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Wet Chemical Synthesis and Characterization of Zirconia : As A Biomaterial

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ABSTRACT

The objective of the following study was synthesis of zirconia by wet chemical method and evaluation of its structural and biological properties. The sample was characterized by powder X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and evaluated the antibacterial property are investigated by spread plate method against *E. coli* bacterial pathogen and studied for degradation using phosphate buffered saline (PBS) solution. The XRD pattern shows that the monoclinic phase of zirconia was obtained. The FESEM images showed that the prepared sample consists homogenous particle size distribution. The sample of zirconia inhibited the bacterial growth. The sample shows stability at physiological condition and does not show degradation.

Keyword: Zirconia, wet chemical synthesis, biocompatibility, biomedical application

I. INTRODUCTION

Zirconia based ceramic materials have received extensive interest in the past decades as important structural ceramic and biomedical materials. The excellent electrical, mechanical, optical and thermal properties of zirconia, makes it a good choice for application such as: structural materials [1], dental crowns [2], femoral heads for total hip replacement [3], solid oxide fuel cell electrolytes [4], air-fuel ratio sensors for automotive applications [5], Catalytic application [6].

There are many synthesis routes have been employed to obtain zirconia particles likes co-precipitation [7], Glycothermal Processing [8] Solid-State Reaction [9], Pechini Method [10], microwave-assisted sol-gel synthesis [11], bio-phase protocol [12], hydrothermal method [13] and sol-gel [14].

In the present work zirconia synthesized using efficient wet chemical method and characterize the sample for their structural and biological properties.

II. METHOD AND MATERIALS

A. Co-precipitation of zirconia

Aqueous zirconium chloride ($ZrOCl_2$) was added drop by drop with sodium hydroxide (NaOH) solution kept at PH around the 10. Then it gets converted into precipitation. It stirs for 1 hrs. The precipitation were allowed to settle over night followed by decantation and washing. The resulting precipitates were dried for 24 hrs and made pellets. The resulting pellets were sintered by using microwave furnace at 800^0C for a 2 hrs respectively.

B. Evaluation of antimicrobial activity

Viable count method, a relatively quick and easily executed semi quantitative test is employed to determine the antibacterial potential of ZrO_2 powder from pellets against bacterial pathogen *E. coli* was used for testing. The agar used is Muller –Hinton agar that is rigorously tested for composition and pH. The stock solution was prepared by mixing 1ml *E. Coil* with 9ml Luria-Bertani broth and incubated at 37^0c for 24 hrs with shaking at 250 rpm. The broth was serially diluted up to 10^5 concentrations for minimizing the load of bacterial cells. 0.01g and 0.1 g ZrO_2 extract were mixed with stock solution. 0.1ml of prepared mixture was then inoculated on Luria-Bertani agar plates followed by incubation 37^0c for 24 hrs. Then, the number of colony-forming units was counted for each dilution.

C. Study of degradation

Degradation test of Zirconia pellets were done by taking phosphate buffered saline solution. The pH of solution was 7.4 at 37^0c. Initially, took the weight of pellets P1, P2 and P3. Then pellets were soaked in phosphate buffered saline solution. The pellets were dried at 120^oc after every one week and final weight of sample was taken. This process was repeated for five weeks.

$$\%\text{Weight loss} = \frac{W_0 - W_t}{W_0} * 100$$

Where, W_0 = initial weight of pellet

W_t = final weight of pellet after soaking in phosphate buffered saline solution

III. RESULT AND DISCUSSION

Powder X-ray Diffraction analysis of sintered samples was carried out in order to study the structural properties of zirconia using a Rigaku diffractometer (XRD, miniflex Rigaku. Field effect scanning electron microscopy (FESEM) technique was also used to observe the surface morphology. For this, a very small amount of powder was placed on an adhesive carbon tape, coated with gold/palladium and then observed in a FE-SEM (HITACHI S-4800).

A. X-ray diffraction (XRD) analysis

The structural properties are studied by X-Ray diffraction technique. The XRD pattern of CP synthesis ZrO_2 as shown in Fig. 1 which is well matched with standard ICCD file no, 01-074-0815). ZrO_2 has a single pure phase with a monoclinic crystal structure which was sintered at 800^0C.

