A Study on the Synthesis, Characterisation, and Pseudocapacitive Behaviour of Fe₂O₃ Nanomaterials

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ABSTRACT

In this work, we studied the synthesis, characterization, and pseudocapacitive performance of Fe₂O₃ nanomaterials produced using a simple co-precipitation method. The as-synthesized nanomaterials were characterized in detail for their structural, morphological, and compositional properties. Conclusively, based on UV-Vis analysis, characteristic optical absorption at ~293.29 nm was confirmed for Fe₂O₃. FTIR investigation revealed the presence of specific Fe–O vibration modes corresponding to the iron oxide phase, as well as surface hydroxy groups. XRD patterns clearly recognized hematite (a-Fe₂O₃) to be the main crystalline phase, accompanied by a slight content of maghemite (γ-Fe₂O₃). Scanning Electron Microscopy (SEM) images revealed the particulate morphology of agglomerated submicron to nanoscale particles, confirming the high purity and stoichiometric Fe₂O₃ content using Energy Dispersive X-ray (EDX) spectroscopy. Cyclic voltammetry (CV) electrochemical investigation revealed a pronounced pseudocapacitive nature of the electrode, characterized by well-developed redox peaks due to the reversible Fe^{2+}/Fe^{3+} transformation. Co-precipitated Fe₂O₃ nanomaterials exhibited a significant specific capacitance of value to 446.34 F/g at a scan

rate of 5 mV/s, implying that the costeffective Fe_2O_3 nanomaterials are a promising, abundant, and environmentally friendly electrode material for highperformance supercapacitor applications.

Keywords: Iron Oxide nanomaterials; polypyrrole (PPy); cyclic voltammetry; specific capacitance optimization

INTRODUCTION

The need for practical, eco-friendly, and high-performance devices is increasing in the rapidly developing field of energy storage. Traditional sources of energy suffer from environmental issues, whereas energy storage methods frequently fail to meet power density, cycle life, or charge/discharge rate requirements [1]. The critical needs have driven substantial studies on advanced materials and novel device structures. Consequently, supercapacitors, also known as electrochemical capacitors, have emerged as promising candidates for next-generation energy storage. In contrast to batteries, where energy is primarily stored through redox reactions, and dielectric capacitors, which store energy electrostatically, supercapacitors fill this role by exhibiting a combination of high-power density (inherent to capacitors) and decent energy density (comparable to that of a battery). This unique feature is derived from their energy storage mechanisms, which first arise from EDLC or pseudocapacitive behavior. EDLCs operate non-Faradaic bv the adsorption and desorption of ions at the electrode-electrolyte surface [2], [3]. This mechanism is entirely electrostatic, with no charge transfer across the interface. Materials such as active carbon, graphene, and carbon nanotubes have been widely used in EDLC due to their high specific surface areas and electrical conductivity. Although they exhibit good power density and cycle durability, the energy density is usually restricted by the specific surface area and the type of electrolyte. On the other hand, pseudocapacitance is a faradaic charge storage mechanism that is controlled by the surface or near-surface region. In contrast to batterytype faradaic reactions, pseudocapacitive reactions are fast and reversible, offering ultrafast charge/discharge performance and high-power densities, but with significantly less energy storage capacity compared to batteries. It occurs through the redox, intercalation, deintercalation, and electrosorption of electroactive materials, which induce significant extra capacitance beyond the capacity of EDLC. Transition metal oxides and conducting polymers are typical materials that exhibit pseudo-capacitance [4]. The fact that these species might undergo reversible redox reactions on the electrode surface enables the storage of more charge per unit mass or volume, thereby increasing the energy density of SCs. Among various pseudocapacitive materials, transition-metal oxides have been recognised as appealing pseudocapacitive electrode materials due to their multiple charge states, adjustable electronic configurations, and inherent pseudocapacitive performance. MnO₂. RuO₂, NiO, and Co₃O₃ are some of the most popular examples. But each has its pros and cons. For instance, RuO₂ is a wellperforming material; however, due to its expense and toxicity, it has limitations. MnO₂ is abundant and environmentally friendly; however, it often exhibits low electrical conductivity and poor power density. As a result, alternative transition metal oxides are being researched for a compromise between performance, cost, and environmental benignity [5], [6], [7].

