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Characterizing Crystallinity and Phase Compounds in Polyvinyl Alcohol–Montmorillonite Nanocomposites Through X-Ray Diffraction and Optical Microscopy

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The aim of this study is to analyse modern advancements in x-ray diffraction and optical microscopy to enhance the characterization of polyvinyl alcohol–montmorillonite 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 are reviewed, and their effectiveness, advantages, and limitations are evaluated. The results demonstrate that digital holography, fluorescence microscopy, and confocal microscopy improve significantly the visualization of microstructural features and provide detailed 3*D* images and quantitative phase contrast. *In situ* x-ray diffraction allows real-time monitoring of structural changes, while small-angle x-ray scattering provides detailed information about the size, shape, and distribution of nanostructured features. Synchrotron x-ray diffraction offers very high resolution and sensitivity, facilitating precise characterization of the nanocomposites' properties. By integrating these advanced techniques, the study establishes a comprehensive framework for understanding the crystallinity and phase composition of polyvi-

nyl alcohol–montmorillonite nanocomposites, paving the way for optimized material development.

Метою даного дослідження було проаналізувати сучасні досягнення в рентґенівській дифрактометрії й оптичній мікроскопії для поліпшення характеризації нанокомпозитів полівініловий спирт–монтморилоніт. Розробка передових нанокомпозитів привертає значну увагу завдяки їхньому потенційному застосуванню в різних галузях. Серед них полівініловий спирт, армований монтморилонітом, став перспективним матеріялом завдяки своїм поліпшеним механічним і термічним властивостям. Однак глибоке розуміння кристалічности та фазових сполук у цих нанокомпозитах є важливим для оптимізації їхньої продуктивности й якости. Для досягнення цієї мети нещодавні дослідження по цих методиках було розглянуто, а також оцінено їхні ефективність, переваги й обмеження. Результати показують, що цифрова голографія, флюоресцентна мікроскопія та конфокальна мікроскопія значно поліпшують візуалізацію мікроструктурних особливостей і забезпечують детальні 3*D*-зображення та кількісний фазовий контраст. Рентґенівська дифракція *in situ* дає змогу моніторити структурні зміни в режимі реального часу, тоді як розсіяння Рентґенових променів під малими кутами надає детальну інформацію про розмір, форму та розподіл наноструктурованих особливостей. Синхротронна рентґенівська дифракція забезпечує дуже високу роздільчу здатність і чутливість, що сприяє точній характеризації властивостей нанокомпозиту. Завдяки інтеґрації цих передових методів дослідження створює комплексну основу для розуміння кристалічности та фазового складу нанокомпозитів полівініловий спирт–монтморилоніт, прокладаючи шлях для оптимізованої розробки матеріялів.

Key words: phase composition, x-ray diffraction, optical microscopy, crystallinity, montmorillonite, polyvinyl alcohol.

Ключові слова: фазовий склад, рентґенівська дифракція, оптична мікроскопія, кристалічність, монтморилоніт, полівініловий спирт.

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1. INTRODUCTION

Polyvinyl alcohol (PVA) is a versatile synthetic polymer renowned for its remarkable properties, including excellent film-forming ability, chemical resistance, and biocompatibility [1–3]. PVA is widely used in various applications such as packaging, textiles, pharmaceuticals, and biomedical fields. Its solubility in water and biodegradability also makes it an environmentally friendly material [4]. The molecular structure of PVA, characterized by hydroxyl groups along its polymer chain, imparts unique physicochemical properties, which are crucial for numerous industrial applications. A critical

aspect of PVA performance is its crystallinity, which influences significantly its mechanical strength, thermal stability, and barrier properties [5–7]. Crystallinity in polymers refers to the degree of structural order within the material, where regions of highly ordered, tightly packed polymer chains (crystalline regions) coexist with disordered, amorphous regions. The extent of crystallinity can affect the polymers' properties, with higher crystallinity generally leading to enhanced strength and stability. In addition to crystallinity, the presence of phase compounds within the PVA can further modify its characteristics. Phase compounds refer to distinct regions or domains within the polymer matrix, which have different compositions or structures. In the context of nanocomposites, the incorporation of nanomaterials such as montmorillonite (MMT) can lead to the formation of new phase compounds, which can enhance the overall performance of the polymer [8]. These phase compounds can influence the mechanical, thermal, and barrier properties of the nanocomposite, making their characterization essential for optimizing material properties.

