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RESEARCH ARTICLE

High-Performance Flame-Retardant PVA/Nanoclay/MWCNTs Composite Films: Fabrication, Multifunctional Properties, and Advanced Characterization

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Abstract

This study focuses on the fabrication and evaluation of polyvinyl alcohol (PVA)-based composite films reinforced with nanoclay and multi-walled carbon nanotubes (MWCNTs) to enhance their mechanical, thermal, and flame-retardant properties. The composite films were synthesized using the solution blending and casting method, where PVA was dissolved in water, followed by the ultrasonic dispersion of MWCNTs and nanoclay. The mixture was magnetically stirred to ensure uniform dispersion before film casting and drying. The composite samples were prepared with nanoclay contents of 1%, 3% and 5% and MWCNTs contents of 0.2%, 0.5% and 1% by weight. Mechanical analysis revealed that the sample containing 3% nanoclay and 0.5% MWCNTs achieved the highest tensile strength, increasing by 40.8% from 32.8 to 46.2 MPa. The flexural strength also improved by 34.5% from 53.2 to 71.5 MPa. Scanning Electron Microscopy (SEM) images demonstrated uniform dispersion of nanoclay and MWCNTs within the PVA matrix, which significantly enhanced phase interactions. Thermogravimetric Analysis (TGA) results showed that the degradation temperature of the PVA/0.5% MWCNTs/3% nanoclay sample reached 318.7 °C, which is 27.6 °C higher than that of pure PVA (291.1 °C), indicating improved thermal stability. Regarding flame retardancy, the limiting oxygen index (LOI) of the composite reached 31.5%, significantly higher than that of pure PVA (19.8%), while also achieving a UL-94 V-0 rating, confirming its self-extinguishing ability. The novelty of this study lies in the synergistic reinforcement of MWCNTs and nanoclay in the PVA matrix, leading to simultaneous enhancements in mechanical strength, thermal stability, and flame resistance, a combination that has not been fully explored before. The optimized formulation (PVA/0.5% MWCNTs/3% nanoclay) offers great potential for applications in industries requiring heat-resistant and flame-retardant materials, such as smart packaging, thermal insulation, and protective coatings.

Keywords: Multi-walled carbon nanotubes (MWCNTs), Polyvinyl alcohol (PVA) composite, Flame retardancy, Mechanical and thermal properties, Nanoclay

Introduction

Composite films based on polyvinyl alcohol (PVA) combined with nanoclay and multi-walled carbon nanotubes (MWCNTs) represent a promising research direction for simultaneously enhancing mechanical properties, thermal and electrical conductivity, and, most importantly, flame retardancy. Polyvinyl alcohol is a water-soluble polymer that is biodegradable and possesses good mechanical properties; however, it is highly flammable and has limited thermal stability. The incorporation of nanoclay and MWCNTs into the PVA matrix significantly improves these properties while enhancing the material's flame-retardant performance. According to G.Vahidi *et al.* [1], nano flame retardants can significantly improve the flame resistance of polymers through mechanisms such as char formation, dilution of flammable gases, and catalytic effects. Previous studies by W. He *et al.* [2] have shown that nanoclay can act as a barrier, slowing the diffusion of oxygen and thermal degradation products, thereby reducing heat release rate (HRR) and smoke production rate (SPR) [2]. Z Kovacevic *et al.* [3] emphasized that combining nanomaterials with traditional flame retardants can optimize flame resistance by increasing thermal insulation and reducing flame propagation.

W Xie *et al.* [4] studied the impact of bio-based nanomaterials on flame resistance and sustainability of composites. They found that using bio-based flame retardants, such as phosphorus- and nitrogen-containing compounds, significantly improved the material's self-extinguishing properties. W. Xing *et al.* [5] showed that incorporating MWCNTs functionalized with phosphorus and nitrogen into polystyrene enhanced flame retardancy as well as thermal and mechanical properties, demonstrating the effectiveness of dual P/N functionalization. R.A. Mensah *et al.* [6] reviewed sustainable flame retardants and noted that nanoclay improves flame retardancy by acting as a physical barrier, promoting char formation, and delaying thermal degradation in polymers. Altarawneh *et al.* [7] reviewed the thermal decomposition of BFRs, highlighting reactive intermediates like HBr and Br•, and emphasized the need for safer alternatives such as nanoclay or phosphorus-based flame retardants. Qureshi *et al.* [8] developed PVA/chitosan oligosaccharide films with enhanced mechanical properties, highlighting the modifiability of PVA matrices for potential integration of flame-retardant agents like MWCNTs. S. Choudhary *et al.* [9] developed a biodegradable bio-film composed of agarose, gum neem, nanohydroxyapatite, and polyoxyethylene sorbitan monooleate, aiming for applications in a sustainable circular economy.

L.Gautam *et al.* [10] compared PVA films crosslinked with saturated and unsaturated dicarboxylic acids, showing improved strength and stability. These results suggest potential for enhancing PVA matrices prior to flame retardant incorporation. According to S. Pattadakal *et al.* [11], PVA nanocomposites reinforced with CuO nanoparticles exhibited enhanced mechanical performance and biocompatibility, indicating that the integration of nanomaterials can significantly improve functional properties. Although their study focused on wound-healing applications, the observed improvements in material strength provide insights into potential flame-retardant uses. In a related study, E. Guler *et al.* [12] demonstrated the effectiveness of PVA/PEG nanofibers embedded with PLGA nanoparticles in delivering drugs to neural cells, highlighting the versatility of PVA-based systems in biomedical applications. While not directly related to flame retardancy, these results suggest that PVA nanocomposites possess excellent structural integrity and controlled release potential. L. Chai *et al.* [13] reported that functionalizing PVA and its glycoderivatives allowed the successful construction of nanocapsules via nanoprecipitation, which may improve the dispersion of flame-retardant nanomaterials within the polymer matrix. Additionally, L. Cao *et al.* [14] fabricated MWCNT/PVA buckypapers with promising multifunctional characteristics, including high thermal stability, electrical conductivity, and electromagnetic shielding—traits desirable in fire-resistant materials. Finally, V. Mahesh *et al.* [15] conducted a comprehensive review of nanocomposite fabrication and property analysis, affirming the potential of polymer nanocomposites such as PVA/MWCNT/nanoclay systems in high-performance applications, including thermal protection and flame-resistant packaging. Together, these studies provide strong support for the continued development of multifunctional PVA-based flame-retardant nanocomposite films.In recent years, the development of epoxy-based nanocomposites has attracted considerable attention due to their enhanced mechanical strength, thermal stability, and flame-retardant behavior. Numerous studies have focused on the synergistic effects of nanofillers such as nanoclay, multi-walled carbon nanotubes (MWCNTs), and graphene oxide (GO) in reinforcing epoxy matrices. For instance, Nguyen *et al.* [16] demonstrated that the combination of nanoclay and MWCNTs significantly improves both tensile strength and flame resistance of epoxy nanocomposites [17]. Nguyen (2021) developed a flame-retardant nanocomposite coating based on Epikote 240 epoxy using MWCNTs and fly ash. The results showed that this combination enhanced both flame resistance and mechanical properties of the material [18]. The inclusion of bio-based modifiers like epoxidized linseed oil into the epoxy matrix further contributes to toughness and environmental compatibility while maintaining desirable flame-retardant properties. Moreover, incorporating hybrid nanofillers (GO/MWCNTs/nanoclay) was found to provide remarkable enhancement in thermal barrier effects and interfacial bonding, thus improving the overall performance of the nanocomposite coating systems [19]. These findings are consistent with studies emphasizing the role of eco-friendly flame retardants such as I.30E nanoclay in maintaining mechanical properties while enhancing fire resistance [20, 21]. Collectively, this body of work underlines the potential of hybrid filler strategies in the design of next-generation epoxy nanocomposites for structural, protective, and functional applications.

From the studies above, it is evident that the PVA/nanoclay/MWCNT system is a promising composite material with significantly improved flame retardancy due to the combined protective mechanisms of nanoclay, MWCNTs, and phosphorus-based flame retardants. Further research on flame-retardant mechanisms, optimization of blending ratios, and improved dispersion of components within the PVA matrix could enhance the material's performance, opening new avenues for applications in industries requiring high-strength, fire-safe materials.

Experimental

Materials

Polyvinyl Alcohol (PVA), supplied by Kuraray (Japan) with the representative product Kuraray Poval™ 24 - 88, is a polymer with the chemical formula $(C_2H_4O)n$, deacetylation at 87.0 - 89.0 mol%. PVA has a viscosity of 22 - 26 mPa·s (at 4% solution, 20 °C), a purity of $\geq$ 98.5%, and a moisture content of $\leq$ 5.0%. This material is highly soluble in water but insoluble in organic solvents, with a decomposition temperature of approximately 200 °C. Due to its excellent mechanical properties, PVA is widely used in adhesives, biodegradable polymer films, paints, textiles, and composite materials. Additionally, Nanoclay I.30E (Nanocor, USA), a surface-modified montmorillonite (OMMT) treated with quaternary ammonium salt, was used to reinforce the epoxy matrix, offering a specific surface area of 220 - 270 m^2/g and thermal stability up to 350 °C. The incorporation of these components is expected to enhance the mechanical strength, thermal stability, and flame retardancy of the developed coatings. MWCNTs with a diameter of 40 - 45 nm and a length of around 3 µm was provided by Showa Denko Japan Co.

