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Research Article

CRYSTALLINITY AND PHASE COMPOUNDS IN POLYVINYL ALCOHOL-MONTMORILLONITE NANOCOMPOSITES THROUGH X-RAY DIFFRACTION AND OPTICAL MICROSCOPY

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Abstract
The aim of this study was to analyse modern advancements in X-ray diffraction and optical microscopy to enhance the characterization of polyvinyl alcohol-montmorillonite (PVA-MMT) nanocomposites. The development of advanced nanocomposites has garnered significant attention due to their potential applications in various fields. Among these, polyvinyl alcohol reinforced with montmorillonite has emerged as a promising material owing to its enhanced mechanical and thermal properties. However, a thorough understanding of the crystallinity and phase compounds in these nanocomposites is essential for optimizing their performance and quality. To achieve this goal, recent studies on these techniques were systematically reviewed, and their effectiveness, advantages, and limitations were evaluated. The results demonstrated that digital holography, fluorescence microscopy, and confocal microscopy significantly improve the visualization of microstructural features and provide detailed 3D images and quantitative phase contrast. *In situ* X-ray diffraction allowed real-time monitoring of structural changes, while small-angle X-ray scattering provided detailed information about the size, shape, and distribution of nanostructured features. Synchrotron X-ray diffraction offered very high resolution and sensitivity, facilitating precise characterization of the nanocomposite's properties. By integrating these advanced techniques, the study established a comprehensive framework for understanding the crystallinity and phase composition of PVA-MMT nanocomposites, paving the way for optimized material development.

Keywords Crystallinity, Montmorillonite, Optical Microscopy, Phase Compound, Polyvinyl Alcohol, X-Ray Diffraction.

INTRODUCTION

Polyvinyl alcohol (PVA) is a versatile synthetic polymer notable for its excellent film-forming ability, chemical resistance, biocompatibility, and solubility in water (Hasimi, Papadokostaki, &

Sanopoulou, 2014; Gaidukov et al., 2015; Nagarkar & Patel, 2019; Antwi, Gyamera, & Abdulshakur, 2025). These properties have made PVA a widely used material in diverse applications such as packaging, textiles, pharmaceuticals, and the biomedical field. Moreover, its biodegradability makes it an environmentally friendly alternative for a variety of industrial uses (Schiessl et al., 2022). The molecular structure of PVA, characterized by hydroxyl groups along its polymer chain, contributes to its unique physicochemical properties and suitability for these applications. However, optimizing PVA for advanced functionalities requires a deeper understanding of its internal structure and the factors influencing its performance. A critical determinant of PVA's performance is its crystallinity, which refers to the degree of structural order within the material (Adekenov et al., 1991; Anugradia, Kruehong, & Alvarez, 2025; Candia et al., 2025). Crystalline regions consist of tightly packed polymer chains, while amorphous regions remain disordered. This balance significantly influences the polymer's mechanical strength, thermal stability, and barrier properties (Bunn, 1948; Li, Li, & Sun, 2021; Pan et al., 2019). Higher crystallinity typically enhances strength and stability, making it a key property for tailoring PVA for specific industrial needs (Tuan et al., 2024; Kuznetsov et al., 2004). In addition to crystallinity, phase compounds – distinct regions or domains with varying compositions or structures – also play a crucial role in defining the material's properties. The introduction of nanomaterials, such as montmorillonite (MMT), into PVA matrices forms nanocomposites with unique phase components that can significantly improve mechanical, thermal, and barrier properties (Shen et al., 2021). Characterizing these phase compounds is therefore essential for optimizing PVA-MMT nanocomposites for advanced applications.

Understanding the crystallinity and phase compounds in PVA-MMT nanocomposites is particularly important for developing materials with tailored properties for high-performance applications. Techniques such as optical microscopy (OM) and X-ray diffraction (XRD) are widely used for this purpose. OM enables direct visualization of the microstructure, providing valuable insights into the spatial distribution of crystalline regions and phase compounds (Fahmy et al., 2020; Musa & Hameed, 2020; Kayış et al., 2021; Beltran. 2025; Intharit, Navarro, & Chanudom, 2025). As a non-destructive technique, OM preserves the sample's integrity, allowing further analysis with other methods. Additionally, OM facilitates real-time observation of dynamic processes such as crystallization or phase transitions under varying environmental conditions, providing critical insights into the behaviour of nanocomposites during processing and service life (Soliman et al., 2021; Ikhsan et al., 2025). Furthermore, its relatively low cost and availability make OM an accessible tool for routine laboratory analyses. However, OM is limited by its resolution, which is constrained by the wavelength of visible light, making it challenging to resolve nanoscale features critical for studying PVA-MMT nanocomposites (Zheng et al., 2020).