Fe₂O₃, also known as hematite, is a promising material for pseudo-capacitive applications, garnering attention as a material with high potential in various fields, including energy. Iron is the most abundant and cheapest element on Earth, making iron oxide very appealing from an economic standpoint. Moreover, Fe₂O₃ possesses specific inherent properties that make it an ideal electrode material for supercapacitors [8], [9], [10]. It displays various oxidation states (Fe^{2+}/Fe^{3+}) that can lead to reversible acting redox reactions, as pseudocapacitance. The theoretical specific capacitance of it is still very high. However, the bulk Fe₂O₃ has inherent drawbacks, such as low electrical conductivity and slow ion diffusion kinetics, which limit its electrochemical performance, especially at high charge/discharge rates. To address these fully limitations and harness the pseudocapacitive performance of Fe₂O₃, researchers have recently focused on controlling its nanostructure and size. Nanostructured materials have several advantages unique in improving electrochemical activity [9], [11]. Due to the high surface area of nanomaterials, the ratio of surface to volume is large, which in turn offers more active sites for electrochemical reactions when compared with other forms of materials and effectively enhances the specific surface area for charge storage. At 16 seconds, the shortened diffusion distances for ions and electrons in nanostructures can charging kinetics. thereby enhance increasing power density. Thirdly, the quantum size effect and surface energy effect at the nano-dimensional level can alter the electronic band structure, enhancing the inherent conductivity of materials that are insulators in bulk form. Several synthetic methods, including hydrothermal synthesis, solvothermal synthesis, co-precipitation, solgel. and electrodeposition, have been employed for the synthesis of variously shaped Fe₂O₃ nanomaterials, such as nanoparticles, nanorods, nanowires, nanosheets, and hollow structures. Each of these morphologies can lend various electrochemical properties to their specific surface area, pore structure, and crystallinity [12]. In this article, a detailed study of the characterisation, synthesis. and pseudocapacitive response Fe₂O₃ of nanomaterials is reported. The objective is to understand the manipulation how of synthesis conditions affects the characterisation of the morphology, crystalline structure, and surface properties of Fe₂O₃ nanomaterials, and then how these structural and morphological features influence the electrochemical behaviour, with application to supercapacitors. The work aims to clarify the subtle correlation between the design of nano-based materials and their corresponding pseudo-capacitance [13]. To this end, we systematically study various synthesis approaches and their impact on the structure of the resultant Fe₂O₃ nanoarchitectures, and develop the most effective strategies for producing Fe₂O₃based super capacitive electrodes. The applied characterisation methods will provide in-depth information on material compositions, crystal structures. morphologies, specific surface areas, and porosities. This detailed knowledge will be complemented by electrochemical characterizations, including cyclic analyze the specific voltammetry, to capacitance, energy density, power density, and cycling stability of the engineered Fe₂O₃ nanomaterials. Ultimately, this work contributes to the corpus of knowledge on pseudocapacitive materials and provides essential information for the rational design and fabrication of next-generation Fe-based supercapacitors, aiming to meet the demands of a sustainable energy future [12], [13], [14].

MATERIALS & METHODS

In this work. the synthesis and electrochemical response of Fe₂O₃ nanomaterials were studied in great detail, primarily utilizing the versatile coprecipitation approach. All chemicals, such as Iron (III) Chloride Hexahydrate (FeCl₃ \cdot 6H₂O), Iron (II) Sulfate Heptahydrate (FeSO₄ \cdot 7H₂O), Ammonium Hydroxide (NH₄OH) and Sodium Hydroxide (NaOH), were of analytical reagent (AR) grade (without further purification) and deionized (DI) water was used throughout the synthesis.

Co-precipitated Preparation of Fe₂O₃ Nanomaterials

The co-precipitation method was used to synthesized the Fe₂O₃ nanomaterials. In this process, 10 mmol FeCl₃.6H₂O was dissolved in 100 mL of DI; the solution was then heated 60 °C under consistent at stirring. Afterwards, the solution was adjusted to the desired pH range (7.0 to 9.0) by the addition of 1 M ammonium hydroxide, resulting in a reddish-brown precipitate of iron (III) hydroxide. The suspended matter was then allowed to age for 1 to 3 hours, and the precipitate was washed with DI water using centrifugation or filtration under vacuum to achieve a neutral pH. The washed precipitate was dried at 80 ^oC for 24 h and then calcined in air using a muffle furnace at 600 °C for 2 h to produce Fe₂O₃ nanomaterial. The optimization of the co-precipitation procedure was performed through а systematic study, where precursor concentration, precipitation pH, reaction temperature. aging time. calcination temperature, and calcination time were varied to determine the optimal conditions and material properties.