Understanding the crystallinity and phase compounds in PVA, especially, in nanocomposites like PVA–MMT, is vital for developing materials with tailored properties for specific applications. Among other methods, the most widely used ones for studying these properties are optical microscopy (OM) and x-ray diffraction (XRD). OM provides several advantages for the study of crystallinity and phase compounds in PVA–MMT nanocomposites. It allows for direct visualization of the microstructure, making it easy to identify and analyse the spatial distribution of crystalline regions and phase compounds [9–11]. As a non-destructive technique, OM preserves the samples' intrinsic properties, enabling further analyses with other methods. Additionally, OM allows for real-time observation of dynamic processes such as crystallization or phase transitions under various environmental conditions, offering insights into the behaviour of the nanocomposites during processing or in-service use [12]. Furthermore, the relatively low cost and widespread availability of optical microscopes make OM a practical choice for preliminary studies and routine analyses in most research and industrial laboratories.

Despite its advantages, optical microscopy has several limitations. It is limited by its resolution, constrained by the wavelength of visible light, which restricts the ability to resolve fine details at the nanoscale, essential for studying PVA–MMT nanocomposites. Additionally, it often suffers from a shallow depth of field, making it challenging to obtain clear images of thick or layered samples. Traditional OM primarily provides intensity-based images, lacking phase-contrast information necessary to differentiate between mate-

rials with similar optical densities, hindering clear identification of different phases within the nanocomposite. Extensive sample preparation, including staining or embedding, can introduce artefacts and potentially alter the samples' native structure, complicating image interpretation and leading to inaccurate conclusions. Moreover, conventional OM lacks the capability to capture dynamic processes in real time, a significant limitation for studying time-dependent phenomena such as crystallization, phase transitions, or mechanical deformation in PVA–MMT nanocomposites [13].

XRD offers several significant advantages for studying the crystallinity and phase compounds in PVA–MMT nanocomposites [14, 15]. One of its primary strengths is its high precision in identifying and quantifying crystalline phases, making it an essential tool for determining the degree of crystallinity within the sample. XRD provides detailed information about the crystal structure, lattice parameters, and phase composition, allowing for a thorough analysis of the materials' properties. Additionally, XRD is a non-destructive technique, preserving the sample for further analysis. Its ability to analyse bulk materials, combined with its quantitative nature, makes XRD a robust method for comprehensive crystallinity characterization [16]. However, conventional XRD struggles with the resolution required to distinguish closely-spaced diffraction peaks in complex materials like PVA–MMT nanocomposites, obscuring detailed crystalline-phase information and hindering accurate crystallinity determination. It can be insensitive to weak diffraction signals, critical for identifying minor phases or poorly ordered regions, leading to incomplete or misleading characterizations. While providing atomic-scale structural information, conventional XRD is less effective at characterizing larger nanostructures such as the size, shape, and distribution of MMT platelets within the PVA matrix, limiting the understanding of their influence on composite properties. Traditional XRD provides static snapshots, failing to capture dynamic processes like real-time structural changes due to temperature variations or mechanical stress. Proper sample preparation and alignment are crucial; misalignment or improper preparation can introduce artefacts or errors in diffraction patterns, resulting in inaccurate structural interpretations.

As discussed earlier, conventional XRD and OM are valuable tools for analysing the crystallinity and phase composition of materials. However, these methods have inherent limitations that can restrict their effectiveness, particularly, when dealing with complex nanocomposites. To address these challenges, this work focuses on analysing recent advancements in the XRD and OM techniques. The goal is to expand the capabilities of these conventional methods, enabling more accurate and detailed characterization of the crystallin-

ity and phase compounds in PVA-based nanocomposites. By integrating modern approaches and technologies, such as enhanced imaging methods and advanced diffraction techniques, it is possible to overcome the limitations of conventional methods and achieve a more comprehensive understanding of the materials' structure and properties.

2. MATERIALS AND METHODS

This review focuses on two primary techniques for studying the crystallinity and phase compounds in polyvinyl alcohol–montmorillonite 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, which presented significant advancements and innovative approaches in imaging techniques. Priority was given to studies, which explored new imaging modalities, enhanced resolution, and improved measurement accuracy. Works, which introduced or reviewed techniques, such as confocal microscopy, fluorescence microscopy, and digital holography, were particularly valued. 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, which provided a comprehensive overview of current techniques and offered practical guidance for their implementation, were also considered.

