Full Paper

Electrochemical Grown of Cd²⁺ Doped Fe₃O₄ Nanoparticles and Study of their Charge Storage Performance

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Abstract- Cd^{2+} doped iron oxide nanoparticles (Cd-IONs) with magnetite crystal structure and 10-20 nm sizes were prepared through cathodic electro-synthesis (CES) method. In this procedure, IONs are galvanostatically deposited on stainless steel cathode with applying simple electrochemical conditions. The prepared both IONs were used as supercapacitor electrode material and their performances were determined through cyclic voltammetry (CV) and galvanostat charge-discharge (GCD) tests. The Cd-IONs sample exhibited specific capacitance as high as 206 F g^{-1} and cycling stability of 93.9% after 2000 cycling at 0.5 A g^{-1}. The electrochemical data confirmed the proper capacitive ability of the Cd-IONs.

1. INTRODUCTION

In all technology areas, there is a growing need for electrochemical energy storage devices like batteries, which exhibit high energy density and electrochemical capacitors (ECs), which deliver high power density. ECs are capable to provide specific power (∼500-10,000 W/kg) and long cycle life (>100,000 cycles) [1]. The electro-active materials are key element of ECs in exhibiting such abilities. Hence, considerable studies are going on the fabrication and development of highly efficient and electro-active materials for ECs. Metal

hydroxides and oxides i.e. nickel hydroxide [2-5], cobalt hydroxide [6-13], manganese oxides [14-19], iron oxides [20-28], and nickel oxide [29-32] are proper electro-active electrode materials in this case. Among the mentioned oxides, Fe_3O_4 has benefits of environmental friendliness, natural abundance, low cost and high specific capacitance (Cs=2299 F/g) [28]. However, it has low electrical conductivity, which is its major obstacle for use in ECs [22-28]. Up now, several works including compositing with CNTs and GO [33-39] and metal cations doping [40-43] have been performed to receive an improvement in the charge storage ability of iron oxide electrode. In this paper, the Cd^{2+} doped iron oxide nanoparticles (Cd-IONs) are prepared though a novel electrochemical platform i.e. cathodic electrosynthesis (CES) and their electrochemical performance is investigated. It was reported that nanostructured metal oxides/hydroxides could be easily synthesized *via* OH^- electro-generation on the cathode [44-50]. However, this method has been rarely applied for the preparation IONs [51-56]. Here, CES was used for the synthesis of Cd^{2+} doped IONs. To the best of our knowledge, CES synthesis of Cd^{2+} ion doped IONs has not been studied until now. The prepared Cd-IONs were examined by XRD, FE-SEM, VSM, cyclic voltammetry (CV) and galvanostat charge-discharge (GCD) techniques. The results of these analyses confirmed suitable capacitive capability of the fabricated Cd^{2+} doped IONs.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Cadmium chloride ($CdCl_2 \cdot 7H_2O$ 99.9%), Ferrous chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$, 99.5%), ferric nitrate nonahydrate ($Fe(NO_3)_3$, $9H_2O$, 99.9%) and polyvinylidene fluoride (PVDF, $(CH_2CF_2)_n$) were purchased from Sigma Aldrich and used as received without any purification.

2.2. Electrosynthesis of CD-IONs

The electrosynthesis cell was assembled using a stainless steel cathode with dimension of 5 cm×5 cm×0.5 mm and two parallel graphite plates as anodes. The deposition solution was iron(III) nitrate (2 g), iron(II) chloride (1 g) and cadmium chloride (0.3 g) dissolved in 1 liter deionized H_2O. The deposition runs was performed using a power supply system and constant current density of 5 mA cm^{-2} was applied for 1 h. After each run, the cathode was washed several times with deionized H_2O. Then, the black film deposited on the steel was scraped and dried. The obtained powder was named Cd-IONs.

2.3. Characterization analyses

Morphology of the prepared IONs powder was observed through field-emission scanning electron microscopy (FE-SEM, Mira 3-XMU with accelerating voltage of 100 kV). X-ray

diffraction (XRD, Phillips PW-1800) using a Co Kα radiation was used to obtain crystal structure of the prepared powders. The magnetic properties of the prepared IONs were recorded in the range of −20000 to 20000 Oe at room temperature using vibrating sample magnetometer (VSM, Meghnatis Daghigh Kavir Co., Iran).

