

SYNTHESIS, CHARACTERIZATION AND SPECIFIC CAPACITANCE STUDIES ON METAL OXIDE NANOPARTICLES

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Article History: Received 2nd August, 2016, Accepted 30th August, 2016, Published 31st August, 2016

ABSTRACT

Zinc Oxide, Magnesium Oxide and Calcium Oxide nanoparticles were synthesized by using chemical precipitation method. The ZnO, MgO, CaO nanoparticles were characterized for crystallinity and crystal size by X-RD, particle size and morphology by SEM. The Electrochemical Supercapacitor property of the synthesized nanoparticles was investigated by Cyclic Voltammetry and it has been found that Magnesium Oxide has high Specific Capacitance.

Keywords: Chemical Precipitation, XRD, SEM, Cyclic Voltammetry, Specific capacitance

1. INTRODUCTION

Nanotechnology is emerging as a rapidly growing field with its various applications in science and technology for the purpose of manufacturing new materials at the nano scale level. At this scale the surface to volume ratio of materials becomes large leading to unique properties (Albrecht et al., 2006). Nanotechnology can provide unparalleled understanding about materials and devices and is likely to have impact in many fields. We can greatly expand the range of performance of existing chemicals and materials by reducing the structure to a nanoscale. Materials reduced to the nanoscale can show different properties compared to what they exhibit on a macro scale, enabling unique applications. In recent years a number of investigations have focused on II-VI group metals. In particular, inorganic oxide nanomaterials like ZnO, MgO and CaO have shown potential as effective alternatives in addressing some of the challenges (Stoimenov et al., 2002; Nasibulin et al., 2009; Zhang, et al., 2012). In particular, inorganic metal oxide nano material like CaO, ZnO and MgO are of particular interest because they are not only stable under harsh process conditions, but also generally regarded as safe materials to human beings.

Electrochemical supercapacitors play an important role as a new class of energy storage device, supercapacitors, also

known as electrochemical capacitors, has received considerable attention that can be used in hybrid electric vehicles, memory backup, computers, short-term power sources for mobile electronic devices (Liu et al., 2010; Wang et al., 2005; Chen et al., 2009) and other emergency power supply devices due to their higher power density, superior cycle lifetime, and low maintenance cost. Supercapacitors are known to possess high power density, whereby large amounts of electrical energy can be provided or captured within short durations. They can be classified into two types based on their charge storage mechanism (i) Electrical double – layer capacitors (EDLCs) and redox supercapacitors (Inamdar et al., 2011a). Compared to the EDLC-based capacitors, redox capacitors based on transition-metal oxides may provide much higher specific capacitances.

In the present study we have reported the synthesis of the ZnO, MgO and CaO nanoparticles by chemical precipitation method and their characterization by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) and we examine the supercapacitance properties of the synthesized nanoparticles by cyclic voltammetry.

2. EXPERIMENTAL PROCEDURE

ZnO, MgO and CaO nanoparticles were synthesized by chemical precipitation method using Zinc Chloride, Magnesium Nitrate Hexahydrate, Calcium Chloride and Sodium Hydroxide as the precursors. The solutions of the

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materials were made by dissolving them in the milliQ water. The precipitation was induced by drop wise addition of the sodium hydroxide into Zinc Chloride, Magnesium Nitrate Hexahydrate and Calcium Chloride solution under constant stirring for 2 hours. After the completion of the reaction, the solution was allowed to settle for overnight. Then the precipitate formed was washed several times with milliQ water to remove the impurities formed during precipitation. Then the precipitate was separated and kept in the hot air oven for overnight to dry away the water. Then the obtained particles were grinded using mortar and pestle and kept for calcination at 450°C for 3 hours. During this process the complete conversion of hydroxide into oxide takes place.

CYCLIC VOLTAMMETRY:

The cyclic voltammetry, were performed at room temperature. The Cyclic Voltammograms (CVs) was recorded in the KOH electrolyte for the synthesized ZnO, MgO, CaO nanoparticle films at four different scan rates (25, 50, 75, and 100 mVs⁻¹).

