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Effect of Annealing on the Preparation of $CuWO_4$ Particles

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Abstract

The article compare the conventional annealing (CA-500 °C and 800 °C) and hybrid microwave annealing (HMA-360s) on $CuWO_4$ particles with varies the solution pH values (3 and 9). It is observe that the CA yield high crystalline Copper tungstate ($CuWO_4$) with CuO, while the HMA showed impurity free $CuWO_4$ nanoparticles.

Keywords: Nanoparticles; Polyol-hydrothermal process; Oxidation

Introduction

$CuWO_4$ is an important semiconductor and used for lithium ion battery [1], NO2 gas sensor [2], water splitting [3], photo catalytic degradation [4] and super capacitor [5]. The W-rich and Cu-rich $CuWO_4$ were also prepared by the hydrothermal process, the furthest one showed more photo catalytic activity than the other and pure $CuWO_4$ nanoparticles [6]. Generally, annealing was focused for solid state reaction, phase transformation, removal of water or additional element and oxidation etc. The annealing process can be performed through either conventional or microwave method. In this work, HMA was adopted, due to drastic reduction of processing time and low energy consumption, for the preparation of $CuWO_4$ particles by polyol-hydrothermal process. For the sake of comparison, the CA also used.

Experimental Procedure

0.5M $Na_2WO_4.2H_2O$ solution was added drop by drop into 0.5M $Cu(NO_3)_2.3H_2O$ solution under stirring followed by the addition of 0.3M of polyethylene glycol (Mw=4000). Sky blue colour of precipitate was drown into 50mL Teflon linked autoclave and subjected into heat at 180 °C for 24h. Then, the product was centrifuged using DW and ethanol. The yellowish green colour product was filtered and dried in oven at 80 °C for 8h. The as-prepared product was annealed in two ways: one was conventional method (for 500 °C and 800 °C) and another was HMA using microwave oven (2.45GHz, 800W) for 360s. Graphite powder was used as susceptor. The prepared samples were characterised by using XRD (X'pert Pro diffracto meter with Cu Kα radiation (λ=1.5406 Å) in the range of 10°-80°) and FTIR spectrum were recorded in the KBr technique using Perkin Elmer spectrometer (Spectrum Two, Model: C92107) with resolution of $4cm^{-1}$ analyses.

Results and Discussion

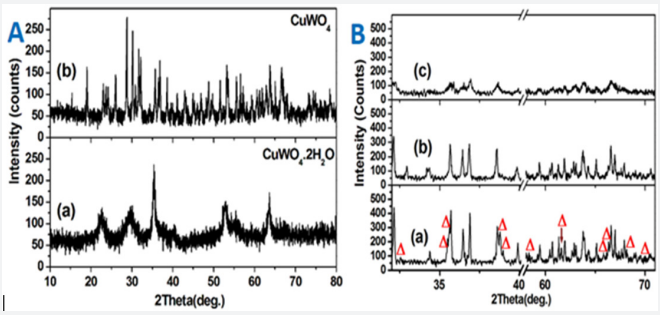

Figure 1: A) XRD patterns of a) as-prepared ($CuWO_4.2H_2O$) at pH 5.2 and b) CA $CuWO_4$ particles at 500 °C for 2h. B) XRD patterns of a) CA at 800 °C for 2h corresponding to as-prepared particles at pH 9 and HMA for 360 s corresponding to as-prepared particles at pH b) 3 and c) 9 (Δ- CuO).

In Figure 1A(a), the as-prepared particles shows amorphous in nature and the 2θ values depicts monoclinic system of wolframite structure $CuWO_4 \bullet 2H_2O$ (JCPDS card: 33-0503. In the CA at 500 °C for 2h (Figure 1A(b)), number of sharp and crystalline peaks observed indicates the formation of triclinic $CuWO_4$ alone (JCPDS card: 73-1823). When increasing annealing temperature to 800 °C for 2h (Figure 1B(a)), the XRD pattern

shows more crystalline peaks of $CuWO_4$ with CuO (JCPDS card: 89-5895). Similar result was reported by Chen and Xu (2015). According to their investigation WO_3 was formed along with $CuWO_4$ at pH ≤3 and CuO was formed at high pH (~8.2) [6]. However, their intensity of crystalline peaks decreased with increase of pH value in HMA samples. By comparing the results with CA, no additional phases such as WO_3 and CuO are formed in HMA samples Average crystallite size is found to be around 102.8nm for CA as calculated from the standard Scherer equation. It is considerably reduced as 39.43nm for pH 3 and 97.43nm for pH 9 in HMA. In the FTIR spectrum of as- synthesized particles (Figure 2), a number of vibrational bands are shown at 896, 795, 730, 584, 510 and 367cm^{-1} belongs to structural and bending vibrations of $CuWO_4$•$2H_2O$ [7].

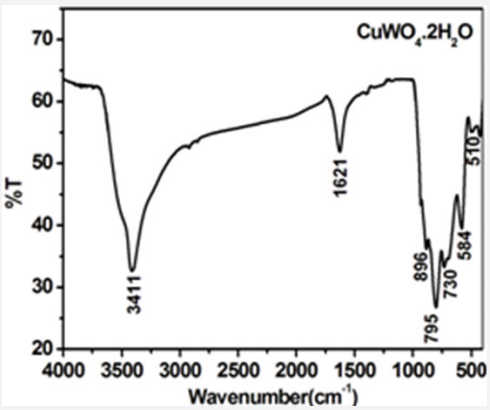

Figure 2: FTIR spectrum of hydrothermally as-prepared $CuWO_4$•$2H_2O$ particles at pH 5.3.

Conclusion

$CuWO_4$ particles were prepared by polyol-hydrothermal process. The samples annealed by conventional method showed highly crystalline $CuWO_4$ with CuO at 800 °C. While the HMA method showed reduce crystallite size. It is concluded from the comparison study, the HMA is useful for synthesising impurity free $CuWO_4$ nanoparticles.

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