Structural, Magnetic, and Optical Properties of Iron-Doped Polyvinyl Alcohol (PVA) Films for Electromagnetic Shielding and Sensor Applications

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Abstract: Pure PVA and Fe-doped composite films were synthesized using the solution casting method in the present work. Doping with iron (Fe) microparticles at weight percentages of 2% and 6%. The morphological, structural, magnetic, and optical properties of the films were investigated using X-ray diffraction (XRD), vibrating sample magnetometry (VSM), and attenuated total reflection (ATR) spectroscopy. XRD analysis revealed that the crystallite size of the iron microparticles, calculated using the Scherrer equation, was approximately 65.5 nm. Magnetic measurements showed a significant enhancement in saturation magnetization (M_s) from 1.6 x10^{-2} emu/g (2 wt% Fe) to 8.1x 10^{-2} emu/g (6 wt% Fe), along with increases in coercivity magnetization. The findings indicate that incorporating iron (Fe) particles into the PVA matrix created a new composite material with typical magnetic behavior. The observations demonstrate the material's potentiality towards sophisticated functional uses, such as sensors, electromagnetic shielding, and specialized electronic devices.

Keywords: PVA, Fe micro particles, VSM, magnetic and structural properties.

INTRODUCTION

Recent advances in nanomaterials research have highlighted their immense potential in various technological applications, including sensors, optoelectronic devices, electromagnetic shielding, and energy storage systems [1-3]. One promising strategy for developing multifunctional materials is the incorporation of nanoparticles (NPs) into polymer matrices, which enables fine-tuning of key properties such as electrical conductivity, magnetic response, optical absorption, and thermal stability [4-6]. This approach has led to the emergence of polymer nanocomposites as highly adaptable materials for next-generation devices [7-9].

Polyvinyl alcohol (PVA) is a widely used synthetic polymer known for its excellent film-forming ability, biodegradability, and compatibility with various additives. It serves as a promising matrix material for developing functional nanocomposites due to its hydroxyl-rich structure, which facilitates strong interactions with nanoparticles [10]. Incorporating magnetic nanoparticles into the PVA matrix can impart tunable magnetic properties, expanding its utility in applications such as electromagnetic shielding, magnetic sensors, and biomedical devices [11 -13]. Iron (Fe), in particular, is a suitable dopant due to its magnetic characteristics, abundance, and biocompatibility.

However, most previous studies have focused on Fe-doped systems using iron oxides (Fe_3O_4, Fe_2O_3), while fewer investigations have explored the use of pure metallic iron powder in PVA matrices. Investigating the magnetic and structural properties of Fe-doped PVA films at varying iron concentrations using a combined XRD, VSM, and ATR approach, which has not been comprehensively reported before. This research provides new insights into the correlation between iron content and magnetic performance in polymer composites, which can inform future development of materials for magnetic sensing and electromagnetic shielding. This work aims to fill this gap by synthesizing PVA films doped with varying concentrations of iron (2 wt% and 6 wt%) and analyzing their morphological, structural, and magnetic properties using XRD, VSM, and ATR techniques.

Experimental Part

Preparation of Fe‑PVA Composite Films

Polyvinyl alcohol (PVA) granules (M_w = 77,000 g/mol, Sigma-Aldrich, purity > 99%) were weighed precisely (1 g) using a digital balance and placed into a 100 mL beaker. Iron (Fe) powder (purity 99 %, particle size: 5 µm, supplied by Sigma-Aldrich was used as the doping agent). The Fe micro particles were ultrasonically dispersed in ethanol and dried at 60°C to remove surface contaminants and enhance dispersion in the polymer matrix. The method used to prepare Fe-PVA composite films is illustrated in Figure **1**.

