



## Short Communication

## Electrochemical investigation of graphene/cerium oxide nanoparticles as an electrode material for supercapacitors

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## ABSTRACT

Mechanisms of charge storage, stability, capacitance, morphology and response current of graphene/cerium oxide (CeO<sub>2</sub>) nanoparticles as an electrode material for electrochemical capacitors have been investigated. Electrochemical properties of the assembled electrodes were studied using cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) techniques in 3 M NaCl, NaOH and KOH electrolytes. Scanning electron microscopy (SEM) is used to characterize the microstructure and the nature of prepared electrodes. SEM images confirm the layered structure (12 nm thickness) of the used graphene. The proposed electrode shows a maximum specific capacitance as high as 11.09 F g<sup>-1</sup> in the potential range between -0.55 and 0.3 (V vs. SCE) at scan rate of 5 mV s<sup>-1</sup>. The charge/discharge cycling test shows a good reversibility and confirms that capacitance will increase after 500 cycles by 37%.

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## 1. Introduction

Energy is currently a vital global issue given the likely depletion of current resources (fossil fuels) coupled with the demand for higher-performance energy storage systems [1]. Such systems require the advantages of portability and energy efficiency whilst being environmental friendly [2,3]. Among different energy storage systems electrochemical capacitors can provide high power capabilities, excellent reversibility (90–95%) and long cycle life (> 10<sup>5</sup>) and exhibit 20–200 times larger

capacitance per unit volume or mass than conventional capacitors [4–6].

Depending on the electrode material and charge storage mechanism, electrochemical capacitors are classified as electrochemical double layer capacitors and pseudocapacitors [7–10]. The electrochemical double layer capacitors arise from the charge separation at the electrode/electrolyte interfaces, whereas pseudocapacitors exhibit electrochemical Faradic reactions between electrode material and electrolyte [11]. Transition metal oxides are considered to be the most suitable candidate materials for electrochemical capacitors. These stem from the high specific capacitance coupled with low resistance resulting in a high specific power which makes them suitable for commercial applications. Because of the direct and fast transformation of Ce(III) and Ce(IV), CeO<sub>2</sub> nanoparticles may be good candidate as an electrode material for

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electrochemical capacitors [12]. In this paper, mechanical pressing as a fast and easy method was used to fabricate the electrodes. Then, prepared nanocomposites were evaluated as a novel electrode material for electrochemical capacitors using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM) techniques.

## 2. Experimental

### 2.1. Materials

High purity (99.98%) cerium oxide nanoparticles (10–30 nm) were purchased from US-nano, USA, graphene nanoflakes (60 nm, multi-layered) with a purity of 98.5% from graphene supermarket, USA and polytetrafluoroethylene (< 2  $\mu\text{m}$ ) from Aldrich, USA. All other chemicals used in this study were purchased from Merck, Germany. The mixture containing 15 wt%  $\text{CeO}_2$ , 75 wt% graphene and 10 wt% polytetrafluoroethylene (PTFE) was well mixed using ultrasonic in ethanol bath and in a paste form for 60 min. Paste form was chosen for better dispersion of cerium oxide nanoparticles between graphene nanoflakes. After drying and powdering, the prepared composite was pressed onto a 316 L stainless steel plate ( $5 \times 10^7$  Pa) which was served as a current collector (surface area was 1.22  $\text{cm}^2$ ). A steel rod and hollow cylinder of epoxy was used for pressing and a Teflon paper was used as a separator at the bottom of the rod during pressing. The typical mass load of electrode material was 45 mg. 3 M NaCl, NaOH and KOH solution was used as electrolyte.

### 2.2. Characterization

The electrochemical behavior of prepared nanocomposites was characterized using CV and EIS tests. Electrochemical measurements were performed using Autolab PGSTAT 302N (Netherlands). CV tests were performed within the range of  $-0.55$  and  $+0.3$  V (vs. SCE), using scan rates of 5, 10, 20, 30, 40, 50, 100, 200, 300, 400 and 500  $\text{mV s}^{-1}$ . EIS measurements were also carried out in frequency range of 100–0.02 Hz at open circuit potential with an AC amplitude of 10 mV. For better understanding the effect of the surface morphology and its nature on the charge storage and charge delivering capability of prepared electrodes scanning electron microscope (TESCAN, USA) was used.

