



Hydrothermal synthesis of Fe₂O₃ nanoparticles and their electrochemical application

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ABSTRACT

In the present investigation, we report on Iron oxide (α -Fe₂O₃) nanoparticles synthesized by simple hydrothermal method with different reaction times as 6 h (H1) and 8 h (H2) for supercapacitor application. The significance of varying the reaction time on structural, morphological, and vibrational properties of α -Fe₂O₃ was explored. XRD, FTIR, and Raman study affirmed that the products consist of only the rhombohedral phase of α -Fe₂O₃ nanoparticles. SEM image infers that with a change in reaction time the surface morphology of α -Fe₂O₃ changed from spherical to octahedra. The size of nanoparticles reduced with reaction time. XPS spectra again confirmed the growth of α -Fe₂O₃ nanoparticles. The electrochemical characteristics of the fabricated H2 electrode exhibited excellent performance in a 2 M KOH electrolyte solution. The specific capacitance (Cs) achieved from CV and GCD curves were 299.4 F g⁻¹, and 351.3 F g⁻¹. The R_{ct} acquired via electrochemical impedance (EIS) reduced from 11.2 to 9.18 Ω demonstrating the rise in the conductivity of the prepared electrodes. Remarkable capacitance retention of 92% was accomplished, even after 1000 cycles, thus making α -Fe₂O₃ nanoparticles a most promising electrode for the fabrication of energy storage devices.

1 Introduction

The supercapacitor (SC), also known as an electrochemical capacitor (EC), has attracted the attention of researchers because of its high power density, environmental friendliness, very fast charge and discharge times, long cycle lives, and excellent energy storage capability, making them ideal for their numerous

possible applications, including portable electronics, digital communications, backup power storage devices, and renewable energy systems [1–3]. Studies reveal that electrode materials are vital in determining the resultant performance of supercapacitors [4].

In recent times, research has concentrated on the advancement of various kinds of electrode materials, like transition metal oxides (TMO), conducting

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polymers, and porous carbon materials [5–8]. As well, there has been great interest in TMO because of their possible applications in energy storage, especially as electrode materials for supercapacitors [5–7]. The evolving requirement for energy storage devices has led to an increased interest in metal oxides, and a number of studies have focussed on metal oxides like RuO_2 , IrO_2 , NiO , PbO_2 , SnO_2 , V_2O_5 , CeO_2 , Co_3O_4 [9–16], etc. Besides, the mentioned metal oxides are of limited usage as a consequence of their availability, cost, and toxicity issues. Amongst the TMO, iron oxide (Fe_2O_3) proves to be an interesting electrode material in virtue of its high corrosion resistance, availability, low cost, and environmentally friendly nature [12]. Hematite ($\alpha\text{-Fe}_2\text{O}_3$), in particular, is a very promising material, with applications including electrodes in rechargeable batteries/supercapacitors [17], magnetic resonance imaging [18], photo-catalytic [17], and in gas sensor [17] applications.

Earlier reports reveal that the electrochemical behaviour of the electrode materials generally relies on the specific surface area, phase purity, morphology, and crystalline structure, etc. [19, 20]. Hence, appropriate morphology and better phase purity can make the $\alpha\text{-Fe}_2\text{O}_3$ electrodes to have better specific surface area. The preparation technique influences the surface effects, morphology, sub particle structures, particle size distribution, and physical properties of the end product [21, 22]. As per previous reports, a diverse range of synthesis techniques has been designated for manufacturing hematite nanoparticles having different morphologies, such as hydrothermal technique [23], chemical precipitation [24], spray pyrolysis [25], chemical vapour deposition [26], sol–gel [27], pulsed laser deposition [28], polyol method [29], and high vacuum evaporation [30]. Of all these preparation methods, the hydrothermal technique is the most preferred for obtaining high-quality nanoparticles with diverse morphologies. It is an easy and economical method for achieving nanoparticles of high quality. By controlling the hydrothermal reaction parameters such as solvents, temperature, precursors, and reaction time, the properties of the produced materials can be tuned [21, 31–39].

The reaction time is a powerful tool for controlling the size and phase of Fe_2O_3 nanoparticle [40, 41]. Ma et al. Synthesized $\alpha\text{-Fe}_2\text{O}_3$ nanostructures with different sizes and shapes by changing reaction time and solvent via a simple hydrothermal process, $\alpha\text{-Fe}_2\text{O}_3$

nanopolyhydra, nanoparticles and microcubes were obtained by different solvents and reaction times [42].

In the current investigation, we employed a hydrothermal technique to prepare octahedral-shaped $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. Only a few investigations have examined at how reaction parameters like reaction time and temperature impact the growth and formation of iron oxide nanoparticles. Due to this, we expended considerable effort in producing $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles with two distinct reaction times, such as 6 h and 8 h, respectively. The effect of reaction time on the crystalline phase and morphology of Fe_2O_3 products was investigated. The $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles were further investigated to study their vibrational, and electrochemical characteristics.

2 Experimental

Every single chemical used was of analytical grade, and it was used directly with no further purification. The $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles were synthesized via a hydrothermal route with distilled water as the solvent and ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) as a reagent. In this experiment, 30 ml (0.45 M) of sodium hydroxide (NaOH) has been mixed with 50 mL (0.1 M) of FeCl_3 aqueous solution and stirred vigorously under room temperature (RT). The solution mixture is then placed inside an autoclave, which is made of stainless steel along with a Teflon coating, with a capacity of 100 mL, and heated for 8 h at 160 °C and 180 °C respectively. Following the completion of the reaction, the autoclave was left to reach room temperature. The red precipitate was separated using centrifugation. The precipitate was repeatedly rinsed using distilled water and absolute ethanol and dried out at an ambient temperature. The obtained precipitation was dried at 120 °C using a hot air oven. The reaction time has been varied as 6 h and 8 h, and their respective samples were termed (H1), and (H2), respectively. And the final product was used for further characterizations.

