Evaluation of polymeric nanocomposites

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ABSTRACT

This article presents a review about the evaluation of polymer nanocomposites' properties, highlighting the importance of integrating various analytical techniques for these materials' characterization. Using a multi-method approach, some of the main techniques for elucidating the properties of nanocomposites were reviewed – Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), nuclear magnetic resonance (NMR), transmission electron microscopy (TEM), and scanning electron microscopy (SEM). It was possible to demonstrate the importance of the material characterization stage not only for industrial competitiveness through validation of the final application and performance optimization but also for a comprehensive and in-depth understanding of properties such as crystalline structure, composition and molecular mobility, thermal stability, and morphology, thus providing a solid foundation for technological innovation and future developments in the field of polymer nanomaterials, while not overlooking crucial aspects such as sustainability and safety.

Keywords: Materials, Nanocomposites, Polymers, Characterization, FTIR, TGA, DSC, XRD, NMR, TEM, SEM.

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INTRODUCTION

Nanotechnology, an emerging field of development with revolutionary promises, intensely stimulates research into advanced materials. In its broadest form, it involves the aspects in which particles and mechanisms of a very small size (*à priori*, with at least one of the dimensions equal to or less than 100 nm or 10^{-7} meters) are distinguished from their larger forms, or " macro". These differences in properties are mainly attributed to its incredibly high surface area relative to their small size. The square-cube law explains this relationship, where, relative to its dimensions, the area of a cube increases squared, while its volume increases cubed. In the same way that it explains the impossibility of building structures that are too large, it also explains the amplified surface properties at this scale (MANSOORI, 2005; THEODORE, 2006; ROCO, 2011; TAHA et al., 2022; Malik et al., 2023).

The issue of nanotechnology's roles is vast and diverse, impacting several technological sectors. Examples of nanotechnology applications include achieving higher conductivity, enabling the creation of metal-oxide-semiconductor transistor circuits below the 10 nm range, which was used in the most advanced CPUs. Furthermore, the acceptance of exogenous particles in living systems, due to their nano-scale and properties similar to molecules already present in the body, is noteworthy. Applying nanoscience concepts allows for everything from creating longer-lasting perfumes and more efficient medications through controlled-release systems to developing new nanocomposite materials with specific properties and exploring the potential of waste-based nanoparticles prepared for waste treatment (RAMSDEN, 2016; DA ROCHA et al., 2019, BINNS, 2021; RAHMANI, 2022; DA ROCHA et al., 2024).

With its nearly limitless potential for innovations, the advantages and challenges we face with nanotechnology are intricately connect to the future of technological innovation. Within this vast domain, polymeric nanocomposites have emerged as a versatile and promising class, driving innovations across a range of applications (ESCHBACH et al., 2007; YABUKI, ARRIFFIN, 2010; MINEA, 2019; BINNS, 2021; RAHMANI, 2022; VERMA et al., 2023).

Nanocomposites are one of the most explored areas in nanotechnology. These are biphasic blends of nanoparticles and a matrix, diverse in raw material choice, arrangement, and proportion, offering new potential for developing materials with unique properties. It is essential to understand that polymeric nanocomposites represent a synergy between the intrinsic properties of polymers and the unique characteristics of the incorporated nanoparticles. This combination offers a diverse range of applications in areas such as packaging, biomaterials, catalysts, electronics, among others (HASSAN et al., 2021; MUHAMMED SHAMEEM et al., 2021). For fully comprehend the role of nanocomposites in the vast field of nanotechnology, it is essential to adopt a multidisciplinary perspective that addresses the various characterization processes involved (ESCHBACH et al., 2007; MITTAL, 2012; NGUYEN-TRI et al., 2018).

As we advance in this promising field, it is vital to consider critical issues related to environmental safety, toxicity, and scalability of manufacturing processes. Adopting responsible approaches and risk management strategies is imperative to ensure not only the effectiveness and efficiency of these materials but also their safety throughout their lifecycle (VOGEL et al., 2014; KARIM et al., 2018).

This review specifically gathers knowledge about the evaluation of polymeric nanocomposites, highlighting the importance of integrating various analytical techniques for a comprehensive characterization of these materials. At the heart of this analysis are fundamental techniques such as Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and nuclear magnetic resonance (NMR). Among these techniques, NMR stands out as a sophisticated and advanced non-conventional tool that allows for detailed analysis of molecular structures and intermolecular interactions of nanocomposites. The advantages of combining NMR analysis in nanocomposites are numerous, especially considering sustainability. NMR is a non-destructive technique, allowing sample analysis without damaging them, thus reducing material waste. Moreover, it can be used to monitor processes in real-time, enabling optimization of synthesis and processing conditions, thereby reducing energy consumption and chemical reagents. The capability to provide detailed information on molecular dynamics and the structure of nanocomposites aids in developing more efficient and durable materials, contributing to the creation of more sustainable products. The integration of these analytical approaches provides a comprehensive understanding of nanocomposite properties, from their molecular structure to their nanoscale and microscopic morphology (MITTAL, 2012; SOARES IL et al., 2015; NIKULIN et al., 2016; SEBASTIÃO et al., 2016; PASHAEI et al., 2019; CHARVANI et al., 2020; CHEN & ZHEN, 2021; MOHAMADI, 2022; DA ROCHA et al., 2024).

