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# Cyclic Voltammetry and Electrochemical Impedance Spectroscopy Analysis of $Cr^{3+}$ doped $Mg_2SiO_4$ Nanoparticles for Supercapacitor Applications

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**Abstract**

Low temperature solution combustion synthesized $Cr^{3+}$ (1- 4 mol%) doped $Mg_2SiO_4$ nanoparticles were analyzed by Powder X-ray Diffraction (PXRD), Fourier Transform Infra-Red (FTIR) spectroscopy, Cyclic voltammetry (CV) and Electrochemical Impedance Spectroscopy (EIS) techniques. PXRD profile shows the samples are crystalline. FTIR spectra show MgO6 octahedral and Si-O bending and stretching modes. It was observed that, CV show excellent semi rectangular shaped voltammograms due to the oxidation reduction reactions and the reversibility of the reaction which suits for electric double layer capacitance. Charge transfer resistance ($R_{ct}$) was found to be 10 Ω indicates the better electron transfer from one phase to another.

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Cyclic Voltammetry,
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## Introduction

For as far back as not many decades a tremendous exertion has been centered around the creation of electrochemical capacitors or supercapacitors for higher force and vitality thickness than batteries and regular capacitors.[1] The carbon-based materials, progress metal oxides, and leading polymer are generally utilized as cathode material for an electrical double-layer capacitor (EDLC) and pseudocapacitors because of their high surface region and different change states. As of late, the progressively organized nanomaterials with a multi-

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scale association pulls in incredible consideration in light of its favorable circumstances, for example,[2,3] amplifying the particular surface region and along these lines expanding its electrical twofold layer and pseudocapacitance conduct, making permeable inside spaces. Along these lines, the various leveled organized nanomaterial is a useful and perfect decision to be utilized as a terminal material for supercapacitors.

Super capacitors are considered as an efficient power energy source and it very well may be, for the most part, applied with different creation/blend techniques. It is outstanding amongst other fundamentally accessible sources of inefficient power energy innovation.[4,5] The high surface zone (1344 $m^2g^{-1}$) and enormous pore volume (0.902 $cm^3g^{-1}$) of nitrogen-doped mesoporous/microporous carbon (NMMC) based cathode utilized in a supercapacitor, which has the conveyed capacitance estimation of 325 Fg-1.[6] Zhang *et al.*,[7] utilized aqueous assisted technique for the blend of RGO/$Ni_3S_2$ on nickel froth anode, the predominantly performed capacitance estimation of 2188.8 Fg-1 at 2.9 $Ag^{-1}$.