2.1 Characterization

The Fe_2O_3 nanoparticles were characterized by X-ray diffractometer (model; X'PERT PRO PANalytical). The diffraction patterns were recorded in the range of 10°–80° for the Iron oxide samples where the monochromatic wavelength of 1.5406 Å was used.

FTIR spectra between 400 and 4000 cm^{-1} were captured using a Perkin-Elmer spectrometer. The morphology was examined with SEM (model: JEOL/EO-JSM-6390), vibrational through Raman (Horiba Jobin Yvon LabRAM HR). The XPS measurements were performed with an XPS instrument (Carl Zeiss) equipped with an Ultra 55 FESEM with EDS, and all the spectra were recorded under ultrahigh vacuum ($p < 10^{-6}$ Pascal) with Al $K\alpha$ (wavelength of 833 Å) excitation at 250 W. XPS spectra was deconvoluted using Gaussian functions. Electrochemical measurements were performed using a three-electrode system with a saturated calomel reference electrode, a platinum plate counter electrode, as-prepared working electrode, and using 2 M (potassium hydroxide) KOH electrolytic solution via CHI-660B electrochemical workstation. CV studies were carried out at varying scan rates from 5 to 50 mV s^{-1} within the potential range of -0.8 to 0.2 V. Electrochemical impedance spectroscopy (EIS) was done with a frequency range between 100 kHz and 10 mHz.

2.2 Electrochemical measurements

The electrochemical data were examined in a three-electrode setup with a 1 M KOH aqueous electrolyte solution at room temperature. Cyclic voltammetry (CV), galvanostatic charge/discharge (GCD) and electrochemical impedance spectroscopic (EIS) tests were performed by using an electrochemical workstation (CHI660e). The working electrode was prepared by using the active materials, activated carbon and polyvinylidene fluoride in weight percentage of 80:15:5 respectively and mixed with N-methyl-2-pyrrolidone (NMP) solvent to form homogenous slurry. Then, the slurry was coated on 1×1 cm² size of nickel foam (NF) with the help of a painting brush. Then, it was dried at 90 °C for 12 h in hot air oven. Finally, the prepared electrode was used as working positive electrode. In addition, the platinum rod and Ag/AgCl were used as the counter and reference electrodes, respectively.

3 Results and discussion

3.1 X-ray diffraction studies

The XRD study for the synthesized products is displayed in Fig. 1. The diffraction peaks were observed

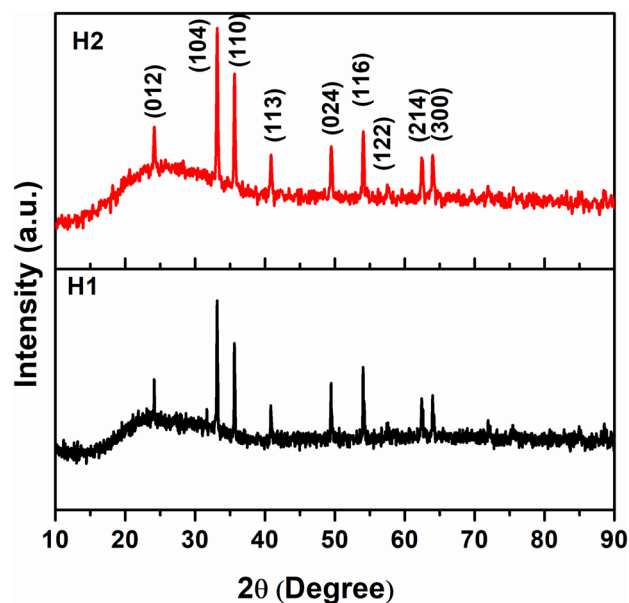


Fig. 1 X-ray powder diffraction patterns of the $\alpha\text{-Fe}_2\text{O}_3$ nanostructures

Table 1 Micro structural parameters of $\alpha\text{-Fe}_2\text{O}_3$ NPs

Samples	FWHM (β) (104)	d-Spac- ing (Å) (104)	Average Crystal- lite size (D) (nm)	Micro strain (ϵ) $\times 10^{-3}$ (lines ⁻² / m ⁴)	Dislo- cation density (δ) $\times 10^{14}$ lines/m ²
H1	0.1004	2.7037	39	1.21	6.53
H2	0.0836	2.7029	52	0.88	3.63

at 2θ range of 24.16°, 33.14, 35.62, 40.85, 49.47, 54.05, 57.52, 62.43, and 63.99, which corresponds to (012), (104), (110), (113), (024), (116), (122), (214), and (300) crystal planes. Every single diffraction peaks are strongly related to rhombohedral phase $\alpha\text{-Fe}_2\text{O}_3$ having R-3c space group (hematite, JCPDS no.01-089-0596) with the lattice constant values of $a = 5.0370$ Å, $b = 5.0370$ Å, and $c = 13.7710$ Å. Further, there is no additional phase peak in the XRD pattern, indicating the formation of a highly pure $\alpha\text{-Fe}_2\text{O}_3$ product. Highly intense (104) peak specifies that the preferential orientation of $\alpha\text{-Fe}_2\text{O}_3$ product is along (104) crystal plane. As seen in Fig. 1, and Table 1, as hydrothermal treatment time increases from 6 to 8 h, all diffraction lines become sharp and narrow (decrease in FWHM). This indicated that the 8 h synthesised product (H2) was well crystallized.

