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PSEUDOCAPACITIVE BEHAVIOR OF Co_3O_4 AND BINARY $NiCo_2O_4$ ELECTRODE MATERIALS FOR SUPERCAPACITOR APPLICATIONS

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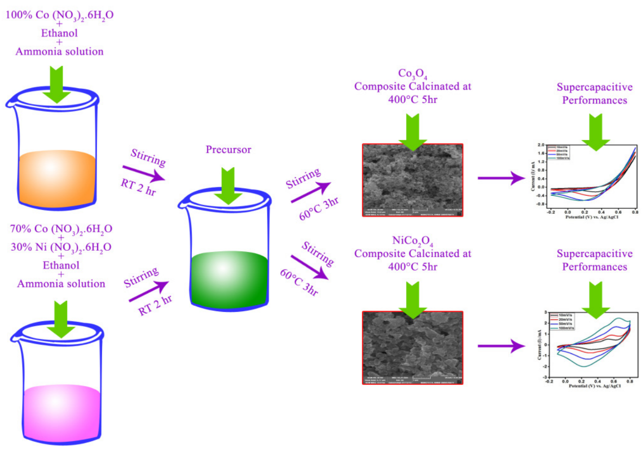
ABSTRACT

The Cobalt oxide and Binary Cobalt-Nickel oxide composite electrode material were prepared by co-precipitation method. The average crystalline size was calculated by using scherrer's formula. The cobalt oxide and cobalt-Nickel oxide composite size were found to be 19.5nm and 15.8nm.The structural, thermal properties and morphology studies were carried out by the XRD, TGA and SEM analyses. The prepared both electrode materials were subjected to analysis the electrochemical property studies of cyclic voltammetry, charging-discharging and impedance spectrum were carried out by three electrode system. These pure and composite electrode materials reveal the pesudocapacitive behavior and the specific capacitance value composite material exhibit 934.53F/g at 1M KOH electrolyte. The capacitive performances of this composite electrode material are great interest for supercapacitor practical applications. The impedance spectrum of composite material indicating higher capacitance and electrical conductivity.

INTRODUCTION



The Electrochemical capacitors (ECs) are called as double-layer capacitors, ultra-capacitors, supercapacitors. It's have been attracting significant interest because, of their high power density, fast power delivery, excellent reversibility, low equivalent series resistance (ESR) and long cycle life instantaneously provide when compared with batteries. It's an outstanding property of excellent candidates for acceleration power electric vehicles, electrical regenerative braking storage for electric drive systems, power assist to hybrid vehicles, starting power for fuel cells, pulse power for mobile telecommunication, computers, mobile electric devices and other applications. [1-3] According to the charge storage mechanism, the electrochemical capacitors basically two types: i.e., (i) electrical double-layer capacitors (EDLCs), based on the capacitance arising from charge separation at an electrode/electrolyte interface, and (ii) redox supercapacitors, based on the pseudocapacitance arising from reversible Faradaic reactions occurring at the active electrode surface. The redox supercapacitors show the higher capacitance than EDLCs [4]. The Electrochemical capacitor electrode materials are three types. (i) Carbon based materials (ii) transition metal oxides (iii) polymer based materials. But these materials are storing the energy through both mechanisms. Unfortunately, each one of these materials may face some challenges i.e., the carbon based materials may only physically store limited charges, causing low specific capacitance (SC), the conducting polymers based materials may swell and shrink during the intercalating/deintercalating processes leading to low cycling stability. The metal oxides may show poor electronic and ion conductivity, low specific surface area, limiting the electrochemical supercapacitor (ES) power density. Making for all these challenges, have been introduced to the composite materials for the electrochemical supercapacitor (ES). [5]. Now days,

