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Synthesis of Mesopores of Zirconia by Using CTAC as Template

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A mesoporous material is a material, which contains pores in a range between 2 and 50 nanometers [1]. This type of material can be used as catalyst for bulky reactant molecules due their large pore size. The first publication about synthesis of mesoporous materials by using a surfactant template was in 1992 by Mobil scientists [2]. After that, several methods have been developed for the synthesis of mesoporous metal oxides [1]. However, the synthesis of mesoporous zirconia by using Cetyltrimetylammonium chloride (CTAC) as template has not investigated. The mesopores of zirconia was prepared by using zirconium oxide chloride octahydrate (Sigma-Aldrich, purity 99.5%), tetramethylammonium hydroxide solution TMAOH (Sigma-Aldrich, 25 wt% in water) and cetyltrimethylammonium chloride solution, CTAC (Aldrich, 25 wt% in water) as source**.** In a first step, 1.611 g of $ZrOCl_2$*$8H_2O$ was dissolved in 7.2 g of destilled water. Then, 3.2 g of CTAC solution was added to the Zr-solution. For the second step, 10.94 g of TMAOH solution was added slowly (drop by drop) to Zr-surfactant solution and stirred. The final suspension was stirred and heat in a hot stir plate at 90 °C to get a material with a molar ratio of $ZrOCl_2$*$8H_2O$:CTAB:TMAOH:H_2O equal to 1:0.5:6:75. The gel obtained was aged in a polypropylene bottle at 80 °C for 1 day. After that, the hydrothermal treatment, the sample was washed with distilled water, centrifuged and dried at 80 °C for 1 day. The powder X-ray diffraction patterns were collected with a siemens D5000 X-ray diffractometer equipped with graphite monochromatized high–intensity CuK_λ (λ=1.54178 Å). The Bragg angle 2θ ranges from 1° to 8° at a scanning rate of 0.02°/s. The surface morphology images of the samples were analyzed by using a scanning electron microscopy FEG-SEM JEOL JSM 7600. Figure 1 shows the XRD pattern of zirconia synthesized. This figure shows a characteristic peak about the presence of mesopores in the material around 1.04 degrees. Figure 2a shows an image of mesoporous zirconia. Figure 2b shows the EDS spectrum of the mesopores. The results shows that the optimal hydrothermal treatment temperature for mesoporous zirconia synthesis is at 80 °C because more temperature of treatment could collapse mesopores formation in zirconia and it was developed an easy procedure for the synthesis, which can be, applied at different materials.

References

[1] D.W. Bruce, *et al*., **Volume** 1 (2010), p. 1.
[2] C. T. Kresge *et al*, Nature **Volume** 359 (1992) p. 710.
[3] The authors acknowledge funding from H. Consejo Técnico of Institute of Physics and Mathematics, and Consejo Nacional de Ciencia y Tecnología (CONACyT), México.

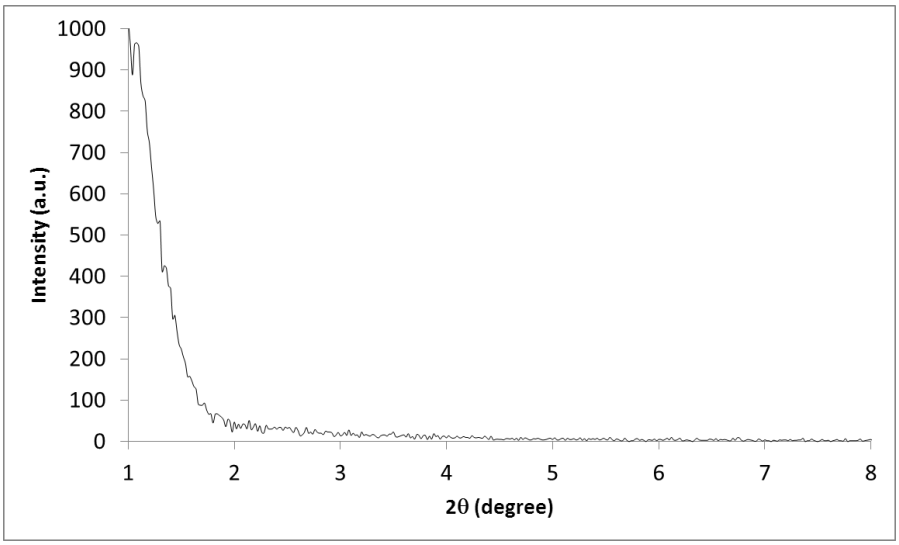

Figure 1. XRD pattern of mesoporous zirconia

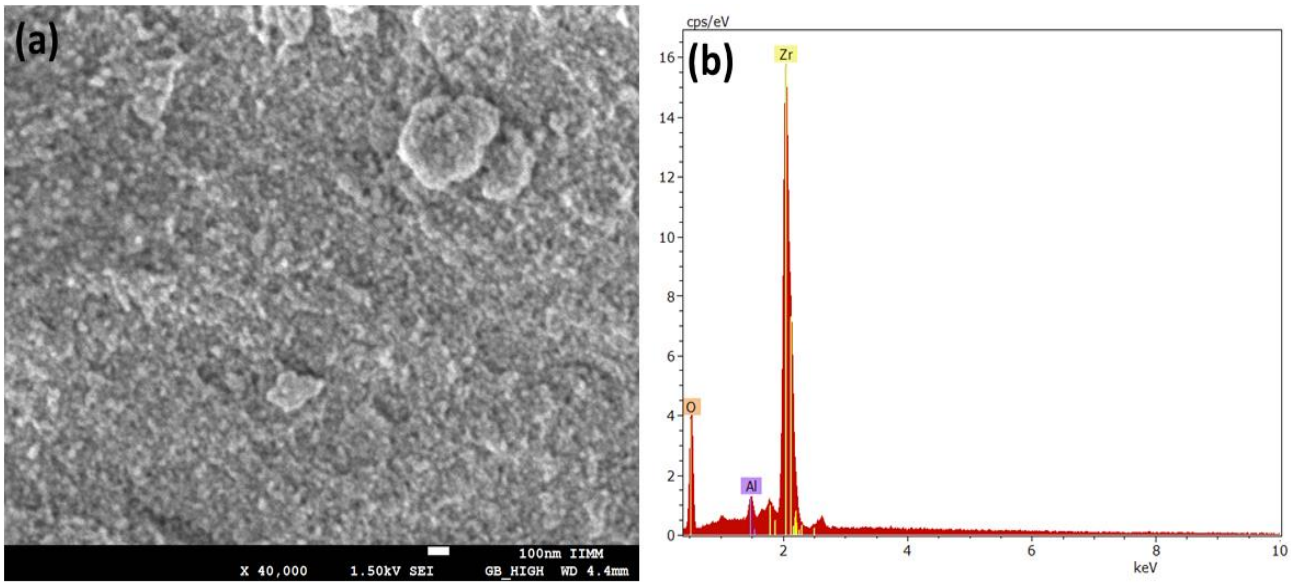

Figure 2. a) SEM image of mesoporous zirconia, b) EDS spectrum of the mesoporous of zirconia.

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