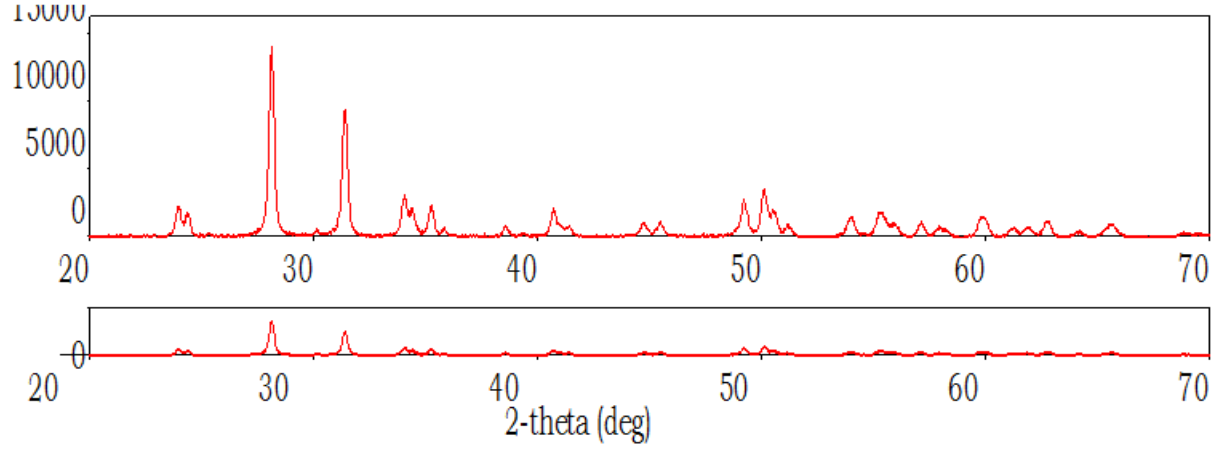

Figure 1: XRD patterns of CP synthesis ZrO_2 sintered at 800°C

B. FESEM analysis

Field Effect scanning Electron Microscope (FESEM) is well known and reliable technique to analyze nanoscale samples. It gives surface morphology of samples. The FESEM image for ZrO_2 Fig. 2at different magnification shows uniform distribution of particles and spherical like morphology.

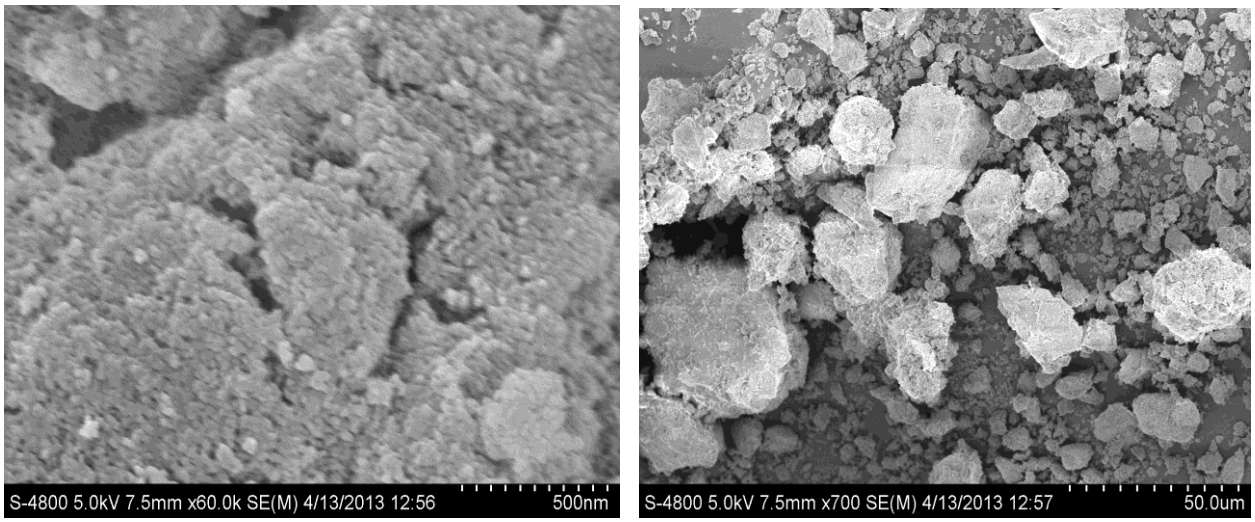

Figure 2: FE-SEM images of ZrO_2 at different magnification

C. Antibacterial assessment of ZrO_2

Extract from ZrO_2 pellets tested for its antimicrobial activity against bacterial pathogen, *E. coli* by viable count method. Fig. 3 (a) is control plate (without extract of ZrO_2) shows growth of bacterial pathogen. Fig. 3 (a-b) shows ZrO_2 pronounced significant growth inhibitory effect against surface area by their size. However ZrO_2 particles possess superior antimicrobial bacterial activity against E. coli bacteria which are clearly visualized in the antibacterial photograph. The antimicrobial performance due to the following assumptions: active oxygen species generated from the ZrO_2 particles actively inhibit the growth of *E. coli* cells by accumulation or deposition on the surface of *E. coli* cells. It is also suggested that ZrO_2 nanoparticles are able to slow down *E. coli* growth due to disorganization of *E. coli* membranes, which increases membrane permeability leading to