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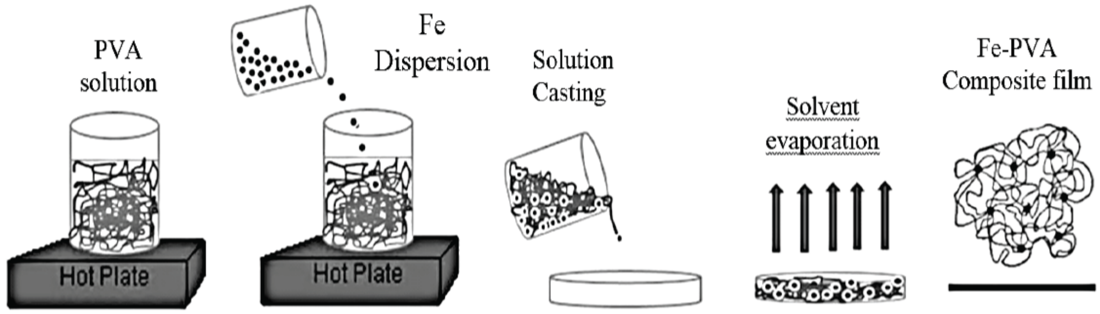

Figure 1: Schematic illustration of the production process for Fe-PVA composite films.

Indeed, Deionized water (50 mL) was added to the beaker, and the mixture was stirred with a magnetic stirrer. The solution was heated in a water bath maintained at 80°C for 2 hours to ensure complete dissolution of PVA, resulting in a clear and colorless solution. Prior to incorporation, the Fe powder was characterized by optical microscopy and XRD to confirm particle size and phase purity. The Fe powder was not subjected to any chemical pre-treatment but was ultrasonically dispersed in deionized water for 30 minutes to prevent agglomeration.

The Fe powder was then added to the PVA solution at weight percentages of 2% and 6%. The mixture was sonicated in an ultrasonic bath for 1 hour to ensure homogeneous dispersion of Fe particles in the polymer matrix. The resulting suspensions were poured onto clean glass Petri dishes and air-dried at room temperature for 24 hours to form uniform Fe-doped PVA films with a brownish appearance.

Characterization Techniques

The morphology of Polyvinyl alcohol (PVA) and its composite films were examined through optical microscopy. X-ray diffraction (XRD) measurements were performed at room temperature using an X'Pert PANalytical (D2 PHASER) diffractometer with CoKα radiation (λ = 1.78 Å), scanning 2θ angles from 5° to 80° for films and 40° to 120° for Fe powder, with a step size of 0.02°.

Magnetic measurements were conducted at room temperature using a MicroSense vibrating sample magnetometer (VSM). The magnetic field was swept from -15000 to 15000 Oe with a stepwise progression of 200 Oe.

ATR-FTIR analysis, PVA and Fe-doped PVA films were cut into small flat sections and directly placed on the diamond crystal of the ATR accessory without any additional treatment. The contact pressure was kept consistent across all samples to ensure reproducibility. Baseline correction was performed automatically using Origin 8 software to each spectrum, in order to remove background drift and enhance peak clarity. The measurements were performed using an Agilent Cary 630 spectrometer. Spectra were recorded over a wavenumber range of 400 - 4000 cm^{-1} with a resolution of 4 $cm^{-1.}$

RESULTS AND DISCUSSION

Morphology

Figure **2** illustrates the micrographic images of pristine PVA and Fe-PVA composite films. The pure PVA film exhibits a smooth and uniform surface topology, while the Fe-PVA composite films show a clearly rough surfaces morphology. This textural variation due to the incorporation of iron (Fe) microparticles into the polymer matrix. The Fe particles are mainly spherical in shape as illustrated in Figure **1**, creating a heterogeneous surface structure that is responsible for the surface roughness of these composite films.