Characterization techniques

The prepared Fe₂O₃ nanomaterials were characterized using a variety of analytical techniques. For the characterization of the crystallographic phase, crystal structure, m, and mean crystallite size, XRD analysis was performed, utilizing the Scherrer equation. SEM-EDX characterized the morphology, particle size distribution, and elemental composition of the powder [15], [16], [17]. N₂ Adsorption-Desorption Isotherms (BET Surface Area and Pore Size Distribution) were used to characterize the specific surface

area, pore volume, and pore size distribution important in electrochemical performance. Functional groups and iron oxide bond formation were characterized by using FTIR Spectroscopy [18], [19], [20]. Lastly, the elemental and surface chemical states of iron and oxygen were analyzed by XPS.

Electrochemical analysis

Electrochemical characterization of the prepared FTCS was performed using a Princeton Applied Research potentiostat, with phosphate-buffered saline and electrochemiluminescence as the electrolyte, in 5% 0.4 M K₂CO₃. A three-electrode electrochemical system was thoroughly evaluated to assess the pseudocapacitive performance. The working electrodes were fabricated by casting a slurry of 80 wt% Fe₂O₃ nanomaterial, 10 wt% Super P carbon, and 10 wt% PVDF binder in NMP onto nickel foam or carbon cloth, followed by vacuum drying. A Pt foil or graphite rod was utilized as the counter electrode, and a saturated calomel electrode (SCE) or Ag/AgCl electrode was employed as the reference electrode. A 3 M aqueous potassium hydroxide (KOH) was chosen as an electrolyte due to its appropriate ionic conductivity and stability of transition metal oxides. The electrochemical measurements were conducted on an electrochemical workstation. CV was conducted in the potential range of -0.2 V to 0.6 V (vs SCE) at different scan rates to study the charge storage mechanism and calculate the specific capacitance [12], [13].

RESULT

UV-Vis analysis

The UV-Vis absorption profile of the films prepared from Fe_2O_3 nanoparticles is illustrated in the fig. 1, revealing interesting information related to their electronic structure and optical features. The spectrum exhibits distinctive atmospheric absorption bands in the ultraviolet (UV) and visible regions.



Figure 1: UV-Vis spectra of Fe₂O₃ nanomaterials

There is an intense absorption peak located at around λ max = 293.29 nm in the UV area. Such strong UV absorption is usually assigned to charge transfer transitions within the Fe₂O₃ lattice, namely the O2– \rightarrow Fe3+ ligand-to-metal charge transfer transitions. Beside the intense UV absorption, a wider absorption range is seen up to visible region including 400 nm, and beyond in the spectrum. This extended absorption at visible wavelengths is typical in optical characteristics for Fe₂O₃. This visible region

absorption is due to d-d electronic transitions of the Fe³⁺ cations in a crystal field, and intervalence charge transfer transitions, when mixed valence iron species could be present, but Fe2O3 is dominantly Fe3+. The broader nature of the absorption in the visible region leads to the characteristic red-brown appearance of Fe_2O_3 materials, since it absorbs blue and green light more strongly and transmits, or reflects, red light [13]. The absorption properties of Fe₂O₃ nanoparticles depend on many factors, such as the size, shape, and crystalline state, as well as purity of the particles. For nano-scale materials, quantum confinement effects can cause a blue-shifting (shifting to shorter wavelengths) or red-shifting (shifting to longer wavelengths) of absorption edges compared to a bulk material. The higher intensity for the peak at 293.29 nm reveals its narrow size distribution or a particular electronic transition of the as-synthesized nanoparticles. The total absorption spectrum of Fe_2O_3 thoroughly confirms the valid formation of Fe_2O_3 and demonstrates their possibility for use as photocatalytic or other optical applications in which light absorption is a crucial parameter. Optical band gap of the as-prepared Fe_2O_3 nanoparticles can be determined by using other analyses, i.e Tauc plots.

FTIR analysis

The FTIR spectrum of the aniline-capped Fe_2O_3 nanoparticles is the key to understand the chemical composition of the synthesized materials,to study the underlying lattice vibrational modes of the iron oxides, and to check for any adsorbed chemical or functional groups on the surface of the study Fe_2O_3 nanoparticles (Fig. 2). The spectrum shows some characteristic absorption bands which agree with the formation of Fe_2O_3 and may provide information for the surface chemistry.