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research focusing on the composite electrode materials for supercapacitor, particularly the cobalt and nickel oxide, because these materials are low cost and environmentally friendly. The Nickel oxide electrode material is used in the potential applications of supercapacitor due to their high electrochemical reaction activity, electronic conduction and ion transport in the electrode/electrolyte. Therefore, it's an enhancing the both energy density and power density. The theoretical specific capacitance value Nickel oxide is 3228 F/g. [6, 7]. The Cobalt species materials are such as Co(OH)2, CoOOH, and Co_3O_4 have an excellent electrochemical reversibility, cycle life, high specific capacitances, good capability retention, low cost and the theoretical specific capacitance value of cobalt oxide is 2600 F/g [8, 9,10]. In an attempt has been made to identify the suitable candidate electrode materials for supercapacitors. The cobalt oxide and cobalt-nickel oxide composite electrode materials were prepared by using the co-precipitation method without using any surfactant, template and high temperature.

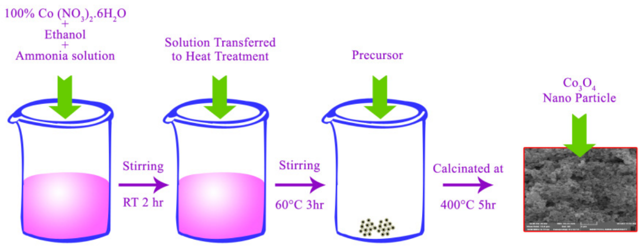

Figure (a)

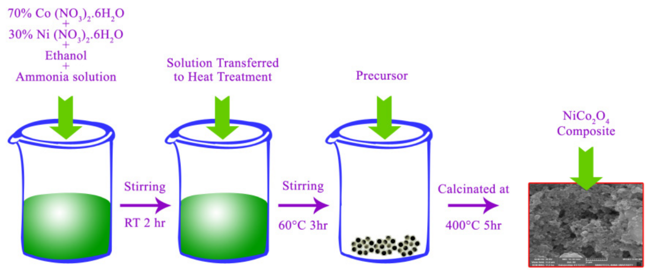

Figure (b)

Figure1 (a). The typical Synthesis procedure for Co_3O_4 Nanoparticle and (b) $NiCo_2O_4$ composite.

Experimental

Typically for the following two step syntheses of cobalt oxide nanoparicle and cobalt –nickel oxide composite. The 0.1m of $Co(NO_3)_2.6H_2O$ was dissolved in 100 mL Ethanol under vigorous stirring at room temperature and we get clear dissolved solution The pH of the solution was adjusted to 9-10 by adding ammonia solution drop by drop and stirred for 2h in room temperature. The adjusted pH solution was heated at 60°C and stirred for 3h and get green precipitate product was separated by filtration and washed with several times of water, ethanol to remove their impurities. The sample was dried in air oven at 100°C for one day. The final product of Co_3O_4 was obtained by calcination at 400°C for 5 h. The same procedure was followed to prepared 70% Co $(NO_3)_2.6H_2O$ and 30% Ni $(NO_3)_2.6H_2O$ of composite material. Figure 1(a). Shows the typical Synthesis procedure for Co_3O_4 Nanoparticle and (b) $NiCo_2O_4$ composite.

CHARACTERIZATIONS

The crystal structure of pure and composite material were characterized by using XRD (XRD, Rigaku miniflux (II)-c). The thermal properties of both materials were carried out using TGA (exstar S(II) TG/DTA 6300), the morphology of the both products were viewed on a SEM by using (VEGA3 SBU,VGB 825111771N).

Electrochemical measurements

The electrochemical properties of cobalt oxide and cobalt-nickel oxide were investigated with cyclic voltammetry (CV), charge–discharge and electrochemical impedance spectrum (EIS) measurements in a conventional three-electrode cell employing a Bio-logistic system electrochemical workstation. The three-electrode cell contained a platinum wire as the reference electrode, Ag/AgCl as the reference electrode, and cobalt oxide and cobalt-nickel oxide samples were prepared on glassy carbon as the working electrodes, with a solution of 1 M KOH as the electrolyte at room temperature. Cyclic voltammograms were performed for cobalt oxide the potential range between -0.1 and 0.5 V vs. Ag/AgCl at various scan rates from 10, 20, 50 and 100mV/s. The Cobalt- nickel oxide was performed in the potential ranges between -0.1 and 0.5 V vs. Ag/AgCl and -0.2 and 0.8V vs. Ag/AgCl at various scan rates from 10, 20, 50 and 100mV/s. Galvanostatic charge–discharge testing were conducted both samples at different current densities and electrochemical impedance spectrum were analyses for both samples in different frequencies.