The crystallite size is evaluated from Debye–Scherrer's formula [43].

$$D = \frac{k\lambda}{\beta_{hkl} \cos \theta} \quad (1)$$

here D = size of the particle, λ = wavelength of $\text{CuK}\alpha$ radiation ($1.5418 \text{ \AA}$), β = full width at half-maximum intensity, and θ = peak position. The line broadening effect is mainly caused from the size and micro-strain [44] of nanoparticles. The micro-strain calculated using the formula [43],

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (2)$$

The calculated values of inter-planer spacing (d), average crystallite size, micro-strain, and dislocation density are shown in Table 1. The FWHM decreases as reaction time increases, suggesting that the crystalline quality has improved [45, 46]. Prolonging the reaction time improved the crystallinity, which resulted in an increase in crystallite size with the reaction time. It is possible that these ions (Fe^{2+} , OOH^{-1}) may organize into the typical sites and/or emerge into the reaction solution as the reaction time increases. Restructuring the interstitial defects may reduce Fe and O vacancies, which would result in larger crystallites. Also, the extended hydrothermal reaction time could result in a particle coalescence effect with a subsequent increasing in the crystallite size, causing relaxation in the unit cell volume [47]. Karade et al. similarly observed that crystallite size increases with an increase in reaction time for green synthesized Fe_3O_4 nanoparticles [48]. When compared to H1, the microstrain value decreases for H2 samples, which are imputed to the relaxation of strain in the respective unit cells, which causes deviations in the shape and size of the particles. For H1 and H2 samples, we evaluated the d (inter-planer spacing) values for different crystal plane (hkl) orientations. The analysed values were found to fluctuate with the crystal plane (hkl) orientations. The investigated d values of H1 for (104), (110), and (116) planes, are 2.703, 2.521, and 1.696 $\text{\AA}$, whereas for (024), (214), and (300) planes of H2, the (d) values are 1.842, 1.486, and 1.455 $\text{\AA}$. The orientation-dependent lattice spacing indicates that these crystalline materials are subject to strain, which arises because of changes in reaction rates [49]. The induced strain is also related to the change in the dislocation density of the nanoparticles [49].

3.2 FTIR spectroscopic studies

The FTIR was carried out to further validate the formation of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. The FTIR spectra for prepared $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles are shown in Fig. 2. As given in Fig. 2, the broad absorptions at 3421 cm^{-1} indicate absorbed water and structural hydroxyl groups of $\alpha\text{-Fe}_2\text{O}_3$. The wide peak at 3424 cm^{-1} belongs to the stretching vibrations of $-\text{OH}$. The absorption picks around 1633 cm^{-1} , 1383 cm^{-1} are due to the asymmetric and symmetric bending vibration of $\text{C}=\text{O}$. The strong band below 700 cm^{-1} is assigned Fe–O stretching mode [50]. The peaks observed at 834 cm^{-1} are imputed to the deformation vibration of Fe–OH groups. The peaks around 535 and 529 cm^{-1} and 445 and 455 cm^{-1} indicate the presence of the Fe–O group. No peaks that would correspond to other phases of iron oxide were observed, indicating the formation of a pure hematite phase [51].

3.3 Scanning electron microscopic studies

A typical SEM image for the samples H1 and H2 is given in Fig. 3. As seen from Fig. 3 (H1), bunches of spherical- shaped nanoparticles with an average size of 60–80 nm were formed. After increasing the reaction time from 6 to 8 h, sample H2 exhibited high uniformity and well-defined shapes. Particles have a truncated nano-octahedral shape and are bound by quasi-triangular facets that are highly symmetric.

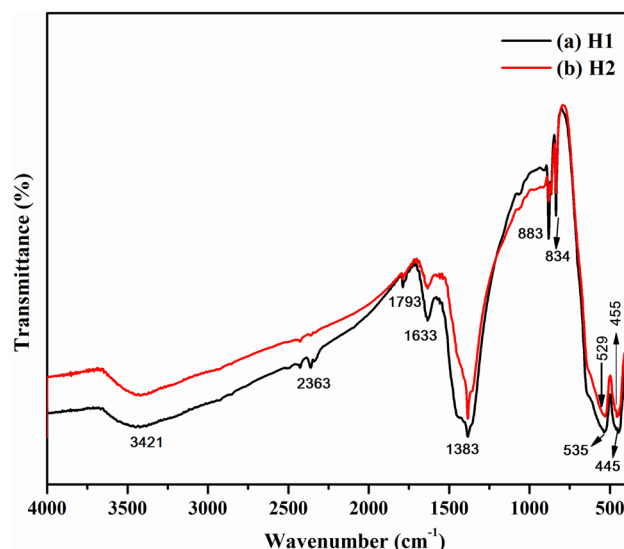
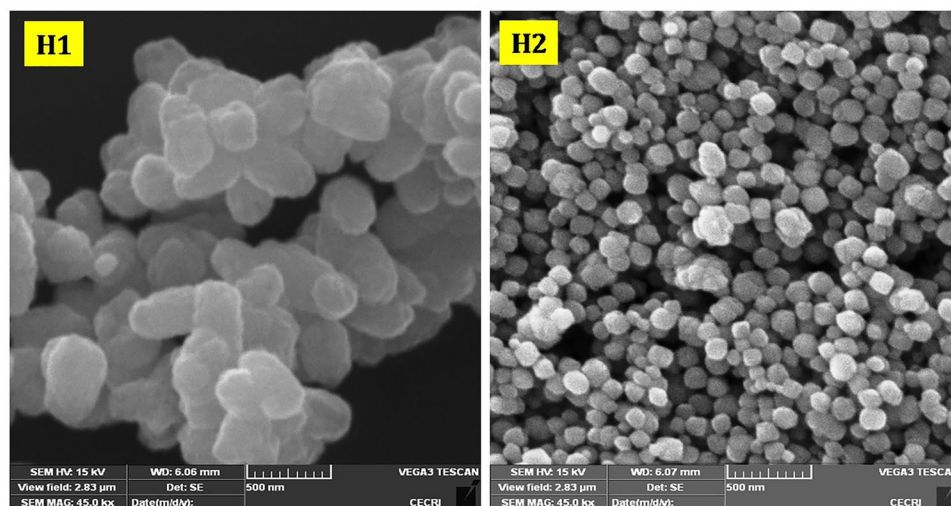