Figure 2: FTIR spectra of Fe₂O₃ nanomaterials

A strong, very broad absorption band appears around 3428 cm⁻¹. This band is the typical presence of O-H stretching vibrations, due to the adsorption of water molecules, the surface hydroxyl group (Fe-OH) or residual moisture from the synthesis and drying. Surface hydroxyl groups of metal oxide nanoparticles have been reported to be widely distributed and strongly contribute to their surface chemistry, wettability and electrochemical interaction in aquatic environments. Moreover, one more peak

observed at around 1632 cm⁻¹ also confirms the existence of water molecules, which may be related to the bending vibrations of adsorbed H₂O. Other peaks observed are usually ascribed to environmental or processrelated effects. The commonly observed peak at around 2923 cm⁻¹ is attributed to C-H stretching vibrations, implying that there exists a small amount of organic impurity, verv likelv. it comes from solvent. atmosphere CO₂ adsorption or contacts with the environment during the experiment. A sharp but weak peak at around 2317 cm⁻¹ is often assigned to absorption due to atmospheric CO₂. The 1033 cm^{-1} band is not often seen for pure hematite and its potential origins could include residual sulfate groups from precursors or iron oxyhydroxide species that did not completely transition to Fe₂O₃ upon calcination, and it requires some attention. Importantly, the very intense absorption bands in the fingerprint region, well below 800 cm–1, down to 400 cm⁻¹, are a strong evidence of iron oxide phase formation. These bands are attributed to the inherent metal oxygen stretching vibration (Fe-O modes) of Fe₂O₃ in the crystal lattice. For α -Fe₂O₃ (hematite), various specific absorption peaks should be located in this low wavenumber region, and it is confirmed that the target material is successfully prepared [12], [13], [14].

XRD analysis

The XRD pattern further verifies the crystalline of the Fe₂O₃ nanomaterial. All of the central diffraction peaks match that of hematite (α -Fe₂O₃, JCPDS No. 33-0664), which is indicated by marked reflections of (012), (104), (110), (113), (024), (116), (018), (214), and (300) (Fig. 3).



Figure 3: XRD spectra of Fe₂O₃ nanomaterials

The highest peak is at $\approx 33.1 \circ$ (104). Deflection of additional peaks (denoted by *), also appearing, is only attributed to another phase, which can be assigned to maghemite(γ -Fe2O3, JCPDS No. 16-0653). These peaks are identified as reflections

(022), (420), and (620), etc. Peaks are sharp, indicating good crystallinity, and broadening is observed, suggesting a crystallite size of approximately 34.5 nm. Overall, the XRD pattern further proves the existence of Fe_2O_3 nanoparticles with a dominant hematite

phase, while maghemite is presented as a minor phase.

SEM-EDX analysis

The morphology and elemental composition of the prepared Fe_2O_3 nanomaterials were investigated by SEM-EDX. According to the SEM images (Fig. 4), the morphology is particulate with sub-micron nature with a tendency to aggregate. At higher magnification (x2000), discrete aggregates can be seen composed of individual nano ranging in size from tens of nm to a few hundred nm. Such a granular 'agglomerated' morphology is characteristic of the coprecipitation and calcination process and may give a high surface area upon dispersion despite agglomeration [5], [21].



Figure 4: SEM images of Fe₂O₃ nanomaterials

The EDX spectrum displays strong peaks for Oxygen (O) and Iron (Fe) with no visible impurity signals, confirmed the high purity (Fig. 5). The elementary composition is also confirmed by the quantitative analysis: O: 28.94 mass% (58.70 atom%) and Fe: 71.06 mass% (41.30 atom%). These data turn out in very good correlation with the empirical stoichiometry of Fe_2O_3 (about 30.06 mass% O; 69.94 mass% Fe) proving the successful synthesis of iron (III) oxide.



Figure 5: EDX spectra of Fe₂O₃ nanomaterials

Electrochemical analysis (CV)

The pseudocapacitive properties of the prepared Fe_2O_3 nanomaterials were well studied using cyclic voltammetry (CV) in a three-electrode system. CV curves were stepwise recorded by changing the scan rate to 2, 5, 10, and 20 mV/s, respectively, between the

potential window from 0 V to ca. 0.45 V (Fig. 6). The distinct characteristics of the CV curves decisively demonstrate the predominance of brain pseudocapacitive behavior in the Fe₂O₃ nanomaterials, in sharp contrast to the ideal double-layered electrode capacitor behavior.