RESULTS AND DISCUSSION

Structural Analysis

The crystal phase and structure information of the both products were obtained by XRD analysis. The fig.2.Shows the typical XRD patterns of the cobalt oxide and cobalt-nickel oxide composite and the structure of the pure and composite system are cubic. Table: 1 shows the comparison of 2ɵ value and particle size between cobalt oxide nanopaticle and cobalt-nickel oxide composite. The average crystalline size was calculated to be 19.5 and 15.8 nm corresponding for cobalt oxide and cobalt-nickel oxide composite respectively. The average crystalline size Dc was determined by using the Debye -Scherrer's equation,

$$\mathbf{D_c} = \frac{0.9\lambda}{B\cos\varphi} \quad (1)$$

Where, Dc is the crystalline size, λ is the wavelength of the X-ray, B is the full width half-maximum and θ is the bragg angle of the diffraction peak. The diffraction peaks of the cobalt-nickel oxide composite materials is little shifting as compared to the cobalt oxide and other there is no other impurities peaks were detected. It's indicating that the high purity of the final products.

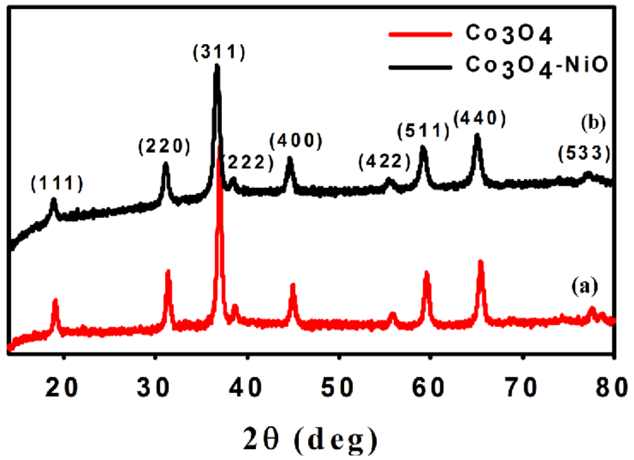

Fig. 2.The typical XRD patterns of (a) Cobalt oxide and (b) Cobalt-Nickel Oxide composite.

Thermal Analysis

The thermo gravimetric (TG) curves of Co = 100% and Co: Ni = 70%: 30% precursor mixtures shows the weight-loss in the multiple stages as shown in Fig.3. The TGA curves were performed from the range of room temperature to 900oC . The Co_3O_4 and Co_3O_4 -NiO composite both are shows three major thermal events. Its first weight loss is attributed to physically absorbed moisture and organic surface impurities at the temperature 120°C. The second weight loss of the temperature is around at 300°C. It's attributed to the formation of basic Co salt to o^3O^4. The third temperature changing at between 850°C and 900°C is to be transformation of Co^3O^4 to CoO [11]. The cobalt-nickel precursor powder is heated at 400oC for five hours to get the mixed of nanoparticles.

Table1. The Comparison 2ω values of Cobalt oxide and Cobalt-Nickel Oxide composite with JCPDS standard file no.