Complementing OM, XRD provides precise quantitative data on the degree of crystallinity, lattice parameters, and phase composition (Siva et al., 2021; Aziz et al., 2020). This non-destructive method is invaluable for bulk material analysis, offering detailed insights into the atomic-scale structural properties of PVA-MMT nanocomposites. However, conventional XRD also has limitations, such as insufficient resolution to distinguish closely spaced diffraction peaks in complex nanocomposites and insensitivity to weak diffraction signals (Deghiedy & El-Sayed, 2020). Moreover, it lacks the capability to capture dynamic structural changes in real time, limiting its use for studying processes such as crystallization or phase transitions. Recent advancements in OM and XRD methodologies, including digital holography, fluorescence microscopy, confocal microscopy, synchrotron XRD, and small-angle X-ray scattering (SAXS), have addressed many of these limitations. These techniques offer higher resolution, enhanced sensitivity, and the ability to monitor dynamic processes under controlled conditions, providing a more comprehensive understanding of the material's structure and behaviour (Fratz et al., 2021; Panagopoulou et al., 2021; Siva et al., 2021; Jalmasco, Loberes, & Lasala, 2025; Jarnawi et al., 2025).

While numerous studies have explored the application of XRD and OM in characterizing PVA-based materials, few have systematically analysed the integration of advanced techniques to overcome the limitations of conventional methods. This study distinguishes itself by critically evaluating modern advancements such as digital holography, fluorescence microscopy, and synchrotron XRD, and their synergistic application in the analysis of PVA-MMT nanocomposites. By bridging the gap between conventional and advanced methods, the work provides a comprehensive framework for characterizing crystallinity and phase compounds with greater precision and detail. This integration not only addresses the limitations of resolution, sensitivity, and real-time monitoring but also establishes methodologies for tailoring material properties to specific industrial applications.

RESEARCH METHOD

This review focuses on two primary techniques for studying the crystallinity and phase compounds in polyvinyl alcohol-montmorillonite (PVA-MMT) nanocomposites: OM and XRD. These methods were selected for their complementary capabilities in providing both qualitative and quantitative insights into the microstructural characteristics of the nanocomposites. The selection of literature for this review was guided by specific inclusion and exclusion criteria to ensure a comprehensive and up-to-date understanding of these methods. For optical microscopy, the inclusion criteria focused on articles published within the last five years that presented significant advancements and innovative approaches in imaging techniques. Priority was given to studies that explored new imaging modalities, enhanced resolution, and improved measurement accuracy. Priority was given to recent advancements and comprehensive reviews that significantly improved the resolution, accuracy, and capabilities of optical microscopy techniques. These articles were selected based on their rigorous experimental design, reproducibility of results, and thorough data analysis, ensuring that only high-quality, scientifically robust studies were included. Additionally, review and tutorial articles that provided a comprehensive overview of current techniques and offered practical guidance for their implementation were also considered.

The selected works on optical microscopy highlight advancements in enhancing resolution, measurement accuracy, and expanding the capabilities of conventional OM methods through recent developments in imaging modalities such as confocal microscopy, super-resolution fluorescence microscopy, and digital holography. Similarly, in the realm of X-ray diffraction, preference was given to recent studies that introduced cutting-edge technologies, such as high-resolution XRD and synchrotron radiation techniques, which improve the precision and depth of crystallographic analysis. These selected articles provide high-quality data and detailed visualizations, ensuring comprehensive structural insights. The reviewed studies emphasize innovative methodologies like in-situ XRD and synchrotron XRD for dynamic and complex analyses of PVA-MMT nanocomposites. By adhering to stringent inclusion criteria, this review consolidates the most relevant and recent advancements in OM and XRD, offering a thorough exploration of their modern applications.