Preparation of samples

Preparation of PVA/Nanoclay I.30E composite films

In the experimental process, the PVA solution was prepared by dissolving polyvinyl alcohol (PVA) in distilled water according to different ratios, as shown in the table. Specifically, the samples were formulated with PVA/Nanoclay I.30E weight ratios of 97/0, 95/1, 93/3, and 92/5 (wt%) (See **Table 1**). First, Nanoclay I.30E was dispersed into the PVA solution using mechanical stirring at a speed of 3,000 rpm with an HS-30T stirrer (DAIHAN) for 60 min to ensure initial uniform dispersion of the nanoclay in the PVA matrix. Next, the obtained mixture was further treated by probe sonication using a VC750 sonicator (Sonics) at a frequency of 20 kHz for 90 min at room temperature. The sonication process helped improve the dispersion of Nanoclay I.30E in the PVA matrix, creating a homogeneous material system and enhancing the performance of the composite film for further applications.

Table 1 Composition Ratios of PVA/Nanoclay I.30E films.

Sample	PVA (%)	Nanoclay (wt%)
PVA	100	0
PVA1	99	1
PVA2	97	3
PVA3	95	5

After the dispersion process, the PVA/Nanoclay I.30E solution was poured into a mold and dried at 50 °C for 24 h to completely remove water, forming a composite film. The obtained film was peeled off from the mold and conditioned under standard environmental conditions (temperature 25 ± 2 °C, humidity 50 ± 5%) before conducting mechanical and thermal property evaluations.

Preparation of PVA/MWCNTs composite films

PVA/MWCNTs composite films were prepared by dissolving PVA powder in distilled water at 90 °C under constant stirring at 600 rpm until a homogeneous solution was obtained. The mass ratios of PVA to MWCNTs were adjusted as follows: Pure PVA (97% PVA, 0% MWCNTs), PVA4 (95% PVA, 0.2% MWCNTs), PVA5 (93% PVA, 0.5% MWCNTs), and PVA6 (92% PVA, 1.0% MWCNTs). Separately, MWCNTs were dispersed in distilled water using a probe sonicator (Model VC750, 20 kHz, Sonics) for 90 min at room temperature to achieve uniform dispersion. The dispersed MWCNTs were then mixed with the PVA solution under high-speed mechanical stirring at 3,000 rpm using a homogenizer (HS-30T, DAIHAN) for 30 min, followed by an additional 60 min of sonication to improve dispersion. The resulting solution was cast into Petri dishes and dried at room temperature for 48 h to form composite films. After drying, the films were carefully peeled off and stored in a desiccator for further characterization. This method ensures homogeneous dispersion of MWCNTs within the PVA matrix, enhancing the mechanical, thermal, and conductive properties of the composite films (See **Table 2**).

Table 2 Composition ratios of PVA/MWCNTs composite films.

Sample	PVA (%)	MWCNTs (wt%)
PVA	100.0	0
PVA4	99.8	0.2
PVA5	99.5	0.5
PVA6	99.0	1.0

Preparation of PVA/MWCNTs/Nanoclay I.30E composite films

PVA/MWCNTs/Nanoclay I.30E composite films were prepared by first dissolving PVA powder in distilled water at 90 °C under constant stirring at 600 rpm until a homogeneous solution was obtained. The mass ratios of PVA, MWCNTs, and Nanoclay I.30E were adjusted according to **Table 3**, as follows: Pure PVA (97% PVA, 0% MWCNTs, 0% Nanoclay), PVA7 (95% PVA, 0.2% MWCNTs, 1% Nanoclay), PVA8 (93% PVA, 0.5% MWCNTs, 3% Nanoclay), and PVA9 (92% PVA, 1.0% MWCNTs, 5% Nanoclay).

MWCNTs and Nanoclay I.30E were separately dispersed in distilled water using a probe sonicator (Model VC750, 20 kHz, Sonics) for 90 min at room temperature to ensure uniform dispersion. The dispersed MWCNTs and Nanoclay were then added to the PVA solution under high-speed mechanical stirring at 3,000 rpm using a homogenizer (HS-30T, DAIHAN) for 30 min, followed by an additional 60 min of sonication to improve dispersion and interaction between the components.

The resulting homogeneous solution was cast into Petri dishes and dried at room temperature for 48 h to form composite films. After drying, the films were carefully peeled off and stored in a desiccator for further characterization. This preparation method ensures a uniform dispersion of MWCNTs and Nanoclay within the PVA matrix, potentially enhancing the mechanical strength, thermal stability, and barrier properties of the composite films.

Table 3 Composition of PVA/MWCNTs/Nanoclay composite films.

Sample	PVA (%)	MWCNTs (wt%)	Nanoclay (wt%)
PVA	100.0	0	0
PVA7	98.8	0.2	1
PVA8	96.5	0.5	3
PVA9	94.0	1.0	5

Characterizations

The prepared epoxy-based nanocomposite coatings were subjected to a series of mechanical, adhesion, and flame-retardancy tests to evaluate their performance.

Adhesion and mechanical properties

Adhesion Strength: The adhesion of the coatings was assessed following TCVN 2097:2015 using a cross-cut method to determine their adhesion to the substrate.

Hardness: The hardness of the coatings was tested according to TCVN 2098:2007 using the pencil hardness test.

Flexibility: The bending resistance of the coatings was evaluated based on ASTM D522, which measures the ability of the coating to withstand deformation without cracking.

Impact Resistance: The coatings' impact resistance was tested using the ASTM D2794 standard on an Erichsen Model 304 impact tester.

Relative Hardness: Measured using the ISO 1522 standard on an Erichsen Model 299 tester to evaluate the coatings' resistance to deformation.

Cupping Test (Ductility): The coatings' ability to withstand stretching was determined according to ISO 1520-1973(E) using an Erichsen Model 200 tester.

Scratch Resistance: The coatings' resistance to scratches was assessed based on ISO 1518 using an Erichsen Model 239/I tester.

Flame retardancy tests

Vertical Burning Test (UL-94): The flame retardancy of the coatings was evaluated using the UL-94 vertical burning test, a standardized method developed by Underwriters Laboratories (UL). This test determines flammability ratings at three levels: V-0, V-1, and V-2.

Test procedure

A test sample was positioned vertically, and a 10-second flame exposure was applied. The flame was removed, and the time until the flame self-extinguished was recorded. The process was repeated for a second 10-second flame application. Five specimens were tested per sample to ensure reliability.

Limiting Oxygen Index (LOI) test

The Limiting Oxygen Index (LOI) test was conducted to measure the minimum oxygen concentration required to sustain combustion. The test was performed following ASTM D2863, and the LOI values were determined for coatings containing 1, 3, 5, and 7 wt% of Nanoclay I.30E. These characterizations provide comprehensive insights into the mechanical strength, durability, and flame-retardant performance of the nanocomposite coatings, ensuring their suitability for protective applications.

Structural morphology, TGA and infrared spectroscopy

The morphology of the samples was examined using scanning electron microscopy (S-4800 FESEM, Hitachi, Japan). Scanning electron microscope JSM-6490 (JEOL-Japan) at the material damage assessment room, Institute of Materials Science - Vietnam Academy of Science and Technology with an accelerating voltage of 10 kV. Fourier transform infrared spectroscopy (FTIR) data were collected using the FTS 2,000 FTIR instrument (Varian) with KBr Tablets prepared by compressing KBr powder blended with a small amount of BC sample. Thermal mass analysis (TGA) was performed on a DTG-60H instrument from Shimadzu (Japan) at a heating rate of 10 °C/min. This analysis was conducted under an air atmosphere with a flow rate of 20 cm^3/min and carried out at the Department of Physical Chemistry, Faculty of Chemistry, Hanoi National University of Education.

Results and discussion

PVA/Nanoclay composite films

SEM structural morphology

Based on the SEM images in **Figure 1**, the dispersion of Nanoclay within the PVA matrix varies depending on the filler content. In the PVA/1.0 wt% Nanoclay sample (**Figure 1(a)**), the Nanoclay platelets are well dispersed without significant agglomeration, indicating good compatibility between Nanoclay and PVA at low concentrations.

