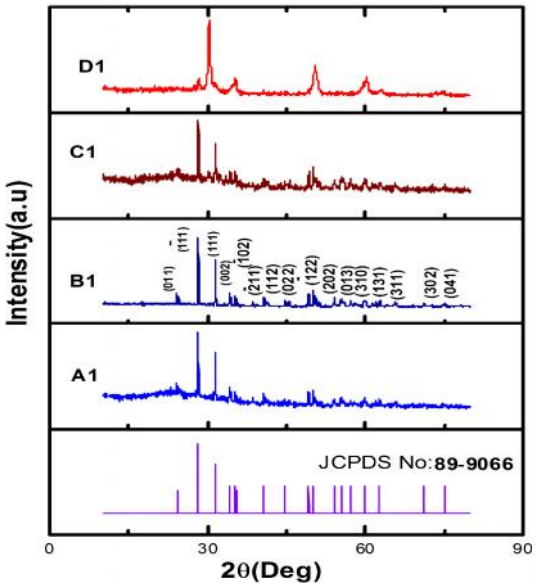

Figure 1- XRD pattern of ZrO_2 nano A1) NaOH B1) KOH C1)Na_2CO_3 D1) NH_3 as a precipitation agent

B. FTIR analysis

The bonding nature and purity of nanoparticles were examined by the FT-IR. Fig.2 shows the FT-IR spectra of ZrO_2nanoparticles using various precipitation agents. The sharp peaks at 510 and 771 cm^{-1} corresponding to Zr-O stretching and Zr-O_2-Zr asymmetric mode respectively of monoclinic structure of ZrO_2 confirm the presence of monoclinic ZrO_2 nano particle and broadness of the band indicated that ZrO_2 powders were nano crystals [7].

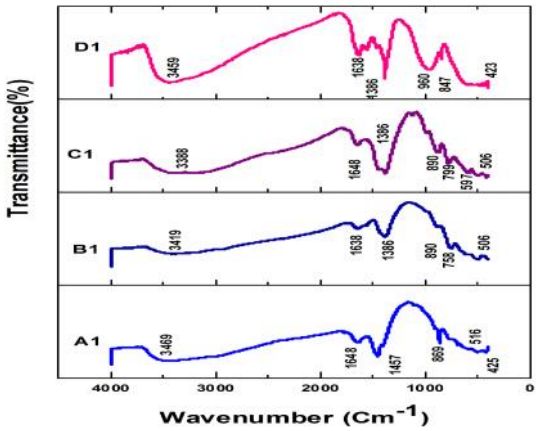

Figure 2- FTIR spectra of ZrO_2 nano particles A1) NaOH B1) KOH C1) Na_2CO_3 D1) NH_3 as a precipitation agent.

The broad peaks located at 3465 and 1656 cm^{-1} revealed the –OH stretching and bending vibration of absorbed water and the peak at 1408 cm^{-1} implied that the hydrated molecular could be in hydroxyl group[8]. The FT-IR studies were in agreement with the XRD pattern of the ZrO_2 as both confirmed the presence of monoclinic ZrO_2 phase.

B. SEM analysis

Fig 3 shows the surface morphological study of the samples A1 to D1. The samples exhibited different morphology. The sample A1 showed bundles of nano rods (0.31× 0.52µm). B1 and C1 showed that microstructure of grains of the sample were ultra-small and agglomerated.

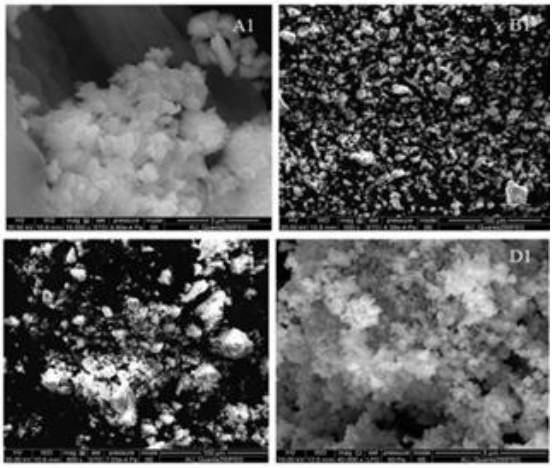

Figure 3 SEM images of ZrO_2 nano particles A1) NaOH B1) KOH C1) Na_2CO_3 D1) NH_3 as a precipitation agent.