JCPDS 65-3103 Co_3O_4	2ω value Cobalt oxide	2 ω value composite	Corresponding Planes for pure	Cobalt oxide (nm)	Cobalt-nickel composite (nm)
19.06	19.10	18.90	111	21.06	17.62
31.38	31.35	31.16	220	22.4	15.43
36.97	36.94	36.69	311	20.42	15.22
38.68	38.61	38.41	222	21.81	20.81
44.97	44.90	44.62	400	22.34	15.18
55.86	55.76	55.47	422	16.61	7.15
59.58	59.46	59.13	511	19.89	15.19
65.49	65.37	65.04	440	19.88	14.50
77.66	77.29	77.08	533	12.07	6.19

Morphological Analysis

The surface morphology of Co_3O_4 and Co_3O_4- NiO composite were investigated by SEM analysis as shown in fig.4. The crystalline structure of cobalt oxide is very low as compared to the composite material of cobalt –nickel oxide and its very high crystalline structure in nature. The average particle size of the composite material is small and particles were well agglomerated as well as shape was destroyed. The product was heated at 400 °C temperature it reveals that the formation of uniform or non-unifrom nanosized spherical particles size within the range of 15 nm (average size). [12]. Cyclic voltammetry Analysis (CV)

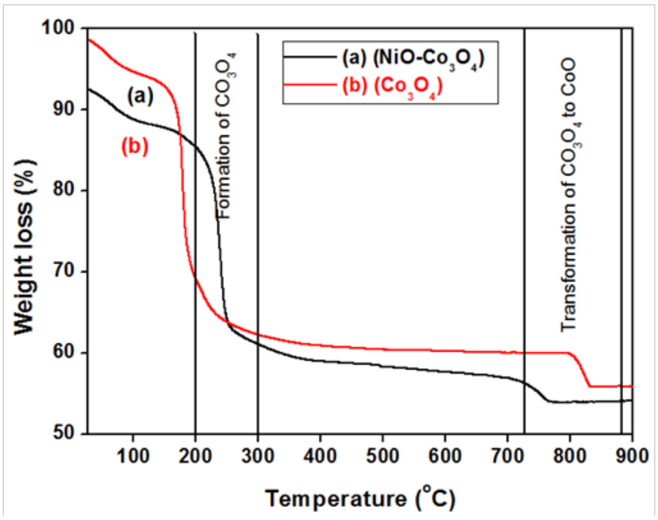

Fig.3.The TGA spectrum of (a) Cobalt oxide and (b) Cobalt-Nickel Oxide composite.

The electrochemical supercapacitor application of the both electrode materials were carried out in the potential range between -0.1 to 0.5V 1M KOH electrolyte at different sweep rates. The Fig. 5(a). Shows all the CV curves of cobalt oxide nanoparticle and its did not show the clear rectangular shape. Fig. 5(b). Shows all the CV curves of cobalt-nickel oxide composite electrode is revealed that the characteristic of capacitance which would produce the ideal rectangular shape. The specific capacitance of the both electrode materials were calculated by using the following equation,

$$C_s = \frac{I \,.\, \Delta t}{\Delta V . m} \quad (2)$$

Where m is the mass of the electro-active materials, ΔV is the potential window, I is the oxidation or reduction current, Δt is the total discharge time, and Cs is the specific capacitance of the electrode. In this way, the cobalt-nickel oxide composite exhibited the specific capacitance of 934.53 F/g at a sweep rate of 10mV/s

The Cobalt-Nickel Oxide (NCO) composite electrode for Supercapacitor Application

The Cobalt-Nickel Oxide composite electrode material for Supercapacitor applications of the CV curves are shown fig.5(c) in the potential range of -0.2 to 0.8 V. It's clearly shows the rectangular shape and its good performances for supercapcitor applications. The two redox peaks (P1/P2) are probably due to the integration of redox couple of NiO/NiOOH and redox couple CoOOH/Co_3O_4 because these couples are similar reaction potentials and its implying better electrochemical reactivity [13].The characteristic of two prominent peaks which indicate capacitive behavior an ideal capacitor shows the rectangular shape with no resistance but that the peaks are occur within the voltage ranges. It's usually evidence of the pseudocapacitor behavior. The redox peaks are shifted in the CV curve as the composition of Co_3O_4-NiO composite were changed. It is observed that higher concentration of Ni with the composites, that the oxidation and reduction peaks are shifted to the positive voltage value within the applied voltage window. The quasi-reversible electron transfer process is shows in all CV curves. It's indicating that the measured capacitances are mainly based on the redox mechanism. [14]

