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Hydrothermal engineering of polyethylene glycol-assisted boron nitride/hematite nanohybrid composites for high-performance supercapacitors

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Developing high-performance energy storage materials is essential to meet the increasing global demand for sustainable energy solutions. In this study, a novel strategy is employed to synthesize polyethylene glycol-assisted boron nitride/hematite (PEG-BN/ α -Fe₂O₃) hybrid composites through a hydrothermal process. Polyethylene glycol (PEG) serves as both a dispersant and a non-covalent linker that bridges hematite nanoparticles and BN sheets. With a combination of van der Waals interaction and hydrogen bonding with the component materials, PEG enables stable and homogeneous dispersion of hematite on the otherwise inert and agglomeration-prone BN surface. This dual interaction approach enables controlled interface engineering, solving one of the major challenges commonly faced in the synthesis of BN-based composites. It also acts as a functional modifier that modulates the interfacial interactions and regulates the nucleation and dispersion of α -Fe₂O₃ nanoparticles within the BN matrix. The incorporation of PEG enhanced the electrochemical and structural properties of the hybrid composite. Structural and morphological characterizations confirmed the uniform dispersion of α -Fe₂O₃ within the BN matrix, with PEG enhancing the interfacial interactions and overall material stability. TGA demonstrated that PEG incorporation significantly improved the thermal stability of the composites, delaying degradation and preserving structural integrity under high-temperature conditions. Electrochemical measurements, including CV and GCD analysis in a 6 M KOH electrolyte, revealed superior charge storage capabilities for PEG-BN/ α -Fe₂O₃ compared to BN/ α -Fe₂O₃. This hybrid composite exhibited a remarkable specific capacitance of 361.6 F g⁻¹ at a current density of 3 A g⁻¹, significantly outperforming the individual components. The GCD studies display an enhanced charge retention capability of the hybrid composite with a coulombic efficiency of 83%, indicating reduced internal resistance and improved kinetics. Additionally, electrochemical impedance spectroscopy indicated a lower charge transfer resistance and enhanced conductivity in PEG-modified composites. The composite also retained 85% of its initial capacitance after 5000 cycles, demonstrating excellent cyclic stability. The improved electrochemical performance of PEG-BN/ α -Fe₂O₃ hybrid composites is attributed to the synergistic effects of BN and α -Fe₂O₃, facilitated by PEG, which acts as a thermal buffer, prevents agglomeration, and enhances electrolyte–electrode interactions. These findings underscore the potential of PEG-assisted BN/ α -Fe₂O₃ composites as advanced electrode materials for next-generation supercapacitors and other electrochemical storage devices.

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1 Introduction

Developing high-performance, affordable materials is crucial for advancing various fields from electronics to energy systems. To drive progress in science and technology, it is essential to discover new materials and composites and explore existing ones for novel applications. Energy storage systems are fundamental for the sustainable and efficient usage of energy resources.¹ These systems play a vital role in addressing the intermittency of renewable energy sources like wind and solar power, ensuring stability and enhancing the overall reliability of the energy infrastructure. Out of different energy systems,

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electrochemical energy storage systems (eg, batteries and supercapacitors) are integral to modern energy applications, including electric vehicles, portable electronics, and grid stabilization.² The development of sustainable and advanced materials at a scalable level using straightforward facile synthesis methods, while ensuring their stability and efficiency, remains a significant challenge. Addressing this issue is critical for creating innovative materials that can meet the growing demands of modern applications without compromising environmental sustainability or economic feasibility. Two-dimensional (2D) materials offer a rich source of functional materials. Their unique structural and electronic properties make them up-and-coming candidates for next-generation devices.³ Among these, hexagonal boron nitride (h-BN) and transition metal oxides (TMO) (eg, iron oxide, cobalt oxide, copper oxides, titanium oxides, *etc.*) stand out due to their abundance, low cost, and exceptional properties like thermal and chemical stabilities.⁴

In recent years, h-BN, a 2D material with alternating boron (B) and nitrogen atoms (N) in a honeycomb lattice arrangement, has been widely used in many energy systems as it indicates an improved efficiency of catalytic processes, particularly throughout the electrochemical reaction.⁵ The layered structure of h-BN ensures the exfoliation into nanosheets, which is a versatile building block for next-generation devices. Chao Chen *et al.* demonstrated that the integration of h-BN to form a composite structure leads to enhanced specific capacitance and stabilities.⁶ TMOs are recognized for their potential in energy storage due to their ability to undergo multiple oxidation states during electrochemical processes. Transition metal oxide composites, when hybridized with 2D materials such as boron nitride, exhibit improved capacitive performance but a suppressed faradaic signature due to enhanced conductivity and surface interactions.⁷ Iron(III) oxide (α -Fe₂O₃/hematite) is a widely studied transition metal oxide and an active material in energy storage. Among the oxides of iron, Fe₃O₄ (Magnetite) is more explored than α -Fe₂O₃. α -Fe₂O₃ is thermodynamically stable under typical surface conditions such as room temperature and atmospheric pressure.⁸ The structural instability of α -Fe₂O₃ in the case of electrochemical operations, such as electrolyte interactions, potential fluctuations, *etc.*, ultimately leads to material degradation.⁹ It is also evident that α -Fe₂O₃ exhibits certain limitations for energy application, including poor rate capability, limited surface area, cyclic stability, and structural stability.¹⁰ To overcome these issues, α -Fe₂O₃ needs to be integrated with other 2D materials like h-BN and graphene, as these materials offer intriguing properties, thus enhancing superior performances. For instance, Yang *et al.* studied the preparation of electrodes of α -Fe₂O₃/rGO nanosheet composites with a specific capacitance of 320 F g⁻¹ and a capacity retention of about 97% after 500 cycles.¹¹ Various synthesis routes, material modifications, and technologies are employed to design composite materials that could significantly impact the ionic and electronic transports in electrochemical systems.^{12,13} Polyethylene glycol (PEG) has emerged as a versatile polymer in energy domains, as it is biocompatible and also ensures high solubility in various solvents. In energy systems, PEG frequently

serves as a matrix for electrolytes, where it enhances ionic conductivity while preserving mechanical stability.¹⁴ Integrating PEG into electrochemical devices like batteries and supercapacitors enables researchers to achieve notable performance, including cyclic stability and greater efficiencies.¹⁵ Moreover, PEG-assisted composite materials support the development of stable interfaces, ensuring the durability and safety of energy systems.¹⁶ Incorporating PEG-assisted modification in BN/ α -Fe₂O₃ enhances the conductivity and electrochemical stability, thus mitigating the drawbacks faced by individual components.¹⁷ Superior thermal conductivity is observed in PEG-assisted hybrid fillers compared to individual fillers, demonstrating the potential usage of PEG in enhancing the performance of hybrid fillers.¹⁸ PEG grafted BN composites indicate significant improvement in the mechanical robustness and stability.¹⁹ Dalal Hasan *et al.* investigated on PEG/ α -Fe₂O₃ composites and ensured that incorporating PEG with α -Fe₂O₃ improves the optical and thermal properties of the composites.²⁰ Hybrid composites depict exceptional electrochemical activities.²¹ The substantial surface area of BN enables the accommodation of a larger quantity of α -Fe₂O₃, thereby increasing the available active sites for electrochemical processes.²² Doping techniques and surface functionalizations introduce more significant enhancements in the electrochemical performance of composites by efficiently improving the active sites, charge transfer mechanisms, and ion diffusion pathways.^{23,24}