Fig. 2 FTIR spectra of the $\alpha\text{-Fe}_2\text{O}_3$ nanostructures

Fig. 3 SEM images of the samples prepared at different time duration of 6 (H1) and 8 h (H2), respectively



Also interestingly, all edges of the octahedron are concave, as presented in Fig. 3 (H2). Further, with variation in reaction time, the nanoparticles transformed from spherical to a nano-octahedral shape with sharp edges of 30–50 nm (Fig. 3 (H2)). Early reports indicate that the formation of hematite particles from FeCl_3 hydrolysis involves phase transfer from akaganeite ($\beta\text{-FeOOH}$) to hematite ($\alpha\text{-Fe}_2\text{O}_3$) [52, 53]. According to our examination of the time-dependent formation process of the truncated octahedral particles, it appears that the process involves three steps. First, a hydrothermal process hydrolyzed the Fe^{3+} cations, which then crystallized as $\beta\text{-FeOOH}$ nano spheres. Following that, the $\beta\text{-FeOOH}$ nanospheres have been dissolved and then re-crystallized to generate the $\alpha\text{-Fe}_2\text{O}_3$ nanooctahedra with pointed edges. The size of the nanoparticles reduced gradually and became uniform.

As a representative, the morphology and structure of the $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles are further characterized by TEM. The typical TEM micrographs for the H2 sample is shown in Fig. 4a. It was clear that the $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles were having nano-octahedral shapes with a mean diameter of ~50 nm. The selected area electron diffraction (SAED) pattern (Fig. 4b) was taken from the whole area of the nanoparticles turned out to be a series of diffraction rings with bright spots which may be attributed to the polycrystalline and well indexed to the corresponding crystal planes of hematite. HRTEM characterizations show the lattice fringes of the H2 sample, and the interfringe distance shown in Fig. 4d is 0.252 nm, which correspond well to {311} planar space of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles,

respectively. Both the lattice fringes and SAED, Fig. 4b, c clearly presents a group of atomic planes within each particle, revealing the highly crystalline nature of these nanoparticles.

3.4 Micro-Raman analysis

Since the H2 sample shows better results than the H1, further analyses were performed only for the H2 product. Micro-Raman study is carried out to further authorize the formation of hematite ($\alpha\text{-Fe}_2\text{O}_3$) because it is an efficient technique for differentiating different oxides of iron. Raman (Fig. 5) spectra for the reaction products indicate all active bands for hematite crystal; i.e., two A_{1g} modes (observed at 222.5 and 497.2 cm^{-1}), four E_g modes (observed at 243.4, 291.1, 407.2, and 610.8 cm^{-1}), and an additional line at 556.7 cm^{-1} attributed to the Raman-forbidden longitudinal optical (LO) E_u mode [54–56]. Hence, from the Raman analysis, the formation of hematite structure is further deep-rooted.

3.5 Compositional analysis

The composition and electronic structure of iron oxide can be determined using XPS analysis. As shown in Fig. 6 XPS survey scan shows strong signals corresponding to O and Fe which confirms the presence of oxygen (O), and iron (Fe) and the purity of prepared nanoparticles. Figure 6a and b shows the deconvoluted XPS spectra for Fe 2p and O 1s. The two major peaks observed at 724.1 (Fe 2p_{1/2}) and 710.9 eV (Fe 2p_{3/2}), represents spin-orbit peaks of Fe_2O_3 [57]. The deconvoluted Fe^{3+} 2p_{3/2} spectra show three peaks at