## 3. Result and discussion

Graphene with flake morphology and very close interlayer distances has a high specific surface area which made it suitable as an electrode material for supercapacitors. Fig. 1 shows the surface morphology of the prepared graphene/cerium oxide nanocomposite. Perpendicular to these nanoflakes it shows no porosity and is completely flat. Fig. 1 confirms that the used material is completely porous in 2D and will be flat in 1D. With this morphology, it seems the charge storage depends directly on the charge separation on the upper part (which is the most accessible surface of the electrode) and on open pore systems (which

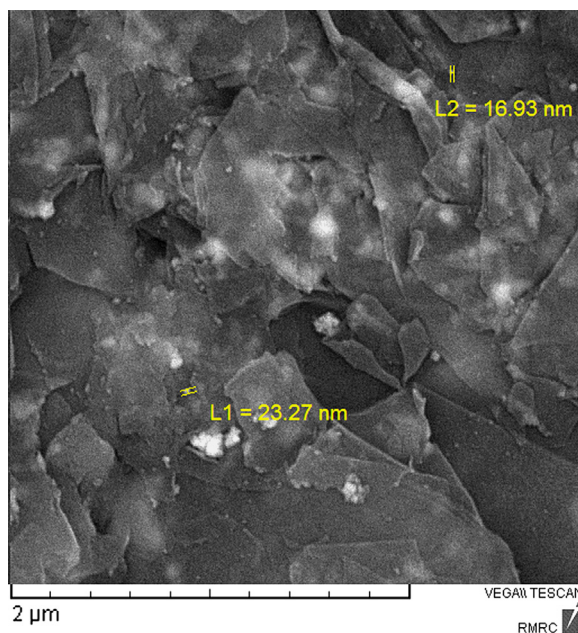


Fig. 1. Scanning electron microscopy image obtained from  $\text{CeO}_2/\text{graphene}$ .

are ion-size-dependent and less accessible) simultaneously. Ion size, ion diffusion and electron transfer sites through these pores would affect the activation of these less accessible surfaces, especially at high scan rates. Although high molarities (3 M NaOH, KOH and NaCl) of these electrolytes can be used but ion mobilities through the nanopores may be decreased and have adverse effect on the capacitance. Increasing the ionic radius would decrease the number of adsorbed ions on the unit surface area of the electrode and would decrease the stored charge on the outer Helmholtz layer. The main difference of these electrolytes is the effective radius of their anions and cations.  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Cl}^-$  and  $\text{OH}^-$  ions have effective radii of 102, 138, 181 and 153 pm, respectively. Therefore the ratio of interlayer distance of the used graphene (3.36 Å) to ionic radius ( $\alpha$ ) of these ions would be 3.29, 2.43, 1.86 and 2.20.

The specific surface area of graphene nanoflakes is high enough for the large accumulation of ions but very close interlayer distances (3.63 Å) will limit ions diffusion to these site. By adding  $\text{CeO}_2$  particles, flat surface of the electrode becomes nearly porous; therefore, the specific surface area of the electrode increases. The  $\text{CeO}_2$  quasi-spherical particles could be homogeneously dispersed across the surface of the graphene nanoflakes, thus preventing the  $\text{CeO}_2$  particles from agglomeration and will effectively accommodate the volume change of  $\text{CeO}_2$  during the charge–discharge process (Fig. 1). Graphene nanosheets possess a high electronic conductivity, which keeps the dispersed  $\text{CeO}_2$  quasi-spheres connected and decreases the contact resistance of the active material in the electrode [13]. As shown in Fig. 2, bare  $\text{CeO}_2$  showed high solution resistance and low charge transfer resistance in 3 M NaCl electrolyte which was improved by the addition of conductive materials such as graphene. Fig. 3

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