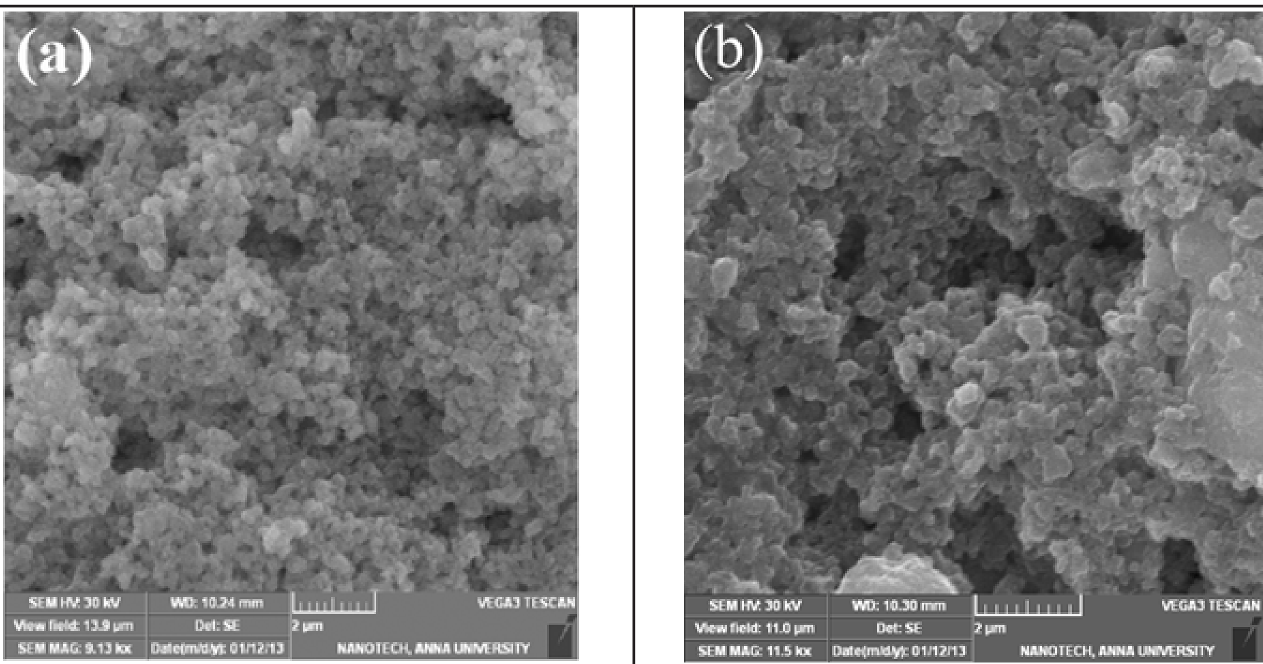

Fig.4. The SEM images of (a) Cobalt oxide and (b) Cobalt- Nickel Oxide composite

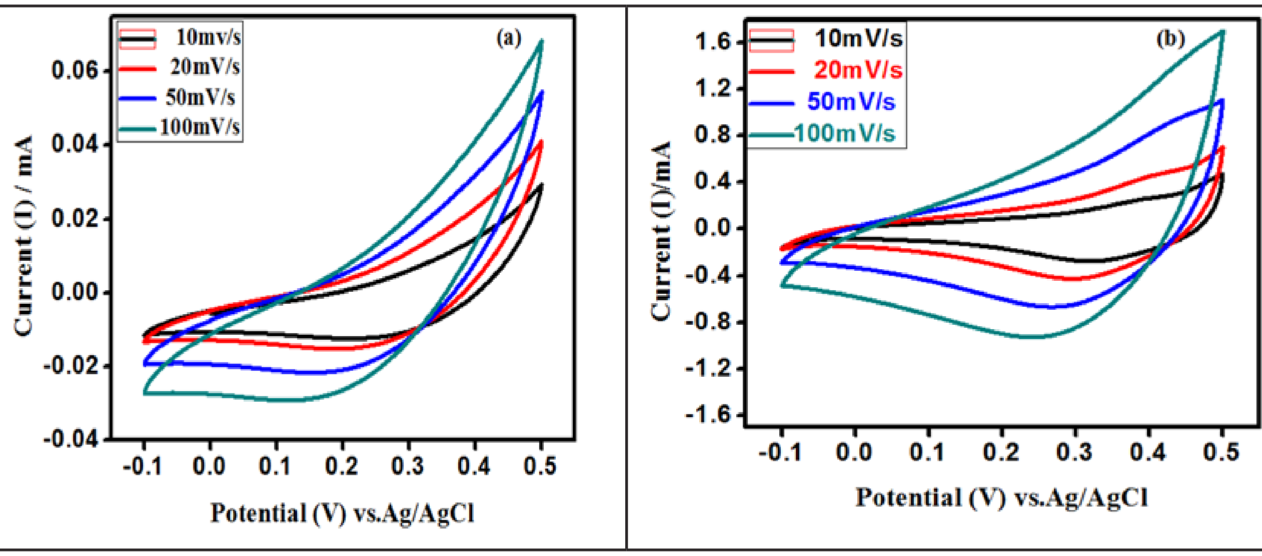

Fig.5. The CV spectrum of (a) Cobalt Oxide and (b) Cobalt-Nickel Oxide composite

Electrochemical Impedance Spectroscopy (EIS) Analysis

The electrochemical impedance analyses were carried out for both electrodes. The both plots are composed of semicircle at high frequency region and straight line at low frequency region. The high frequency medium of semicircle is attributed to charge transfer process at electrode and electrolyte interface On other hand the straight line is should be ascribed to diffusion process of solid. From the point intersecting with the real axis in the range of high frequency, which is composed to three terms i.e., (i) the resistance of the electrolyte, (ii) the intrinsic resistance of the electro-active material, (iii) the contact resistance between electro-active material and electrolyte interface. From the XRD results shows that the crystalline size of the Co_3O_4 –NiO composite is smaller than Co_3O_4. On the other hand due to the small size of Co_3O_4 –NiO composite, the electrode material and the electrolyte could contact with each other