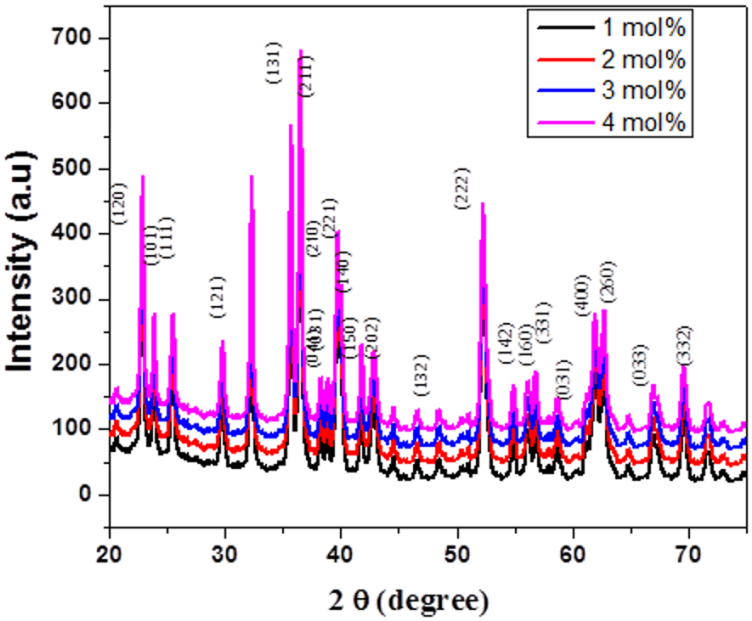


**Fig. 1: PXRD Pattern of $Mg_2SiO_4$:$Cr^{3+}$ (1-4 mol%) nanoparticle**

Silicates are an attractive class of materials among inorganic phosphors for a wide scope of uses because of their uncommon properties, for example, water, chemical resistance, and visible light transparency. Specifically, inorganic nanophosphors with the fuse of trivalent rare-earth cations uncovered significant iridescence impacts. Further, different opening and imperfections present in the host matrix result in different luminescence features.[3] Improved electrical, luminescent and optical properties of nano phosphors were brought about by the quantum size impact, which was created by an expansion in the bandgap because of a diminishing in the quantum permitted state and the high surface-to-volume ratio.[10] Among silicate family, the $Mg_2SiO_4$ have doped with rare earth and transition metal ions show some fascinating applications, for example, long-lasting phosphor, X-ray imaging, light-emitting diode (LED), environmental monitoring, and so forth.[11–14]

## Experimental

$Mg_2SiO_4$: $Cr^{3+}$ (1-4 mol%) nanoparticles have been prepared using solution combustion method as per the stoichiometry and by maintaining the fuel to oxidizer ratio unity. Raw materials taken are magnesium nitrate (Mg $(NO_3)_2{\cdot}6H_2O$ (Sigma Aldrich, 99.99%), fumed silica ($SiO_2$ (Sigma Aldrich, 99.99%), laboratory prepared oxalyl dihydrazide

(ODH; $C_2H_6N_4O_2$) fuel and chromium nitrate ($Cr(NO_3)_3$: 99.9%, Sigma Aldrich). The mixture of these in 1-4 mol% of $Cr^{3+}$ is dissolved in a minimum quantity of doubled distilled water in a petri dish and mixed thoroughly using a magnetic stirrer for about 5 min. The petri dish was introduced into the muffle furnace set at 350°C. The solution turns out as a gel and then foam continues catching fire and finally forms a powder. The powder was ground and used for characterizations without calcination.[15]

**Results and Discussion**

PXRD and FTIR analysis of as-prepared samples were carried out for structural confirmation. All the PXRD peaks were indexed and well-matched with JCPDS card No. 78-1371 with space group pbnm (No.62) and orthorhombic structure (Fig. 1). The lattice parameters and unit cell volume for (211) plane were estimated using the following relations and was found to be 2.471 Å and 289.92 x 10-30 $m^3$ respectively.

$$2dSin\theta=n\lambda \quad ...(1)$$

$$d_{hkl}=1/\sqrt{(h^2/a^2+k^2/b^2+l^2/c^2)} \quad ...(2)$$

$Cr^{3+}$ peaks were not observed which indicates that it is homogeneously mixed and effectively doped in the host lattice in $Mg^{2+}$ sites.[15] The average crystallite size (D) was estimated from the line broadening in X-ray powder using Scherrer's formula[39]

$$D = K\lambda/\beta\cos\theta \quad ...(3)$$

where, 'K' ; constant, '$\lambda$' ; wavelength of X-rays, and '$\beta$' ; FWHM] was found to be in the range 25-35 nm. Using the FTIR analysis method, samples have been scanned by infrared light to determine bonds and confirm the prepared material (Fig. 2). The peaks at 425, 525, 625, 685, 885, 1015, 1255 and 1405 $cm^{-1}$ were assigned to $MgO_6$ octahedral, Si–O ($\nu$4), Si–O (bending), Mg–O, Si–O ($\nu$3, stretching), (CO and Si–O), C–H and $NO_3$ respectively, where $\nu$3 was asymmetric stretching and $\nu$4 was asymmetric deformation vibrations.[15]