RESULTS AND DISCUSSION

This section presents the comprehensive findings from review of advanced characterization techniques for studying the crystallinity and phase composition of PVA-MMT. The analysis focuses on two primary methodologies: optical microscopy and X-ray diffraction, with particular attention to advanced approaches within each method. To overcome limitations of traditional methods, advanced OM techniques such as Digital Holography, Fluorescence Microscopy, and Confocal Microscopy provide higher resolution, enhanced phase contrast, and 3D imaging capabilities. These techniques facilitate detailed and accurate visualization of the PVA-MMT nanocomposite structure. Similarly, advanced XRD techniques like SAXS, *in situ* XRD, Synchrotron XRD, and offer enhanced resolution, sensitivity to weak signals, and the ability to monitor dynamic processes. These methods provide comprehensive insights into the crystallinity and phase composition of PVA-MMT nanocomposites, enabling the development of materials with optimized properties for specific applications.

Digital Holography

Digital Holography (DH) is an advanced optical microscopy technique that captures the entire optical field, including both amplitude and phase information, in a single exposure (Htwe & Mariatti, 2020). This method can significantly enhance the study of crystallinity and phase composition in PVA reinforced with MMT nanocomposites by providing detailed three-dimensional (3D) imaging and quantitative phase contrast (Schnars et al., 2015; Jumaera, Blessing, & Rukondo, 2025; Kheang, Hankhuntod, & Wesonga, 2025). Digital Holography involves recording a hologram of the sample using a digital sensor and then reconstructing the image computationally (Htwe & Mariatti, 2020; Nou et al., 2025; Obenza et al., 2025). One of the key advantages of DH is its ability to provide quantitative phase contrast. The phase information is directly related to the optical path length changes caused by variations in the refractive index and thickness of the sample. The basic setup includes a coherent light source, typically a laser, which is split into two beams: the reference beam and the object beam (Figure

1). In Figure 1, each component of the system is illustrated, showing how the beams are directed and recombined to create an interference pattern on the CCD camera. This pattern encodes the optical characteristics of the sample, enabling precise 3D reconstruction and phase analysis. Both beams are then recombined to create an interference pattern (hologram) on the digital sensor. The hologram contains all the information needed to reconstruct the 3D image of the sample, including phase and amplitude variations.

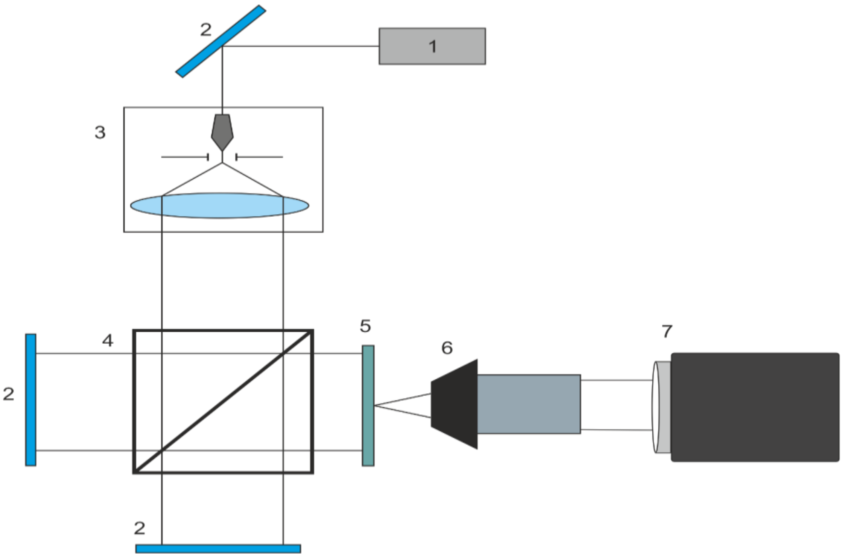

Figure 1. Digital holographic microscope: 1 – laser, 2 – mirrors, 3 – beam expander, 4 – beamsplitter cube, 5 – object under study, 6 – microobjective, 7 – CCD camera