X-Rays Diffraction Characterization

X-ray diffraction (XRD) patterns of pure PVA, Fe particles and Fe-filled PVA films are shown in Figure **3**. Analysis of the Fe particles reveals intense, well-defined crystal peaks at the following 2θ positions 52.7°, 77.4° and 99.8°corresponding the different diffraction planes (110), (200) and (211) respectively [13,17]. These characteristic peaks correspond to a cubic-centered crystal structure (BCC) with the lattice parameters a = b = c = 2.866 Å with angles α = β = γ = 90°. The average crystallite size of Fe particles was estimated using the Scherrer formula [13] applied to the (110) diffraction peak at 2θ = 52.6°, based on Co Kα radiation (λ = 0.1789 nm). The calculated size was approximately 65.5 nm.

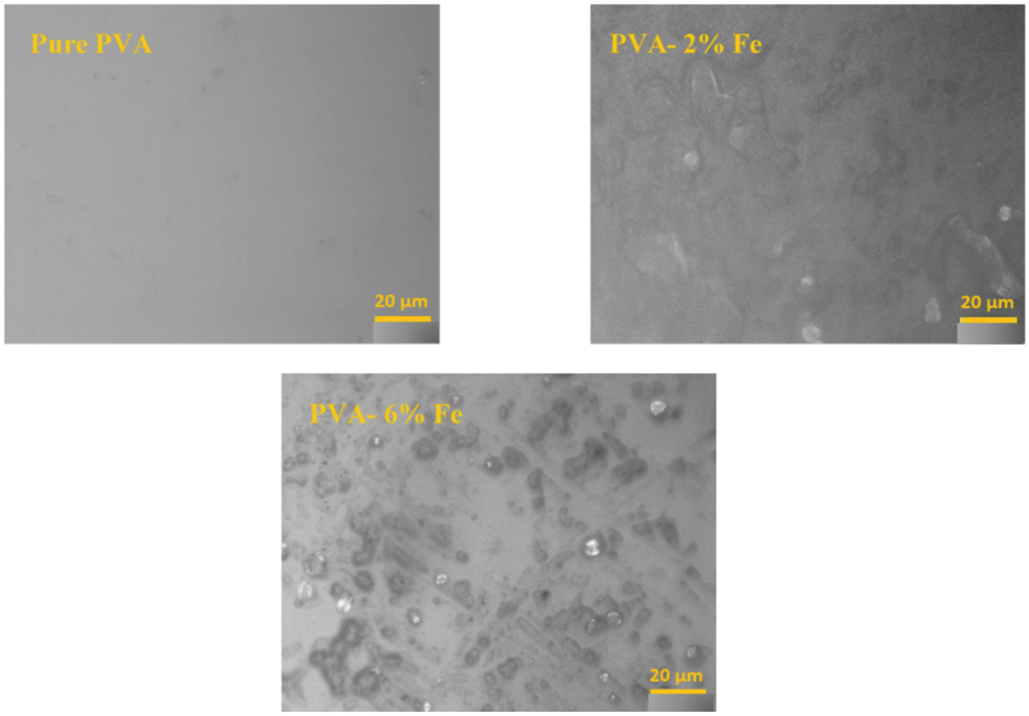

Figure 2: Surface Morphology of Pure PVA and Fe-PVA Composite Films Observed by Optical Microscopy.