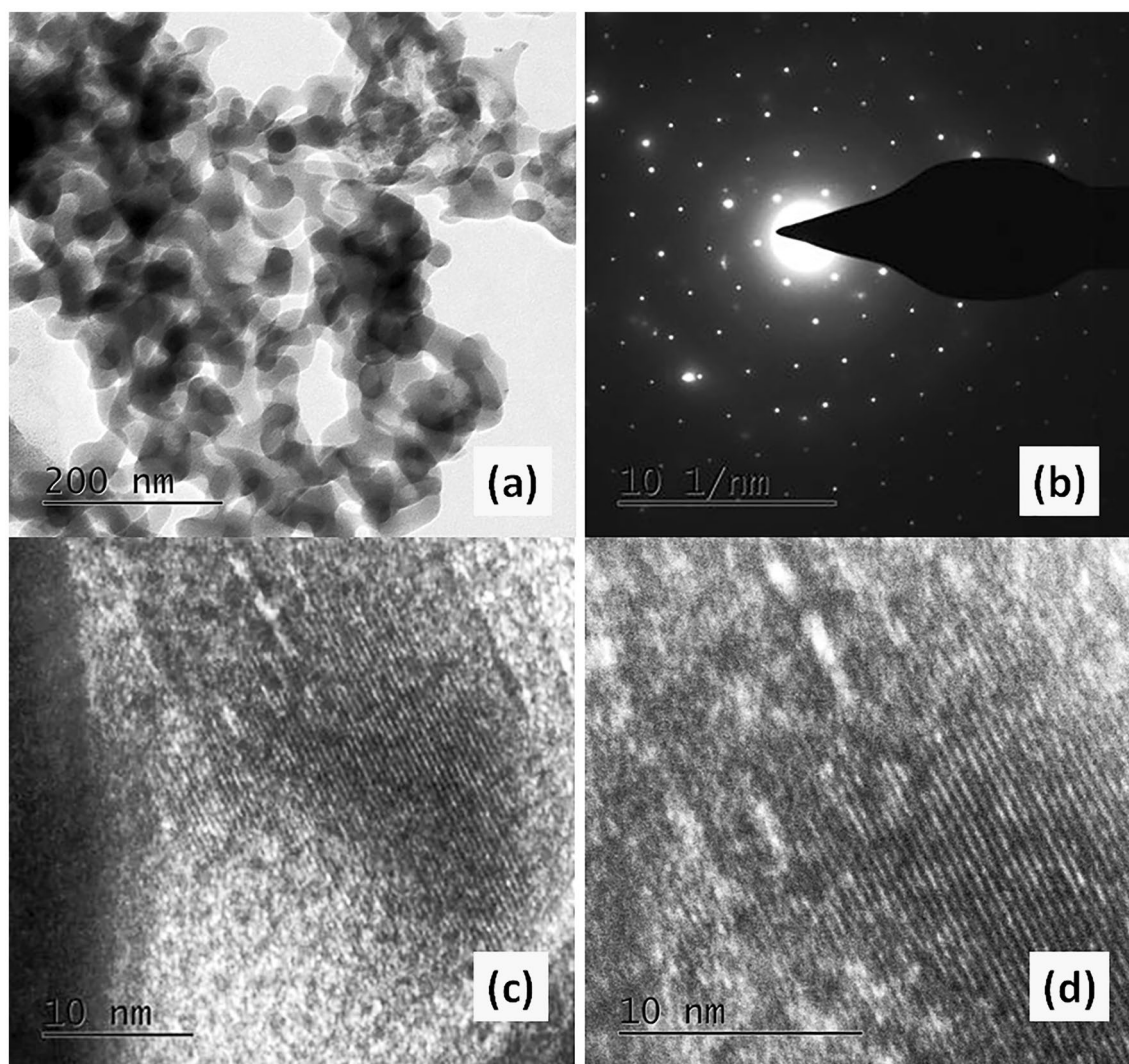


Fig. 4 **a** TEM image of H2 sample, **b** SAED, **c**, **d** HRTEM image

712.9, 711.3, and 710.3 eV, while the peaks at 724.1 and 726.2 eV are associated with $\text{Fe}^{3+} 2p_{1/2}$. These peaks represent the formation of the Fe–O bond [58, 59]. In between, the observed broad peak at 734.2 eV is accredited to the satellite peak of Fe. Also, the O 1s spectra showed three peaks located at 533.4 (C–O–C), 531.1 (C=O), and 529.8 (Fe–O) [58]. These values confirmed the formation of Fe_2O_3 nanoparticles.

Deconvolution into Gaussian functions was performed on the peaks. Three peaks at 285.1 eV, 286.6 eV, and 289.0 eV were found in the deconvolved high resolution C 1s spectra of the film, and they are ascribed to the sp^3 bulk bonded carbon C–C, C–O chemical bonds, and C=O bonds (Fig. 4). C–C sp^3 hybridization is linked to the first peak at about 285.1 eV. This is

explained by sp^3 C–H bonding or a structure like a diamond. Peak 286.6 eV detected may represent C–O–C or C–O–H. A second sub-peak, attributed to the C(O) OH or HO–COH bonds, was seen at roughly 289.0 eV [60].

3.6 Electrochemical properties

To examine the electrochemical properties of the prepared H2 electrode, the $\text{Fe}_2\text{O}_3/\text{NF}$ was tested by a three-electrode system including 2 M (potassium hydroxide) KOH electrolytic solution. The Fig. 7a displays the CV curves at varying scan rates from 5 to 50 mV s^{-1} within the potential range of -0.8 to 0.2 V. The appearance of two pairs of faradaic redox reaction peaks at the

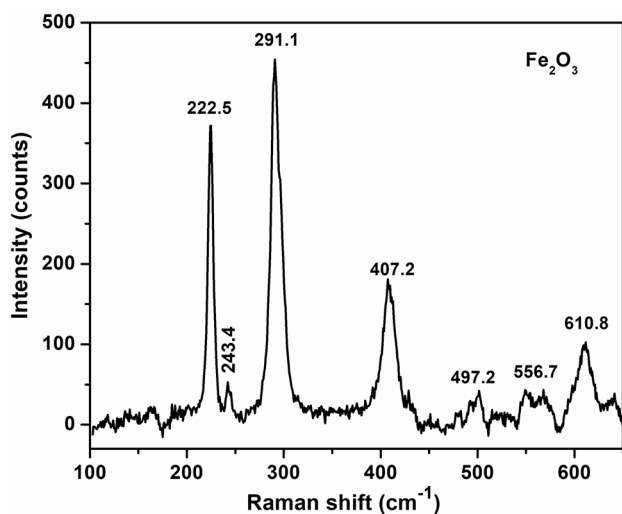
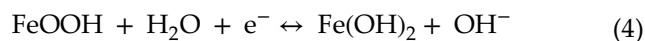
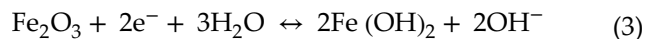


Fig. 5 Raman analysis for H2 sample

potential is because of the pseudo-capacitance characteristics of the electrodes at high scan rates, denoting outstanding electrochemical performance.