Digital Holography can capture the 3D structure of PVA-MMT nanocomposites with high precision (Maripov, 1994; Ahmadov et al., 2022; Mabeza et al., 2025). This capability allows for detailed visualization of the distribution and morphology of crystalline regions and phase compounds. Crystalline regions and phase compounds typically have different refractive indices compared to the amorphous matrix, resulting in distinct phase contrasts. By analysing the phase contrast images, the distribution and number of crystalline regions can be quantitatively assessed. This quantitative data is crucial for understanding the degree of crystallinity in the nanocomposite. The 3D reconstruction capabilities of DH can reveal the spatial arrangement of MMT platelets within the PVA matrix, providing insights into the degree of dispersion and exfoliation of the clay nanoparticles. As a non-destructive technique, it is well-suited for real-time observations of dynamic processes in PVA-MMT nanocomposites. For instance, the crystallization process can be monitored *in situ* by observing the changes in phase contrast as the sample is heated or cooled. This real-time capability provides valuable information on the kinetics of crystallization and phase transitions, which are essential for tailoring the material properties. Digital Holography offers enhanced resolution and depth of field compared to conventional optical microscopy. The ability to focus through the entire thickness of the sample without mechanical sectioning allows for comprehensive analysis of the internal structure. This is particularly beneficial for studying thick or opaque samples, where traditional optical microscopy techniques might be limited.

Fluorescence Microscopy

Fluorescence microscopy is an advanced imaging technique that leverages the fluorescence properties of certain materials or dyes to provide high-contrast images. This method can be particularly useful for presented problem, offering detailed insights into the distribution and interaction of different phases within the material (Fratz et al., 2021; Rachmatika & Salighehdar, 2024; Putri et al., 2025). Fluorescence microscopy involves exciting a sample with a specific wavelength of light, causing fluorescent molecules within the sample to emit light at a longer wavelength (Lyubchyk et al., 2015). These fluorescent molecules can either be inherent to the sample or introduced via fluorescent dyes or markers. The emitted light is then captured to create high-contrast images that highlight the regions of interest within the sample (Qiang & Wang, 2020; Fratz et al., 2021; Rubio et al., 2025; Shagembe et al., 2025). This technique provides high-contrast images by highlighting only the fluorescently labelled regions. This selective imaging is particularly useful for studying the phase composition, as it allows for the clear identification of different phases within the nanocomposite. Crystalline regions and phase compounds can be visualized with high specificity, providing detailed insights into their morphology and spatial distribution (Dinzhos et al., 2005; Merkhatuly et al., 2023; Somantri, 2024; Salim et al., 2025). Such capabilities are particularly valuable for PVA-MMT nanocomposites, where understanding the interaction between PVA and MMT platelets is crucial. Fluorescence microscopy enables researchers to study these interactions at the molecular level, including how the dispersion and alignment of MMT platelets influence the overall crystallinity and mechanical properties of the nanocomposite. By tagging specific components, this technique allows for the visualization and quantification of spatial relationships between crystalline and amorphous regions, providing deeper insights into microstructural organization and guiding the optimization of material properties.

To apply fluorescence microscopy to PVA-MMT nanocomposites, specific components of the nanocomposite can be tagged with fluorescent dyes (Fratz et al., 2021). For example, fluorescent dyes can be attached to MMT platelets or to specific crystalline regions of PVA. This labelling allows for the selective visualization of these components, making it easier to distinguish between crystalline and amorphous regions and to observe the dispersion and distribution of MMT within the PVA matrix. Fluorescence intensity can be quantitatively analysed to estimate the concentration and distribution of fluorescently labelled components. This quantitative capability is valuable for assessing the degree of crystallinity and the extent of MMT dispersion within the PVA matrix. By analysing fluorescence intensity, researchers can obtain quantitative data on the amount and distribution of crystalline regions, enhancing the understanding of the material’s microstructure. It also is well-suited for dynamic studies, allowing real-time observation of processes such as crystallization, phase transitions, and the interaction between different components within the nanocomposite. By monitoring changes in fluorescence intensity and distribution, researchers can gain insights into the kinetics of these processes, which are critical for optimizing the material properties.

Advanced fluorescence microscopy techniques, such as confocal fluorescence microscopy and super-resolution fluorescence microscopy (e.g., stimulated emission depletion microscopy, photoactivated localization microscopy, stochastic optical reconstruction microscopy), can provide even higher resolution and depth selectivity (Panagopoulou et al., 2021; Stelzer et al., 2021; Siddique et al., 2025). Confocal fluorescence microscopy uses spatial pinholes to eliminate out-of-focus light, resulting in sharper images and 3D reconstruction capabilities. Super-resolution techniques surpass the diffraction limit of light, offering nanometre-scale resolution for detailed structural analysis.