2.4. Electrochemical tests

All electrochemical tests were conducted on an electrochemical station (AUTOLAB®, Eco Chemie, PGSTAT 30) in a three-electrode electrochemical cell. This cell was assembled using Ag/AgCl reference electrode (saturated with 1 M KCl), and a counter electrode (platinum wire) and Cd-IONs paste electrode. The working electrodes (WEs) were fabricated through well-known paste procedure reported in Refs. [35,44]. First, the Cd-IONs powder was physically mixed with acetylene black (>99.9%) and conducting graphite (with rations of 75:10:10), and the mixture was homogenized properly. Then, 5%wt polyvinylidene fluoride (PVDF) dissolved in N-Methyl-2-pyrrolidone (NMP) was added into the mixture. The resulting paste Ni foams were dried for 5 min at about 150 °C in oven. The electrolyte was selected to be aqueous solution of 1 M Na_2SO_3. Two electrochemical tests of cyclic voltammetry (CV) and galvanostatic charging/discharging (GCD) were applied for evaluation of supercapacitive performances of the fabricated Cd-IONs electrode. Mass loading Cd-IONs powder onto the Ni foam was 2.5 mg. CVs of the fabricated electrode was recorded in the potential windows of -1.0 to +0.1 V *vs*. Ag/AgCl with applying different potential scans of 2, 5, 10, 20, 50 and 100 mV s^{-1}. The GCD profiles were provided at the different current loads of 0.25, 0.5, 1, 2, 3 and 5 A g^{-1} within potential range of -1.0 to 0V *vs*. Ag/AgCl. After these tests, the specific capacitances and cyclability of Cd-IONs were calculated from their CV and GCD profiles.
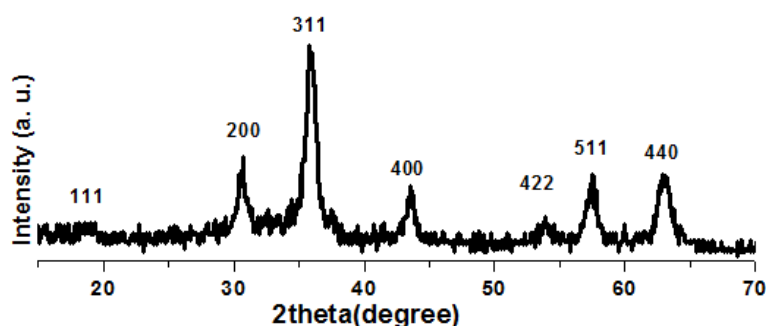

Fig. 1. XRD pattern of the Cd-IONs powder

3. RESULTS AND DISCUSSION

3.1. Structural and morphological characterizations

The X-ray diffraction pattern of the electro-synthesized powder is shown in Fig. 1. This pattern was well conceded with cubic Fe_3O_4 (JCPDS no. 65-3107), and the (220), (311), (400), (422), (511) and (440) diffraction planes are located at about 18.11°, 29.05°, 35.09°, 43.12°, 53.41°,57.21° and 63.44°, respectively. Hence, the Cd-IONs sample has Fe_3O_4 crystal structure.

The average crystallite size (D) of Cd-IONPs was calculated using the Debye–Scherrer formula (i.e. D=0.9 $\lambda/\beta cos(\theta)$), where λ is the X-ray wavelength, β is the full width at half maximum of the diffraction line, and θ is the diffraction angle. From the diffraction line-width of (311) peak, the average crystallite size of Cd^{2+} doped IONs were obtained to be 9.9 nm.
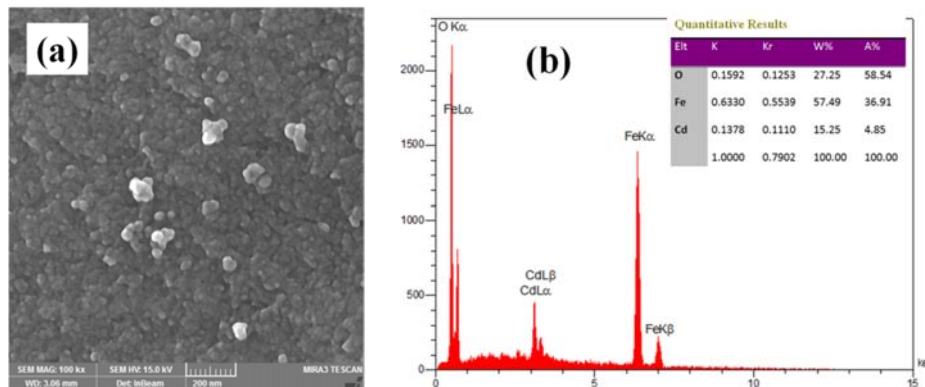
Fig. 2. (a) FE-SEM image and (b) EDS data of the Cd-IONs

