JPSE (Journal of Physical Science and Engineering), Vol. 9, No. 2, 2024, Page 94–99.
DOI: 10.17977/um024v9i22024p94-99

JPSE
(Journal of Physical Science and Engineering)

http://journal2.um.ac.id/index.php/jpse
EISSN: 2541-2485

Synthesis and Characterization of Magnetite Nanofiber for Magnetic Field Sensor

Received
12 September 2024

Revised
17 October 2024

Accepted for Publication
19 October 2024

Published
31 October 2024

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Abstract

This study reports the performance of a magnetite sensor using magnetite nanofibers. Magnetite nanoparticles, synthesized from natural iron sand via coprecipitation and integrated into a polyvinylidene fluoride matrix by electrospinning. Structural analysis confirmed by x-ray diffraction (XRD), scanning electron microscopy (SEM), and fourier transform infrared (FTIR) characterizations. The nanofiber had an average diameter of 618.4 nm. The results of the magnetic property analysis showed that the nanofiber was superparamagnetic with a saturation magnetization value of 15.04 emu/g. The sensor exhibited good sensitivity and resolutions with a value 3.31 mV/mT and 0.015 mT with excellent stability.

Keywords: Magnetite, PVDF, Superparamagnetic, Magnetic Field Sensor

1. Introduction

The ubiquity of magnetic field sensors in scientific and industrial domains belies their persistent technological limitations [1], [2], [3] Conventional sensor architectures are fundamentally constrained by challenges in miniaturization, power consumption, cost-efficiency, and long-term operational stability [4], [5]. Furthermore, their intrinsic susceptibility to electromagnetic interference (EMI) and inadequate sensitivity to external magnetic fields often precipitate high signal-to-noise ratios. This technological gap underscores the imperative for novel materials and device architectures engineered for superior reliability.

In response, nanofiber-based membrane development has been proposed as a alternative[6], [7], [8]. This topology offers distinct advantages, particularly its very high surface area-to-volume ratio [9], which plays a key role in enhancing sensitivity to magnetic field variations. Furthermore, the structure reportedly exhibits superior mechanical properties compared to conventional ferrites and superior EMI resistance. Material selection has prioritized magnetite nanoparticles, renowned for their potent magnetic properties[10], particularly a high saturation magnetization (approx. 92–100 Am²/kg) [11]. Its implementation in sensors has resulted in a sensitivity of up to 0.51 mV/Oe. However, magnetite is prone to oxidation and particle aggregation, which degrades its performance.

To overcome these limitations, stabilization through polymer matrix compositing is used. Polyvinylidene fluoride serves as an ideal matrix, as it is a semi-crystalline ferroelectric polymer characterized by high elasticity, chemical stability, and ease of processing [12], [13], [14], [15]. The integration of magnetite nanoparticles into the polyvinylidene fluoride matrix engenders a multiferroic composite. This system synergistically couples the high magnetization of the magnetite filler with the ferroelectric response of the polyvinylidene fluoride matrix, yielding a pronounced magnetoelectric (ME) coupling[16]. The ME phenomenon, which facilitates the interconversion of magnetic and electrical energy is pivotal for high-performance sensing [17], [18]. Magnetite/polyvinylidene fluoride nanocomposite membranes thus demonstrate significant potential, exhibiting a more robust response to external magnetic fields than their monophasic counterparts. Augmenting the magnetite concentration within the matrix directly enhances the composite's total saturation magnetization, which is strongly correlated with an amplification of sensing sensitivity.

2. Methods

Magnetite nanoparticles were synthesized from natural sand via magnetic separation [19], followed by chemical co-precipitation using HCl (12 M) and NH_4OH (14 M). Magnetite/polyvinylidene fluoride nanofibers were fabricated via electrospinning. A polyvinylidene fluoride solution (15 wt% in 3:1 DMF/Acetone) was combined with varied magnetite concentrations (11,2 wt%) and dispersed using sonication. Electrospinning was conducted using a 13 kV voltage, a 13 cm collector distance, and a 5.0 mL/h flow rate onto a rotating drum collector. Sensitivity characterization evaluated the membrane's magneto-optical response; transmitted light through the membrane under a magnetic field was detected to measure voltage variations.

