Synthesis of Mesoporous Ceria by Using CTAB as Template

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A material which contains pores in a range between 2 and 50 nm is considered as mesoporous material [1]. The first publication about the synthesis of this kind of material by using an organic template was in 1992 by Mobil scientist [2]. In the case of the synthesis method of mesoporous ceria, different organic templates have been used; for example, cetyltrimethylammonium bromide (CTAB) [3] and triblock copolymer non ionic surfactant [4-6]. However, there is not enough research about the synthesis of mesoporous ceria by using CTAB as template. Mesoporous ceria was synthesized using both Sol – Gel method with a hydrothermal soft treatment. The mesopores of ceria was prepared by using cerium chloride heptahydrate (Sigma-Aldrich), Sodium hydroxide (J.T.Baker) and hexadecyltrimethyl ammonium bromide, CTAB (Sigma, purity) as source, alkaline material and template, respectively. In a first step, two solutions were prepared. The first one was obtained by dissolving 1.863 g of CeCl₃·7H₂O and 0.911 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 Ce-solution and stirred. The final solution was stirred and heat in a hot stir plate at 90 °C to get a material with a molar ratio of CeCl₃·7H₂O:CTAB:NaOH:H₂O equal to 1:0.5:8:150. 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. After that, the material was dried at 80 °C for 1 day. After the synthesis procedure showed above, the calcination of the sample is necessary at 560 °C for 6 hrs in order to obtain complete formation of cerium oxide and complete elimination of organic template. The surface morphology and the structural characteristics images of the as-synthesized samples were analyzed by using a scanning electron microscopy FEG-SEM JEOL JSM 7600 and a Tecnai F20 microscope with a field emission gun, respectively. Figure 1a shows an SEM image of ceria synthesized. This figure shows particle size in nanometric scale (<100 nm). Figure 1b shows the EDS spectrum taken over material analyzed. Figure 2 shows the presence of mesopores in the material with a pore size > 2 nm. In this case, the results showed that the molar ratio between cerium and CTAB and hydrothermal treatment temperature could be critical parameters in the formation of mesoporous ceria. Also, medium amount of water in the synthesis procedure allow the formation of mesopores in the material. The synthesis procedure of ceria presented in this research work is easy with a low cost.

References

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Figure 1. a) SEM image of synthesized material, b) EDS spectrum taken from over one particle.

Figure 2. TEM image of mesoporous material after synthesis.