Developing new strategies to enhance the electrochemical performance of boron nitride remains a crucial challenge. In this context, integrating BN and α -Fe₂O₃ has emerged as a promising approach, offering synergistic effects for improved energy storage and conversion. However, the area remains unexplored, necessitating innovative material designs. To address this gap, we present the development of hybrid nanocomposites specifically, BN/ α -Fe₂O₃ and PEG-assisted BN/ α -Fe₂O₃ composites, engineered to optimize the electrochemical properties. Additionally, the incorporation of polyethylene glycol (PEG) further enhances the dispersion and material stability of the composites. This research work investigates the synthesis and electrochemical characterization of a novel composite material: PEG-assisted BN/ α -Fe₂O₃ for supercapacitor application. The composite is prepared using the facile hydrothermal method and ultrasonication technique. The ultrasonication technique refines and homogenizes the components, enhancing their structural and electrochemical properties. PEG-BN/ α -Fe₂O₃ mixture underwent hydrothermal treatment to facilitate the formation of a composite material with superior properties. The hydrothermal method is employed as it provides a controlled environment, high-pressure, and high-temperature systems under sealed conditions, thus enabling the growth of well-defined nanocomposites with precise morphologies. This synthesis approach is advantageous in synthesizing composites, as it ensures good dispersion and interaction between the components. A comprehensive analysis, including structural, morphological, and electrochemical studies, is conducted on the prepared samples, and the obtained results are thoroughly discussed to

understand their characteristics and performance. This work represents a significant contribution to advancing functional nanomaterials for electrochemical energy storage and conversion.

2. Experimental section

2.1 Materials

Hexagonal boron nitride powder (AR grade, 99%), iron(III) oxide nanopowder (AR grade, 99%), and polyethylene glycol (PEG) with a molecular weight of 4000 g mol⁻¹ were procured from Sigma Aldrich, USA. Deionized water and dimethyl formamide (DMF) were sourced from Merck.

2.2. Synthesis of BN, PEG-BN/ α -Fe₂O₃, and BN/ α -Fe₂O₃ composites

The liquid-phase exfoliation process is introduced to exfoliate h-BN.²⁵ Bulk h-BN is dispersed in DMF and undergoes stirring for about 45 minutes, followed by an ultrasonication process to disrupt the interlayer forces between the h-BN structure, promoting the formation of individual BN layers. This mixture is then subjected to a centrifugation process (3500 rpm) to eliminate unexfoliated bulk h-BN material. The exfoliated BN was oven-dried at a temperature of 70 °C for 48 hours to remove the residual solvent. Separately, a PEG solution of a 1 : 3 volume ratio is prepared with deionized water. To this PEG solution, α -Fe₂O₃ nanoparticles are added, and the mixture undergoes vigorous stirring for about 1 hour to ensure the homogeneity of the suspended particles. Following this, previously prepared BN is added to the PEG- α -Fe₂O₃ solution and stirred for an additional 1 hour to facilitate the mixing between the components. This well-mixed suspension is then subjected to microwave-assisted ultrasonication for 2 hours. This method initiates rapid and efficient heating, further enhancing the dispersion and thus minimizing agglomerations. The final synthesis step involves the hydrothermal treatment of the solution. The mixture is then transferred to a Teflon-lined autoclave and subjected to hydrothermal conditions (120 °C, 5 hours). The elevated temperature, pressure, and time duration within the autoclave aid in the formation of well-defined nanocomposites and also improve the interaction between the subsequent components. The autoclave is then allowed to cool at room temperature. After hydrothermal treatment, the mixture is subjected to centrifugation (4000 rpm), washed with distilled water and ethanol, and oven-dried at 100 °C for 7 hours. The resultant product (PEG-BN/ α -Fe₂O₃) is carefully removed, ground into fine powders, and sealed in zip-lock covers for further analysis. A control sample (BN/ α -Fe₂O₃) is also prepared by following the same procedure, excluding the addition of PEG to the synthesis process. This control sample also provides a comparative analysis of the composites, enabling a better understanding of the composite and its performance.

2.3 Material characterization

The thermal stability of the samples is evaluated using SIINT 6300 T, a German thermogravimetric (TGA) analysis system.

The measurements were carried out under a nitrogen atmosphere. Approximately 5 mg of the sample is used for each experiment. To determine the crystalline structure and particle size of the composites, X-ray diffraction (XRD) is performed using X'Pert Pro-PANalytical. The analysis is conducted with CuK α radiation ($\lambda = 1.5406$ Å) over a 2θ range of 10–80°. The structural and morphological characteristics of the synthesized nanoparticles were examined using Field emission scanning electron microscopy (FESEM) by SIGMA HV-Carl Zeiss with Bruker Quantax 200-ZOEDS detector instrument. Raman spectroscopy is further employed to analyze the structural properties of the material. The Raman spectra were recorded using an XPLORA PLUS, Horiba, France, Raman spectrometer. Additionally, the chemical bonds and functional groups present in the sample are determined by Fourier transform Infrared spectroscopy (FTIR) with a Bruker Alpha T, Germany FTIR instrument. The electrical properties are analyzed through current–voltage (I – V) measurements and are conducted using a Keithley 2401 system. UV-visible (UV-vis) analysis technique is implemented to determine the electronic structure, bandgap energy, *etc.* of the synthesized composites with the help of Systronics, 2202, India UV-visible spectrophotometer instrument. These comprehensive characterization techniques ensured a detailed understanding of the material's structural, morphological, and functional properties, essential for optimizing its performance in various applications.

2.4. Electrochemical characterization

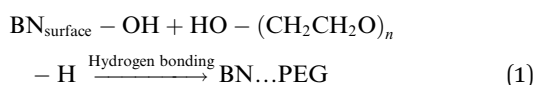
The electrochemical characteristics of the samples are analyzed using cyclic voltammetry (CV) analysis, galvanostatic charge–discharge (GCD) testing, and Electrochemical impedance spectroscopy (EIS). These experiments were conducted in a conventional three-electrode system, controlled by an Origalys OGF01A electrochemical workstation. In this setup, an Ag/AgCl electrode functions as a reference electrode, and a platinum wire is used as a counter electrode. The following methods were utilized to synthesize the working electrode. Initially, a homogeneous slurry is formulated by thoroughly mixing 80wt% of the electroactive material (PEG-BN- α -Fe₂O₃ and BN- α -Fe₂O₃), 15wt% of conductive carbon and 5wt% polyvinylidene fluoride (PVDF) in a 10% solution of *N*-methyl-2 pyrrolidone (NMP) solvent. This composition ensures optimal electron transport, mechanical stability and effective binding of the active material. 1 mg of this prepared slurry is then uniformly deposited onto a graphite sheet, serving as the current collector, *via* the drop-casting technique. The mass of active material deposited per electrode is approximately 0.80 mg. The coated electrodes were subsequently subjected to vacuum drying at 80 °C overnight to eliminate the residual solvent and enhance the adhesion between the active material and the substrate. This method also improves the uniformity and stability of the electrode surface for electrochemical characterization. All analyses were carried out at room temperature. Aqueous electrolytes are often preferred due to their availability, low toxicity, cost-effectiveness, and ease of handling. To investigate the electrochemical behavior of the synthesized samples, including their

capacitive behavior and efficiencies, a 6 M potassium hydroxide (KOH) aqueous electrolyte is utilized. The CV scans are recorded at different scan rates of 10 mV s^{-1} , 25 mV s^{-1} , 50 mV s^{-1} , and 100 mV s^{-1} consecutively. The GCD studies are also carried out at different current densities varying from 3 A g^{-1} to 6 A g^{-1} , respectively. A potential window of 0.1 to 0.5 V is maintained for all electrochemical measurements.