closely, which can increase the electrical conductivity of electrode. At high frequency region, the semicircle behavior of Co_3O_4–NiO composite is smaller than that of Co_3O_4 electrode, which indicated that the electrochemical reaction resistance of Co_3O_4 –NiO smaller than Co_3O_4 and its suggesting that Co3O4–NiO small diffusion impedance and large capacitance than Co_3O_4 [15-17].

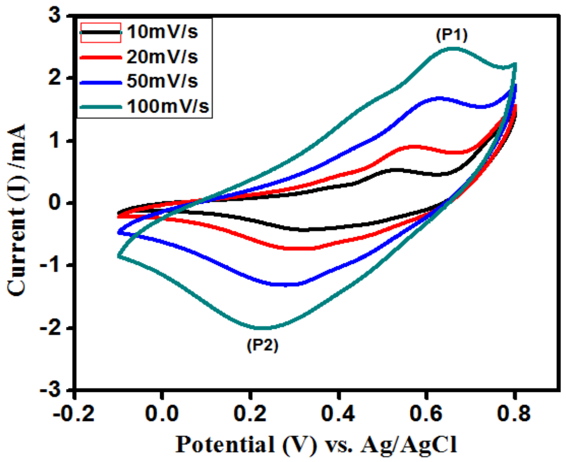

Fig.5(c). The CV spectrum of Cobalt-Nickel Oxide composite at the potential window of -0.2 to 0.8V.

CONCLUSION

The cobalt oxide and cobalt-Nickel oxide composite possesses the cubic structural phase and confirmed by XRD technique with evaluated the average crystalline sizes for both materials. The morphology of the both materials was spherically agglomarateted in uniform and non-unifrom nanosized particles. The composite material was reveals pseudocapacitive behavior and high specific capacitance in 1M KOH electrolyte and its shows the rectangular shape. The impedance spectrum of composite material exhibits the high electrical properties and electrochemical reaction resistance. An identified as the great capacitance performance of composite electrode material is a suitable candidate for supercapacitor applications.