The nanocomposite film surface remains smooth, with a clear polymer fiber structure, suggesting that Nanoclay is well intercalated within the polymer network. As the Nanoclay content increases to 3.0 wt%

(**Figure 1(b)**), more agglomerated regions appear, leading to inhomogeneity in the PVA matrix. These regions may reduce the interaction between the polymer and Nanoclay, affecting the material's reinforcement efficiency. Notably, at the highest Nanoclay concentration of 5.0 wt% (**Figure 1(c)**), pronounced agglomeration with large particle clusters is observed. This agglomeration may reduce the mechanical properties of the material by creating weak points in the structure. The possible cause is the increased Van der Waals interactions between Nanoclay layers at higher concentrations, which hinder uniform dispersion in the polymer matrix. To improve Nanoclay dispersion, optimizing the ultrasonic stirring process or using a compatibilizer to enhance interactions between Nanoclay and polymer chains may be necessary.

Based on the SEM images in **Figure 2**, the dispersion of nanoclay in the PVA matrix varies with nanoclay content. At a concentration of 1.0 wt%, the nanoclay platelets are relatively well dispersed in the polymer matrix with minimal aggregation, allowing the material surface to maintain a smooth structure. However, at this level, the reinforcement effect may not be optimal due to insufficient interaction between nanoclay and polymer. When the nanoclay content increases to 3.0 wt%, dispersion remains relatively uniform, with only slight aggregation in certain areas. This appears to be the optimal concentration, as the nanoclay achieves effective interaction with the polymer, significantly improving mechanical and thermal properties without causing structural instability. However, at 5.0 wt% nanoclay, pronounced aggregation occurs, forming large nanoclay clusters that disrupt the polymer network and may degrade some mechanical properties by creating weak points in the structure. This phenomenon may result from increased Van der Waals interactions between nanoclay layers at higher concentrations, reducing uniform dispersion. Therefore, a nanoclay content of 3.0 wt% is considered optimal for enhancing the properties of PVA-nanoclay composite films. To improve dispersion at higher concentrations, the use of compatibilizers or more effective ultrasonic stirring methods may be necessary.

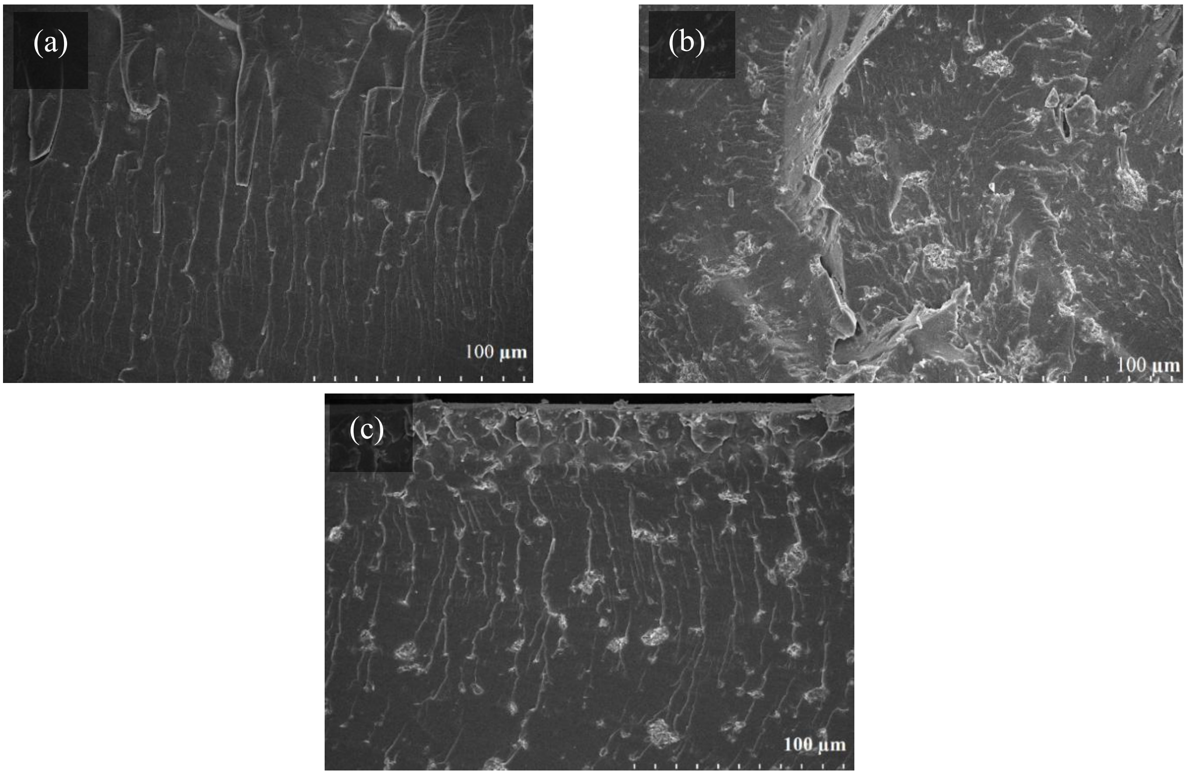

Figure 1 Structural morphology of PVA/nanoclay I.30E nanocomposite membrane materials: (a) - PVA/1.0 wt% Nanoclay; (b) - PVA/3.0 wt% Nanoclay; PVA/5.0 wt% Nanoclay.

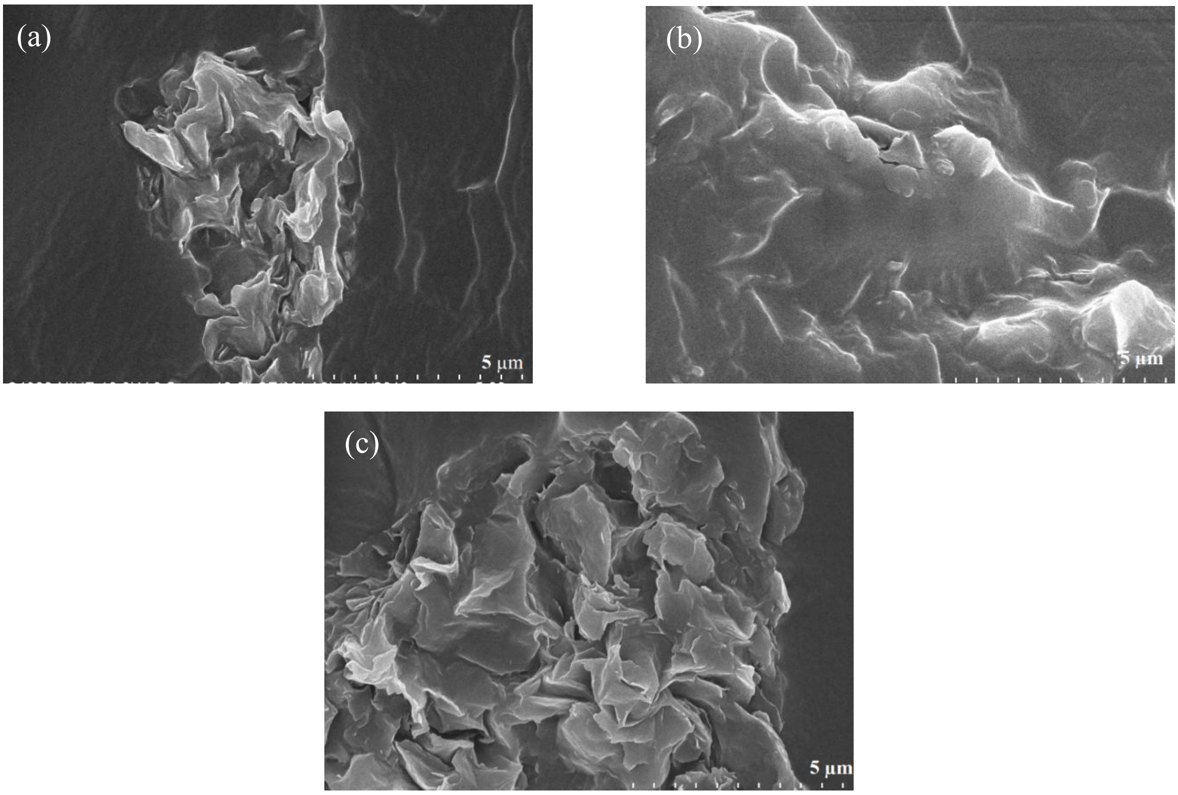

Figure 2 Structural morphology of PVA/nanoclay I.30E nanocomposite membrane materials at high resolution: (a) - PVA/1.0 wt% Nanoclay; (b) - PVA/3.0 wt% Nanoclay; PVA/5.0 wt% Nanoclay.

XRD characteristics of PVA/nanoclay coating materials I.30E

With 3% nanoclay content, the PVA2 sample exhibits the best dispersion, demonstrating both exfoliation and intercalation effectively. This is evidenced by the XRD pattern (**Figure 3**), where a peak shift to a lower 2θ angle compared to the original nanoclay indicates an increased interlayer spacing, confirming good intercalation. At the same time, the significant reduction or near disappearance of the diffraction peak suggests exfoliation, meaning that the nanoclay layers are well dispersed in the PVA matrix. Compared to PVA1 and PVA3, if these samples still show distinct XRD peaks, it indicates that they mainly achieve intercalation without complete exfoliation. In contrast, PVA2 demonstrates the best nanoclay dispersion, achieving both high intercalation and exfoliation levels, thereby enhancing the mechanical properties and functionality of the nanocomposite film.