FE-SEM image of the IONs are presented in Fig. 3a. The particle morphology with size range of 10–20 nm is observed. Fig. 3b indicates the energy-dispersive X-ray (EDX) data. The composition of elements 57.49%wt Fe, 27.25%wt O and 15.25%wt Cd was detected for the doped iron oxide, where these percentages are very close to those of Fe_3O_4 composition, which have elemental analysis of 72.36%wt Fe and 27.64%wt O. These results clearly proved composition of 15%wt Cd^{2+} doped IONs for our CES product.

The hysteresis loop of cadmium cations doped IONs powders was measured by VSM analysis between -2 and +2 kOe at room temperature. The S like form and absent of hysteresis showed the superparamagnetic behavior of Cd-IONs powder. The values of saturation magnetization (*Ms*), remanent magnetization (*Mr*) and coercivity (*HCi*) for IONs sample are listed in Table 1, and compared with related data in literature.
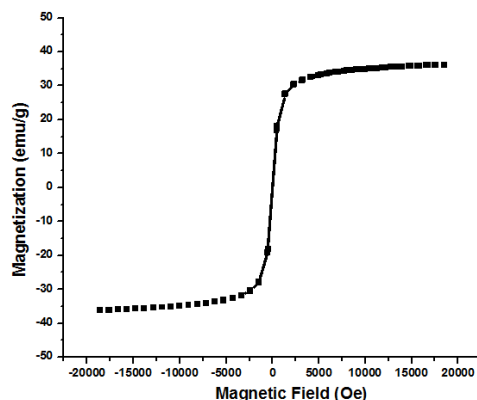
Fig. 3. Magnetization profile of Cd^{2+} doped IONs

**Table 1.** Magnetic data for doped IONs reported in the literature

Sample name	Ms (emu/)g	Coercivity (Hci)G	Retentivity Mr(emu/g)	Refs.
Cd^{2+} -IONs	36.1	8.7	0.33	This work
pristine IONs	72.96	14.6	0.95	[57]
Sm^{3+} -IONs	41.89	10.1	0.51	[58]
La^{3+} -IONs	49.75	10.7	0.58	[59]
Gd^{3+}-IONs	32.9	--	--	[60]
Cu^{2+}-IONs	53.2	--	--	[61]
Mn^{2+}-IONs	47.25	15.85	0.22	[62]

For Cd-IONs, the magnetic data are obtained to be Ms=52.04 emu g^{-1}, Mr=0.73 emu g^{-1} and H_{Ci}=7.56 G. The negligible coercivity and remanent magnetization demonstrated that the Cd-IONs have superparamagnetic behavior. As reported in Refs. [57-59], the pristine IONs prepared through electrochemical route exhibited the vales of Ms, Mr and H_{Ci} of 72.96 emu g^{-1}, 0.95 emu g^{-1} and 14.61 G, respectively. Comparing these magnetic data with those of our sample revealed smaller remanent magnetization and coercivity values doped iron oxide, implicating improvement of superparamagnetic behavior of Fe_3O_4 due to Cd^{2+} doping, where lower Ms may be related to the lower magnetism of cadmium element compared to iron. From data listed in Table 1, it is seen that Cd-IONs have better superparamagnetic behavior (i.e. higher Ms and lower Mr and Hci values) as compared with magnetic data reported for doped IONs in the literature. Hence, it is specified that Cd^{2+} doping provide better superparamagnetic performance for iron oxide.