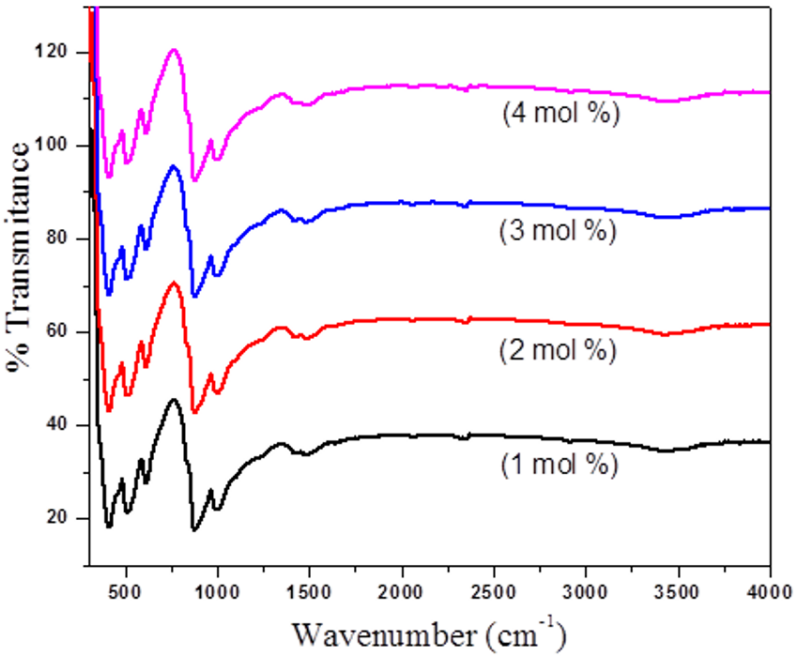


**Fig. 2: FTIR pattern of $Mg_2SiO_4$:$Cr^{3+}$ (1-4 mol%) nanoparticle**

CV and EIS were recorded at room temperature for all the prepared $Mg_2SiO_4$:$Cr^{3+}$ nanoparticles which shows remarkable electrochemical performance. CV analysis usually done to understand mass transportation and electron conduction. Shape of the curve is observed to be nearly rectangular, which

is the characteristic of the electrical double layer capacitor. Increase of scan rates did not influence the mass transportation and electron conduction as observed in the diagram. Reversibility of the reaction, quantification of charge efficiency was understood with the help of CV analysis. By taking the difference between the oxidation potential (EO) and the reduction potential (ER) at the scan rate of 10 mV/s reversibility of the reaction was analyzed. Reversibility is high for smaller EO-ER. CV of $Mg_2SiO_4$: $Cr^{3+}$ (1, 2, and 4 mol %) recorded at scan rates from 0.01 V/s to 0.05 V/s were shown in Fig. (3-5). Semi rectangular voltammograms are observed for all the samples. It was observed that, redox peak current increases with an increase in scan rate due to overpotential[16], and it was found maximum for 4 mol% doped sample. Redox peak shifted to lower potential due to the perfection in the reversibility of the electron transfer process and surface area of the modified electrode. At the extreme potential, current is found maximum because of electro-oxidation and reduction processes. The area under the 4 mol% curve is found to be maximum among all synthesized materials. This was mainly due to reduce in the ion diffusion path ensuring high utilization of surface pseudocapacitive reactions.[17]