3. RESULTS AND DISCUSSION

X-RAY DIFFRACTION:

X-ray diffraction is a non-destructive and analytical method for identification and quantitative analysis of various crystalline forms of nanoparticles, also known as phases of the compound present in the samples. XRD analysis was carried out on dried sample of the prepared ZnO, MgO, and CaO nano particle. Fig.1 shows that all the diffraction peaks are well indexed to the hexagonal ZnO wurtzite structure with lattice parameters $a=3.248 \text{ \AA}$ and $c=5.205 \text{ \AA}$. Fig.2 shows that the diffraction peaks are well indexed to the phase centered cubic MgO structure with lattice parameters $a = 4.211 \text{ \AA}$. Fig.3 shows that the diffraction peaks are well indexed to the monophasic cubic CaO structure. The XRD pattern of MgO, CaO nanoparticles shows some of the unidentified peaks, the broadness of the peak and amorphous nature pointed out the presence of slight impurities in the synthesized product.

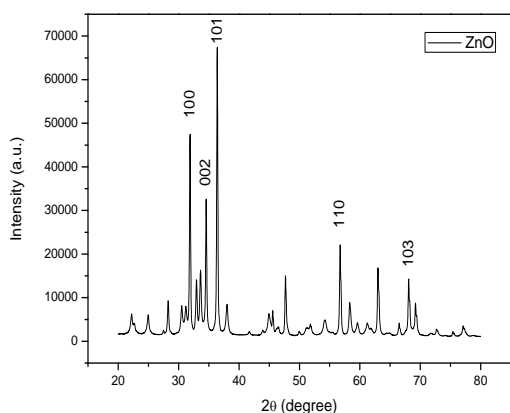


Fig.1. X-RD Pattern of ZnO Nanoparticles

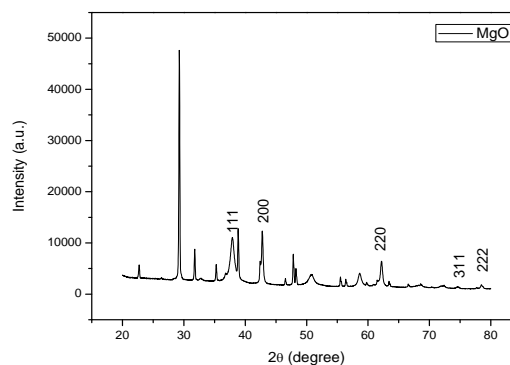


Fig.2. X-RD pattern of MgO Nanoparticle

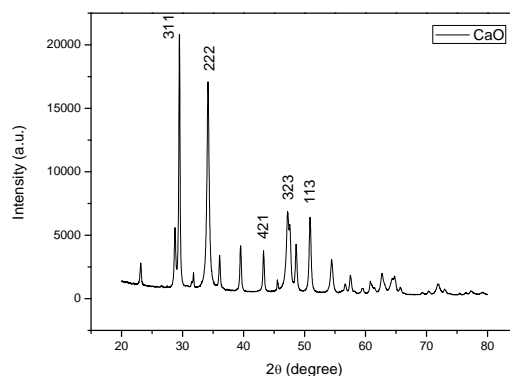


Fig.3. X-RD Pattern of CaO nanoparticle

The average size of the particle was calculated using the Debye Scherrer formula

$$D = K\lambda / \beta \cos\theta$$

D = the particle size, k = a constant of (0.9), λ = the X-ray wavelength (1.54 Å), θ = the Bragg's angle in radians, β = the full width at half maximum of the θ peak used.

The average size of the synthesized ZnO, MgO, and CaO nanoparticle were calculated to be 40.89 nm, 22.70nm, 31.38 nm respectively.

SCANNING ELECTRON MICROSCOPE:

It is important to know the exact nature of nanoparticles formed and this can be deduced from the SEM image of the sample. The morphology, structure and size of the samples can be investigated by SEM. Fig.4 shows that the synthesized ZnO nanoparticles were flower shaped and the Fig.5 shows that the MgO nanoparticles were irregular in shape and the Fig.6 shows that the CaO nanoparticles were spherical in shape. The average size was calculated to be 2.1 μm , 2.25 μm and 1.38 μm for the ZnO, MgO, CaO nanoparticles respectively.