The selected works on optical microscopy highlight key advancements and comprehensive reviews of modern techniques. These studies demonstrate innovations in enhancing resolution, improving measurement accuracy, and increasing the non-destructive analysis capabilities of OM. By focusing on recent developments in imaging modalities, including confocal microscopy, super-resolution fluorescence microscopy, and digital holography, these works contribute significantly to the field by expanding the capabilities of conventional OM methods. In the realm of x-ray diffraction, the selection criteria emphasized articles, which focused on non-destructive analysis techniques and detailed structural analysis without damaging the sample. Preference was given to recent publications from the last five years, which introduced cutting-edge technologies or significant improvements in existing methods. This included high-

resolution XRD, synchrotron-radiation studies, and other advanced techniques, which enhance the precision and depth of crystallographic analysis. The selected articles were required to present high-quality data, with clear visualizations such as detailed diffraction patterns and comprehensive analyses of crystallographic information.

The selected works on x-ray diffraction showcase advanced methodologies and innovations in materials' characterization. These studies emphasize the non-destructive nature of XRD techniques and highlight significant improvements in the resolution and accuracy of XRD methods such as high-resolution and synchrotron x-ray diffraction. They explore complex structural analyses and *in situ* studies, which provide valuable insights into the properties and behaviour of PVA–MMT nanocomposites under various conditions. This review, by adhering to stringent inclusion criteria and focusing on the most recent and relevant literature, ensures a thorough exploration of modern advancements in the OM and XRD techniques.

3. RESULTS

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 convenient methods, advanced OM techniques such as digital holography, fluorescence microscopy, and confocal microscopy provide higher resolution, enhanced phase contrast, and 3*D*-imaging capabilities. These techniques facilitate detailed and accurate visualization of the PVA–MMT-nanocomposite structure. Similarly, advanced XRD techniques like small-angle x-ray scattering (SAXS), *in situ* XRD, synchrotron XRD 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.

3.1. 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 [17]. This method

can significantly enhance the study of crystallinity and phase composition in polyvinyl alcohol (PVA) reinforced with montmorillonite (MMT) nanocomposites by providing detailed three-dimensional (3*D*) imaging and quantitative phase contrast [18]. Digital holography involves recording a hologram of the sample, using a digital sensor and then reconstructing the image computationally [17]. 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 (Fig. 1). The object beam interacts with the sample, while the reference beam bypasses it. 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 3*D* image of the sample, including phase and amplitude variations.

Digital holography can capture the 3*D* structure of PVA–MMT nanocomposites with high precision. 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 3*D*-reconstruction capabilities of DH can reveal

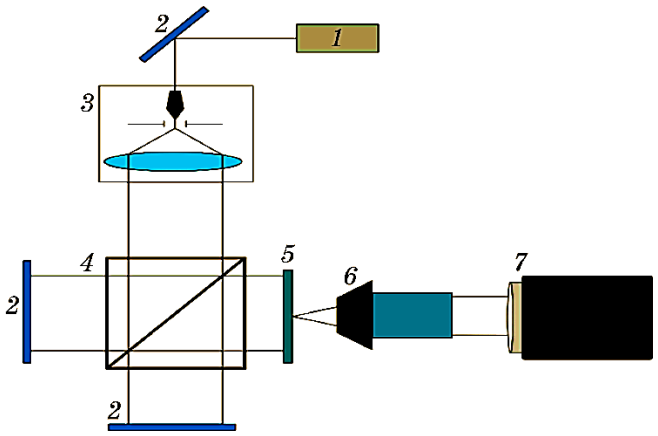

Fig. 1. Digital holographic microscope: *1*—laser, *2*—mirrors, *3*—beam expander, *4*—beam splitter cube, *5*—object under study, *6*—microobjective, *7*—CCD camera.

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.

3.2. 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 [19]. 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. These fluorescent molecules can either be inherent to the sample or be introduced *via* fluorescent dyes or markers. The emitted light is then captured to create high-contrast images, which highlight the regions of interest within the sample [19, 20]. 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.

To apply fluorescence microscopy to PVA–MMT nanocomposites, specific components of the nanocomposite can be tagged with fluorescent dyes [19]. 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 ana-

lysed 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 materials' 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 materials' 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 [21, 22]. Confocal fluorescence microscopy uses spatial pinholes to eliminate out-of-focus light, resulting in sharper images and 3*D*-reconstruction capabilities. Super-resolution techniques surpass the diffraction limit of light, offering nanometre-scale resolution for detailed structural analysis.