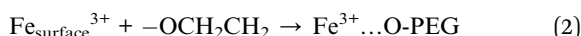
3. Results and discussion

3.1 Mechanistic insights into the formation of PEG-BN/ α - Fe_2O_3 composites

The stepwise formation of PEG-BN/ α - Fe_2O_3 and BN/ α - Fe_2O_3 hybrid composites is illustrated in Scheme 1. The exfoliation process of BN increases the surface area and exposes active sites for the attachment of α - Fe_2O_3 nanoparticles. The aqueous PEG solution stabilizes the BN/ α - Fe_2O_3 composites, preventing their agglomeration through steric hindrance. The PEG molecules adsorb onto the boron nitride (BN) surface. The α - Fe_2O_3 particles are combined with the PEG chains *via* coordination of Fe^{3+} ions. Resulting in the formation of the final hybrid composite structure. The process can be represented as:



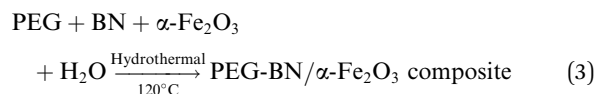
The α - Fe_2O_3 nanoparticles possess Fe^{3+} ions on their surface that coordinate with the lone pair of the ether oxygen groups present in PEG.



This indicates a strong anchor of α - Fe_2O_3 nanostructures with PEG. The well-dispersed PEG-BN/ α - Fe_2O_3 suspension undergoes hydrothermal treatment at 120°C in an autoclave, resulting in the nucleation and controlled growth of α - Fe_2O_3 nanoparticles on the BN surface. The BN nanosheets provide nucleation sites, as they possess a high surface area and chemical stability, further improving the adhesion and subsequent growth of α - Fe_2O_3 nanoparticles.²⁶ At 120°C (hydrothermal treatment), α - Fe_2O_3 epitaxially bonds on the BN

surface, resulting in controlled nucleation.²⁷ PEG is used as a stabilizing and structure-directing agent that helps in the uniform dispersion of α - Fe_2O_3 , thus favoring nucleation and controlled growth.²⁸ The electrostatic interactions facilitate the attachment of Fe^{3+} on the negatively charged BN surface.^{29,30} The zeta potential measurements of boron nitride range between -26 mV to -52 mV , respectively, providing experimental evidence on the negativity of boron nitride^{31–33}. The hydrolysis reaction in the aqueous solution leads to minimal $\text{Fe}(\text{OH})_3$ precipitation, which further undergoes dehydration to form α - Fe_2O_3 nanoparticles upon heating. It is also evident that PEG formed hydrogen bonds with both BN and α - Fe_2O_3 , enhancing adhesion and further contributing to the stability of the composite.

The overall reaction pathway for the composite synthesis is summarized as:



3.2 Morphology and structure of the composites

The X-ray diffraction analysis (XRD) is a powerful technique for characterizing the crystalline structure, phase composition, and degree of crystallinity in materials. Fig. 1 represents the XRD pattern of BN/ α - Fe_2O_3 composites and composites, respectively. The XRD peaks of α - Fe_2O_3 nanoparticles exhibit characteristic peaks at 24.20° , 33.23° , 35.70° , 49.57° , 54.20° and 64.15° following the (012), (104), (110), (024), (116), (300) crystal planes, respectively. These peaks align well with the crystal structure of

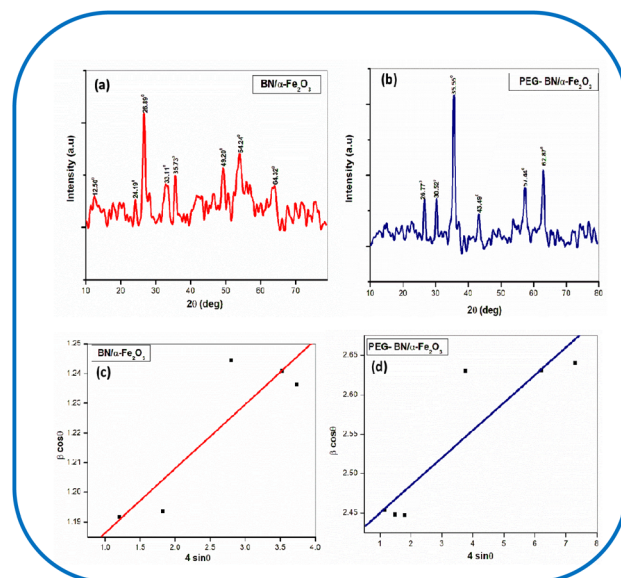
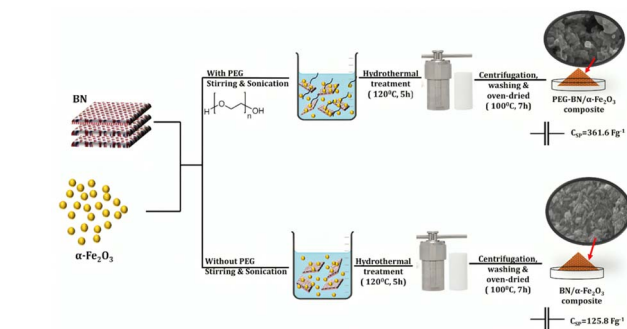


Fig. 1 XRD analysis of hydrothermal method-assisted synthesis of (a) BN/ α - Fe_2O_3 composites, (b) PEG-BN/ α - Fe_2O_3 composites. Williamson–Hall analysis plotted between $4 \sin \theta$ against $\beta \cos \theta$ of (c) BN/ α - Fe_2O_3 and (d) PEG-BN/ α - Fe_2O_3 produced using the hydrothermal synthesis route.



Scheme 1 Experimental setup for the hydrothermal synthesis of PEG-BN/ α - Fe_2O_3 and BN/ α - Fe_2O_3 hybrid composites.

α -Fe₂O₃, as referenced in the JCPDS card no 01-089-05961.¹⁵ The XRD pattern exhibits a high-intensity peak at 26.61°, consistent with the (002) *hkl* plane of boron nitride as indexed in the JCPDS database (Reference no: 34-0421).³⁴ This confirms the presence of exfoliated boron nitride within the composite material.

Fig. 1(a) illustrates the XRD analysis of BN/ α -Fe₂O₃. The characteristic peaks are obtained at 12.500, 24.190, 26.890, 33.110, 35.730, 49.610, 54.010, and 64.320 respectively. The peaks represent the characteristic diffractions corresponding to both α -Fe₂O₃ and exfoliated boron nitride phases. The XRD pattern of PEG-BN/ α -Fe₂O₃ composites depicts the peaks at 26.350, 30.520, 35.550, 43.490, 57.300, and 62.870 (Fig. 1(b)). The distinct peaks depict the crystalline structure, indicating the successful incorporation of exfoliated BN with α -Fe₂O₃ without disrupting the fundamental phase structure of either component. Minor changes in the peak intensity and slight shifts in the peaks are observed in PEG-BN/ α -Fe₂O₃ composites due to the influence of PEG in the synthesis of the composite. The PEG molecules absorb onto the BN surface, causing a disruption, thus weakening the intensity of the BN characteristic peak as compared to that of BN/ α -Fe₂O₃ composites.