Confocal Microscopy

Confocal Microscopy is an advanced optical imaging technique that provides high-resolution and depth-selective images, making it particularly effective for studying the PVA reinforced with MMT nanocomposites. This method utilizes spatial pinholes to eliminate out-of-focus light, producing sharp and detailed images that can be reconstructed into three-dimensional structures (Chen et al., 2021). Confocal Microscopy operates by scanning a focused laser beam across the sample and collecting emitted or reflected light through a pinhole aperture that is conjugate to the focal point of the objective lens. This setup ensures that only light from the focal plane is detected, significantly enhancing the resolution and contrast of the images. The confocal microscope typically uses point-scanning or spinning disk techniques to achieve rapid imaging. One of its key advantages is its ability to obtain optical sections at different depths within the sample. It allows for quantitative analysis of the structural features within the nanocomposite (Teng, Li, & Lu, 2020; Syahrul et al., 2025). Image analysis software can be used to measure the size, shape, and distribution of crystalline regions and MMT platelets (Boyko et al., 2023; Vasetska, 2024). By quantifying these parameters, researchers can gain insights into the degree of crystallinity and the effectiveness of MMT dispersion within the PVA matrix. By acquiring a series of optical sections, it is possible to reconstruct three-dimensional images of the nanocomposite, providing a comprehensive view of its internal structure. This depth selectivity is particularly useful for studying thick or layered samples, where traditional microscopy techniques might be limited.

This technique provides high-resolution images that can reveal fine details of the crystalline and amorphous regions within PVA-MMT nanocomposites and can be used for real-time observations of dynamic processes such as crystallization, phase transitions, and mechanical deformations. By using a heating or cooling stage, the effects of temperature on the crystallinity and phase composition can be monitored *in situ* (Jonkman et al., 2020). This capability provides valuable information on the kinetics and mechanisms of these processes, aiding in the optimization of the material properties. The improved resolution, often down to sub-micrometre levels, allows for the precise characterization of the morphology and distribution of MMT platelets and crystalline domains within the PVA matrix.

In Situ X-Ray Diffraction

In situ XRD is an advanced technique that allows for the real-time monitoring of structural changes in materials under varying conditions, such as temperature, pressure, or mechanical stress (Sai et al., 2020; Goergens, Manninger, & Goetz-Neunhoeffer, 2020). This method is particularly useful for studying the crystallinity and phase composition, providing insights into the dynamic processes and transformations occurring within the material. *In situ* XRD involves performing XRD measurements while the sample is subjected to controlled environmental changes (Sai et al., 2020; Goergens, Manninger, & Goetz-Neunhoeffer, 2020; Chang et al., 2020). This setup typically includes a sample holder that can be heated, cooled, or mechanically manipulated, along with a detector that continuously collects diffraction data. The resulting diffraction patterns reflect the structural changes occurring in the sample in real time, allowing for the analysis of phase transitions, crystallization, and other dynamic processes (Luo et al., 2022; Yulianti & Awingan, 2024).

One of the key applications of *in situ* XRD in the study of PVA-MMT nanocomposites is monitoring the crystallization process and phase transitions. By heating or cooling the sample, researchers can observe the formation or dissolution of crystalline regions within the PVA matrix. This real-time monitoring provides valuable information on the kinetics of crystallization, including the nucleation and growth rates of crystalline domains. In addition to kinetic insights, the XRD analysis of PVA-MMT nanocomposites reveals a clear enhancement in crystallinity with increasing MMT content. This improvement reflects the alignment and interaction of PVA chains around the MMT platelets, contributing to the material's mechanical and thermal properties. The quantitative crystallinity values for different MMT concentrations are summarized in Table 1. This capability allows researchers to study the effects of temperature and mechanical stress on the crystallinity and phase composition of the nanocomposites. For example, by applying a mechanical load to the sample while performing XRD measurements, researchers can investigate how stress influences the alignment and orientation of MMT platelets and the development of crystalline regions. Similarly, temperature variations can reveal the stability of different phases and the conditions under which phase transitions occur. Understanding these processes is crucial for optimizing the thermal and mechanical properties of the nanocomposite. The real-time data collection capability of *in situ* XRD provides continuous information on structural changes, enabling the detailed analysis of transient phenomena that would be difficult to capture with ex situ methods. This continuous monitoring allows for the identification of intermediate phases and transient states, providing a more comprehensive understanding of the material's behaviour under different conditions.