The succeeding equations might be employed to explain the potential redox reactions for Fe_2O_3 :



The specific capacitance (C_s) for the Fe_2O_3 electrodes from CV curves was determined by the subsequent Eq. [46]:

$$C_s = \frac{\int \text{IdV}}{\text{mv} - V}$$

where $\int \text{IdV}$ is the surface area enclosed by the CV curve, v is the scan rate (V s^{-1}), V is the difference of

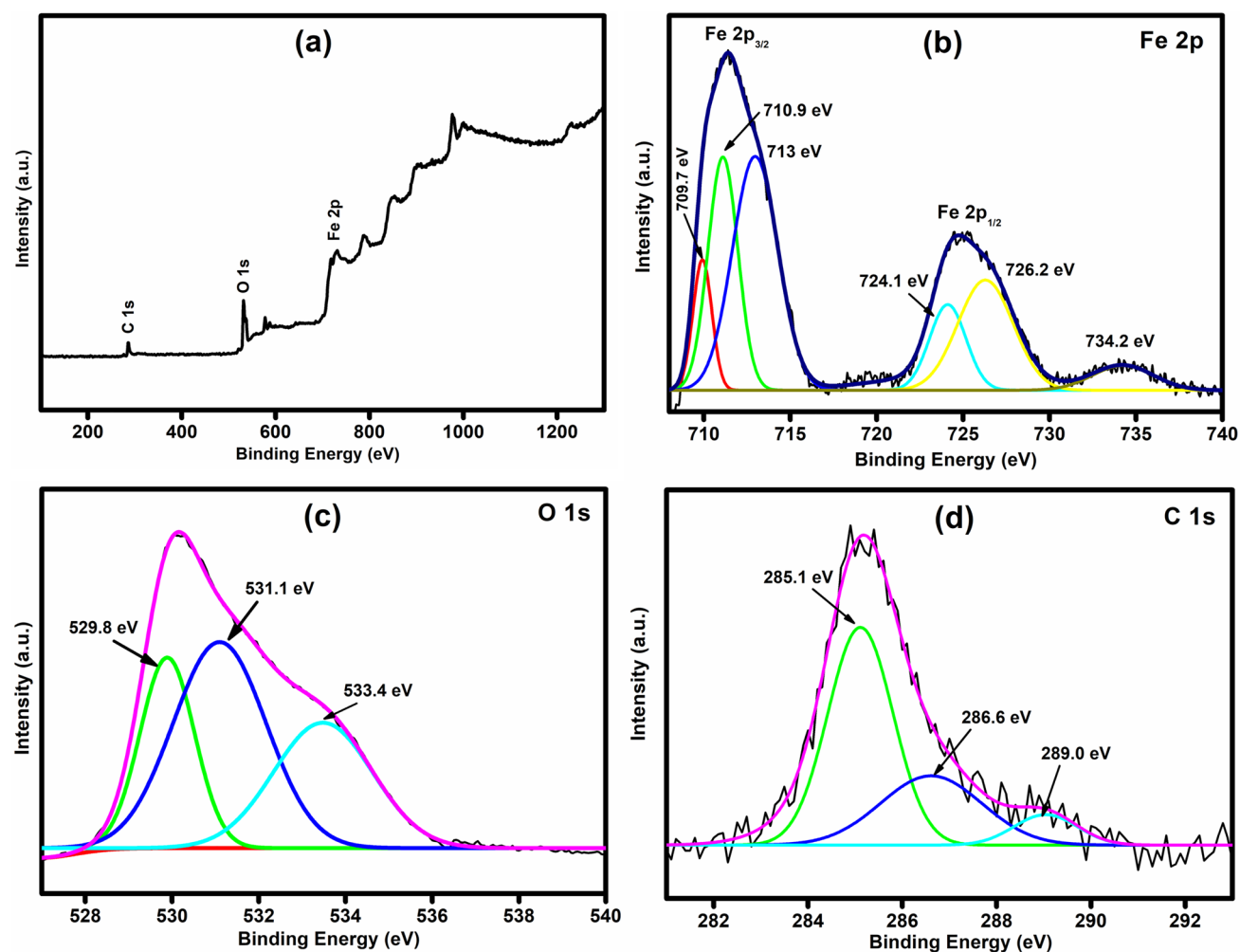


Fig. 6 XPS spectra for Fe_2O_3 **a** full scan, **b** Fe 2p and **c** O 1s, **d** C 1s