Figure 6: CV analysis of Fe₂O₃ nanomaterials

The CV curves vividly illustrate the sharp anodic (oxidation) and cathodic (reduction) peaks that represent the specific characteristics of faradaic redox reactions. These peaks, observed at around 0.3-0.4 V on the reverse scan and 0.1-0.2 V on the forward scan, can be attributed to the reversible phase transformation between the Fe2O3 and Fe3O4 states on the surface of the Fe2O3 nanomaterials. This electrochemical behavior is known to be related to the reversible intercalation and deintercalation of cations (K+ from the KOH electrolyte), accompanied by variations in the iron oxidation states. In addition, as the scan rate is incremented from 2 mV/s to 20 mV/s, the resulting current density also increases correspondingly, indicating the material's ability to store charge at different rates, as expected. However, at higher scan rates, the redox peaks become broader and shift apart, denoting increased polarization and kinetic restriction. This typical is of pseudocapacitive materials, in which ion diffusion and charge transfer processes are hindered at high scan rates. Nevertheless, the curves consistently maintain their nonrectangular shape, indicating that the charge storage is primarily of a pseudocapacitive nature [13], [14]. Values of the specific capacitance obtained from the integrated charge (Area) of the CV curves also indicate the good pseudocapacitive nature of the Fe₂O₃ nanomaterials. Specific capacitance is found to be extremely high at extremely low scan rates. It reaches a maximum value of 438.91 F/g at 2 mV/s and 446.34 F/g at 5 mV/s, and a general decrease in specific capacitance taken place as the scan rate increases to 10 mV/s (with an SC value of 282.25 F/g) and 20 mV/s (with an SC value of 242.34 F/g), which is commonly observed to occur in the pseudocapacitive materials and attributed to the inability to utilize all the active sites when faster scan rates limit ion diffusion time. It shows that, though there is a rise at 40 mV/s (295.84 F/g), the capacitance decreases at a higher rate of voltage from 5 to 50 mV/s (189.17 F/g). However, the high specific capacitance and good retention of capacitance at higher scan shown by the present rates Fe₂O₃ highlight their promising nanomaterials for high-performance potential energy storage devices.

CONCLUSION

The prepared Fe₂O₃ nanomaterials were synthesized using a simple, low-cost coprecipitation method, with systematic tuning of the synthesis conditions to control their pseudocapacitive energy storage performance. In-depth characterizations revealed precise details about the structure, morphology, and composition of the material. UV-Vis spectroscopy revealed the characteristic optical absorption of Fe2O3, with absorption peaks at ~approximately 293.29 nm, indicating the electronic transitions characteristic of iron oxides. FTIR spectra confirmed the Fe₂O₃ phase with characteristic Fe-O vibrational modes in the low wavenumber region as well as the presence of surface hydroxyl groups and adsorbed water, which are essential for the electrode-electrolyte interactions. XRD studies confirmed the crystalline nature of the as-prepared material, which was found to be composed of hematite (α -Fe2O3) as the main phase (JCPDS No. 33-0664) along with a minor presence of maghemite y-Fe2O3 (JCPDS No. 16-0653). Analysis by SEM revealed a particulate nature, with particles in the nm size range, indicating aggregates with a high surface area. EDX analysis also revealed the stoichiometric and high purity of Fe₂O₃, with the elemental percentages in agreement with the theoretical values. CV was used for the electrochemical study, revealing the excellent pseudocapacitive performance of the Fe₂O₃ nanomaterials. The CV curves showed pronounced redox peaks, representing reversible faradaic reactions of the Fe2+/Fe3+ redox pair. The specific capacitance value is significantly higher at lower scan rates, reaching 446.34 F/g at 5 mV/s. It exhibits the classical drop in specific capacitance at higher scan rates due to kinetic limitations, while still preserving high charge storage capability, demonstrating good rate performance. The studies show that nanomaterials with high crystalline purity and pseudocapacitive applications could be produced using the co-precipitation route. The results provide insight into Fe_2O_3 as a promising, abundant, and environmentally friendly electrode material for highperformance supercapacitors. Future work will focus on further improving the combination of morphology and conductivity to enhance cycling stability and power density.

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