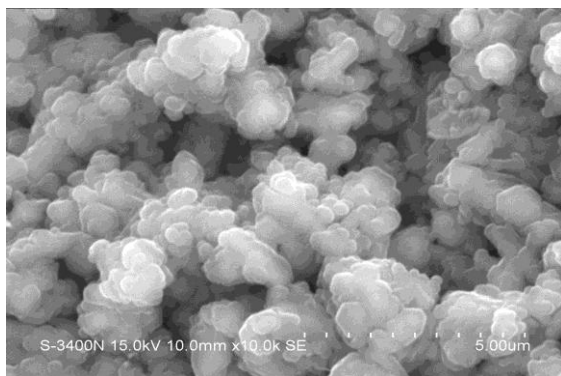


Fig.4. SEM Image of ZnO Nanoparticles

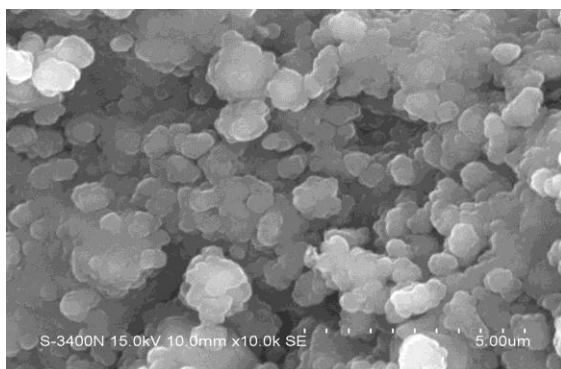


Fig.5. SEM Image of MgO Nanoparticles

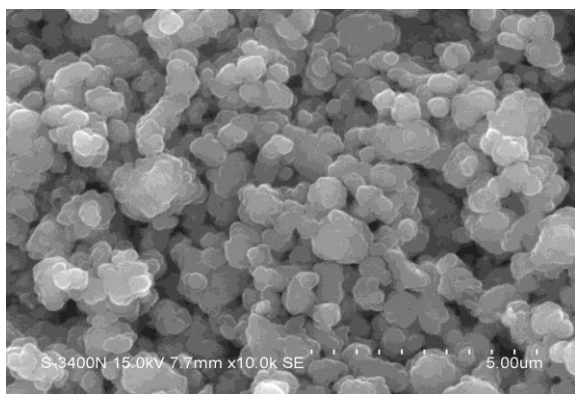


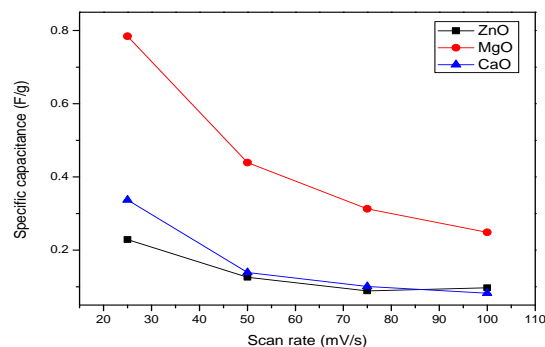
Fig.6. SEM Image of CaO Nanoparticles

CYCLIC VOLTAMMETRY RESULTS:

CVs provide valuable information on reduction oxidation (charge-discharge) behaviour. Here, the capacitance was mainly based on the redox reaction because the shape of the CVs is distinguished from the shape of electric double-layer capacitance, which is normally close to an ideal rectangle (Inamdar et al., 2011b). As the scan rate was increased, the current response, which is a measure of the capacitance, increased. The similar shape of the CVs recorded at different scan rates indicates excellent electrochemical reversibility of the synthesized ZnO, MgO, CaO nanoparticles. The specific capacitance of the electrode can be calculated from the CV curves according to the following equation.

Fig.7 shows the high specific capacitance for MgO nano particles than CaO, ZnO nano particles.

$$C = \frac{1}{ms(v_a - v_b)} \int Idv$$



4. CONCLUSION

ZnO, MgO and CaO nano particles was synthesised by chemical precipitation method. The XRD results reveals that the ZnO has hexagonal wurtzite structure, MgO has phase centered cubic structure, CaO has mono phasic cubic structure and the average size was calculated to be 40.89 nm, 22.70 nm, 31.38 nm respectively. The SEM image results shows that the synthesized ZnO nanoparticles were flower shaped and the MgO nanoparticles were irregular in shape and the CaO nanoparticles were spherical in shape. The average size was calculated to be 2.1µm, 2.25µm and 1.38µm respectively for the ZnO, MgO, CaO nanoparticles. CV results show the high specific capacitance for MgO nano particles than CaO, ZnO nano particles.

5.ACKNOWLEDGEMENT

This work has been completed with the support Nano Science lab, Anna University, Chennai. The author wishes to acknowledge the Centre for Nano Science and Technology for this research work and also thankful to Dr.R.Jayavel, Director Research, AnnaUniversity, Chennai for provding the cyclic voltommetry meaurment.

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