3.2. Electrochemical evaluation

3.2.1. Cyclic voltammetry

Cyclic voltammetry was used to evaluate the supercapacitive performance of the Cd^{2+} doped IONs. Fig. 4a presents the CV profiles of the electrode within the potential range of -1.0 to +0.2 V *vs*. Ag/AgCl with applying the scan rates of 2-100 mV s^{-1}. The shapes of the CV curves clearly reveal the pseudocapacitive characteristics of the Cd-IONs, which is different from the electric double-layer capacitance. In Refs. [63-66], a combination of both EDLC and pseudocapacitance involving the reduction/oxidation of adsorbed SO_3^{2-} anions on the iron oxide surface has been reported for the capacitance behavior of iron oxide electrode in sulfite sodium solution:

$$2SO_3^{2-} + 3H_2O + 4e^- \leftrightarrow S_2O_3^{2-} + 6OH^- \tag{1}$$
$$S_2O_3^{2-} + 3H_2O + 8e^- \leftrightarrow 2S^- + 6OH^- \tag{2}$$
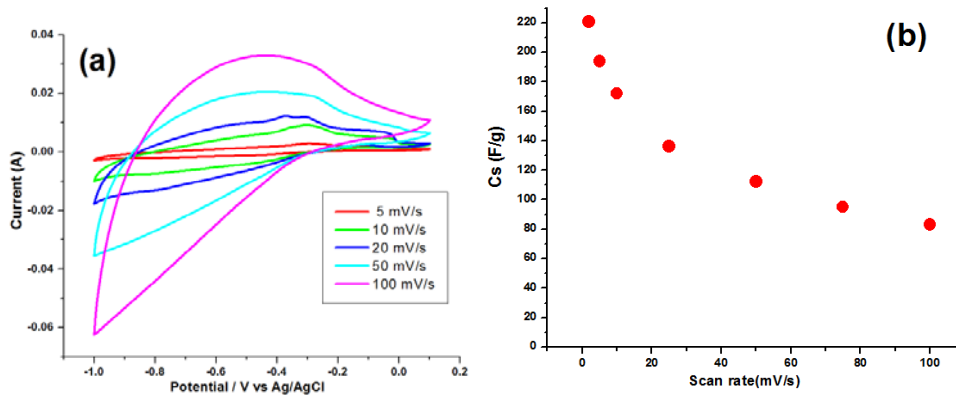
Fig. 4. (a) CVs and (b) calculted capacitances for the fabricated Cd-IONs at the various scan rates

The *Cs* values of the Cd-IONs were calculated from their CV profiles by integrating the area under the current–potential curves using Eq. (3) [67]:
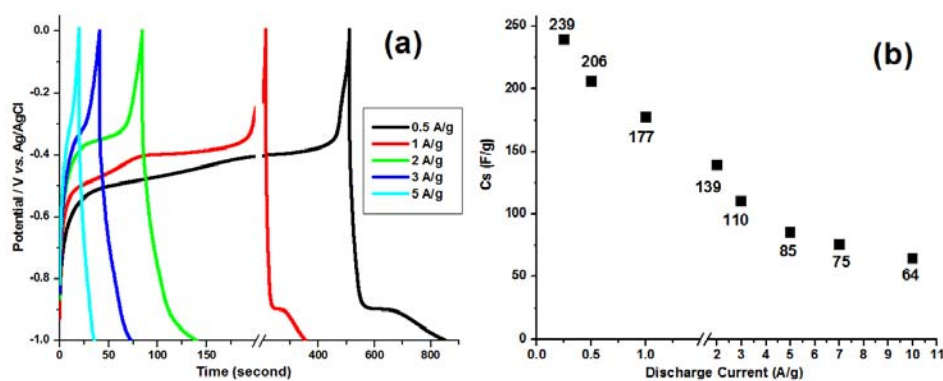
$$Cs(F\ g^{-1}) = \frac{Q}{m\Delta\ (V)}, \quad Q = \int_{V_a}^{V_a} I(V)dV \tag{3}$$

Where *Cs* is the capacitance of prepared Cd-IONs powder (F g^{-1}), Q is the total charge, ΔV is the potential window, *m* is the mass of Cd-IONs powder (g), v is the scan rate (V s^{-1}) and I(V) is the current response during the potential scan. Then, the *Cs* values were plotted *vs*. scan rate, as shown in Fig. 4c. The calculations revealed that the Cd-IONs electrode are capable to give *Cs* values as high as 221, 194, 172, 136, 112, 95 and 83 F g^{-1} at the scan rates of 2, 5, 10, 20, 50, 75 and 100 mV s^{-1}, respectively. These *Cs* data confirmed the proper charge storage abilities of the electro-synthesized Cd-IONs for use in supercapacitors. The

electrochemical behavior of Cd^{2+} doped IONs was also investigated by GCD measurements, which are discussed below.