The average crystallite size of the synthesized material is calculated using the Debye–Scherrer formula,

$$D = k\lambda/\beta \cos \theta \quad (4)$$

The XRD patterns of both samples exhibit broad diffraction peaks. The assessment of the crystallite size, derived from the peak broadening in the XRD patterns, suggests that the crystal domains fall within the nanoscale range. A significant reduction in the crystallite size is observed for PEG-BN/ α -Fe₂O₃ composites, 9.3 nm compared to the composite without PEG, 14.45 nm. This indicates that PEG plays a crucial role as a surfactant and dispersing agent by hindering the agglomeration of BN and α -Fe₂O₃ nanoparticles. To quantitatively distinguish the contributions of crystallite size and strain to the observed peak broadening and intensity variations, the Williamson–Hall(W–H) plot approach is employed. This method utilizes the following equation given below:

$$\beta \cos \theta = k\lambda/D + 4\varepsilon \sin \theta \quad (5)$$

where ' β ' is the full width at half maximum, FWHM value, ' θ ' is the Bragg angle, and ' ε ' is the microstrain, respectively. A linear relationship graph is obtained when $\beta \cos \theta$ is plotted against $4\varepsilon \sin \theta$. The crystallite size (D) is determined from the y -intercept ($k\lambda/t$), and the microstrain is obtained from the slope of the line. Fig. 1(c and d) shows the W–H plot of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites. The analysis mentions that the W–H plot of the samples demonstrates positive slopes. The positive slope indicates a small amount of tensile strain in the composites.³⁵ It also increases the interplanar distances, potentially increasing the conductivity of the prepared composites. Fig. 1(c), exhibits the W–H plot of BN/ α -Fe₂O₃. The W–H plot of PEG-BN/ α -Fe₂O₃ is shown in Fig. 1(d) and it reveals a lower lattice strain, as PEG aids in the reduction of internal strains within the composite material.

The dislocation density(ρ), a measure of the defects (specifically dislocations), in the crystal lattice, is calculated using the formula,

$$\rho = 1/D^2 \quad (6)$$

where ' D ' displays the corresponding crystallite size of the sample. The defect concentration in the sample is reflected by the magnitude of the dislocation density values, which are presented in Table 1. A lower dislocation density signifies an enhanced crystalline quality.³⁶ The mechanical and electronic properties of the as-synthesized composites are significantly affected by the internal strain that develops within the material. The presence of α -Fe₂O₃ nanoparticles reinforces the BN matrix, improving the overall integrity and mechanical stability of the composites.

The FTIR analysis is performed to investigate the functional groups, molecular interactions, and structural modifications of α -Fe₂O₃, BN/ α -Fe₂O₃, and PEG-BN/ α -Fe₂O₃ composites as depicted in Fig. 2(a). The Fe–O stretching vibrations in α -Fe₂O₃ are observed at 432.9 cm^{−1} and 533.6 cm^{−1}.^{37,38} The absence of significant peaks in the higher wavenumber region indicates that the sample consists primarily of α -Fe₂O₃ without any organic functional groups. A strong adsorption band at 1388.8 cm^{−1} and 1401.9 cm^{−1} in BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites corresponds to the B–N stretching vibrations, confirming the incorporation of boron nitride into the composite. A broad adsorption band at 3454.8 cm^{−1} depicts the hydrogen bonding interactions between PEG and BN/ α -Fe₂O₃ components in the composite, corresponding to hydroxyl groups (O–H stretching vibrations) in PEG. The shift in peak positions and broadening effects exhibit strong molecular interactions that enhance the composite's stability. The higher peak intensity in PEG-BN/ α -Fe₂O₃ is due to the presence of PEG polymer, as it has numerous C–H, C–O, and O–H bonds that contribute well to the FTIR spectrum consecutively.³⁹

The Raman spectra elucidate the molecular interactions, vibrational modes, and phase composition of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites and are shown in Fig. 2(c and d). The most intense peaks in α -Fe₂O₃ appear between 1320 cm^{−1} and 1350 cm^{−1} respectively. In Fig. 2(c), BN and α -Fe₂O₃ interact through surface bonding (Fe–O–B) interactions at the interface. The lower frequency peak at 387.1 cm^{−1} is attributed to the E_g symmetric vibrations of the Fe–O bonds in hematite. Compared to pure hematite, the Fe–O stretching vibrations in the composite are slightly shifted. The in-plane B–N stretching vibrations in BN correspond to the 1365 cm^{−1} adsorption peaks. A newer peak at 1609.4 cm^{−1} corresponds to the C–H bending

Table 1 Structural parameters of PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites prepared using the hydrothermal synthesis method

Sample	Average crystallite size, D (nm)	Strain, ε (%)	Dislocation density, $\rho \times 10^{14}$ lines per m ²
PEG-BN/ α -Fe ₂ O ₃	9.3	1.716	0.0115
BN/ α -Fe ₂ O ₃	14.45	2.415	0.0048

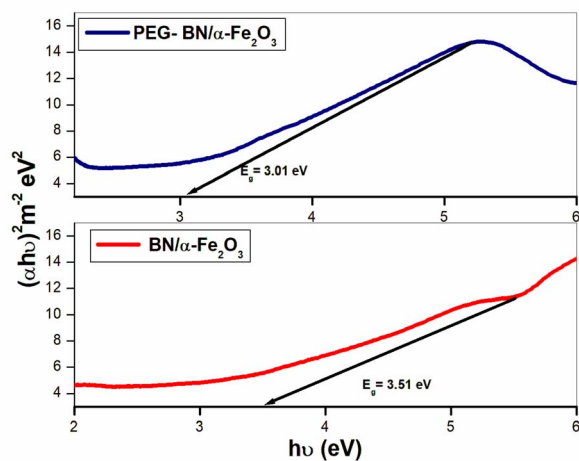


Fig. 3 Tauc plot analysis of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites synthesized via the hydrothermal method, illustrating their optical bandgap.

decomposition (Fig. 4(a)). Fig. 4(b) exhibits that α -Fe₂O₃ specifies a progressive weight loss of 10.93% over the temperature range studied, primarily due to the removal of surface-bound water and other residuals. The ODT of α -Fe₂O₃ is observed at 100.87 °C and the ODT for BN/ α -Fe₂O₃ is 114.2 °C, showing improved stability compared to that of α -Fe₂O₃ but reduced stability relative to BN (Fig. 7(c)). PEG-BN/ α -Fe₂O₃ demonstrates an ODT OF 304.98 °C as shown in Fig. 4(d). The degradation involves the breaking of C–C bonds and C–O bonds in the polymer chain, leading to the formation of volatile products and resulting in significant weight loss in the TGA curve. It is also noted that PEG-BN/ α -Fe₂O₃ composites show enhanced thermal stability out of all the composites, including BN, as PEG acts as a thermal buffer, delaying the degradation and preventing the rapid decomposition of BN and α -Fe₂O₃, thus enhancing the interfacial bonding, which stabilizes the composite structure under high-temperature conditions.

The morphology and size of the particles are analyzed using FESEM analysis as displayed in Fig. 5. The FESEM images of BN/ α -Fe₂O₃ illustrate the surface morphology at different magnifications in Fig. 5(a–c) respectively. The particles predominantly display a rod-like elongated morphology, characteristic of α -Fe₂O₃ while BN provides a supporting matrix, potentially enhancing the structural integrity and stability. The estimated particle size with an average diameter falls between 20–40 nm for PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites. The uniform

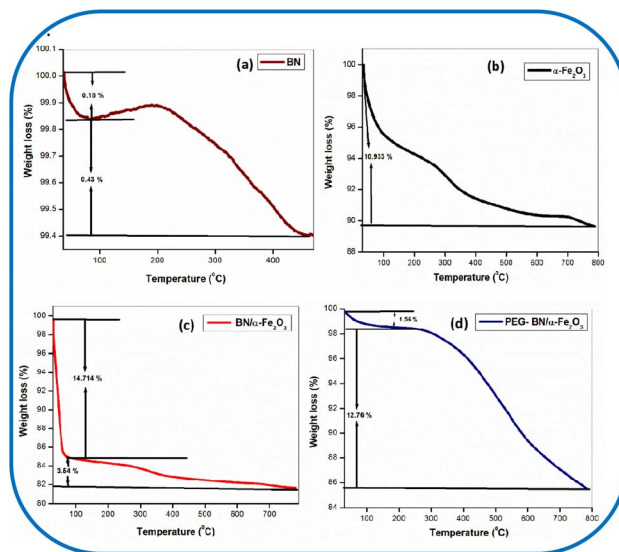


Fig. 4 TGA plot (Temperature v/s Weight loss), of (a) BN, (b) α -Fe₂O₃, (c) BN/ α -Fe₂O₃ and (d) PEG-BN/ α -Fe₂O₃ composites prepared using the hydrothermal technique.