3. Results and Discussion

X-ray diffraction (XRD) analysis (Fig. 1) of magnetite/polyvinylidene fluoride nanofibers showed a pattern dominated by reflections at $2\theta = 20.2°, 36.1°$, which definitively confirmed the presence of the β-phase of polyvinylidene fluoride [20]. The magnetite peaks were not clearly identified in the composites, this phenomenon is due to the coexistence of the phases due to the high concentration of polyvinylidene fluoride during the electrospinning process [21]. This β-phase is greatly facilitated by the high electrical voltage during electrospinning, which enhances the ferroelectric and piezoelectric properties [22], [23], which are fundamental for sensing performance [13], [22].

Fourier Transform Infrared (FTIR) spectroscopy (400–4000 cm^{-1}) was utilized to identify the functional groups within the magnetite/polyvinylidene fluoride nanocomposite membranes (Fig. 3). The presence of magnetite nanoparticles was validated by characteristic Fe-O vibrations corresponding to octahedral (~430 cm^{-1}) and tetrahedral (~705 cm^{-1}) sites, consistent with previous reports [24]. The polyvinylidene fluoride spectrum confirmed a mixture of crystalline phases. Bands at 612 cm^{-1} and 979 cm^{-1} were attributed to the α-phase, where as a series of absorption bands at 842 cm^{-1} (CH_2), 887 cm^{-1}, 1080 cm^{-1} (CF_2), 1286 cm^{-1} (CF), and 1417 cm^{-1} (CH_2) definitively confirmed the presence of the β-phase. The CH_2 stretching vibrations were also observed as a doublet at 3020 cm^{-1} and 2978 cm^{-1}. Bands corresponding to O-H (1664 cm^{-1}, 3292 cm^{-1}) and C=O (1745 cm^{-1}, 2310 cm^{-1}) indicated the presence of adsorbed water molecules or residual solvents. Significantly, the incorporation of magnetite nanoparticles was found to promote the formation of the β-phase as ferroelectric properties of the composite.

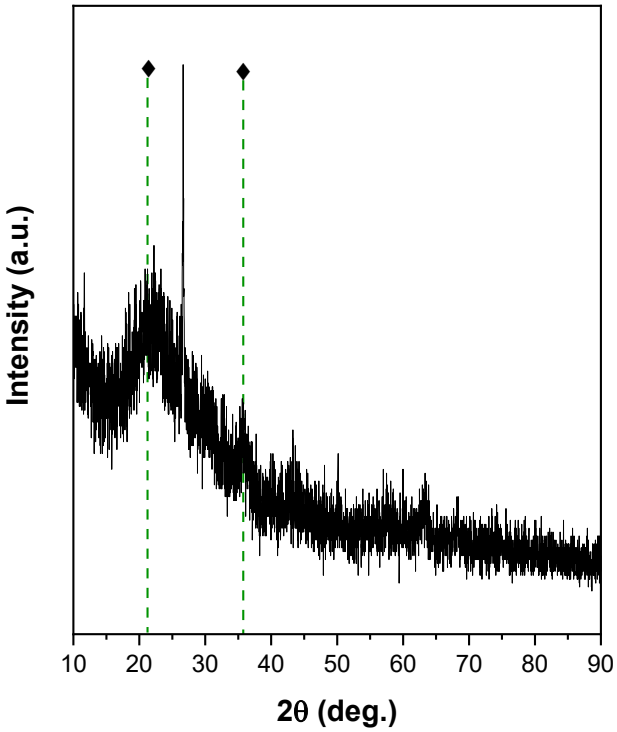

Figure 1. The XRD Pattern of Magnetite/Polyvinylidene Nanofiber.