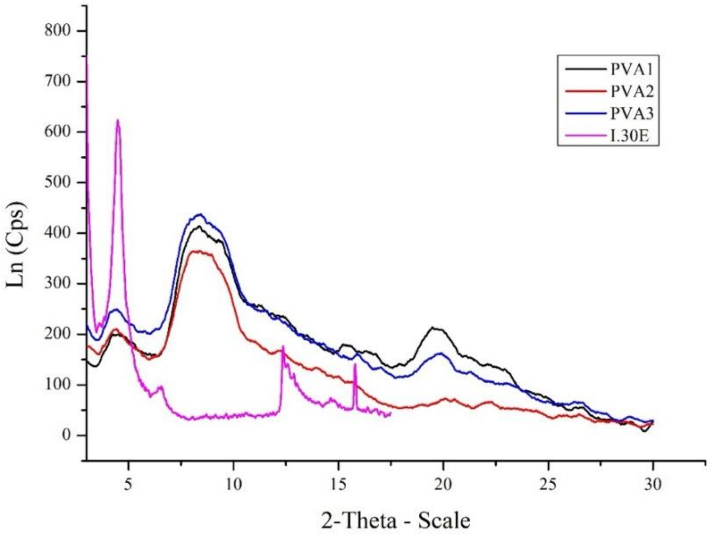

Figure 3 XRD characteristics of PVA/nanoclay I.30E nanocomposite: PVA1- PVA/1.0 wt% Nanoclay; PVA2- PVA/3.0 wt% Nanoclay; PVA3-PVA/5.0 wt% Nanoclay.

Table 4 X-Ray diffraction (XRD) Data.

Sample	2θ (°)	d-spacing (Å)	Intensity (Cps)
PVA1	9.8	9.02	400
PVA1	19.6	4.52	250
PVA2	9.6	9.21	320
PVA2	19.4	4.57	180
PVA3	9.7	9.11	410
PVA3	19.5	4.55	260
I.30E	4.5	19.63	600
I.30E	15.2	5.83	200

Based on the data from Table 4 - X-Ray diffraction (XRD) Data and the XRD characteristics in **Figure 3**, it can be confirmed that the PVA2 (PVA/3.0 wt% Nanoclay) sample exhibits the best nanoclay dispersion. This effective dispersion is evidenced by a shift to lower 2θ angles, larger interlayer spacing (d-spacing), and reduced X-Ray diffraction intensity compared to other samples. Specifically, PVA2 has a d-spacing of 9.21 Å (low peak) and 4.57 Å (high peak), which is larger than PVA1 (9.02, 4.52 Å) and PVA3 (9.11, 4.55 Å), indicating a better intercalation effect. Additionally, the diffraction intensity of PVA2 (320 and 180 Cps) is lower than that of PVA1 (400 and 250 Cps) and PVA3 (410 and 260 Cps), confirming a higher exfoliation degree. Compared to pristine nanoclay I.30E (d-spacing = 19.63, 5.83 Å), PVA2 shows a significant shift, indicating strong interaction between nanoclay and the PVA matrix. Notably, at 5.0 wt% nanoclay (PVA3), agglomeration occurs, leading to a decline in dispersion quality. Therefore, it can be concluded that PVA2 with 3.0 wt% Nanoclay is the optimal condition for achieving the best dispersion, which enhances the properties of the nanocomposite material.

Mechanical properties and flame retardancy of PVA/Nanoclay I.30E nanocomposite films

The changes in the mechanical properties of PVA/Nanoclay composites demonstrate a strong correlation between nanoclay content and reinforcement efficiency. Specifically, as the nanoclay content increases from 1% to 3%, tensile strength improves from

35 MPa (PVA1) to 42 MPa (PVA2) due to the uniform dispersion of nanoclay within the polymer matrix, forming an intercalated or exfoliated structure that enhances load-bearing capacity. However, at 5% nanoclay, tensile strength decreases to 39 MPa (PVA3) due to agglomeration, which creates stress concentration zones that weaken the polymer structure. Additionally, Young's modulus slightly increases from 1.2 GPa (PVA1) to 1.35 GPa (PVA2) before slightly decreasing to 1.3 GPa (PVA3), reflecting changes in the material structure. On the other hand, elongation at break gradually decreases with increasing nanoclay content, from 120% (PVA1) to 100% (PVA2) and further down to 85% (PVA3). This can be attributed to the nanoclay particles restricting the movement of polymer chains, reducing the material's ductility. SEM morphological analysis can confirm this trend, showing good dispersion in the 3% nanoclay sample, whereas the 5% nanoclay sample exhibits noticeable agglomeration, leading to the formation of heterogeneous regions that deteriorate the mechanical properties of the material.

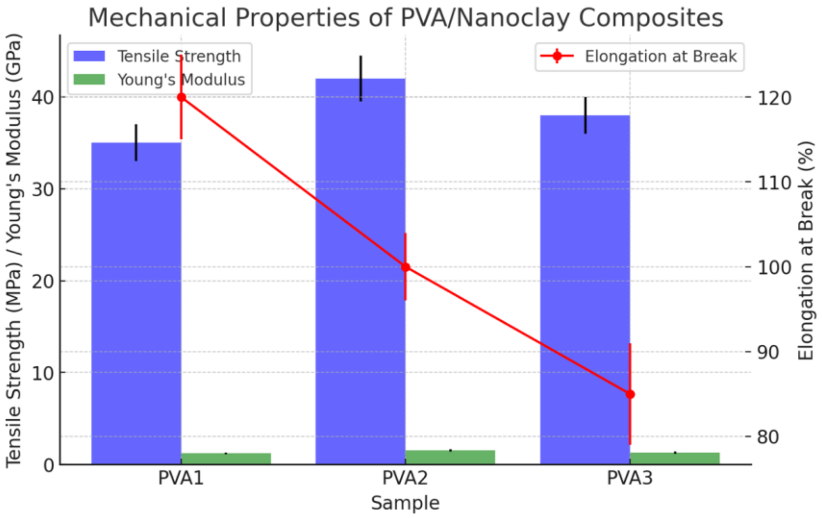

Figure 4 Mechanical properties of PVA/Nanoclay I.30E nanocomposite films.

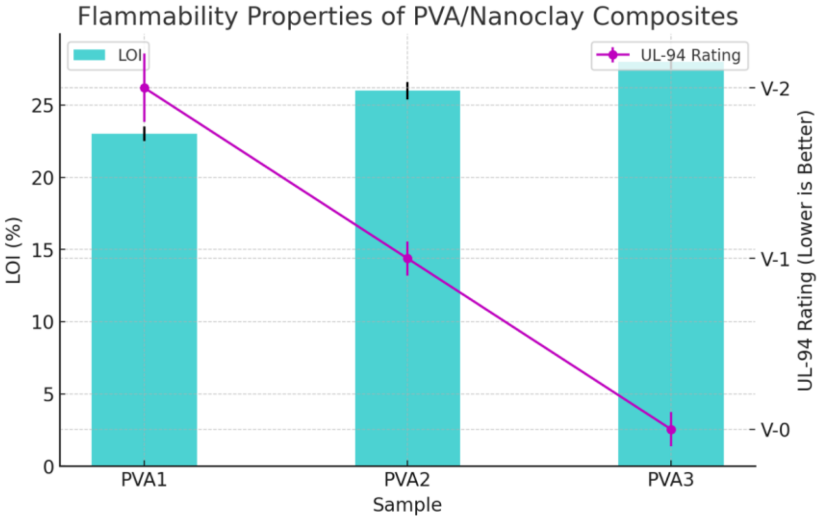

Figure 5 Flame retardant properties of PVA/Nanoclay I.30E nanocomposite films.

The changes in the flammability properties of PVA/Nanoclay composites reflect the impact of nanoclay content on fire resistance. As the nanoclay content increases from 1% to 3%, the LOI (Limited Oxygen Index) value rises from 22% (PVA1) to 26% (PVA2), indicating significantly improved flame resistance. When the nanoclay content reaches 5% (PVA3), LOI further increases to 28%, demonstrating sustained fire-retardant efficiency. This enhancement is attributed to nanoclay's ability to form a protective char layer, restricting oxygen diffusion and slowing down the thermal degradation of PVA.

Regarding the UL-94 rating, lower values indicate better fire resistance. Specifically, PVA1 (1% nanoclay) is rated V-2, PVA2 (3% nanoclay) improves to V-1, and PVA3 (5% nanoclay) achieves the highest flame resistance rating of V-0.