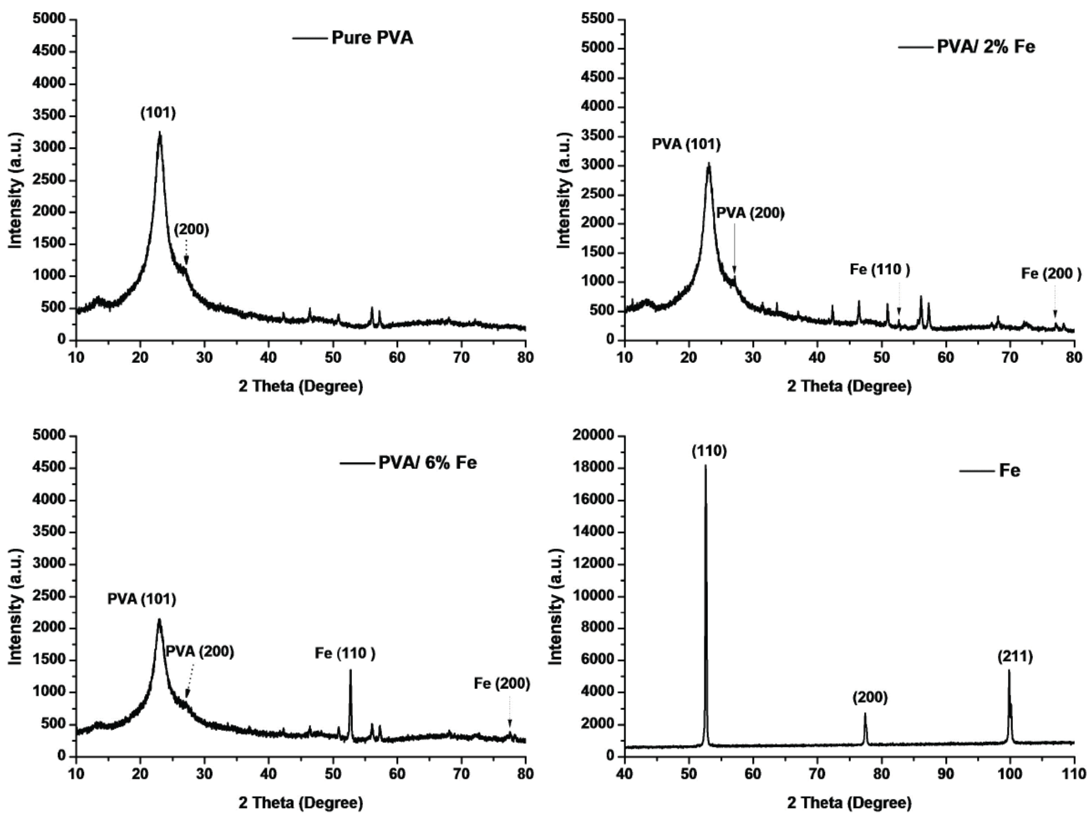

Figure 3: XRD patterns of Fe powder and Fe-PVA composite films.

The XRD curve of pure PVA film, shows a relatively broad peak centered at 22.9° corresponds to the diffraction plane (101) attributed to the crystalline plane indicating the semi crystalline nature of PVA, and also the presence of crystalline and amorphous regions [14-16]. The Fe-filled composite films show a combination of peaks at 22.9° (101) for pure PVA, and 52.7° (110) and 77.5° (200) to iron particles, confirming the presence of iron particle into the PVA matrix.

It was observed that increasing the Fe particles content in the polymer matrix resulted in a noticeable decrease in the intensity of the characteristic PVA diffraction peak. This reduction suggests a decline in the crystallinity of PVA, likely caused by the disruption of its regular molecular packing due to the presence and dispersion of iron particles within the matrix [20].

Attenuated Total Reflection (ATR) Analysis

Figure **4** depicts the recorded infrared spectroscopic spectra of pure PVA and Fe-PVA composite films. A typical -OH stretching band of PVA functional group was observed at 3257.7cm^{-1} [18]. The C-H asymmetric and symmetric stretching bands of PVA was detected at 2916.6 cm-1 and 2849.5 cm^{-1}, respectively. The Acetyl (C=C) group is located at 1641.9 cm^{-1}. The bending vibration of CH_2 is observed at 1412.6 cm^{-1}, while the wagging vibration CH_2 was detected at 1321.3 cm^{-1}. The C-O stretching band of acetyl groups was observed at 1082.7 cm^{-1}. The C-C stretching of backbone at 915 cm^{-1}, the absorbance band situated at 829 cm^{-1} is assigned to the CH_2 stretching [18].

The introduction of iron (Fe) particles into the PVA matrix, increases the peak intensities of the composite spectra. Indeed, we observed an increase in the intensity of the O-H stretching band of PVA/ 6% Fe composite film and shifted towards lower wavenumbers to 3250.4 cm^{-1}. This increase in intensity is attributed to the formation of strong hydrogen bonds between PVA hydroxyl groups (-OH) and Fe particles, as well as water molecules potentially adsorbed on the Fe surface.