3.3. 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 montmorillonite nanocomposites. This method utilizes spatial pinholes to eliminate out-of-focus light, producing sharp and detailed images, which can be reconstructed into three-dimensional structures [23]. 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 [24]. Image-analysis software can be used to measure the size, shape, and distribution of crystalline regions and MMT platelets. By quantifying these parameters, researchers can gain insights into the degree of crystallinity and the effective-

ness 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, which can reveal fine details of the crystalline and amorphous regions within the 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* [25]. This capability provides valuable information on the kinetics and mechanisms of these processes, aiding in the optimization of the materials' properties. The improved resolution, often down to submicrometre levels, allows for the precise characterization of the morphology and distribution of MMT platelets and crystalline domains within the PVA matrix.

3.4. *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 [26, 27]. 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 [26–28]. 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 [29].

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. This allows for the study of the effects of temperature and mechanical stress on the crystallinity and phase composition of studied nanocomposites. For example, by applying a mechanical load to the sample, while per-

forming 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 materials' behaviour under different conditions.

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 [30]. *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 [31]. These complementary techniques can help elucidate the relationship between thermal transitions and structural changes, enhancing the interpretation of the XRD data.

3.5. Small-Angle X-Ray Scattering

Small-angle x-ray scattering (SAXS) is a powerful technique for analysing the nanoscale structure of materials [32]. 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 [33]. 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 [32]. 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. By combining SAXS data with mechanical-testing results, researchers can develop models to predict the mechanical behaviour of the nanocomposite based on its nanostructure.

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 [33]. 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 materials' microstructure, spanning both the nanoscale and atomic scale. This dual approach is essential for fully characterizing the hierarchical structure of PVA–MMT nanocomposites.

3.6. Synchrotron X-Ray Diffraction

Synchrotron XRD is an advanced technique that utilizes highly intense and tuneable x-rays produced by synchrotron radiation [34]. This method offers several advantages over conventional XRD. Synchrotron XRD provides exceptional resolution and sensitivity, enabling detailed structural analysis of complex materials [35]. Synchrotron radiation is produced by accelerating electrons to near the speed of light and deflecting them through magnetic fields [36]. 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. The high intensity and brightness of synchrotron x-rays enable the collection of high-resolution diffraction data. 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 [35]. 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 [37]. 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 the larger specimens, providing a more comprehensive understanding of the materials' heterogeneity.

4. DISCUSSION

The review of improved methods shows that advancements in XRD and OM techniques can significantly enhance the ability to characterize accurately crystallinity and phase composition in nanocomposites. These improvements are important because they allow for more precise analysis of materials' properties, leading to better understanding and optimization of nanocomposites for various applications. Digital holography, fluorescence microscopy, and confocal microscopy are powerful and versatile techniques, which enhance significantly the study of crystallinity and phase composition in PVA–MMT nanocomposites. Digital holography offers high resolution and 3*D*-imaging capabilities, enabling detailed visualization of microstructural features and phase compounds in polyvinyl alcohol–montmorillonite nanocomposites. Fluorescence microscopy provides high contrast and specificity by using fluorescent labels that is ex-

cellent for identifying different phases and components, though it requires careful preparation to avoid photobleaching. Confocal microscopy combines high resolution and 3*D* imaging with the ability to investigate optically section samples, providing detailed spatial information about the distribution of crystalline regions. Each method offers non-destructive analysis and real-time observation of dynamic processes, although sample preparation is critical to avoid artefacts and ensure accurate results. The cost and complexity of these methods vary, with digital holography and confocal microscopy typically requiring equipment that is more specialized and expertise compared to fluorescence microscopy. Together, these advanced optical-microscopy techniques enhance the characterization of crystallinity and phase compounds, overcoming the limitations of conventional methods.