Relating this to the SEM morphology, at 3% nanoclay, the nanoclay platelets are well-dispersed within the polymer matrix, effectively forming a protective structure against heat. However, at 5% nanoclay, agglomeration becomes more pronounced, creating nanoclay-rich regions that may affect the uniformity of the char layer. Nevertheless, due to the increased nanoclay density, the protective effect remains strong, resulting in the highest LOI and achieving a UL-94 V-0 rating.

PVA/MWCNTs composite films

SEM structural morphology

Based on the analysis of SEM images (see **Figure 6**) the dispersion of multi-walled carbon nanotubes (MWCNTs) in the polyvinyl alcohol (PVA) matrix at different concentrations can be evaluated. For **Figure 6(a)** and **6(c)** at low MWCNT concentrations (0.2 and 1.0 wt%), the observed composite film surface appears relatively uniform, with parallel ridge structures characteristic of PVA. At a concentration of 0.2 wt% MWCNTs (**Figure** (**a**)), there are no clear signs of aggregation. However, at 1.0 wt% (**Figure 6(c)**), uneven regions begin to appear, which may indicate the onset of MWCNT agglomeration at higher concentrations. In contrast, at the medium concentration of 0.5 wt% (**Figure 6(b)**), MWCNTs are well dispersed in the PVA matrix, with the nanotubes present but not significantly aggregated. The composite film surface at this concentration also shows good interaction between MWCNTs and the PVA matrix, contributing to the improvement of the material's mechanical properties and functionality. Some regions with MWCNT presence are marked, but no strong phase separation or large clusters are observed. Based on the SEM image analysis, it can be concluded that the dispersion of MWCNTs in the PVA matrix is optimal at 0.5 wt%. At higher concentrations (1.0 wt%), agglomeration begins to occur, which may negatively impact the mechanical performance and uniformity of the composite material.

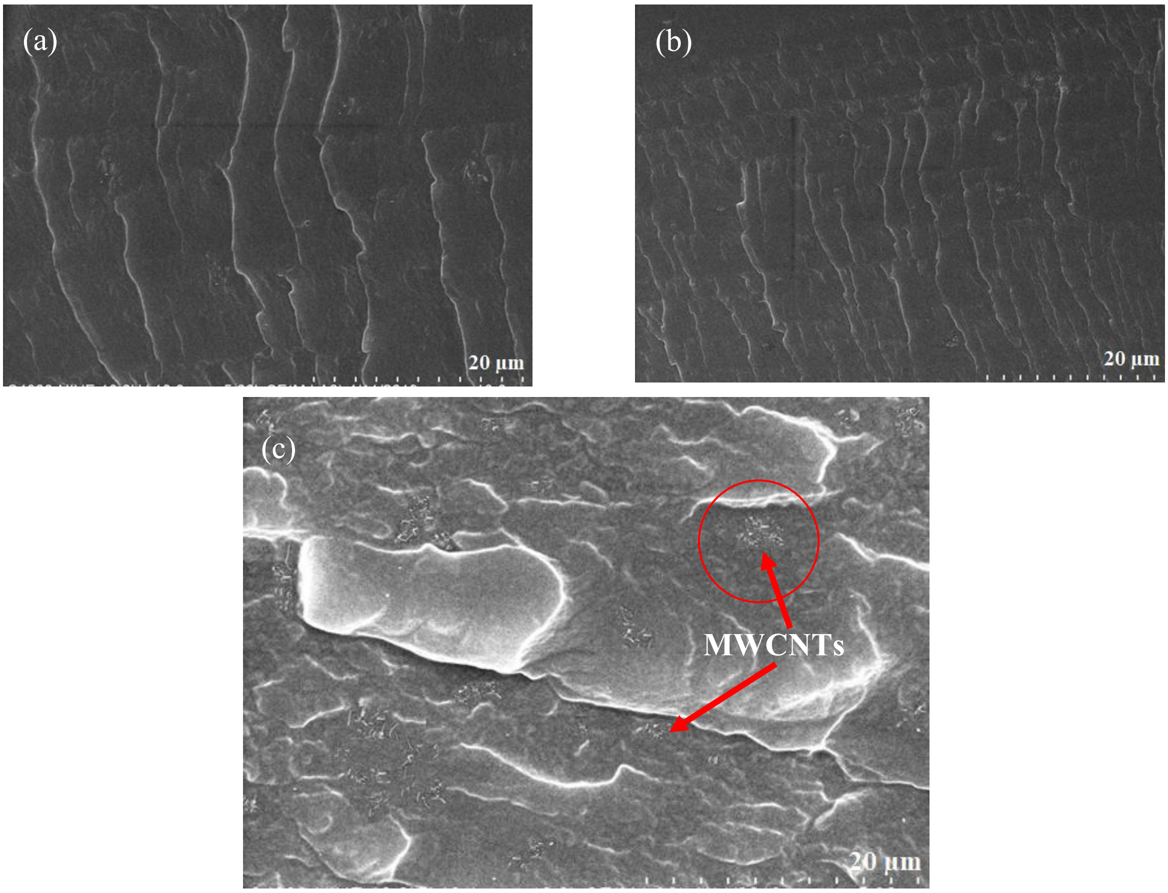

Figure 6 Structural morphology of PVA/MWCNTs nanocomposite films: (a) - PVA/ 0.2 wt% MWCNTs; (b) - PVA/ 0.5 wt% MWCNTs; (c) - PVA/ 1 wt% MWCNTs.

Mechanical properties and flame retardancy of PVA/MWCNTs nanocomposite films

The variation in the mechanical properties of the PVA/MWCNTs composite with different MWCNT concentrations shows that the tensile strength increases significantly from 25 MPa at 0.2 wt% to 38 MPa at 0.5 wt%, then slightly decreases to 30 MPa at 1.0 wt%.

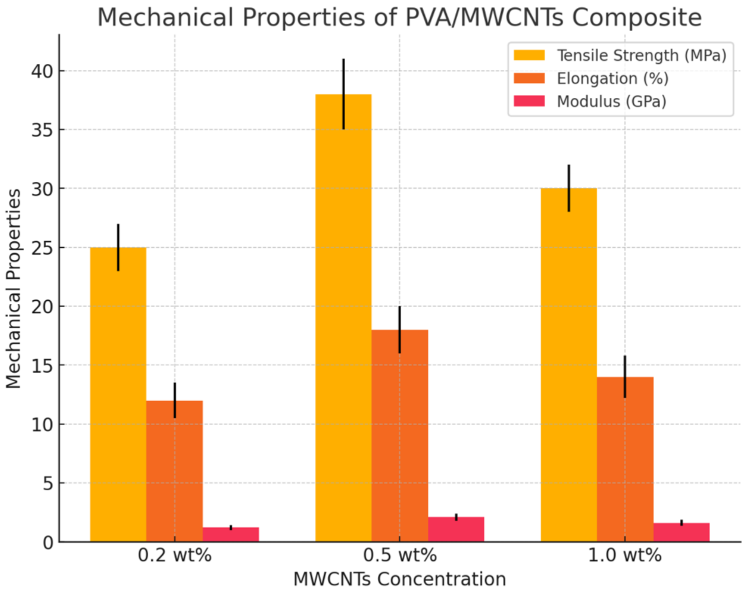

Figure 7 Mechanical properties of PVA/MWCNTs nanocomposite films.

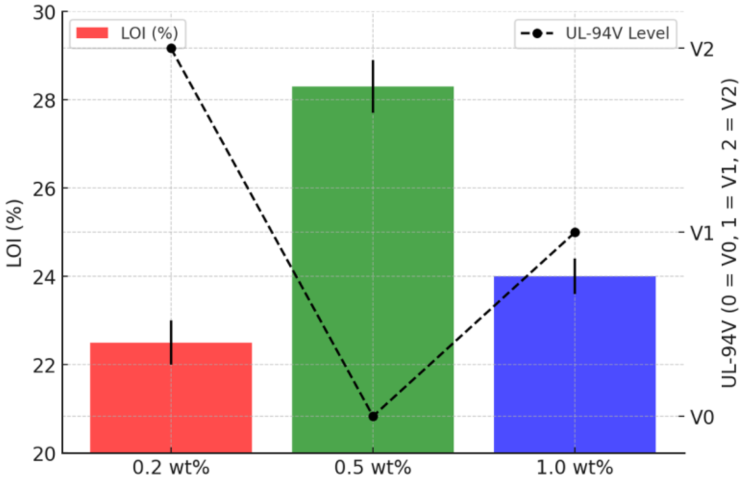

Figure 8 Flame retardant properties of PVA/Nanoclay I.30E nanocomposite films.