The interaction between Fe nanoparticles and the PVA matrix, suggesting the formation of Fe-O-C bonds, is expected to produce a stretching vibration around 1087 cm^{-1} [20]. However, in this study, this signal is likely masked by the strong absorption band of the C-O-C stretching vibration. A similar observation was reported by Su Y. *et al*. [20]. Additionally, the weak transmittance peak around 650 cm^{-1}can be attributed to Fe-O stretching vibrations.

Vibrating Sample Magnetometer (VSM) Measurements

Magnetic properties of the Fe-PVA composite films were measured at room temperature using a vibrating sample magnetometer (VSM). Figure **5** presents the magnetization (M) versus applied magnetic field (H)

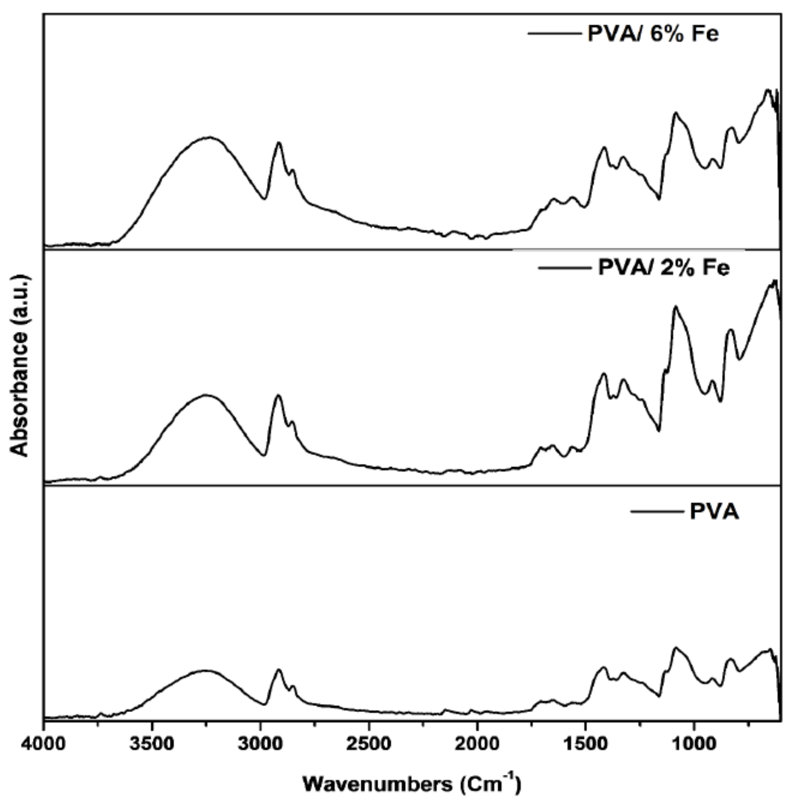

Figure 4: ATR-FTIR Spectra Showing the Chemical Structure of PVA and Fe-PVA Composite Films.