dispersion of hematite within BN exhibits a strong interfacial interaction likely due to hydrogen bonding and electrostatic forces developed during hydrothermal synthesis. The FESEM micrographs of PEG-BN/ α -Fe₂O₃ showcase the surface morphology progressively at higher magnifications (Fig. 5(d–f)) consecutively. The FESEM analysis demonstrates that PEG-BN/ α -Fe₂O₃ significantly improves the morphology and dispersion of BN/ α -Fe₂O₃ composites. Well-dispersed spherical-shaped nanoparticles are observed in PEG-BN/ α -Fe₂O₃ composites. The polymer chains of PEG produce a steric hindrance effect, which resists the random elongation of crystals, leading to spherical structures.⁴⁶

3.3 Electrochemical analysis

The cyclic voltammetry (CV) is conducted to analyze the electrochemical behavior of PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites. The experiments were performed in 6 M KOH electrolyte, within a set voltage ranging from 0 to 0.5 V. Cyclic voltammetry curves of PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites at different scan rates are depicted in Fig. 6. The CV curves display a quasi-rectangular shape reflecting a capacitive charge storage process that derives considerable contributions from electric double-layer capacitance and pseudocapacitance. Even though the CV curves lack well-defined, sharp redox peaks,

Table 3 UV-visible and Tauc plot analysis PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites synthesized using the hydrothermal route

Samples	Absorption onset wavelength, λ_{onset} (nm)	Bandgap Energy (eV) from λ_{onset} calculations (eV)	Tauc plot, bandgap energy (eV)
BN	210–215	—	5.9 (ref. 43)
α -Fe ₂ O ₃	496–590	—	2.1–2.5 (ref. 44)
PEG-BN/ α -Fe ₂ O ₃	405.1	3.06	3.01
BN/ α -Fe ₂ O ₃	378.1	3.28	3.51

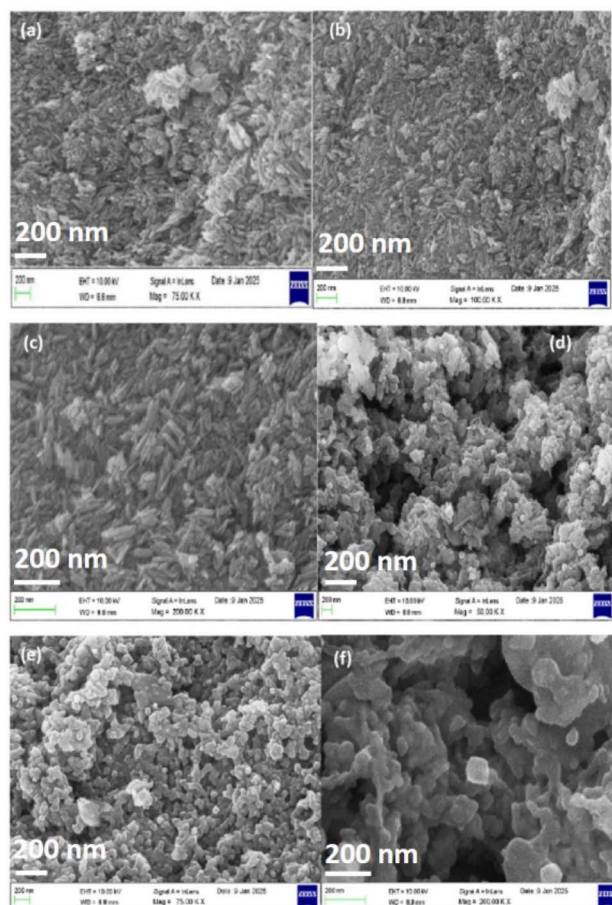


Fig. 5 SEM micrographs of (a), (b) and (c) BN/ α -Fe₂O₃ and (d), (e) and (f) PEG-BN/ α -Fe₂O₃ at different magnifications produced using the hydrothermal process.

vague broad redox features related to faradaic reactions of α -Fe₂O₃ exist below the rectangular profile, which is an indicator of surface-confined redox activity, a capacitive charge storage process. Fig. 6(a) shows the CV curve of BN/ α -Fe₂O₃ composites. BN provides surface area for the accumulation of more charge, enhancing the capacitance of the composite. The PEG-BN/ α -Fe₂O₃ exhibits superior electrochemical performance to BN/ α -Fe₂O₃ composites, as displayed in Fig. 6(b).

The larger CV area of PEG-BN/ α -Fe₂O₃ indicates higher capacitance and charge storage capabilities. PEG also improves the electrode–electrolyte interaction, leading to better stability and cyclic performance than BN/ α -Fe₂O₃ composites. PEG contains an ether functional group that configures hydrogen bonds with polar electrolytes and water molecules, thus enhancing the wettability and dispersibility of the electrode material in the electrolyte, enabling effective penetration of the electrolyte in the electrode structure.⁴⁷ This improves the ion transport and reduces interfacial resistance, ultimately resulting in enhanced electrochemical performance.⁴⁸ The redox mechanisms in this composite involve the faradaic charge storage mechanism, facilitated by α -Fe₂O₃, and are further enhanced by the synergistic effects of BN and PEG, respectively.

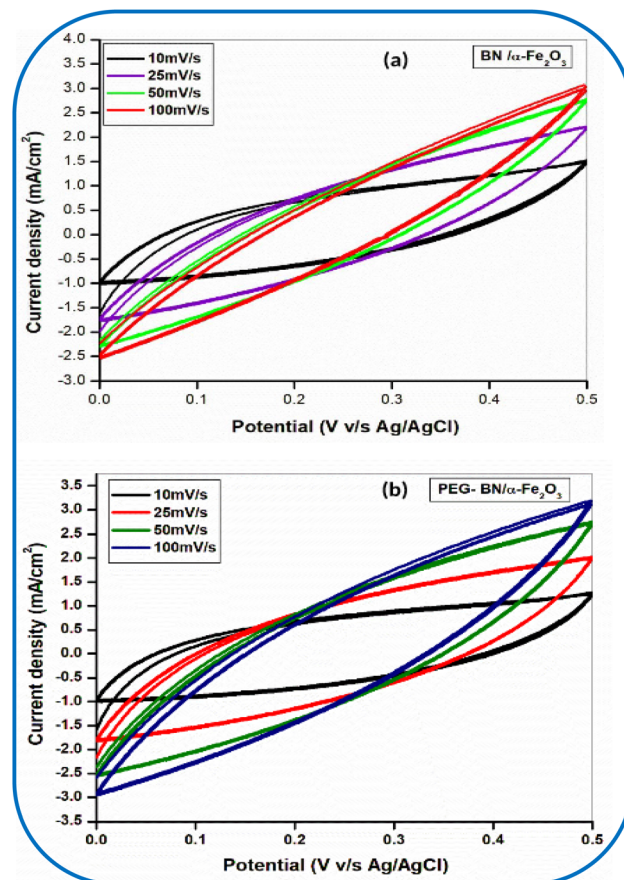
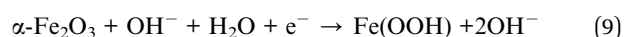


Fig. 6 Cyclic voltammetry analysis of (a) BN/ α -Fe₂O₃, (b) PEG-BN/ α -Fe₂O₃ composites produced by the hydrothermal procedure.

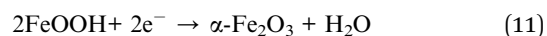
In an alkaline electrolyte (KOH), α -Fe₂O₃ undergoes two-step redox reactions:

During the charging process,



This reaction shows that α -Fe₂O₃ reacts with hydroxide ions (OH[−]) from the KOH electrolyte and water molecules to form FeOOH (iron oxyhydroxide) and release hydroxide ions.

