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Investigations on Copper Nano-Particles Synthesis by Pulsed Electrochemical Dissolution (PECD) Process

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EXTENDED ABSTRACT

This article reports on development of a simple and cost effective electrolytic synthesis of copper nanoparticles using copper sulphate as metal precursor. An in-house setup of pulsed-electrochemical dissolution (PECD) process for the synthesis of nano-particles has been conceptualized and developed. Generated nano-particles were characterized using X-ray diffraction (XRD), scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) to analyze the crystal size, morphology and chemical composition of the particles. Experiments were conducted in two phases, namely pilot and main experiments. The pilot experiments were done to study the effects of electrolyte concentration, inter electrode gap (IEG) and processing time on the nano-particles shape and size. Average minimum size of the particles obtained during the pilot experiments were found to be 150 nm at identified optimum parameters i.e. 5 wt.% as electrolyte concentration; 30 minutes as processing time; and 10 mm as IEG. These identified optimum values of the input parameters were used during the main experiments. The main experiments were conducted to optimize the values of applied voltage, pulse-on time and pulse-off time. Average minimum size of the particles obtained during the main experiments was found to be 70 nm at the identified optimum value of the parameters; 8 V as voltage; 4 ms as pulse-on time and 8 ms as pulse-off time.

Figure 1a and 1b depicts schematic and photograph of the experimental apparatus developed for the generation of nano-particle by PECD. The apparatus consists of four sub-systems namely: (i) power supply; (ii) electrodes; (iii) mechanism to adjust inter electrode gap (IEG); and (iv) housing. The apparatus has two electrodes made of copper. The positive terminal of DC pulsed power supply was connected to first copper electrode to make it anode while the negative terminal was connected to second electrode to make it cathode. These two electrodes were submerged in copper sulphate electrolyte. A threaded screw with supporting clip was used to adjust the inter-electrode gap. The whole apparatus was enclosed in a housing made of perspex sheets.

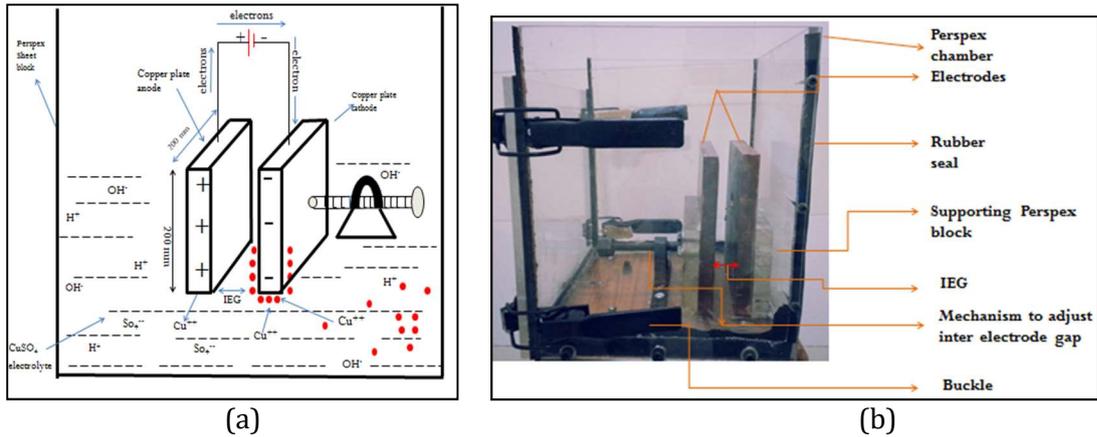
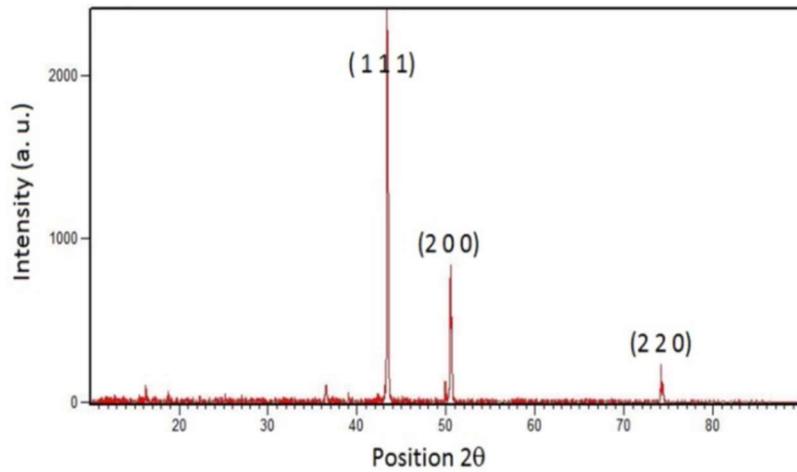
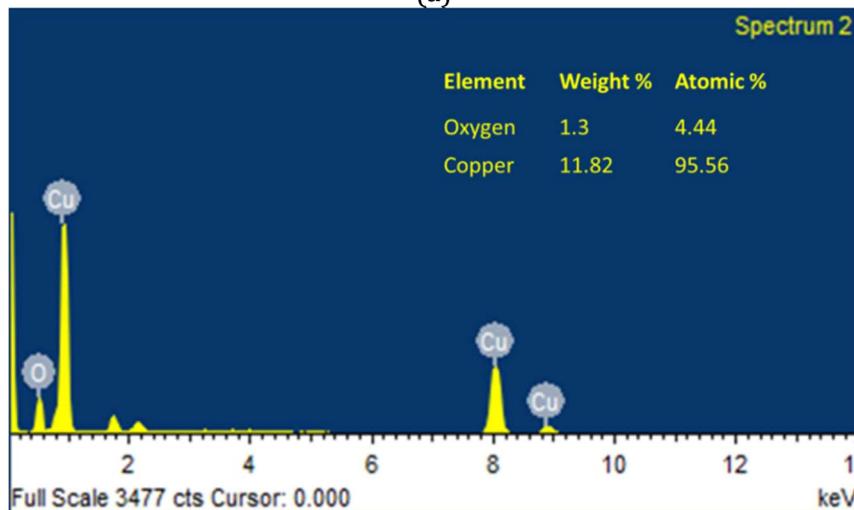


Figure 1: Developed experimental apparatus: (a) schematic diagram; (b) photograph.

It was observed that generated copper particles peaks found at 2θ values of 44, 51, and 74 degrees corresponding to (111), (200), and (220) planes of copper as depicted in Fig. 2(a). It shows that generated peaks obtained are sharp which indicates that copper particles are pure and highly crystalline [1, 2]. EDX results depicts (Fig. 2b) the synthesised particles consist of copper with very less amount of oxygen impurity.



(a)



(b)

Fig 2: (a) XRD analysis of copper nano-particles; and (b) Elemental distribution of the material.

Keywords: nano-particles; copper; electrochemical; PECD

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