Table 1. Crystallinity of PVA-MMT Nanocomposites at Different MMT Concentrations

Sample ID	MMT Content (wt%)	Crystallinity (%)
PVA	0	52.3 ± 1.2
PVA-MMT-1	1	58.7 ± 1.4
PVA-MMT-5	5	65.1 ± 1.7

Source: compiled by the authors based on Aziz et al. (2020), Gaidukov et al. (2015) and Shen et al. (2021).

By correlating the structural changes observed during experiment with macroscopic properties such as mechanical strength, thermal stability, and barrier properties, researchers can establish relationships between the microstructure and the overall performance of the PVA-MMT nanocomposite. This correlation is essential for designing materials with tailored properties for specific applications, such as packaging, coatings, and biomedical devices (Zhu et al., 2021). *In situ* XRD can be combined with other analytical techniques, such as Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (TGA), to provide a more comprehensive understanding of the thermal behaviour of PVA-MMT nanocomposites (Green et al., 2021). These complementary techniques can help elucidate the relationship between thermal transitions and structural changes, enhancing the interpretation of the XRD data.

Small-Angle X-Ray Scattering

SAXS is a powerful technique for analysing the nanoscale structure of materials (Saadatkhah et al., 2020). SAXS provides detailed information about structures at the nanoscale, which complements the data obtained from other XRD methods. It measures the scattering of X-ray at small angles (typically less than 10°). The scattering intensity as a function of angle provides information about the size, shape, and distribution of nanostructures within the material (Gräwert & Svergun, 2020). Unlike wide-angle XRD, which focuses on atomic-scale structures and crystalline phases, SAXS is sensitive to larger structures in the range of 1 to 100 nm, making it ideal for studying the distribution of nanoparticles and other nanostructured features.

One of the primary applications of SAXS in the study of PVA-MMT nanocomposites is analysing the dispersion of MMT platelets within the PVA matrix. SAXS can reveal whether the MMT platelets are well-dispersed, intercalated, or exfoliated. This information is critical for understanding the mechanical, thermal, and barrier properties of the nanocomposite, as the degree of dispersion directly influences these properties. It also can be used to characterize nanostructured phases within the PVA matrix. For example, SAXS can detect the presence of nanocrystalline regions and provide information about their size and distribution (Saadatkhah et al., 2020). This is particularly useful for studying the formation of crystalline phases during processing and how they are affected by the presence of MMT. The nanostructural information obtained from SAXS can be correlated with the mechanical properties of the nanocomposite. For example, the degree of dispersion and the size of MMT aggregates can influence the tensile strength and modulus of the material. Mechanical testing further corroborates these observations, revealing a substantial increase in tensile strength with higher MMT loading. This reflects the reinforcing effect of well-dispersed nanoclay platelets within the PVA matrix. Table 2 presents tensile strength data for PVA-MMT nanocomposites with varying MMT contents. By combining SAXS data with mechanical testing results, researchers can develop models to predict the mechanical behaviour of the nanocomposite based on its nanostructure.

Table 2. Tensile Strength of PVA-MMT Nanocomposites at Varying MMT Concentrations

Sample ID	MMT Content (wt%)	Tensile Strength (MPa)
PVA	0	39.5 ± 2.3
PVA-MMT-30	30	153.4 ± 4.7
PVA-MMT-50	50	178.2 ± 3.9
PVA-MMT-70	70	219.0 ± 5.1

Source: compiled by the authors based on Bouchard et al. (2013).

SAXS is well-suited for *in situ* studies, allowing researchers to monitor structural changes in real time as the nanocomposite is subjected to various conditions, such as temperature changes or mechanical deformation (Gräwert & Svergun, 2020). This capability is valuable for studying processes like crystallization, phase separation, and the response of the nanocomposite to external stresses. *In situ* SAXS can provide insights into the kinetics of these processes and the mechanisms driving structural changes. While wide-angle XRD provides information about the atomic-scale crystalline structure, SAXS complements this by providing data on larger-scale structures. The combination of SAXS and wide-angle XRD offers a comprehensive understanding of the material's microstructure, spanning both the nanoscale and atomic scale. This dual approach is essential for fully characterizing the hierarchical structure of PVA-MMT nanocomposites.