accumulation of nanoparticles in the bacterial membrane and cytoplasmic regions of the cells. From the above discussion we clearly came to know about the enhanced antimicrobial activity of ZrO_2 particles.

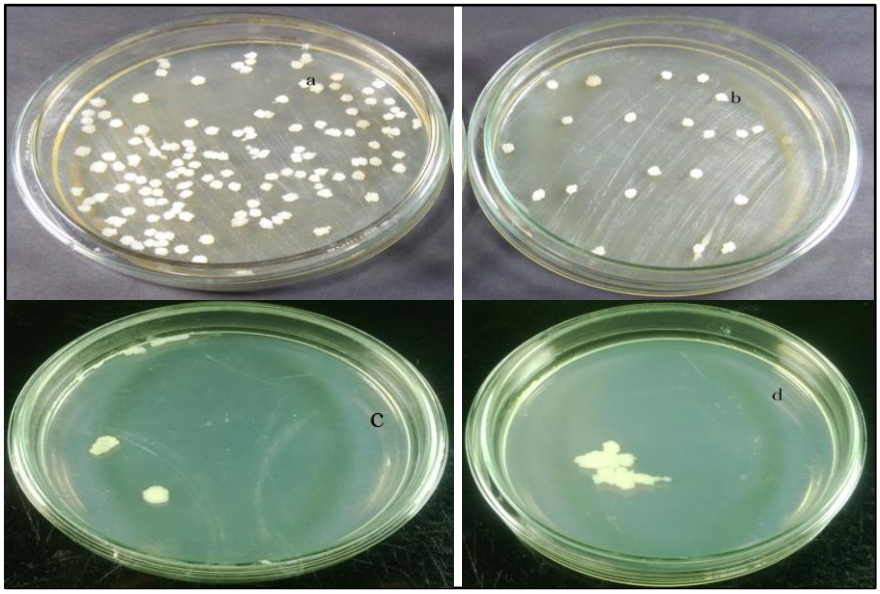

Figure: 3 photographs of (a) in absence of ZrO_2 powder, (b-c) ZrO_2 powder from pellet

D. Degradation of ZrO_2

Fig. 4 shows the degradation of ZrO_2 materials. The Pellets of ZrO_2 powder shows that 0.0021% for five weeks which is very negligible. It means that a zirconia material does not show any degradation and stable at physiological condition in PBS solution. zirconia is inert bioceramic that way it does not shows any degradation and physiologically stable.

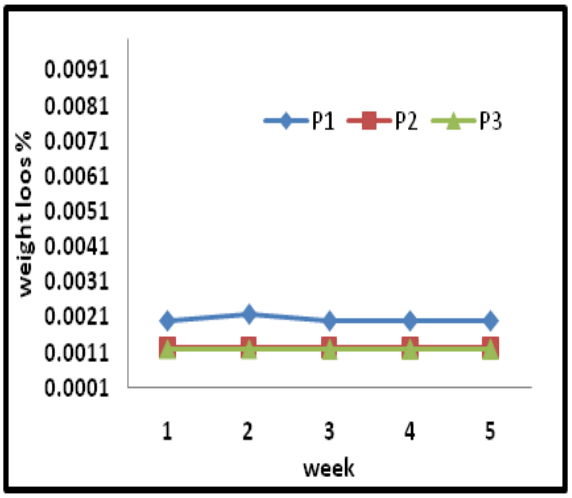

Figure: 4 Degradation of ZrO_2 pellets in PBS

IV. CONCLUSIONS

Zirconia was synthesized by co-pracipretation method and its structural and biological properties were studied. The formation of monoclinic crystalline phase was confirmed by powder XRD. The morphology, particle size were analyzed using field effect scanning electron microscopy. zirconia shows inhibition of bacterial growth. Since, the sample is stable at physiological condition; it can be used for biomedical applications.

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