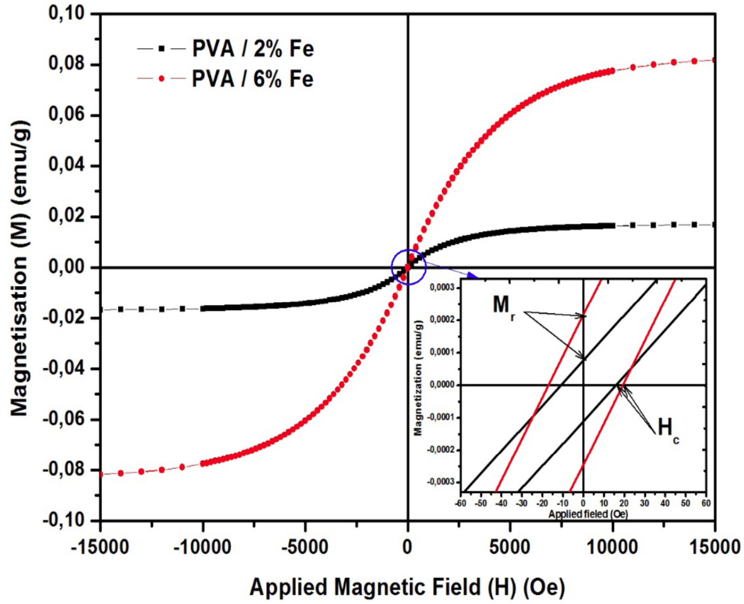

Figure 5: Magnetization Hysteresis Curves of Fe-Doped PVA-Based Composite Films.

curves for PVA films doped with 2 wt% and 6 wt% iron particles. The samples exhibit a visible magnetic property, and the magnetic hysteresis magnetization shows a clear improvement. The addition of iron particles to PVA films significantly enhances their magnetic properties. For a composite containing 2 wt% Fe, the saturation magnetization (M_s) is 1.6×10^{-2} emu/g, while increasing the Fe content to 6 wt% increases M_s to 8.1×10^{-2} emu/g. This substantial improvement reflects the strong influence of iron content on the magnetic behavior of the polymer. The enhancement can be attributed to the higher number of magnetic particles, which provide more sites for magnetic moment alignment, resulting in a stronger overall magnetic response.

The coercivity (H_c) and remanent magnetization (M_r) of the composite films are illustrated in sight of Figure **5**. The magnetic behavior of the Fe-doped PVA films was found to depend strongly on the iron content. For the 2 wt% Fe sample, the low coercivity ($H_c \approx 14.91$ Oe) and near-zero remanent magnetization ($M_r \approx 0.06 \times 10^{-3}$ emu/g) suggest a superparamagnetic-like behavior, likely due to well-dispersed Fe nanoparticles at low concentration. In contrast, the 6 wt% Fe composite exhibited increased H_c (18.15 Oe) and M_r (0.23×10^{-3} emu/g), indicating the emergence of weak ferromagnetic interactions due to higher particle density and potential agglomeration. These results demonstrate a transition from superparamagnetic to soft ferromagnetic behavior with increasing Fe content.

We can explain the results obtained at 2% and 6% Fe maybe show a gradual transition towards a regime of magnetic interactions between particles. Indeed, at 2% the iron particles are mainly isolated and only interact weakly. However, at 6%, the interparticle distance decreases, which promotes the emergence of initial collective interactions, without yet reaching a complete percolation threshold. This behavior indicates a nonlinear evolution of the magnetic properties, highlights the importance of exploring higher concentrations to observe a phase change or a significant enhancement of the composite's functional properties, and has potential applications in various fields such as electronics, sensors, and magnetic materials.

CONCLUSION

Polyvinyl alcohol (PVA) and iron-doped PVA films were synthesized successfully using the solution casting process, with structural integrity of the resulting materials ensured by X-ray diffraction (XRD) analysis. Attenuated total reflectance spectroscopy (ATR) recognized a full series of vibrational bands and functional groups characteristic of the PVA polymer, providing a clear understanding of its molecular structure. Introduction of iron (Fe) particles into the polymer matrix makes it possible to form a new class of composite material possessing unique magnetic characteristics. Such an original approach completely alters the properties of the material. The magnetic behavior of these composites shows potential applications in high-performance functional materials with adjustable properties and inexpensive fabrication, Fe- PVA composite films have high potential for largescale applications in flexible electronics, electromagnetic shielding, and low-frequency sensors.

A limitation of the present study is the lack of systematic thermal and mechanical characterization, as necessary to comprehensively evaluate the performance and stability of the PVA/Fe composite films. Future studies should include the examination of such properties in order to have a clearer picture of their viability for use in real-world applications, especially in flexible and multifunctional devices.

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