3.2.2. Charge-discharge tests

Galvanostatic charge-discharge profiles of Cd-IONs were recorded at different current loads (0.5-10 A g^{-1}). The resulted GCD curves and the calculated Cs data are given in Figs. 5a and b.

**Fig. 5.** (a) GCD profiles and Cs values of the Cd-IONs at the different current loads of 0.2 to 10 A g^{-1}

Two different potential-time behaviors are observed in Fig. 5a. The first one at the potentials larger than -0.4 V, implicating the pure EDLC behavior and the second at the potentials lower than -0.4 V, revealing the faradic reactions of sulfite ions. The *Cs* values were calculated using Eq. (4) [68], and the data is presented in Fig. 5b:

$$Cs \ (F \ g^{-1}) = \frac{Q}{m \times \Delta V} \ , Q = I \times \Delta t \qquad (4)$$

Where *Cs* is the capacitance of prepared Cd-IONs powder (F g^{-1}), Q is the total charge, ΔV is the potential window, *m* is the mass of Cd-IONs powder (g), and I is the applied current load (A) and Δt is the time of a discharge cycle. The calculations revealed that the Cd^{2+} doped IONs are capable of delivering *Cs* values of 239 F g^{-1}, 206 F g^{-1}, 177 F g^{-1}, 139 F g^{-1}, 110 F g^{-1}, 85 F g^{-1}, 74 F g^{-1} and 64 F g^{-1} at the discharging loads of 0.2, 0.5, 1, 2, 3, 5, 7 and 10 A g^{-1}, respectively. These values are close to those calculated based on the CVs (Fig. 4b), confirming the excellent super-capacitive behavior of Cd-IONs. Furthermore, the charge storage performance of our sample are comparable with the reported *Cs* data for the nanostructured pristine and doped IONs electrodes in the literature [57-59,64-68].
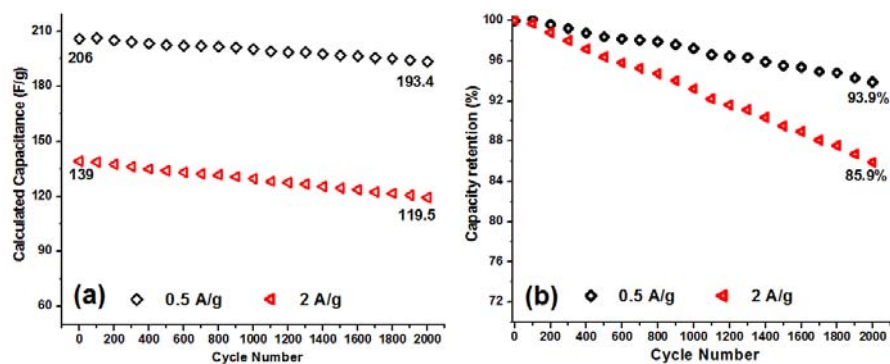
Fig. 6. (a) Cs values and (b) capacity retention for the fabricated Cd-IONs electrode during 2000 chrage-discharge cycling at the current loads of 0.5 and 2 A g^{-1}

The cycle stability of the fabricated electrode was also investigated through their 2000 times cycling at the current loads of 0.5 and 2 A g^{-1}. The Cs values and capacity retentions were calculated and presented in Figs. 6a and b. It was observed that the Cd-IONs exhibited Cs and cycling stability values of 193.4 F g^{-1} and 93.9% after 2000 cycling at 0.5 A g^{-1}, as seen in Fig. 6. Also, Cs of the electrode was reduced from 139.2 F g^{-1} to 119.5 F g^{-1} during 2000 cycling at 2 A g^{-1}, which had 85.9% cycle stability (Fig. 6). These results proved a suitable cycling performance of Cd-IONs electrode.

4. CONCLUSION

In summary, an easy electrochemical method was for first time proposed for the fabrication of Cd^{2+} doped iron oxide nanoparticles. The structural and morphological data provided by XRD, FE-SEM and EDS analyses confirmed the electrodeposited iron oxides have magnetite crystal structure and nano-particle morphology (size≈10 nm). It was also proved that the prepared Fe$_3$O$_4$ has 15%wt Cd^{2+}cations in its composition. VSM analysis revealed the superparamagnetic behavior of Cd-IONs sample. Furthermore, a proper capacitive performance was observed for the electrode assembled using Cd-IONs.

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