During the discharging process,



Unlike iron oxides like Fe₃O₄, α -Fe₂O₃ is preferred due to its relative stability and well-defined electrochemical behaviors in alkaline electrolytes.⁴⁹ BN also contributes to the structural stability of the composite materials, improving the electron mobility, chemical stability, and electrical conductivity, and preventing particle agglomeration, enhancing the electrolyte accessibility to α -Fe₂O₃.⁵⁰ The minimal variation in CV curve shape with increasing sweep rate indicates the outstanding charge storage capability and improved capacitive performance of the synthesized materials.⁵¹

The galvanostatic charge–discharge analysis is shown in Fig. 7 for BN, α -Fe₂O₃, BN/ α -Fe₂O₃, and PEG-BN/ α -Fe₂O₃ composites, conducted at different current densities, 1 A g^{−1}, 3 A g^{−1}, 5 A g^{−1}, and 5.5 A g^{−1}, respectively, within a potential window between 0 to 0.5 V. The chosen current densities between 1 A g^{−1} to 5.5 A g^{−1} are common standard values in electrochemical investigations of composite electrode materials, especially for transition metal oxides and boron nitride systems, to assess rate capability and cycling stability.⁵² Fig. 7(a) depicts the GCD curve for BN and α -Fe₂O₃. The GCD curves of both BN and α -Fe₂O₃ indicate a non-faradaic capacitive behavior with low electrochemical interactions. A main indicator used to evaluate the effectiveness of a material in electrochemical applications is its capacity to store and release charge, denoted as the specific capacitance (C_{SP}). The C_{SP} value is directly related to the charge–discharge duration observed in the GCD curves. Quantitatively, specific capacitance is determined by the formula;

$$C_{SP} = i \times \Delta t / m \times \Delta V \quad (12)$$

where ' C_{SP} ' is the specific capacitance value (F g^{−1}), ' m ' is the mass of the active material (gm), ' i ' represents the discharge current (in amperes(A)), ' ΔV ' corresponds to the potential difference (in Volts(V)), ' Δt ' denotes the discharge time (in

seconds(s)) correspondingly. A larger C_{SP} value directly reflects an enhanced capacity for charge storage and delivery per unit mass of the electrode. A nearly triangular shape in the GCD curve signifies an electric double-layer capacitance (EDLC) nature.⁵³ In contrast, a deviation from the triangular shape and a plateau-like region indicates a pseudocapacitive behavior.⁵⁴ BN contributes an EDLC behavior (electrostatic interactions), and α -Fe₂O₃ provides pseudocapacitive behaviors and faradaic charge transfer *via* redox reactions, representing a hybrid supercapacitor. The C_{SP} of BN and α -Fe₂O₃ at a current density of 1 A g^{−1} is at 12.18 F g^{−1} and 17.64 F g^{−1} respectively. Fig. 7(b) and (c), present the GCD curve of BN/ α -Fe₂O₃ composites at varying current densities. The composite exhibits specific capacitance values of 125.8 F g^{−1} at 3 A g^{−1} and 90.44 F g^{−1} at 5 A g^{−1} indicating a notable improvement over the individual components. Additionally, Fig. 7(d and e) illustrates the GCD curves of PEG-BN/ α -Fe₂O₃, which demonstrate significantly enhanced capacitance, achieving 361.6 F g^{−1} at 3 A g^{−1} and 303.50 F g^{−1} at 5.5 A g^{−1}. These C_{SP} values highlight the superior electrochemical performance of the PEG-BN/ α -Fe₂O₃ composites. The GCD measurements also revealed that the PEG-BN/ α -Fe₂O₃ composite exhibits an energy density of 18.08 Wh per kg, indicating its suitability for supercapacitor applications. The interconnected network of BN and α -Fe₂O₃ also reduces charge transfer resistance, ensuring more efficient electron movement and improved electrochemical performance. The composite structure of PEG-BN/ α -Fe₂O₃ enhances mechanical stability, preventing electrode degradation during repeated charge–discharge cycles. The graphical representation of the composites is depicted in Fig. 7(f). A comparison study of the electrochemical performances of BN and α -Fe₂O₃ material-based composites is shown in the Table 4.

The higher coulombic efficiency of PEG-BN/ α -Fe₂O₃ (83%) shows lower energy losses during charge–discharge cycles, indicating reduced internal resistances and improved charge transfer kinetics.

The BN/ α -Fe₂O₃ with 73.2% efficiency experiences greater energy dissipation than the PEG-assisted composite. It is also evident that the PEG-BN/ α -Fe₂O₃ composite retains about 85% of its capacitance after 5000 cycles, whereas BN/ α -Fe₂O₃ retains only 75%, displaying better cycling stability in the PEG-assisted composite. This improved retention is attributed to PEG's role in maintaining electrode integrity and eliminating capacity loss over time.

The cyclic stability of PEG-BN/ α -Fe₂O₃ and BN/ α -Fe₂O₃ composites is evaluated by monitoring the capacity retention (%) over multiple charge–discharge cycles (5000 cycles) as shown in Fig. 8. The long-term electrochemical performance of electrode materials is crucial in determining their viability for energy storage applications. Both the composites exhibited an initial decrease in capacitance in the first few cycles due to electrolyte penetration and surface redox transitions.⁶³ The PEG-BN/ α -Fe₂O₃ composites show a slower decline in capacity retention over time indicating improved cyclic stability than BN/ α -Fe₂O₃ composites. At 4000 cycles, PEG-BN/ α -Fe₂O₃ composites retain approximately 85% of their initial capacitance, whereas BN/ α -Fe₂O₃ composites stabilize around 75%.

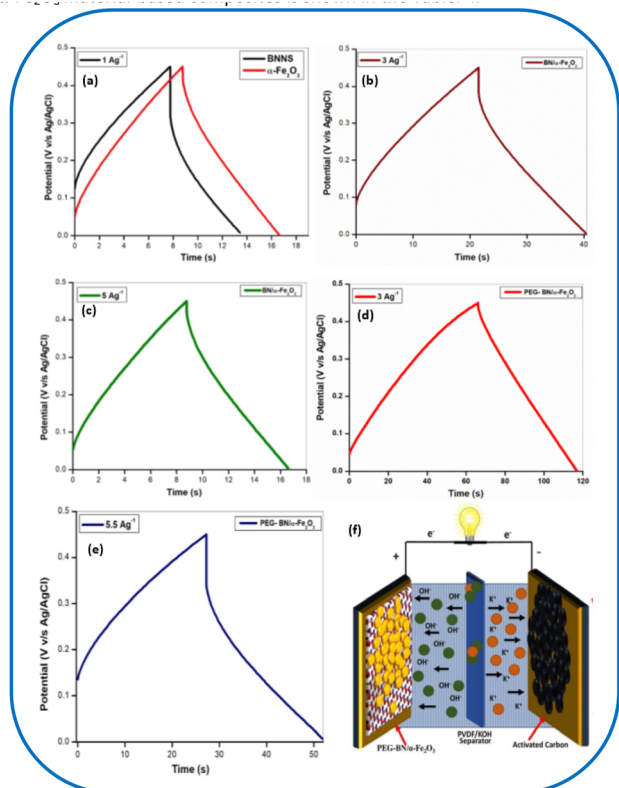
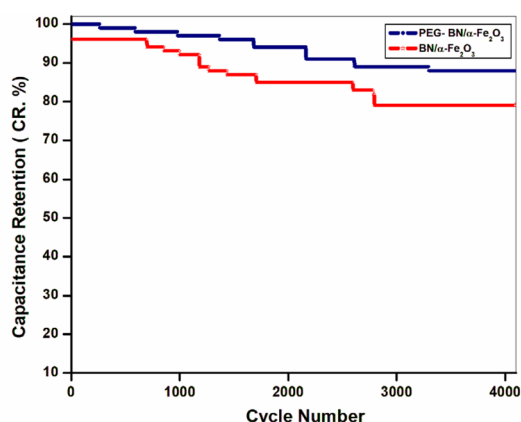


Fig. 7 Galvanostatic charge–discharge analysis at varying current densities (a) BN & α -Fe₂O₃ at 1 A g^{−1}, (b) BN/ α -Fe₂O₃ at 3 A g^{−1}, (c) BN/ α -Fe₂O₃ at 5 A g^{−1}, (d) PEG-BN/ α -Fe₂O₃ at 3 A g^{−1}, and (e) PEG-BN/ α -Fe₂O₃ composites that 5.5 A g^{−1}, (f) graphical representation of charge–discharge process of composites.