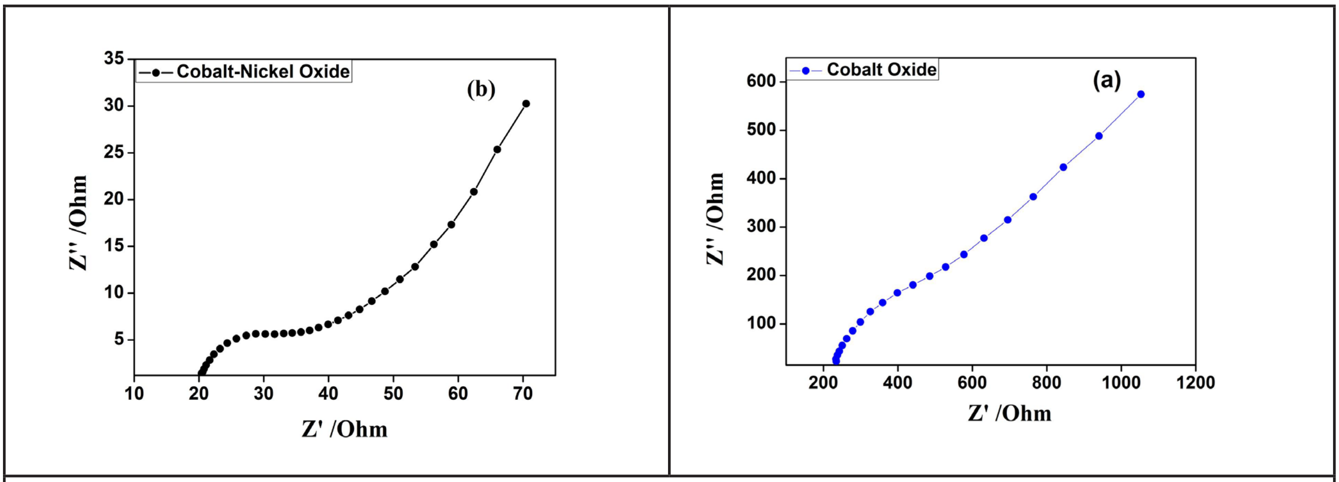

Fig.6. The impedance spectrum of (a) Cobalt Oxide and (b) Cobalt-Nickel Oxide composite

Charge–Discharge Analysis (CD)

The galvanostic charge- discharge studies of cobalt oxide and cobalt-nickel oxide composite were performed in the potential raange 0.0 to 0.5 V at 1M KOH electrolyte solution at 1Ag-1 constant current density as shown in fig.7. The charge-discharge time of cobalt-nickel oxide composite is much smaller than that of cobalt oxide material. The charge –discharge curve were observed that linear and symmetrical and its implies that the cells have excellent electrochemical reversibility and capacitive characteristics.

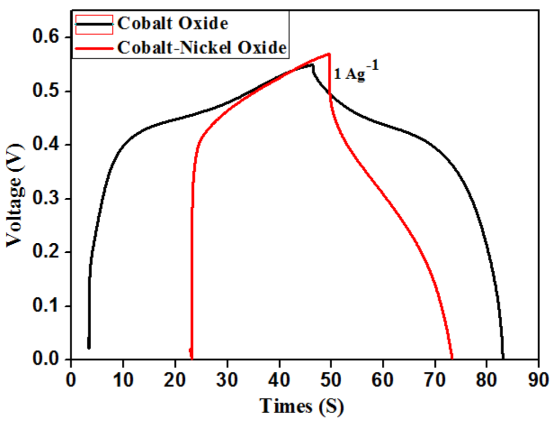

Fig.7. The Charge-Discharge curves of (a) Cobalt oxide and (b) Cobalt-Nickel oxide

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