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Synthesis and Characterization of ZnO/CTAB Nanoflowers

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Abstract – ZnO/CTAB nanoflowers were synthesized by wet chemical route at room temperature with the use of cetyltrimethylammonium bromide (CTAB) as structure directing agent in a solution containing zincate ions. The samples were characterized by XRD, FT-IR, SEM and TG/DTG. The XRD dates were analyzed by Rietveld refinement using the FULLPROF program. Information on the isotropic microstrain and cristallyte size broadening are obtained using a Thompson-Cox-Hasting-pseudo-Voigt line shape function.

ZnO is a semiconductor with a wide band gap (3.37eV) and large exciton binding energy (60 meV) at room temperature, piezoelectricity, and chemical and thermal stability [1]. In this study, for synthesis to ZnO/CTAB nanoflowers were used: 5,5g of CTAB was dissolved in 60ml deionized water to form a transparent solution. 40mL of NaOH 5M and 20mL of $Zn(NO_3)_2.6H_2O$ 0.5M were added to the above solution CTAB under continuous stirring and submitted to 2 hours of agitation in room temperature; the product was washed with distilled water and calcined for two hours at 500ºC.

The formation of the hexagonal phase of the zinc oxide can be easily observed in the DRX patterns. Typical reflectances of pure hexagonal wurtzite could be observed (fig. 1), PDF 361451. The FT-IR spectrum of the of as-synthesized sample showed bands in the regions of 2800-3000 cm^{-1} and 1400-1500 cm^{-1} referent to the modes of stretching and deformation of the CH_2 e CH_3 groups. Bands in the regions of 800-400 cm^{-1} attributed to binding Zn-O. The TG/DTG curves for the ZnO/CTAB presented 3 mass losses in the ranges of 25-184, 184-276, 276-600ºC respectively due to the removal of absorbed water, desorption and decomposition of the surfactant template and the third to dehydroxylation of the surface and removal of little residual surfactant. The images of SEM of the calcinated sample show structures resembling flowers. These nanoflowers are composed by very thin structures in the shape of needles. J Ge et al [2] evaluated the influence of use than surfactant CTAB on the mechanism of growing sisal-like nanostructures of ZnO. The element Zn exists as $Zn(OH)_2^{-4}$ as negatively charged tetrahedrons that are formed, whereas CTA^+ is positively charged with a tetrahedral head and a long hydrophobic tail.

The results of Rietveld refinement show a decrease in the size of crystallites of 682.62 Å to 570.56 Å after heat treatment for removal of surfactant. The values of FWHM also decreased after calcination. The lattice parameters a and c for samples remain constant (a,b = 3.2503824 Å and 3.2502272 Å, c = 5.2063174 Å and 5.2036839 Å, for ZnO as-synthesized and calcined respectively).

Table 1: Parameters of Rietveld refinement

Parameters	ZnO as-synthesized	ZnO calcined
Crystallite size (Å)	682.62	570.56
FWHM (degrees)		
(100)	0.1156	0.0812
(002)	0.1188	0.0850
(101)	0.1211	0.0877
R_{Bragg}	3.35	2.50

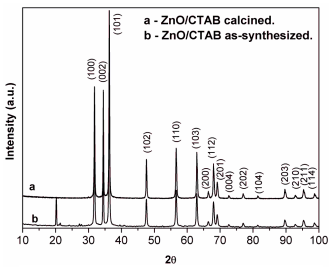

Figure 1: DRX of samples: a) calcined and b) as-synthesized.

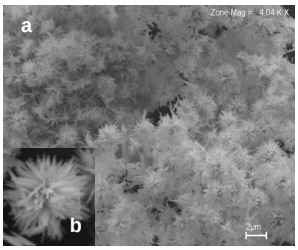

Figure 2: SEM images (a) low magnification, (b) high magnification

References

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