

## Synthesis of Mesoporous Zirconia by Using Alkoxide Precursor and Triethanolamine as Hydrolysis Stabilizer

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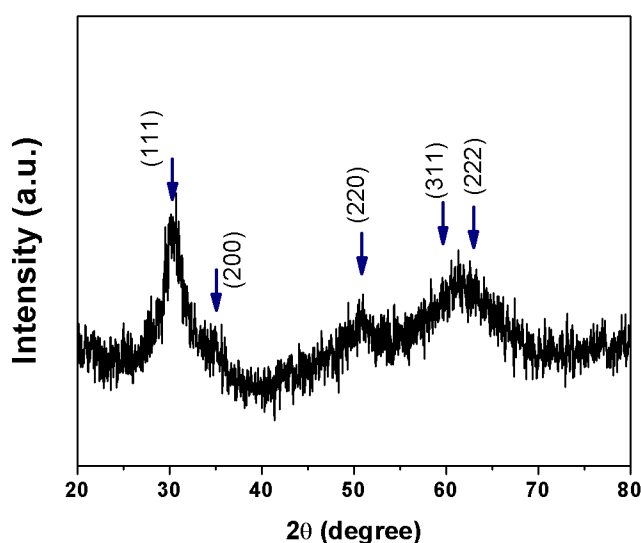
A mesoporous material is one that has pores in a range between 2 and 50 nm. At present, there are several kinds of mesoporous ceramic metal oxides that have been synthesized by different template methods like “Soft template method” and “hard template method” [1]. In the case of the synthesis of mesopores of zirconia many procedures have been developed using zirconium salts as precursor [1-3]. However, there is not enough research about the synthesis of mesopores of zirconia with alkoxide precursors using TEAOH (triethanolamine) as hydrolysis stabilizer. In this work, mesoporous zirconia was synthesized by using a combination of sol-gel method and soft hydrothermal method. The mesoporous of zirconia was prepared by using zirconium butoxide (Aldrich), TEAOH (J. T. Baker), hexadecyltrimethyl ammonium bromide, CTAB (Sigma), sodium hydroxide (J. T. Baker) as Zr-source, hydrolysis stabilizer, template and alkaline material, respectively. In a first step, a stock Zr-solution was prepared by mixing 0.2 mol of TEAOH with 0.1 mol of Zr-source with vigorous stirring for two days. After that, two additional solutions were prepared. The first one was obtained by dissolving 3.4103 g of stock Zr-solution and 3.6445 g of CTAB in 20 g of distilled water. For the second solution, 1.6 g of NaOH was dissolved in 10 g of distilled water. In a second step, the Na-solution was added slowly (drop by drop) to Zr-CTAB-solution and stirred. The final solution was stirred and heat in a hot stir plate at 90 °C in order to evaporate the excess of water and to obtain a material with a molar ratio of Zr-source:TEAOH:CTAB:NaOH:H<sub>2</sub>O equal to 1:2:2:8:150. The gel obtained was aged in a polypropylene bottle at 80 °C for 1 day. After the hydrothermal treatment, the sample was washed with 100 ml of distilled water and centrifuged at 12000 rpm. Then, the material was dried at 80 °C for 1 day. On the other hand, it is necessary the calcination of the sample at 560 °C for 6 h to eliminate the residual organic template. The phase composition of the sample was analyzed using X-ray diffraction (XRD, D2 Phaser Bruker), with a Bragg-Brentano geometry and Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) using the following scan: step size = 0.02°, t = 5 s, 20° ≤ 2 $\theta$  ≤ 90°. The morphology of as-synthesized sample was corroborated using a high resolution scanning electron microscopy (HR-SEM, Jeol JSM-7800F).

Figure 1 shows the XRD pattern of the as-synthesized sample, which corresponds to the formation of zirconium dioxide in a cubic phase of ZrO<sub>2</sub> nanoparticles, where the characteristic peaks are in agreement with the standard data of ICDD #00-049-1642. It is important to remark that no impurity

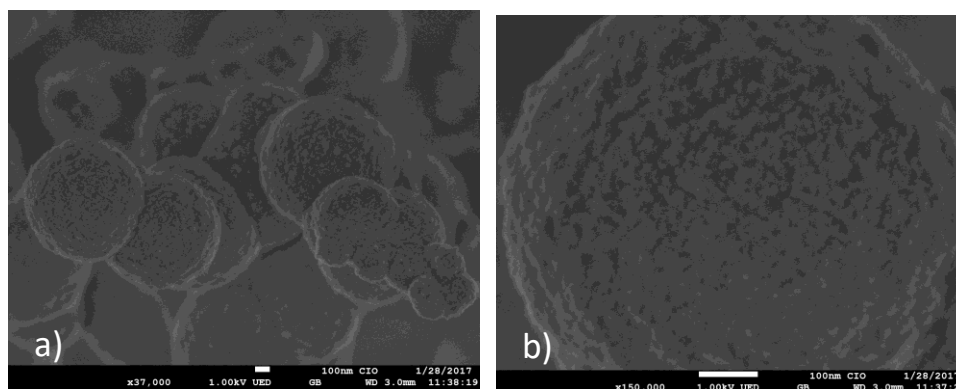
phases were observed. Figure 2 shows images of as-synthesized mesoporous zirconia samples with a pores size around 10 nm. There are several parameters of synthesis that allows shaping the mesopores in zirconia; for example, the amount of water, the temperature of the hydrothermal treatment, the amount of mineralized material, the source of zirconium and the amount of template material. According to our results, the critical synthesis conditions are the temperature of the hydrothermal treatment and the molar ratio between the template and zirconium. Also, the kind of alkaline material and its amount during the synthesis allows controlling the pH that could be a critical factor to obtain mesoporous zirconia. It is important to mention that we stabilize zirconium butoxide with TEAOH in order to make the slower formation of gel and increase the possibility to form mesopores [4].

#### References:

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**Figure 1.** XRD pattern of the as-synthesized sample



**Figure 2.** SEM images of mesoporous zirconia.