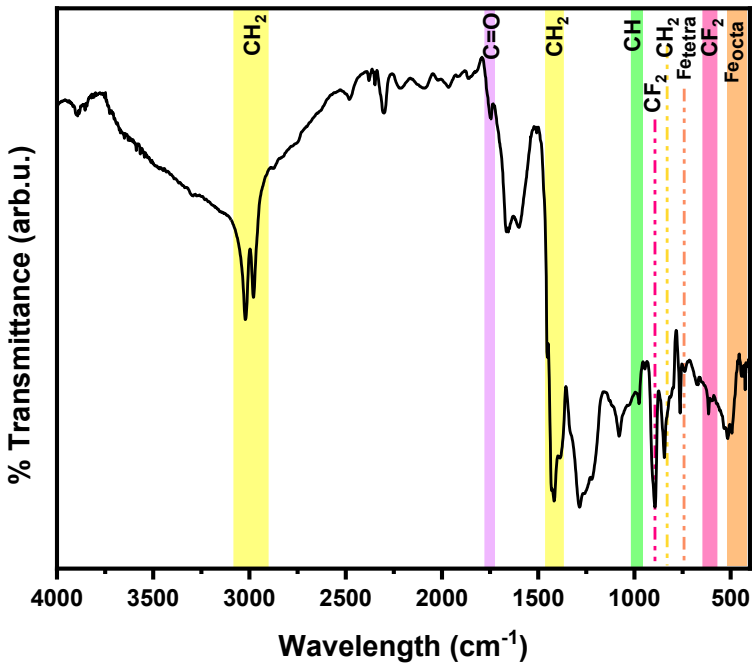

Figure 2. FTIR Spectra of Magnetite/Polyvinylidene Nanofiber.

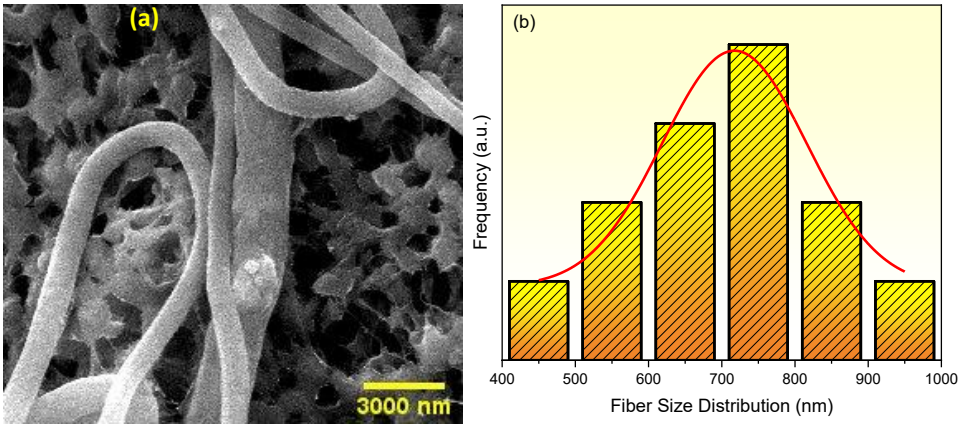

Figure 3. SEM Images (a) and Particle Size (b) of Magnetite/Polyvinylidene Nanofiber.

Scanning Electron Microscopy (SEM) analysis (Fig. 3) confirmed the fibrous morphology of the magnetite/polyvinylidene fluoride nanocomposite membrane. Magnetite resulted in morphological homogenization, characterized by the appearance of bead structures and twisting. This morphological inhomogeneity was also caused by uneven magnetite dispersion and flow instability during the electrospinning process [25]. The average fiber diameter was 618.4 ± 26.3 nm. Precise control of the diameter is essential, as larger fibers potentially contain more magnetite nanoparticles, which can amplify the sensory signal.

The magnetic properties of the magnetite/polyvinylidene fluoride nanofibers were investigated using magnetization curves measured over an external field range from -3 to 3 T (Fig. 4). The composite nanofibers exhibited a characteristic S-shaped hysteresis loop, which is a definitive feature of superparamagnetic behavior [26]. This attribution was quantitatively validated by the negligible (~0) values of the coercivity (Hc). This confirms that the material undergoes complete demagnetization upon removal of the external field. Significantly, the composite nanofibers exhibited a high saturation magnetization (Ms) value, reaching 15.04 emu/g.