Synchrotron X-Ray Diffraction

Synchrotron XRD is an advanced technique that utilizes highly intense and tunable X-rays produced by synchrotron radiation (Haubold et al., 2001). This method offers several advantages over conventional XRD. Synchrotron XRD provides exceptional resolution and sensitivity, enabling detailed structural analysis of complex materials (Ishige, 2020). Synchrotron radiation is produced by accelerating electrons to near the speed of light and deflecting them through magnetic fields (Rathmann et al., 2021). The resulting X-rays are highly intense and can be finely tuned across a wide range of wavelengths. This sensitivity is beneficial for studying the dispersion and interaction of MMT platelets with the PVA matrix, as well as for identifying any new phases formed during processing or thermal treatment. It allows for the collection of high-resolution diffraction data with greater accuracy and speed compared to conventional XRD. The intensity and tunability of synchrotron X-rays enable the detection of weak diffraction signals and subtle structural features (Kutsova et al., 2008). The exceptional intensity and brightness of synchrotron X-rays allow for detailed diffraction data collection. This allows for the detailed analysis of the crystalline structure of PVA-MMT nanocomposites, including the identification of minor crystalline phases and the resolution of closely spaced diffraction peaks. High-resolution data are crucial for understanding the degree of crystallinity and the distribution of different crystalline phases within the nanocomposite.

This method is well-suited for *in situ* and time-resolved studies, allowing researchers to monitor structural changes in real time under various conditions, such as heating, cooling, or mechanical deformation (Ishige, 2020). This capability is valuable for studying dynamic processes such as crystallization, phase transitions, and stress-induced structural changes. Time-resolved synchrotron XRD can provide detailed information on the kinetics and mechanisms of these processes, facilitating the optimization of processing conditions and material properties. The high brightness and focused beam of synchrotron X-rays make it possible to analyse thin films and small sample volumes with high precision (Wiedemann, 2003). This is particularly useful for studying the structural properties of thin PVA-MMT nanocomposite films used in applications such as coatings and membranes. The ability to analyse small samples also enables the study of localized regions within larger specimens, providing a more comprehensive understanding of the material's heterogeneity.

Advancements in XRD and OM techniques have significantly enhanced the ability to accurately characterize the crystallinity and phase composition of PVA-MMT nanocomposites. These improvements are critical for achieving precise material analysis, leading to better understanding and optimization of nanocomposites for diverse applications. Notably, advanced methods like DH, FM, and CM have emerged as powerful tools for studying microstructural features and phase compounds in PVA-MMT systems.

Digital holography provides high-resolution 3D imaging, enabling detailed visualization of crystalline regions and phase compounds (Maripov & Ismanov, 1994). The technique's quantitative phase contrast capabilities facilitate a comprehensive analysis of the spatial distribution and morphology of crystalline regions, making it particularly useful for real-time monitoring of dynamic processes like crystallization. Fluorescence microscopy offers high specificity and contrast through fluorescence labelling, which helps identify and visualize distinct phases and components within the nanocomposite. However, it requires careful sample preparation to prevent photobleaching. Confocal microscopy, combining high resolution and optical sectioning, enhances depth-selective imaging, allowing precise characterization of internal structures like crystalline regions and MMT dispersion.

The integration of these advanced optical microscopy techniques significantly strengthens their individual capabilities. For instance, DH can be combined with FM to correlate quantitative phase data with fluorescence intensity measurements, providing a holistic view of the material's structure. Similarly, integrating CM with DH offers detailed depth-selective visualization, further elucidating the distribution and morphology of MMT platelets and crystalline regions. These combined approaches facilitate a multifaceted and comprehensive analysis of PVA-MMT nanocomposites, enabling a deeper understanding of their structure-property relationships.