Table 4 The comparison of electrochemical performances of BN and α -Fe₂O₃ material-based hybrid composites

Composites	Synthesis routes	Electrolyte used	Voltage window (V)	Specific capacitance (F g ⁻¹)	Cyclic stability retention rate (cycles) with (ref.)
PEG-BN/ α -Fe ₂ O ₃ (as reported in this work)	Hydrothermal	6M KOH	0 to 0.5	361.6 F g ⁻¹ at 3 A g ⁻¹	85% after 5000 cycles
h-BNNS/rGO	Hydrothermal	1M Na ₂ SO ₄	-0.9 to 0.9	48.5 at 2 A g ⁻¹	107% after 5000 cycles (ref. 55)
BN/rGO/Co ₃ O ₄	Hydrothermal	1M KOH	0 to 3	145.7 at 6 A g ⁻¹	78% after 5000 cycles (ref. 56)
BN/CNT/PANI	Chemical oxidative polymerization	1M H ₂ SO ₄	0 to 2	387.5 at 1 A g ⁻¹	87% after 6000 cycles (ref. 57)
BN/Graphene/MOS ₂	Ball milling and ultrasonication	6M KOH	-0.4 to 0.5	423 at 1 A g ⁻¹	96.4% after 10 000 cycles (ref. 58)
α -Fe ₂ O ₃ /rGO	Hydrothermal	1M KOH	-1.1 to 0.3	908 at 2 A g ⁻¹	76% after 2000 cycles (ref. 59)
α -Fe ₂ O ₃ /Graphene	Ion adsorption	2M KOH	-1 to 0	264 at 2.5 A g ⁻¹	85.7% after 1000 cycles (ref. 60)
CNT/ α -Fe ₂ O ₃	Hydrothermal	2M KCl	-1 to 0	296 at 5 mV s ⁻¹	60% after 1000cycles (ref. 61)
α -Fe ₂ O ₃ /Graphene oxide/Polypyrrole	Electrodeposition	1M KCl	-0.1 to 0.7	442.7 at 1 A g ⁻¹	88% after 8000cycles (ref. 62)

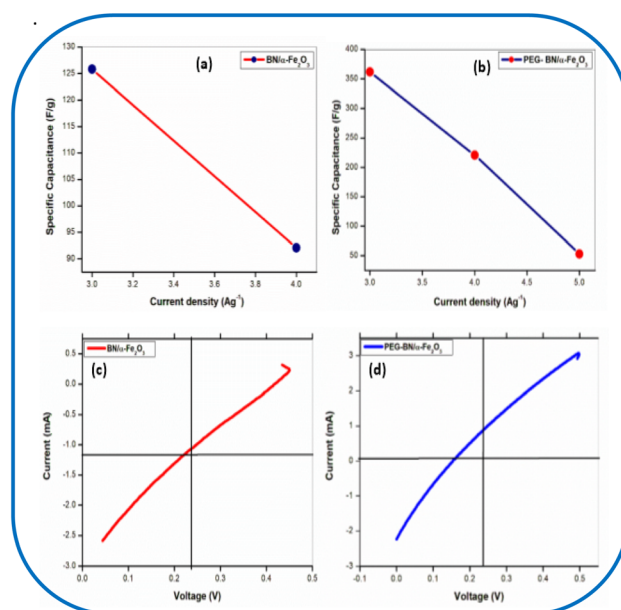
Fig. 8 Cyclic stability determination of hydrothermally prepared BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites obtained after 1st and 5000 cycles, capacitance retention (%) versus cycle number.

This enhanced stability is attributed to the improved dispersion and interfacial contact facilitated by PEG, which minimized the electrode degradation during repeated charge-discharge cycles. It is noted that during repeated charge-discharge cycles, volume expansion and contraction lead to structural stress within any composite material, further reducing the efficiency and capacitance.⁶⁴ PEG forms a flexible network within the composite, accommodating volume fluctuations, thus preventing structural breakdown. The rate capability of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites is investigated by measuring the specific capacitance at different current densities ranging from 3 to 5 A g⁻¹ as denoted in Fig. 9(a and b). Both the composites exhibited a decrease in specific capacitance with increasing current densities, a common characteristic observed in electrochemical capacitors due to kinetic limitations of ion diffusion at higher rates.⁶⁵

However, the PEG-BN/ α -Fe₂O₃ composite consistently demonstrated a higher specific capacitance value compared to the BN/ α -Fe₂O₃ composite across all current densities. At a current density of 3 A g⁻¹ PEG-BN/ α -Fe₂O₃ delivered a specific

capacitance of 361.6 F g⁻¹, while BN/ α -Fe₂O₃ exhibited a capacitance of 125.8 F g⁻¹.

A two-probe method is employed for *I-V* measurements. Solid pellets with a thickness of 1 mm and a diameter of 4 mm were prepared using a hydraulic press. The *I-V* characteristics of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ are depicted in Fig. 9(c) and (d) respectively. The *I-V* curve of α -Fe₂O₃ shows a nonlinear S-shaped behavior curve, indicating its semiconductor nature.⁶⁶ The *I-V* curve of BN appears flat, exhibiting insulating or highly resistive behavior due to its wide bandgap.⁶⁷ The PEG-BN/ α -Fe₂O₃ composites show a higher current response under the same voltage window than BN/ α -Fe₂O₃ indicating better electrical conductivity and improved charge carrier transport. BN

Fig. 9 Illustrates the variation of specific capacitance with an increase in current density for (a) BN/ α -Fe₂O₃ and (b) PEG-BN/ α -Fe₂O₃ composites produced by hydrothermal process, and the *I-V* measurements of hydrothermally synthesized (c) BN/ α -Fe₂O₃ and (d) PEG-BN/ α -Fe₂O₃ composites to analyze their electrical behavior.

with a large band gap acts as an insulator, but when combined with α -Fe₂O₃ influences the charge transport by further modifying the interface and facilitating electron flow. The presence of PEG enhances electron mobility by reducing charge trapping and improving interfacial connectivity, making it a better-conducting material.⁶⁸ Electrochemical impedance spectroscopy (EIS) measurements are performed to evaluate the charge transfer properties of BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites. The resulting Nyquist plots represent the relationship between the real Z_r and imaginary Z_i components of impedance for BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites are denoted in Fig. 10(a) and (b) respectively.