The combined use of advanced optical methods can be performed to increase the capabilities of optical study even more. The combination of DH with advanced optical-microscopy techniques, such as fluorescence microscopy and confocal microscopy, can significantly enhance the study of PVA–MMT nanocomposites. DH captures the entire optical field, providing detailed 3*D* images and quantitative phase contrast. When integrated with fluorescence microscopy, it leverages high specificity fluorescence labelling to enhance contrast and selectively visualize different phases within the nanocomposite. Fluorescently labelled montmorillonite platelets and crystalline regions within the PVA matrix can be distinctly imaged, clearly differentiating various components. The quantitative phase data from digital holography can be correlated with fluorescence-intensity measurements for a comprehensive understanding of the materials' structures, revealing morphology and distribution of crystalline regions alongside concentration and spatial distribution of labelled components. Both techniques allow real-time observations of dynamic processes, enabling simultaneous monitoring of phase changes and fluorescence signals to understand crystallization and phase transitions. Confocal microscopy adds high-resolution, depth-selective imaging, enhancing the detailed visualization of the internal structure of PVA–MMT nanocomposites, including the morphology and spatial distribution of crystalline regions and montmorillonite platelets. This combined approach offers a multifaceted and comprehensive analysis of the crystallinity and phase composition in these materials.

The work by Y. Lin *et al.* [38] reviews various microscopy and spectroscopy techniques used for the characterization of polymeric membranes. The study focuses on the application of these techniques to analyse the structural, morphological, and chemical properties of membranes, with an emphasis on their performance in fil-

tration and separation processes. In contrast, this review focuses on the characterization of polyvinyl alcohol–montmorillonite nanocomposites, specifically using x-ray diffraction and optical microscopy to examine crystallinity and phase composition. The key difference lies in the material of Y. Alqaheem *et al.* [39], where the authors focused on polymer membranes, while the current work is centred on polymer-clay nanocomposites. Additionally, the present review emphasizes the integration of XRD with microscopy techniques, which is less emphasized in the membrane-focused study.

In situ XRD, SAXS, and synchrotron x-ray diffraction are powerful techniques for studying the crystallinity and phase composition of PVA–MMT nanocomposites. *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. *In situ* XRD offers high resolution suitable for identifying crystalline phases and excellent real-time monitoring of structural changes, while SAXS provides high resolution for nanoscale structures and is effective for time-resolved dynamic processes. Synchrotron XRD excels with very high resolution, detailed structural analysis, and sensitivity to minor phases, making it ideal for both bulk materials and thin films. All methods provide high penetration depths and are generally non-destructive. Proper sample preparation and alignment are crucial, with minimal artefacts, if done correctly. However, the cost and accessibility vary, with synchrotron XRD being the most expensive and requiring specialized facilities, whereas *in situ* XRD and SAXS are more accessible, but still require specialized equipment. Overall, these methods complement each other, providing a comprehensive framework for characterizing the crystallinity and phase composition in polyvinyl alcohol–montmorillonite nanocomposites.

Work by A. Ali *et al.* [40] provides a comprehensive review of XRD techniques specifically for mineral characterization, focusing on the fundamentals, practical applications, and future research directions. The review is tailored for engineers, emphasizing how XRD can be applied to analyse various minerals, their crystalline structures, and phase compositions. On the contrary, this work focuses on the characterization of PVA–MMT nanocomposites, particularly, through the combined use of XRD and optical microscopy. While both reviews discuss XRD, this work is more specialized in the context of polymer nanocomposites and explores the synergy between XRD and microscopy techniques, which are less emphasized in the mineral-focused study by A. Ali *et al.*

A. Ali *et al.* [40] also conduct a comparative study on the crystallinity of polyetheretherketone (PEEK), using various techniques, including density measurements, DSC, XRD, and Raman spectros-

copy. The study aims to provide a comprehensive understanding of the crystallinity of PEEK by evaluating and comparing the results from these different analytical methods. Conversely, the present review focuses on the characterization of polyvinyl alcohol–montmorillonite nanocomposites, specifically utilizing XRD and optical microscopy to assess crystallinity and phase composition. The primary difference lies in the work of M. Doumeng *et al.* [41], where PEEK, a high-performance polymer, is investigated, while the current review centres on PVA–MMT nanocomposites. Additionally, the present work emphasizes the combined use of XRD and microscopy techniques, whereas M. Doumeng *et al.* incorporate a broader range of characterization methods. Overall, the combined use of these advanced optical-microscopy and x-ray diffraction techniques provides a more comprehensive understanding of the crystallinity and phase composition in PVA–MMT nanocomposites, paving the way for the development of materials with optimized properties for specific applications. Future research could focus on further refining these techniques and exploring their application in new types of nanocomposites, as well as integrating them with other analytical methods to provide a more comprehensive understanding of materials' properties [42].

5. CONCLUSIONS

This review 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 3*D* 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 montmorillonite 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 nanocomposites' properties. While this study provides a robust framework for character-

izing 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. 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 materials' properties.

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