Compared to prior studies, such as those by Lin et al. (2020) and Alqaheem & Alomair (2020), which focus on membrane materials, this work emphasizes the synergy between XRD and OM techniques specifically for PVA-MMT systems. The integration of advanced XRD methods like in situ XRD, small-angle SAXS, and synchrotron XRD complements the capabilities of OM. In situ XRD enables real-time monitoring of structural changes during dynamic processes such as crystallization and phase transitions, providing critical kinetic insights. SAXS, on the other hand, offers nanoscale structural information, including particle dispersion and size distribution, which are vital for understanding mechanical and thermal properties. Synchrotron XRD, with its exceptional resolution and sensitivity, allows for the identification of minor crystalline phases and detailed structural analysis, particularly for thin films and localized regions within larger samples.

This study also contrasts its findings with other works. For example, Ali, Chiang, & Santos (2022) provide a review of XRD techniques for mineral characterization, while Doumeng et al. (2021) compare different methods for assessing the crystallinity of polyetheretherketone. Unlike these studies, the current review focuses on polymer-clay nanocomposites, highlighting the integration of XRD and OM techniques to overcome limitations of conventional methods. Furthermore, research by Aziz et al. (2020) specifically addresses the role of MMT content in enhancing the crystallinity of PVA-MMT nanocomposites, aligning with this study's findings on the critical influence of nanoparticle dispersion.

Similarly, Shen et al. (2021) discuss the synergistic effects of polymer and clay interactions in improving barrier properties, providing additional context for understanding the performance implications of structural analysis. While prior works often focus on isolated techniques or properties, this study's integration of XRD and OM methodologies offers a broader framework for characterization and optimization.

CONCLUSION

This review systematically analysed advanced optical microscopy and X-ray diffraction techniques to enhance the characterization capabilities of conventional methods and overcome their limitations in studying the crystallinity and phase composition of PVA-MMT nanocomposites. By evaluating the unique strengths and limitations of digital holography, fluorescence microscopy, confocal microscopy, *in situ* XRD, SAXS, and synchrotron XRD, a comprehensive framework for material characterization was established. Digital holography provides detailed 3D images and quantitative phase contrast, making it an excellent tool for visualizing microstructural features and dynamic processes in real time. *In situ* XRD enables real-time monitoring of structural changes under controlled environmental conditions, providing valuable insights into dynamic processes such as crystallization and phase transitions. SAXS complements other XRD methods by offering detailed information about the size, shape, and distribution of MMT platelets and other nanostructured features. Synchrotron XRD, with its very high resolution and sensitivity, is ideal for detailed structural analysis and detecting minor phases, facilitating a precise characterization of the nanocomposite's properties. While this study provides a robust framework for characterizing PVA-MMT nanocomposites, there are certain limitations to consider. The resolution and sensitivity of the techniques used may vary depending on the specific experimental setup and the properties of the material being analysed. Furthermore, the accuracy of the results could be influenced by factors such as sample preparation, environmental conditions during *in situ* measurements, and the inherent complexity of the nanocomposite structure.

Future studies should aim to address these limitations by refining the methodologies and exploring alternative approaches for improved characterization. The integration of advanced techniques, such as combining digital holography with synchrotron XRD or SAXS, could provide synergistic insights into both microstructural and nanostructural features, offering a holistic view of material behaviour. Such approaches would also enable real-time monitoring of phase transitions and crystallization processes under varying environmental conditions, paving the way for a deeper understanding of the relationship between structure and properties in PVA-MMT nanocomposites. Future research could focus on optimizing the conditions for using the discussed methods, such as digital holography and other microscopy and X-ray diffraction techniques, to enhance accuracy and efficiency in studying PVA-MMT nanocomposites. Particular attention should be given to the integration of multiple techniques simultaneously to achieve a more comprehensive and multifaceted analysis of the material's properties.

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AUTHOR CONTRIBUTIONS

Conceptualization, B.P. and A.L.; Methodology, E.C. and A.H.; Formal Analysis, B.P. and E.C.; Investigation, A.H.; Writing – Original Draft Preparation, B.P. and A.L.; Writing – Review & Editing, E.C. and A.H.; Visualization, A.H.; Supervision, A.L.

CONFLICTS OF INTEREST

The author(s) declare no conflict of interest.

USE OF ARTIFICIAL INTELLIGENCE (AI)-ASSISTED TECHNOLOGY

The authors declare that no artificial intelligence (AI) tools were used in the generation, analysis, or writing of this manuscript. All aspects of the research, including data collection, interpretation, and manuscript preparation, were carried out entirely by the authors without the assistance of AI-based technologies.

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