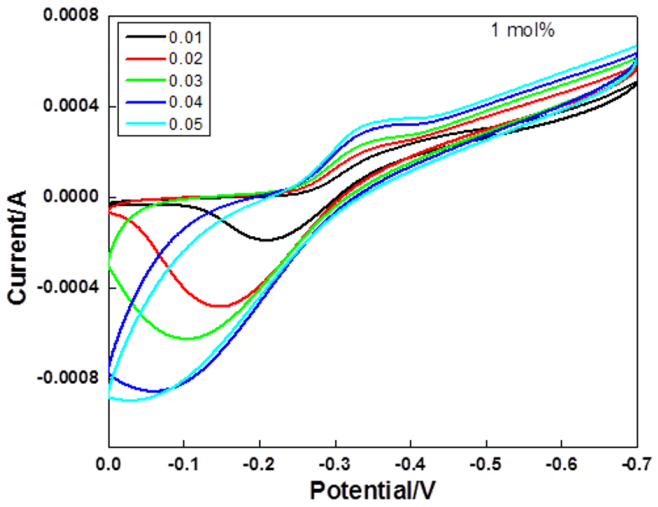


**Fig. 3: CV pattern of $Mg_2SiO_4$:$Cr^{3+}$ (1 mol%) nanoparticle**

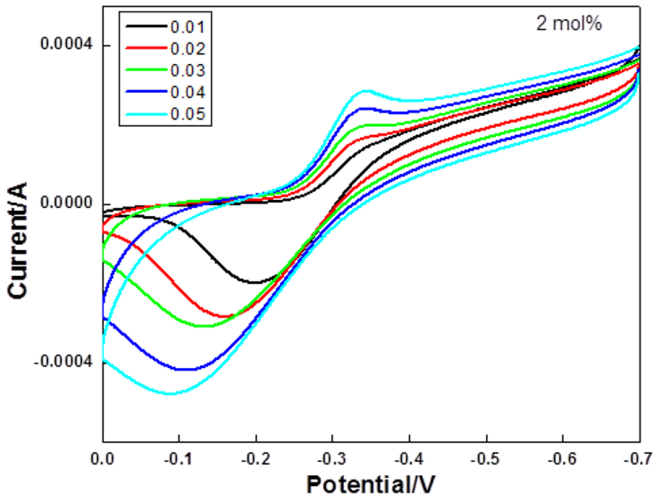


**Fig. 4: CV pattern of $Mg_2SiO_4$:$Cr^{3+}$ (2 mol%) nanoparticle**

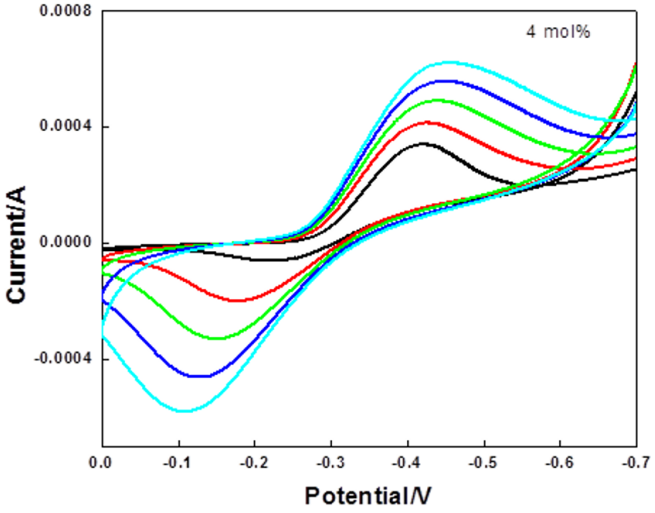


**Fig. 5: CV pattern of $Mg_2SiO_4$:$Cr^{3+}$ (4 mol%) nanoparticle**

Fig (6-8) shows the EIS (Nyquist plot) of $Cr^{3+}$ (1, 2, and 4 mol %) doped $Mg_2SiO_4$ nanomaterials. The electrochemical impedance estimations were done in the recurrence go 1 Hz to 1 MHz with AC sufficiency of 5 mV at a consistent state. The semi-circle district in the Nyquist plot implies a high-frequency part and a linear area signifies a low-frequency part. The diameter of the semi-circle speaks to charge transfer resistance (Rct) and it was accounted for that littler the measurement of the circle, higher the charge transfer rate which offers a significant contribution to the improvement of photocatalytic action of the readied test. It was observed from the diagram that, Rct values for the $Cr^{3+}$ (1, 2, and 4 mol %) doped $Mg_2SiO_4$ nanomaterial was found to be 38 Ω, 11 Ω, and 10 Ω individually.[18-20]