Most of the grains containing a large number of atoms were very small in dimensions, having average size of 3µm and 6µm. The size distribution was extraordinary narrow. D1 revealed cauliflower like morphology and the particles were in agglomeration nature. The particle size was detected as 76nm. This process of particle morphology evolution can be described in terms of oswald-ripening [9].

CONCLUSION

The Zirconia (ZrO_2) was successfully synthesized by co-precipitation method using various precipitation agents such as NaOH, KOH, Na_2CO_3 and NH_3. The monoclinic structure with Fm-3m phase group was confirmed by XRD analysis. All the peaks were exactly correlated to the JCPDS card no.89-9066. The Zr-O stretching vibration and Zr-O_2-Zr bending vibrations were confirmed through FTIR analysis. The different morphology of the powder was observed through SEM analysis. The bundles of nano rod with the size of (0.31× 0.52µm) were observed for NaOH as precipitation additive. With NH_3 as precipitation agent, the cauliflower like morphology and agglomeration of particles were observed. Some particles were visualized and size was detected as 76nm. The ZrO_2 nano particle was tuned in terms of structural, morphology and size. The as prepared nano ZrO_2 could be used as filler in Lithium polymer battery application.

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REFERENCES

[1]. Z Shu, X Jiao, D.Chen “Hydrothermal synthesis and selective photocatalytic of tetragonal star-like ZrO_2 nano structures”, Cryst Eng comm, vol.15, pp.4288-4294, Mar.2013.

[2]. D.S.S.Padovini, D.S.L.Pontes, C.J.Dalmaschio,F.M.Pontes, E.Longo,”Facile synthesis and characterization of ZrO_2 nanoparticles prepared by the AOP/hydrothermal route”, RSC Adv, vol.4, pp.38484-38490, Sep 2014.

[3]. K.Anandan, V.Rajendran, “A low-temperature synthesis and characterization of tetragonal-ZrO_2 nanoparticles via simple

Hydrothermal process" j.of Phy sci, vol.17, pp-179-184, Dec 2013.

[4]. M.Rezaei, S.M.Alavi, S.Sahebdelfar, Zi-Feng yan, "Effect of process parameters on the synthesis of mesoporous nanocrystalline Zirconia with triblock copolymer as template" J.porous mater, vol.15, pp-171-179, 2008.

[5]. F.J.Pereira, M.T.Diez, A.J.Aller, "Effect of temperature on the crystallinity, size and fluorescent properties of Zirconia-based nanoparticles", Materials chem and phy, vol.152, pp-135-146, Feb 2015.

[6]. K.Boopathy, A.Samson Nesaraj, V.Rajendran, "Self-propagating combustion synthesis of Pb_{1-x} Sr_x ZrO_3 ($0<x<0.20$) ceramics and their dielectric properties, Bull.mater.sci, vol.31, pp-149-153, April 2008.

[7]. H.Ranjan Sahu, G.Rangarao,"Characterization of combustion synthesized Zirconia powder by UV-vis IR and other tehniques, Bull.mater.sci,vol.23, pp349-354, Oct 2000.

[8]. Eskanda kolvari, Nadvia koukabi, Maliheh, M.Hosseini, Mitra vahidian, Elhan ghobadi, "Nano-ZrO2 suluric acid: a heterogenous solid acid nano catalyst for Biginelli reaction under solvent free conditions, RSC Adv, vol.6, pp-7419-7425, 2016.

[9]. Nyugen T.K.Thanh, N.Maclean, S.Mahiddine,"Mechanism of nucleation and growth of nanoparticles in solution, Chem-Rev, vol.114, pp-7610-7630, 2014