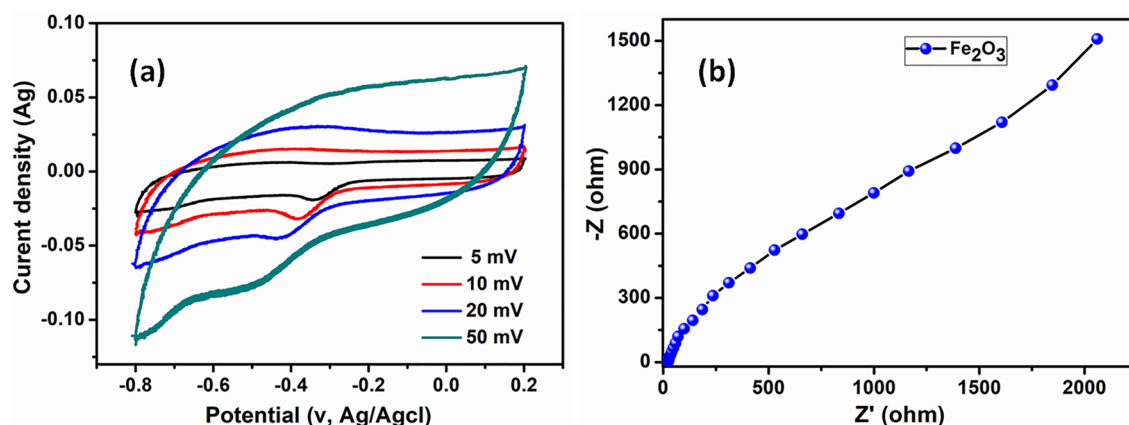


Fig. 7 **a** Typical cyclic voltammograms of Fe_2O_3 , **b** Nyquist plots of Fe_2O_3

potential window (V), and m refers to the mass of the active materials upon the electrodes (g). The C_s values of $\text{Fe}_2\text{O}_3/\text{NF}$ electrode were 299.4, 222.6, 203.8, and 178.8 F g^{-1} at scan rates of 5, 10, 20, and 50 mV s^{-1} , respectively. Increasing the scan rate minimizes the involvement of the electrode's active sites and the diffusion of electrolyte ions on the surface, lowering the C_s values of the electrodes [44]. According to the work of Cornell et al. [61], the pseudo-capacitance reaction mechanisms of magnetite in KOH may result from the surface redox reaction of hydroxide surface-layer of the nanocrystalline might facilitate the intercalation reaction, as well as the redox reactions between Fe II and Fe III accompanied by intercalation of hydroxide ions to balance the extra charge with the iron oxide layers.

Electrochemical impedance spectroscopy (EIS) of both electrodes is obtained with a frequency range between 100 kHz and 10 mHz at an open circuit potential, which the Nyquist plots of electrodes are demonstrated in Fig. 7b. The x-axis intercept at high frequencies, the depressed semicircle at high-medium frequencies and the linear line at lower frequencies are assigned to solution resistance (R_s), charge transfer resistance (R_{ct}) at the electrodes/electrolyte interface and Warburg impedance (W), respectively [62]. Also, the constant phase element (CPE) denotes the double-layer capacitance in simulating the behaviour of imperfect dielectrics. The R_{ct} value reduced from 11.2 to 9.18Ω , which demonstrates an increase in the conductivity of the electrodes.

Galvanostatic charging-discharging is a complementary method for evaluating the electrochemical capacitors at constant current. Figure 8a shows

galvanostatic charging-discharging curves of Fe_2O_3 at different current densities with potential windows ranging from -0.8 and 0.2 V with various current densities were given in Fig. 8a. The pseudo-capacitive performance of the two electrodes was confirmed by potential plateaus in the GCD curves, which correspond to the CV curves in Fig. 9a.

The C_s (specific capacitance) value is also computed from GCD curves from the succeeding equation [46]:

$$C_s = I \times \Delta t / m \times \Delta V$$

where, I is discharge current (A), and t is discharge time (s). The graph of the C_s measurements at various current densities for $\text{Fe}_2\text{O}_3/\text{NF}$ electrodes is displayed in Fig. 8b. The maximum values of C_s for $\text{Fe}_2\text{O}_3/\text{NF}$ electrodes were 351.3, 231.8, 211.1, 195.7, and 152.3 F g^{-1} at current densities of 1, 2, 3, 4 and 5 Ag^{-1} with 70.7% capability. The active sites of the electrode at low current densities can appropriately react with electrolyte ions, but at high current densities, the redox reactions only occur on the surface of the active materials as a result of the limitation of ion diffusion which leads to a decrease of the C_s values [63]. Figure 8a demonstrates the cycling stabilities of $\text{Fe}_2\text{O}_3/\text{NF}$ with capacitance retention of 82.9% electrode maintained 91.7% of its initial capacitance, which points out the excellent stability of the $\text{Fe}_2\text{O}_3/\text{NF}$ electrode. As illustrated in Fig. 9b, the capacitance retention rate of the electrode remained at a very high value of 92%, after 1000 cycles, revealing the excellent electrochemical stability of the Fe_2O_3 electrode. Table 2 compares the values calculated in this work with other reports, including Fe_2O_3 and their composites with different materials.

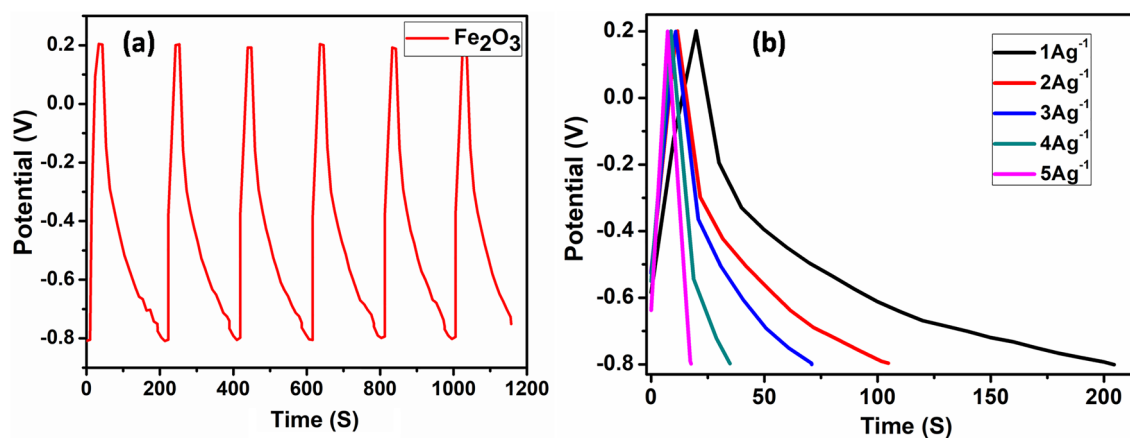


Fig. 8 **a** Galvanostatic charge–discharge behaviour of Fe_2O_3 supercapacitors 5 Cycle, **b** galvanostatic discharge curves at different current densities