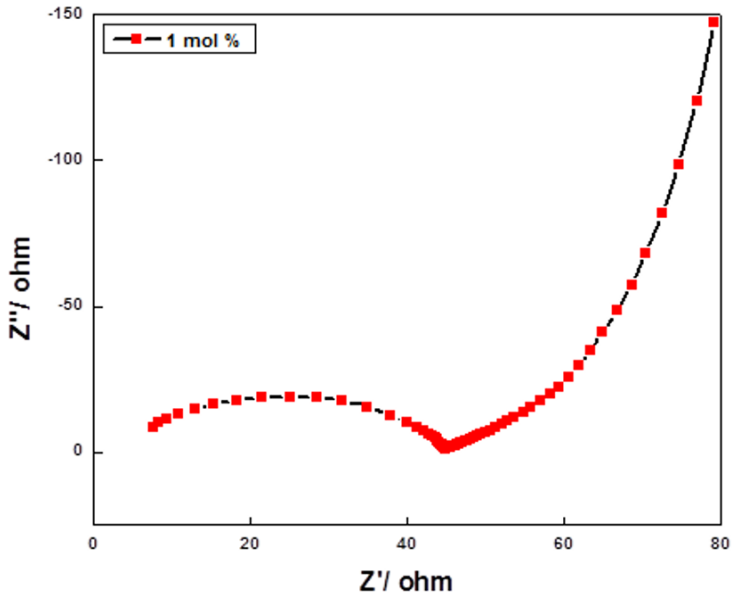


**Fig. 6: EIS pattern of $Mg_2SiO_4$:$Cr^{3+}$ (1 mol%) nanoparticle**

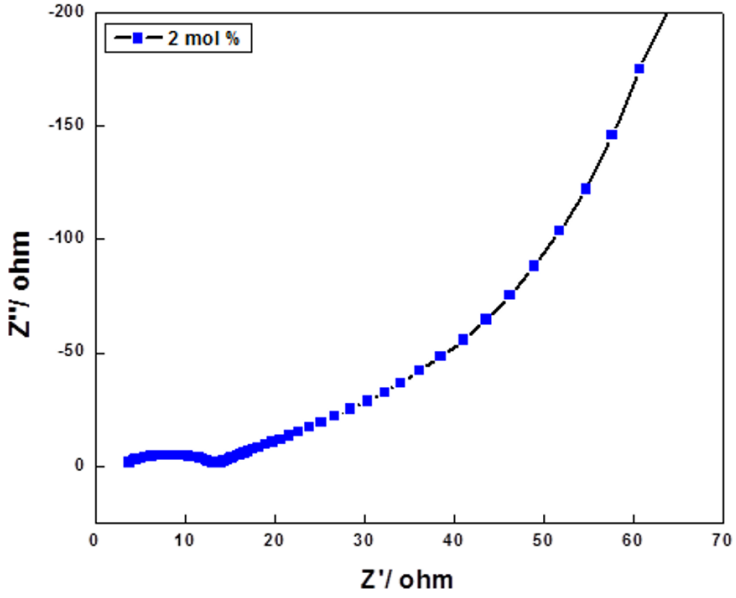


**Fig. 7: EIS pattern of $Mg_2SiO_4$:$Cr^{3+}$ (2 mol%) nanoparticle**

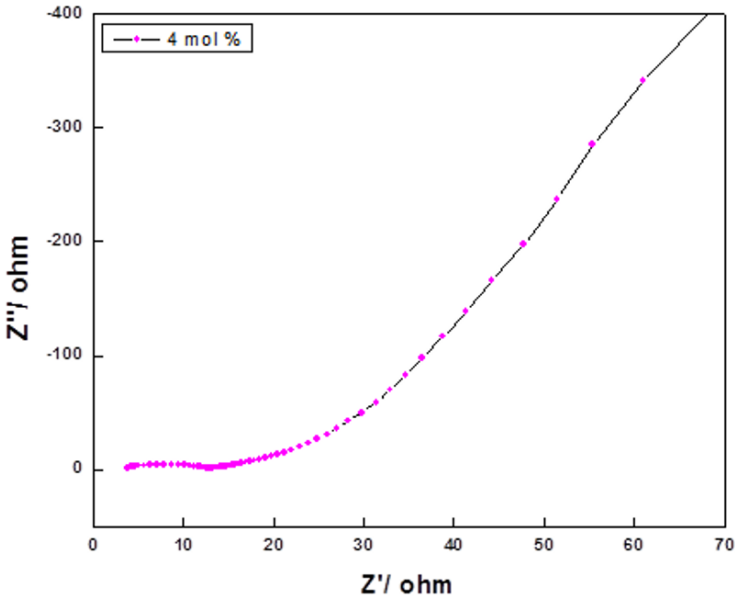


**Fig. 8: EIS pattern of $Mg_2SiO_4$:$Cr^{3+}$ (4 mol%) nanoparticle**

## Conclusion

Low temperature (350°C) initiated solution combustion technique used to prepare $Cr^{3+}$ doped $Mg_2SiO_4$ results in as formed nanoparticles with no further calcination. The prepared samples exhibit excellent electrochemical performance in the wide potential range from 0 to - 0. 7 V. The Rct value for 4 mol% material is found to be the lowest among all presently synthesized nanomaterials (10 Ω). These outcomes show that the sample is suitable for solid state electrochemical sensor development applications.

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**Conflict of Interest**
The authors do not have any conflict of interest.

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