The BN/ α -Fe₂O₃ composites exhibited a large distorted semicircle followed by a sloped line at higher frequencies, suggesting the presence of both resistive and capacitive components. The PEG-BN/ α -Fe₂O₃ composites display a smaller semicircle and a more gradual slope, indicating reduced charge transfer resistance. The potential IR drop in electrochemical applications determines the voltage caused by the resistive components within the system, usually including the charge transfer, electrolyte, and contact resistance.⁶⁹ Equivalent circuits are developed to evaluate the potential of these composites for supercapacitor applications.⁴⁸ A potential drop of 82 mV and 39.70 mV, respectively, at a current density of 3 A g⁻¹ is observed on BN/ α -Fe₂O₃ and PEG-BN/ α -Fe₂O₃ composites. The substantial reduction of IR drop in PEG-BN/ α -Fe₂O₃ composite indicates a minimal energy dissipation during fast charge-discharge cycles, improving the efficiency under practical working conditions. This also corresponds to minimal heating during operation, thus contributing to the long-term stability as well as the durability of the electrode system,

addressing the key challenge faced in supercapacitor applications. In BN/ α -Fe₂O₃ composites, the equivalent circuit consists of charge transfer resistance (R_{CT}) ($R_{CT} = 27.378 \text{ m}\Omega$) in parallel with a single capacitor ($C = 103.38 \text{ F}$). This configuration indicates a fundamental charge storage mechanism with moderate capacitance. The equivalent circuit of PEG-BN/ α -Fe₂O₃ composites includes two capacitors, $C1 = 149.09 \text{ mF}$ and $C2 = 103.75 \text{ F}$, along with a charge transfer resistance ($R_{CT} = 13.234 \text{ m}\Omega$) and storage capability, further enhancing the supercapacitor performance. The additional series capacitance $C1$ in PEG-BN/ α -Fe₂O₃ composites contributes to higher charge storage efficiency and lower internal resistance, compared to BN/ α -Fe₂O₃ composites, factors crucial for long-term stability. The Bode plot represents the relation between impedance ($\log Z$) and frequency, depicting the phase angle variation as demonstrated in Fig. 10(c) and (d) consecutively. The $\log Z$ value decreases with increasing frequency, for both composites indicating a transition from resistive to capacitive behavior. The $\log Z$ value of PEG-BN/ α -Fe₂O₃ composites is higher at lower frequencies, but it decreases more rapidly, indicating enhanced conductivity. At lower frequencies, the impedance of both composites remains high, indicating limited charge transfer mobility and a significant contribution from charge transfer resistance (R_{CT}).

However, PEG-BN/ α -Fe₂O₃ composites exhibit a relatively higher impedance in this region, depicting a more effective charge accumulation mechanism due to improved interfacial interactions and electrolyte accessibility. The sharp decrease in impedance at higher frequencies is attributed to the ability of PEG to improve electrode wettability, ion diffusion, and efficient charge transport mechanisms. A higher phase angle shift also suggests that PEG-BN/ α -Fe₂O₃ composites elucidate efficient capacitive behavior making it a superior choice for supercapacitor applications.⁷⁰ These experimental results highlight that the strategic integration of BN and α -Fe₂O₃ assisted by PEG modification serves as an excellent approach for tailoring material properties and broadening the applicability of these materials in energy storage and conversion technologies.

4. Conclusion

A novel hybrid nanocomposite composed of boron nitride (BN), and hematite (α -Fe₂O₃), assisted by PEG modification, has been successfully synthesized using a combination of ultrasonication and hydrothermal treatment methods. This hybrid takes advantage of affordable materials-polyethylene glycol (PEG), boron nitride (BN), and hematite (α -Fe₂O₃)—and efficient synthesis to generate an economically affordable material system that is not compromised in its performance.^{71–76} The TGA analysis confirms the exceptional stability of the prepared nanocomposites. Notably, PEG-BN/ α -Fe₂O₃ composites demonstrate the highest thermal stability among all composites, as PEG acts as a thermal buffer, enhancing interfacial buffer bonding and delaying decomposition at elevated temperatures. The crystallite size and corresponding lattice strain are calculated from the XRD analysis, validating the

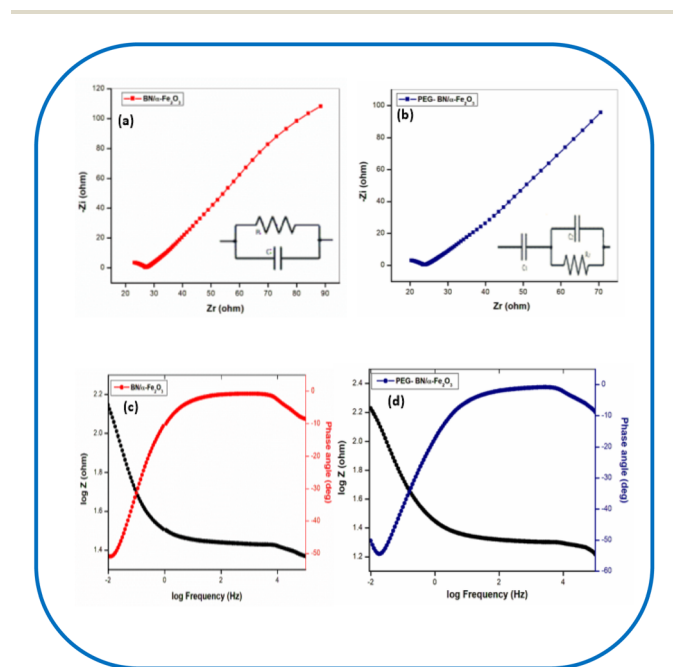


Fig. 10 Nyquist plots for (a) BN/ α -Fe₂O₃ and (b) PEG-BN/ α -Fe₂O₃ composites and bode plots for (c) BN/ α -Fe₂O₃ and (d) PEG-BN/ α -Fe₂O₃ composites synthesized via the hydrothermal method.

successful formation of the nanocomposite and providing clear evidence of its phase composition. FTIR studies substantiate the presence of Fe–O and B–N bonds in the composites, along with O–H stretching vibrations from PEG, indicating successful composite formation and strong molecular interaction. Raman spectroscopy further verified the presence of BN and α -Fe₂O₃, revealing a shift in Fe–O stretching vibrations in the composites, suggesting Fe–O–B interactions. The presence of PEG is confirmed by the observation of C–H bending vibrations. UV-visible spectroscopy affirms a reduction in the bandgap of both BN/ α -Fe₂O₃ (3.28 eV) and PEG-BN/ α -Fe₂O₃ (3.06 eV) compared to BN (5.9 eV), with a further decrease observed upon the addition of PEG. This reduction is attributed to improved charge transfer and electronic interactions facilitated by PEG, which acts as a surfactant and modifies the electric environment. Tauc plot analysis corroborated the bandgap values obtained from UV-visible analysis. The CV and GCD studies revealed that PEG-BN/ α -Fe₂O₃ composites exhibit a higher specific capacitance value (361.6 F g⁻¹ at 3 A g⁻¹) compared to BN/ α -Fe₂O₃ (125.8 F g⁻¹ at 3 A g⁻¹) and the individual components. BN provides increased surface area and improved mobility, while PEG enhances the electrode–electrolyte interaction, leading to better stability and cyclic performance. This is evident in the long-term cyclic stability tests, where PEG-BN/ α -Fe₂O₃ composites retained 85% of their capacitance after 5000 cycles, compared to 75% for BN/ α -Fe₂O₃ composites. The electrochemical analysis indicates that PEG-assisted composite significantly shows an enhancement in their charge storage capability and conductivity, making them more suitable for supercapacitor applications. The reduction in charge transfer mechanism, the presence of additional capacitance elements, and the improved phase angle shift collectively demonstrate the superior capacitive behavior of PEG-BN/ α -Fe₂O₃ composites. This work presents a promising new pathway for developing cost-effective, highly active materials with diverse functionalities. Furthermore, the successful synthesis and characterization of innovative hybrid composites (BN/ α -Fe₂O₃ & PEG-BN/ α -Fe₂O₃ composites) pave the way for advancements in material science, potentially leading to more efficient and affordable energy technologies.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of interest

There are no conflicts to declare.

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