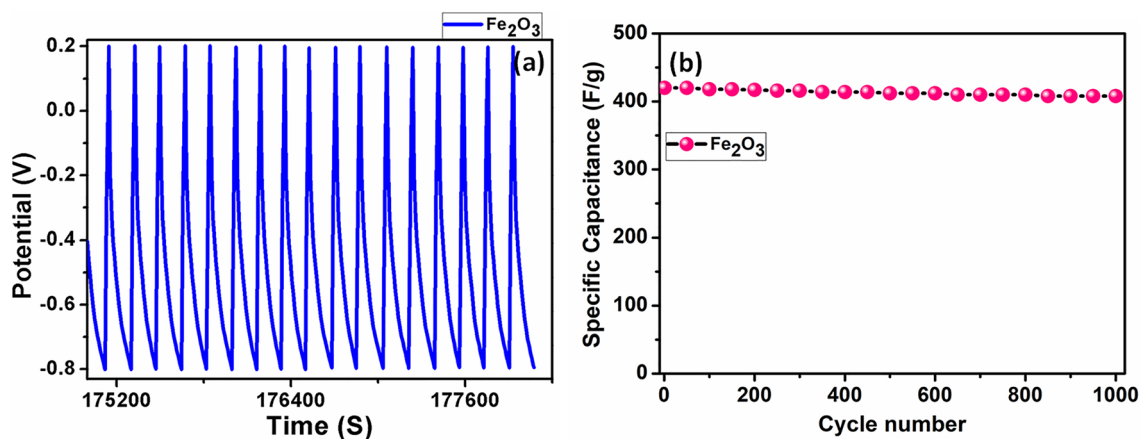


Fig. 9 **a** Galvanostatic charge–discharge behaviour of Fe_2O_3 supercapacitors 1000 Cycle, **b** cycling stability of Fe_2O_3 1000 cycle

4 Conclusions

In summary, homogeneous hematite nanooctahedra have been synthesized using an easy, additive-free hydrothermal method. The experimental results show that the reaction time plays an important role in the formation of the products. The hematite nanoparticles undergo a transfer process from $\beta\text{-FeOOH}$ nanospheres to $\alpha\text{-Fe}_2\text{O}_3$ nanooctahedra and then to corner and edge-etched truncated nanooctahedra. XRD measurements validated the rhombohedral phase of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. The size of the particles from SEM can be systematically tailored over a wide range,

from 52 to 39 nm. Peaks in the FTIR spectra of $\alpha\text{-Fe}_2\text{O}_3$ at 535 and 529 cm^{-1} and 445 and 455 cm^{-1} indicated the presence of the Fe–O group. Raman studies also confirmed the stretching vibration of the $\alpha\text{-Fe}_2\text{O}_3$. The XPS spectra again established the formation of Fe_2O_3 . The maximum specific capacitance (C_s) value of 351.3 F g^{-1} at a current density of 1 Ag^{-1} for the $\text{Fe}_2\text{O}_3/\text{NF}$ electrode with 70.7% capability was achieved. The capacitance retention rate of the electrode remained at a very high value of 92%, after 1000 cycles, revealing the excellent electrochemical stability of the Fe_2O_3 electrode. Thus, the nanooctahedral $\alpha\text{-Fe}_2\text{O}_3$ material is ideal for fabricating bio-sensor and energy storage devices.

Table 2 Comparison of the calculated specific capacitances at a current density of 1 A g⁻¹ on Ni foam in this work with other reports

Active materials	Electrolyte	Specific capacitance Fg ⁻¹	References
Fe ₂ O ₃ /G composite	2 M KOH	315	[56]
Fe ₂ O ₃	1 M H ₃ PO ₄	240	[62]
Fe ₂ O ₃	1 M KOH	145.9	[63]
α-Fe ₂ O ₃	1 M NaOH	277	[64]
Fe ₂ O ₃	3 M KOH	409	[65]
α-Fe ₂ O ₃ @C	6 M KOH	354.4	[66]
Fe-SnO ₂ @CeO ₂	2 M KO	348	[67]
α-Fe ₂ O ₃	0.5 M Na ₂ SO ₃	300	[19]
α-Fe ₂ O ₃	1 M KOH	145	[63]
α-Fe ₂ O ₃	1 M KOH	249	[68]
Porous Fe ₂ O ₃	1 mol dm ³	147	[69]
α-Fe ₂ O ₃	0.1 M Na ₂ SO ₄	200	[70]
α-Fe ₂ O ₃	1 M KOH	351	Present work

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Author contributions

All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by JV, GVP, MS, and KSU. The first draft of the manuscript was written by MS, and JV. KSU and GR, GV edited the entire draft. GV supervised the whole work and edited the manuscript. All authors read and approved the final manuscript.

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Data availability

The datasets generated during the current study are available from the corresponding author on reasonable request. Compliance with Ethical Standards.

Declarations

Competing interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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