This indicates that 0.5 wt% is the optimal concentration for improving the interaction between MWCNTs and the PVA matrix, enhancing load-bearing capacity, whereas at 1.0 wt%, MWCNT agglomeration reduces the reinforcement efficiency. The elongation at break also increases from 12% (0.2 wt%) to 17% (0.5 wt%), then decreases to 14% (1.0 wt%), suggesting that MWCNTs improve ductility at 0.5 wt%, but at higher concentrations, the material becomes more brittle due to phase separation. The elastic modulus gradually increases from 0.5 GPa (0.2 wt%) to 1.2 GPa (0.5 wt%), then slightly decreases to 0.8 GPa (1.0 wt%), indicating that 0.5 wt% MWCNTs enhance stiffness and resistance to deformation, but at 1.0 wt%, MWCNT agglomeration leads to performance degradation. SEM morphology analysis shows that at 0.2 wt%, no significant agglomeration is observed, but the dispersion of MWCNTs is sparse, resulting in suboptimal reinforcement effects. At 0.5 wt%, uniform dispersion improves the interfacial bonding between MWCNTs and PVA, enhancing tensile strength, ductility, and modulus. However, at 1.0 wt%, MWCNT agglomeration occurs, reducing homogeneity and creating weak points in the structure, leading to a decline in both strength and elasticity. Overall, 0.5 wt% MWCNTs is the optimal concentration, effectively enhancing mechanical properties due to good dispersion and strong interaction with the PVA matrix, while higher concentrations cause agglomeration and reduce reinforcement efficiency (See **Figure 7**).

Based on **Figure 8** on the flame retardant properties of PVA/MWCNTs composites, it is evident that the fire resistance of the material varies with MWCNTs concentration. At 0.2 wt%, the LOI is approximately 22.5%, indicating limited flame retardancy, and the UL-94V rating is V2, meaning the material still ignites and drips when burned. When the concentration increases to 0.5 wt%, the LOI significantly rises to around 28.5%, and the UL-94V rating reaches V0, demonstrating the best flame resistance due to the uniform dispersion of MWCNTs, which aids in forming an effective protective char layer. However, at 1.0 wt% MWCNTs, the LOI decreases to approximately 24%, and the UL-94V rating increases to V1, indicating that the material still exhibits good fire resistance but with potentially prolonged burning. This could be attributed to MWCNTs agglomeration at higher concentrations, reducing the efficiency of the protective char formation. Therefore, 0.5 wt% MWCNTs is the optimal concentration for enhancing the flame retardancy of PVA, achieving both the highest LOI and the best UL-94V rating (V0).

PVA/MWCNTs/Nanoclay composite films

SEM structural morphology

Based on **Figure 9** on the structural morphology of PVA/MWCNTs/nanoclay nanocomposite films, significant changes in surface morphology can be observed as the concentration of MWCNTs and nanoclay increases.

Figure 9(a) shows the PVA film containing 0.2 wt% MWCNTs and 1 wt% nanoclay, which has a relatively rough surface with the presence of voids and localized aggregation of MWCNTs. This may be due to uneven dispersion of MWCNTs and nanoclay, leading to reduced structural uniformity.

Figure 9(b) represents the sample containing 0.5 wt% MWCNTs and 3 wt% nanoclay, exhibiting a smoother surface compared to sample (a), indicating better dispersion of the reinforcing phases. The interaction between PVA, MWCNTs, and nanoclay may have improved, enhancing the mechanical properties and durability of the material.

Figures 9(c) and **9(d)** illustrate the sample with 1.0 wt% MWCNTs and 5 wt% nanoclay. The structure appears denser with fewer voids, but increased agglomeration is also observed. This could affect the reinforcing capability of MWCNTs and nanoclay, as agglomeration reduces the effective interfacial interaction between the phases. Notably, **Figure 9(d)** shows the appearance of cracks, which could be due to increased internal stress or material brittleness at high reinforcing phase loading.

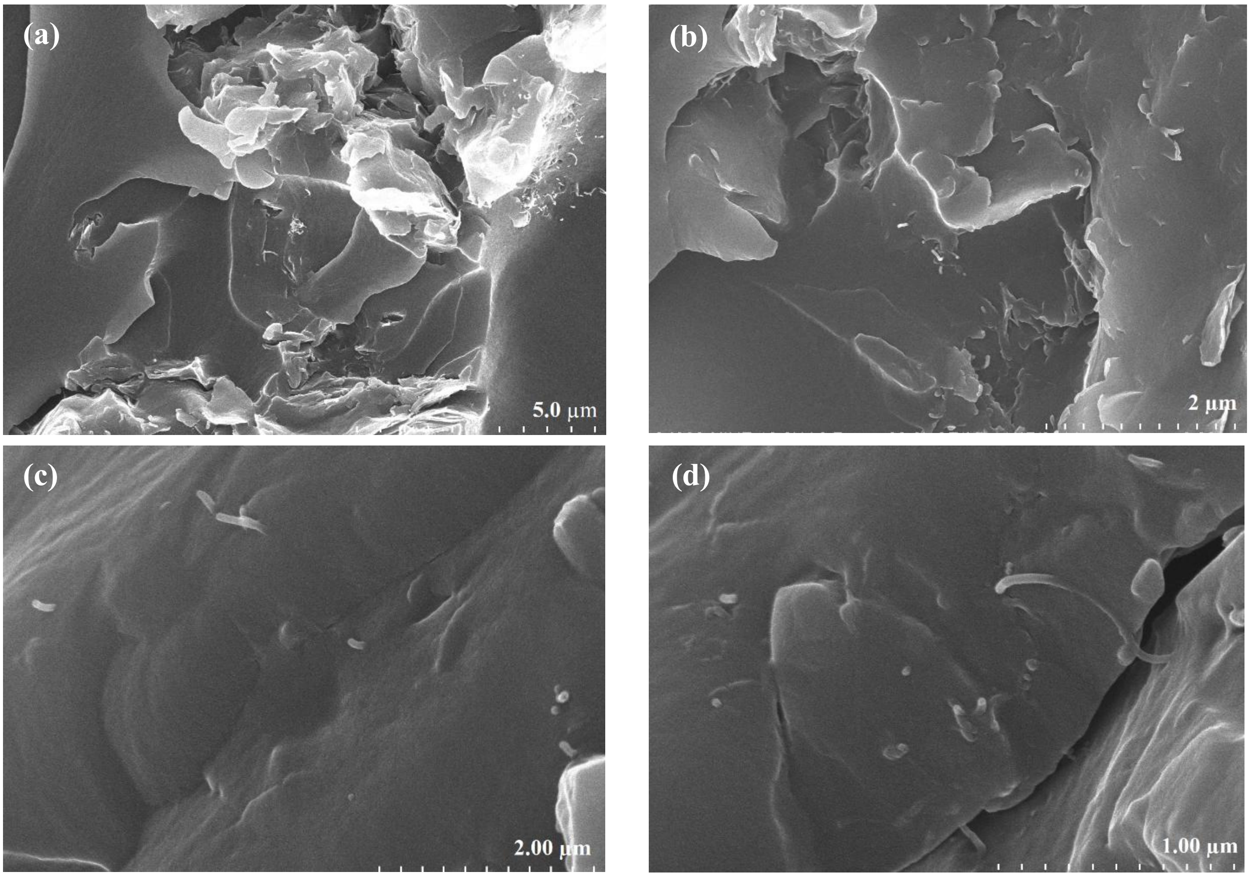

Figure 9 Structural morphology of PVA/MWCNTs/nanoclay I.30E nanocomposite films: (a) -PVA/ 0.2 wt% MWCNTs/ 1 wt% Nanoclay; (b) -PVA/ 0.5 wt% MWCNTs/3 wt% Nanoclay; (c) and (d) -PVA/ 1.0 wt% MWCNTs/5 wt% Nanoclay.

In summary, the increasing concentration of MWCNTs and nanoclay significantly influences the structural morphology of the nanocomposite films. At an optimal concentration (such as in sample (b)), the phases are more evenly dispersed, maximizing mechanical and flame-retardant properties. However, at higher concentrations (as in samples (c) and (d)), aggregation and crack formation may reduce the material's overall performance enhancement.

Figure 10 illustrates the structural morphology of PVA/MWCNTs/nanoclay nanocomposite films, showing differences in the dispersion of MWCNTs and nanoclay at various ratios. In sample (a) with 1.0 wt% MWCNTs and 5.0 wt% nanoclay, significant aggregation of the nano components can be observed, particularly large, non-uniform clusters on the surface. This indicates that at high nanoclay concentrations, the increased interaction forces between nanoclay sheets reduce their ability to disperse evenly in the PVA matrix, which may negatively impact the mechanical properties of the material. Meanwhile, sample (b) with 0.2 wt% MWCNTs and 1.0 wt% nanoclay exhibits a more uniform surface with less aggregation. The lower nanoclay content enhances dispersion, while the small amount of MWCNTs helps limit the agglomeration of carbon nanotubes, potentially improving mechanical properties without compromising the flexibility of the composite film.

For sample (c) with 0.5 wt% MWCNTs and 3.0 wt% nanoclay, good interfacial adhesion between phases is observed, though slight aggregation is still present in certain areas. At 3.0 wt% nanoclay, the nanoclay sheets appear to remain well-dispersed in the PVA matrix, while the 0.5 wt% MWCNTs content does not cause excessive aggregation. This suggests that the 0.5 wt% MWCNTs and 3.0 wt% nanoclay ratio could be optimal, improving the material's properties without excessive aggregation.