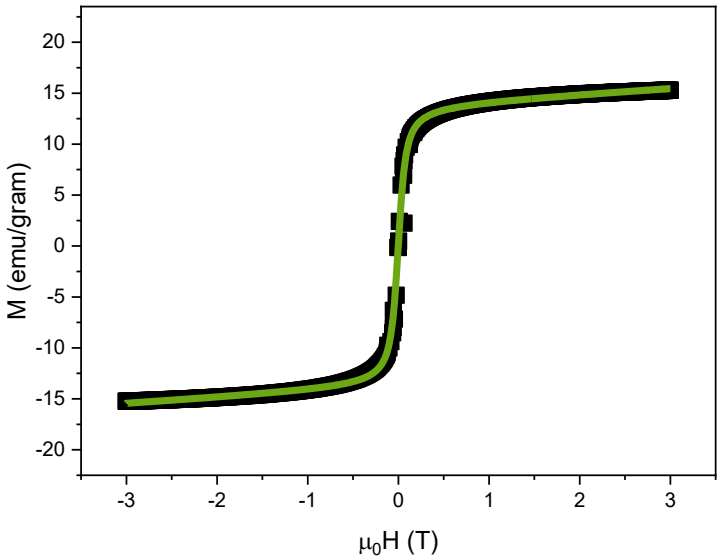

Figure 4. The Hysteresis Loop of Magnetite/Polyvinylidene Nanofibers

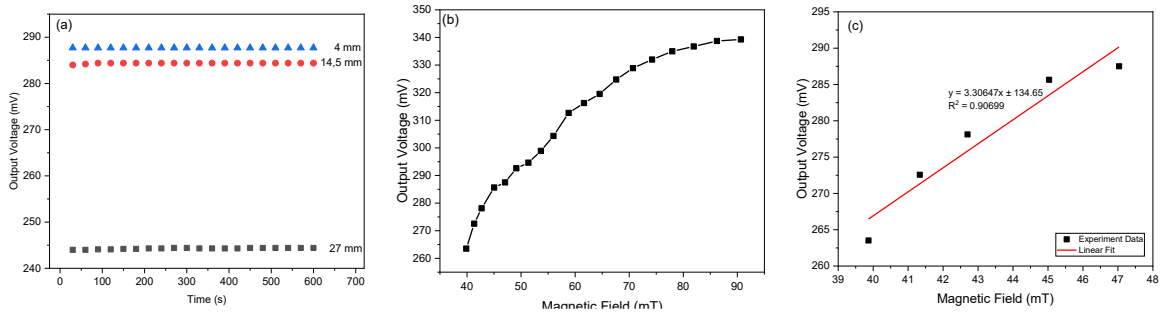

Figure 5. (a) Stability, (b) Range, (c) Sensitivity of Magnetite/Polyvinylidene Nanofibers Sensor.

The sensor performance evaluation focused on sensitivity, stability, and resolution. Analysis of the output voltage response versus the applied magnetic field (Fig.5) was used to determine the linear operating range, which was found to be ~40–91 mT.The sensitivity, determined from the gradient of the linear fit [27], was 3.31 mV/mT. This value represents a substantial improvement over previously reported literature (0.3 mV/mT), indicating a high responsiveness to small magnetic field changes. Stability testing confirmed that the sensor maintained a consistent and stable voltage output over time. The sensor's resolution, calculated as the smallest detectable field change, was 0.015 mT.

4. Conclusion

Magnetite/Polyvinylidene Nanofibers has been successfully fabricated. Morphological analysis confirmed a distinct fibrous membrane structure, characterized by an average nanofiber diameter of 618.4 ± 26.3 nm. The successful formation of the nanocomposite was validated structurally, with the PVDF matrix dominating the XRD diffraction patterns. FTIR spectroscopy confirmed the incorporation of the magnetite filler via its characteristic Fe-O octahedral and tetrahedral vibration bands. Functionally, the resulting sensor demonstrated a sensitivity of 3.31 mV/mT.

Acknowledgement

This research was financially supported by DRTPM-PTM 2024 with Grant Number 11.6.50/UN32.14.1/LT/2024.

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