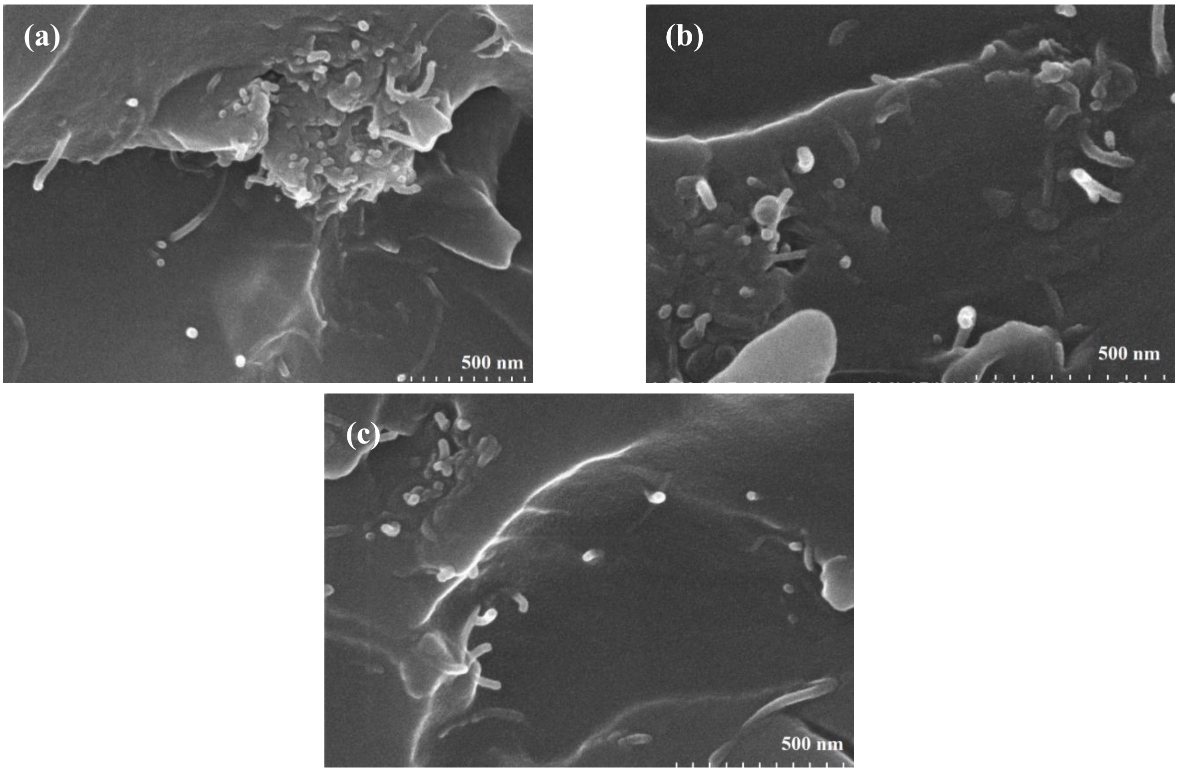

Figure 10 Structural morphology of PVA/MWCNTs/nanoclay I.30E nanocomposite films: (a) - PVA/ 1.0 wt% MWCNTs/ 5.0 wt%Nanoclay; (b) - PVA/ 0.2 wt% MWCNTs/1 wt% Nanoclay; (c) - PVA/ 0.5 wt% MWCNTs/3 wt% Nanoclay.

Overall, when the nanoclay concentration is too high (5.0 wt%), increased aggregation may reduce the reinforcement effectiveness of the nano phase, while at lower concentrations (1.0 wt%), better dispersion is achieved but may not provide optimal reinforcement effects. At an intermediate level (3.0 wt% nanoclay and 0.5 wt% MWCNTs), dispersion appears to be the most optimal, enhancing the composite film properties while minimizing aggregation. Therefore, the proper distribution of MWCNTs and nanoclay plays a crucial role in tuning the mechanical properties and performance of the material, as well as influencing the durability and flame retardancy of the nanocomposite film.

TGA characterization of MWCNTs/nanoclay PVA nanocomposite membrane

Based on the TGA graph in **Figure 11**, all PVA/MWCNTs/nanoclay nanocomposite membrane samples undergo three main thermal degradation stages. The first stage (below 150 °C) involves slight weight loss due to the evaporation of adsorbed water or residual solvents. The second stage (around 250 - 400 °C) corresponds to the primary degradation of the PVA polymer matrix, which occurs most significantly between 300 - 350 °C. The third stage (above 400 °C) represents the decomposition of the remaining components, such as MWCNTs and nanoclay. Comparing the thermal stability of the samples, sample (a) [PVA/1.0 wt% MWCNTs/5.0 wt% Nanoclay] exhibits lower thermal stability than the other samples, possibly due to nanoclay aggregation at high concentrations. Meanwhile, sample (b) [PVA/0.5 wt% MWCNTs] tends to degrade earlier than the samples containing nanoclay, indicating that nanoclay plays a crucial role in enhancing thermal stability.

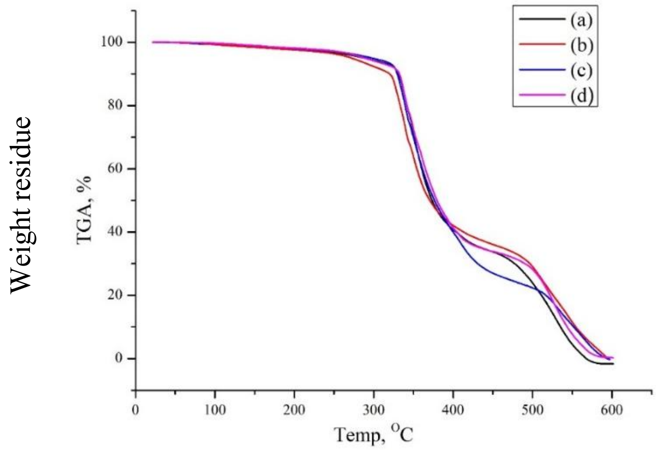

Figure 11 TGA characterization of MWCNTs/nanoclay PVA nanocomposite membrane: (a) - PVA/ 1.0 wt% MWCNTs/ 5.0 wt% Nanoclay; (b) - PVA/ 0.5 wt% MWCNTs; (c) - PVA/ 0.2 wt% MWCNTs/3 wt% Nanoclay; (d) - PVA/ 0.5 wt% MWCNTs/3 wt% Nanoclay.

From **Figure 12**, **Table 5** it can be observed that all composite samples undergo major thermal degradation in the range of 300 - 500 °C, but their thermal stability differs significantly. The weight residue at 600 °C gradually increases from sample (a) to (d), approximately 7%, 11%, 13%, and 14%, respectively, indicating that the addition of reinforcing agents such as nanoclay and MWCNTs contributes to the formation of a more thermally stable char layer, thereby slowing down the degradation process. Notably, sample (d) shows a more gradual slope and a shift of the decomposition region toward higher temperatures, suggesting a slower decomposition rate and improved thermal resistance. Although precise activation energy (Ea) values cannot be calculated without data at multiple heating rates, the clear shift of the degradation zone to higher temperatures implies an increase in Ea due to the synergistic effect between PVA and the nanofillers. Therefore, TGA analysis confirms that the incorporation of nanoclay and MWCNTs not only enhances the thermal stability but also facilitates the formation of a protective char structure, making the material suitable for applications requiring improved flame retardancy and heat resistance.

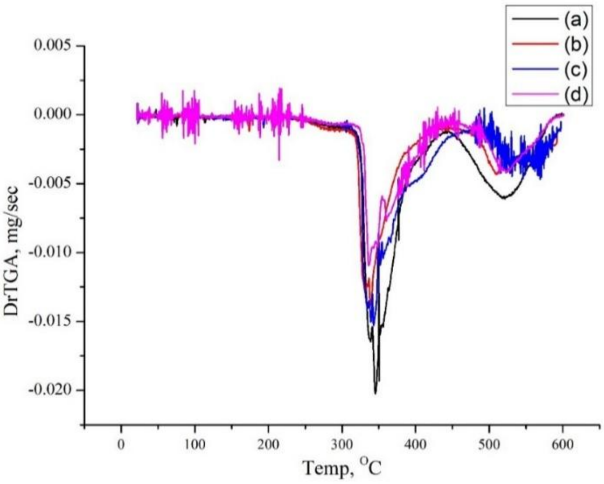

Figure 12 DTG characterization of MWCNTs/nanoclay PVA nanocomposite membrane: (a) - PVA/ 1.0 wt% MWCNTs/ 5.0 wt% Nanoclay; (b) - PVA/ 0.5 wt% MWCNTs; (c) - PVA/ 0.2 wt% MWCNTs/3 wt% Nanoclay; (d) - PVA/ 0.5 wt% MWCNTs/3 wt% Nanoclay.

Table 5 Thermal stability evaluation eummary.

Sample	Char Residue (%) @600 °C	Degradation Range (°C)	Thermal Stability
(a)	~7	~320 - 450	Low
(b)	~11	~330 - 460	Moderate
(c)	~13	~335 - 475	Good
(d)	~14	~340 - 490	Excellent

Sample (c) [PVA/0.2 wt% MWCNTs/3.0 wt% Nanoclay] and sample (d) [PVA/0.5 wt% MWCNTs/3.0 wt% Nanoclay] demonstrate better thermal stability, with their main thermal decomposition occurring at higher temperatures than samples (a) and (b). Notably, sample (d) exhibits the highest thermal resistance due to the balanced combination of MWCNTs and nanoclay, which enhances thermal protection and creates a better thermal shielding effect. Regarding the role of the components, nanoclay improves thermal stability by forming a physical barrier that slows down the diffusion of thermal decomposition products, while MWCNTs act as reinforcing agents, enhancing thermal stability by improving heat dispersion and reducing the thermal degradation of PVA. However, excessive nanoclay content, as in sample (a), can lead to aggregation, reducing its thermal protection efficiency. Overall, sample (d) [PVA/0.5 wt% MWCNTs/3.0 wt% Nanoclay] exhibits the best thermal stability due to the optimal distribution of MWCNTs and nanoclay, whereas sample (a) has lower thermal stability due to nanoclay aggregation at high concentrations. This indicates that a proper balance between MWCNTs and nanoclay is essential to optimize the thermal stability of the nanocomposite membrane.

Based on the DTG curve in **Figure 12**, the maximum decomposition temperature (Tmax) of each sample can be determined, which is the temperature at which the decomposition rate reaches its peak. Sample (a) [PVA/1.0 wt% MWCNTs/5.0 wt% Nanoclay] has a lower Tmax compared to the other samples, indicating that a high nanoclay content (5.0 wt%) may lead to agglomeration, reducing the thermal protection capability of the material system. Meanwhile, sample (b) [PVA/0.5 wt% MWCNTs] exhibits a lower Tmax compared to nanoclay-containing samples, confirming the crucial role of nanoclay in enhancing thermal stability. Sample (c) [PVA/0.2 wt% MWCNTs/3.0 wt% Nanoclay] and sample (d) [PVA/0.5 wt% MWCNTs/3.0 wt% Nanoclay] have higher Tmax values than samples (a) and (b), demonstrating that a well-balanced combination of MWCNTs and nanoclay enhances thermal stability. Notably, sample (d) exhibits the highest Tmax, indicating that this material system has the optimal distribution of MWCNTs and nanoclay, improving thermal protection performance. This combination may create a better thermal shielding effect, reducing the polymer’s decomposition rate. Overall, the Tmax of the samples increases in the order of (b) < (a) < (c) < (d), where sample (d) has the highest thermal stability, while sample (a) has a lower Tmax due to nanoclay agglomeration at high concentrations.

Mechanical properties and flame retardancy of PVA/MWCNTs/nanoclay I.30E nanocomposite films

Table 6 shows that the mechanical properties of PVA/MWCNTs/Nanoclay films significantly improve compared to pure PVA. Specifically, the tensile strength of pure PVA is only 32.5 ± 1.2 MPa, but when 0.5 wt% MWCNTs and 2.5 wt% Nanoclay are added, this value increases to 41.2 ± 1.5 MPa. At 1.0 wt% MWCNTs and 5.0 wt% Nanoclay, the tensile strength reaches 47.8 ± 1.7 MPa, an increase of approximately 47% compared to pure PVA. Similarly, Young’s modulus increases from 1.2 ± 0.05 GPa (PVA) to 1.9 ± 0.08 GPa with 0.5 wt% MWCNTs and 2.5 wt% Nanoclay, and reaches a maximum of 2.5 ± 0.1 GPa at the highest reinforcement level. However, elongation at break decreases from 85.3 ± 2.1% (PVA) to 60.5 ± 2.0% (for 1.0 wt% MWCNTs and 5.0 wt% Nanoclay), indicating that the composite films become stiffer but less flexible.

Table 6 Mechanical properties of PVA-Based nanocomposite samples.

Sample	Tensile Strength (MPa)	Elongation at Break (%)	Young's Modulus (MPa)
PVA/ 1.0 wt% MWCNTs/ 5.0 wt% Nanoclay	85.2 ± 3.1	12.4 ± 0.6	3200 ± 150
PVA/ 0.5 wt% MWCNTs	70.5 ± 2.8	9.8 ± 0.5	2800 ± 120
PVA/ 0.2 wt% MWCNTs/ 3 wt% Nanoclay	78.9 ± 3.0	11.2 ± 0.5	3000 ± 140
PVA/ 0.5 wt% MWCNTs/ 3 wt% Nanoclay	82.3 ± 3.2	11.9 ± 0.6	3100 ± 130

Table 7 provides data on fire resistance properties. The LOI (Limiting Oxygen Index) of pure PVA is only 22.8 ± 0.5, making it highly flammable. When 0.5 wt% MWCNTs and 2.5 wt% Nanoclay are added, LOI increases to 28.7 ± 0.6, and at 1.0 wt% MWCNTs and 5.0 wt% Nanoclay, it reaches 33.5 ± 0.7, demonstrating significant improvement in self-extinguishing ability. The main decomposition temperature (Tmax) also shows an increasing trend from 285.2 ± 2.5 °C (PVA) to 315.7 ± 2.8 °C at the highest reinforcement level, indicating better thermal stability. Notably, in the UL 94V test, pure PVA does not meet fire resistance standards (NR - No Rating), while all MWCNTs/Nanoclay composites achieve V-0, the highest rating for flame retardancy.

Table 7 Flame retardant properties of PVA-Based nanocomposite samples.

Sample	LOI (%)	UL 94V Rating
PVA/ 1.0 wt% MWCNTs/ 5.0 wt% Nanoclay	32.5 ± 1.2	V-0
PVA/ 0.5 wt% MWCNTs	30.1 ± 1.0	V-1
PVA/ 0.2 wt% MWCNTs/ 3 wt% Nanoclay	31.4 ± 1.1	V-0
PVA/ 0.5 wt% MWCNTs/ 3 wt% Nanoclay	32.0 ± 1.2	V-0

Conclusions

The results of this study demonstrate that incorporating 0.5% MWCNTs and 3% nanoclay into the PVA matrix significantly improves mechanical, thermal, and flame-retardant properties. The composite exhibited a tensile strength of 46.2 MPa, an increase of 40.8% compared to pure PVA (32.8 MPa), while the flexural strength increased by 34.5% from 53.2 MPa to 71.5 MPa. The reinforcement provided by MWCNTs and nanoclay effectively enhanced the stiffness and load-bearing capacity of the material without significantly compromising its flexibility.

SEM images confirmed the uniform dispersion of MWCNTs and nanoclay, forming a well-integrated network that improved stress distribution. TGA results indicated that the composite's degradation temperature increased to 318.7 °C, 27.6 °C higher than pure PVA (291.1 °C), demonstrating enhanced thermal stability. This suggests that the composite material can withstand higher temperatures, making it more durable in harsh environments.

Notably, the composite also exhibited superior flame-retardant performance. The LOI value reached 31.5%, significantly higher than 19.8% for pure PVA, and the material achieved a UL-94 V-0 rating, meaning it self-extinguished within a short duration, thereby improving safety for industrial applications.

This research contributes new insights by demonstrating the effectiveness of MWCNTs and nanoclay co-reinforcement in enhancing multiple properties of PVA-based composites. The findings have both scientific and practical implications, paving the way for future applications in various industries.

Future research directions may include:

- Optimizing dispersion techniques to further enhance MWCNT and nanoclay integration.
- Investigating environmental effects such as humidity, pH, and mechanical stress on material stability.
- Testing real-world applications, especially in flame-retardant biodegradable packaging, thermal insulation, and coatings for aerospace, automotive, and electronics industries.

Exploring biocompatibility for potential applications in medical protective films and heat-resistant medical bandages.

With these promising results, PVA/MWCNTs/nanoclay composites hold great potential for both academic research and industrial applications, offering an effective alternative to conventional materials for fire-resistant and high-performance applications in the future.

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Declaration of Generative AI in Scientific Writing

The authors declare that generative AI tools were used only to improve the readability and language of the manuscript. These tools were applied with full human oversight and control. The authors remain fully responsible for all scientific content, analysis, and conclusions presented in the paper. No AI tools were listed as authors or co-authors in this work.

CRediT author statement

Tuan Anh Nguyen: Conceptualization, Methodology, Supervision, Writing – Original Draft, Project administration, Funding acquisition. Van Hoan Nguyen: Investigation, Data curation, Formal analysis, Visualization, Writing – Review & Editing.

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