Finally, this review not only highlights recent advancements and current trends in the assessment of polymeric nanocomposites but also outlines the challenges and opportunities shaping the future of this fascinating area of science and technology. By integrating multidisciplinary knowledge and advanced analytical techniques in nanotechnology, we are paving the way for an era of innovative and sustainable materials with the potential to drive significant advancements across a variety of industrial, biomedical, electronic, environmental, aerospace, and agricultural applications.

METHODOLOGY

This study focused on the classic stages that characterize the development of an integrative literature review, aiming to synthesize, relate, and critically analyze the findings from various previously published scientific studies in the field of polymeric nanocomposites characterization. This approach enabled the compilation and analysis of established research to achieve a comprehensive understanding of the current state of the art in this field. Following the identification of studies on the evaluation of polymeric nanocomposites using techniques such as FTIR, TGA, DSC, XRD, NMR, TEM, and SEM, their findings were compared and contrasted to identify patterns, trends, and discrepancies in the interpretation and presentation of results.

Appropriate search mechanisms were employ to compile scientific literature, including the platform Scientific Electronic Library Online (SciELO) and the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (Capes) portal. The online search engine Google Scholar served as the primary starting point for gathering e-books, academic papers, newspapers, journals, and indexed articles. This study thus substantiates the clear and objective presentation and discussion of the physical principles, objectives, advantages, and limitations of each aforementioned analytical technique in the evaluation of polymeric nanocomposites. The main objective was conceptually review the topic in an accessible manner, particularly for students and beginners in the field, without requiring a high level of specific technical expertise to understand the content, thereby contributing to the dissemination of scientific knowledge.

DISCUSSION OF RESULTS

It is possible to observe in a range of studies that nanotechnology is being apply in an increasing number of areas of knowledge. It can be described as the manipulation of matter on a smaller scale, with at least one of its dimensions below 100 nanometers (nm), or 10^{-7} meters. As a constantly growing field, nanotechnology can be define as a revolution in all technological sectors (BATISTA et al., 2010; LEOPOLDO; VECHIO, 2020; SAHOO et al., 2021).

With its broad range of applications, nanotechnology is highly interdisciplinary, impacting fields such as chemistry, physics, biology, and medicine, among others. Consequently, its applications vary according to each involved area. For instance, nanotechnology can optimize the creation of materials with ideal properties tailored for specific purposes, whether it's for packaging materials or aircraft engines (BATISTA et al., 2010; CADIOLI e SALLA, 2015; BINNS, 2021)

As another example of applications for nanotechnology, nanoelectronics has pushed the final boundaries of complexity limits for its components, with the creation of transistors at scales never seen before. In the pharmaceutical sector, the development of controlled release systems is revolutionizing drug application by reducing side effects and the number of doses required for treatment (SIMONE; BOCHNER; FERREIRA, 2013; CADIOLI e SALLA, 2015; LEOPOLDO; VECHIO, 2020).

Nanocomposites are materials in which nanoparticles are dispersed within a matrix, imparting combined properties from both the matrix and the added nanoparticles. The selection of nanoparticles as fillers in composites involves diverse criteria including composition, shape, and size, chosen based on cost-effectiveness and desired material properties. Common examples include metal oxides such as aluminum oxide (Al_2O_3), titanium oxide (TiO_2), and zinc oxide (ZnO). Silica nanoparticles are frequently utilize to enhance the strength and thermal stability of polymers. Carbon nanotubes, including single-walled (SWCNTs) and multi-walled (MWCNTs), serve to reinforce mechanical strength and improve electrical conductivity. Carbon nanofibers are renowned for enhancing mechanical properties, while clay nanoparticles like montmorillonite enhance gas and vapor barrier properties. Noble metal nanoparticles such as silver (Ag) and gold (Au) were employed for their antimicrobial properties in nanocomposites. The distribution, size, and quantity of these nanoparticles significantly influence the final properties of nanocomposites. Nanocomposites can be classified according to matrix type, such as polymeric, metallic, or ceramic nanocomposites (ABRAHAM et al., 2009; BOGUE, 2011; RATHOD; KUMAR; PANDI BOOMI et al., 2014; JAIN, 2017; MOHD NURAZZI et al., 2021).

The assessment, correlation, and understanding of nanomaterial properties are crucial due to their direct influence on material activity. Surface functional groups of nanomaterials dictate their chemical reactivity and interaction with other substances and biological systems. Dimensions and shapes significantly affect their physical and chemical properties, with smaller particles exhibiting higher reactivity due to a greater surface area-to-volume ratio. The high relative surface area of nanomaterials enhances their capacity to interact with other substances, thereby improving catalytic and adsorption properties. Particle diameter and aspect ratio influence distribution within the medium, penetration into cells and tissues, and efficiency in controlled drug release processes. Uniformity in particle size and shape is critical to ensure consistency in properties and performance. The crystalline structure and chemical composition of nanomaterials determine their electronic, optical, and mechanical properties, with different compositions exhibiting distinct toxicities and varying responses to external stimuli. These characteristics directly affect particle toxicity, with highly reactive surface nanoparticles potentially generating reactive oxygen species, leading to oxidative stress in biological cells. Moreover, their ability to penetrate and accumulate in specific organs can increase the risk of adverse health effects (BATISTA et al., 2010; DONALDSON; POLAND, 2013; VOGEL, 2014; ABBAS et al., 2020; EL-ZAHED et al., 2021).

Therefore, a detailed understanding of the properties of nanomaterials is essential for developing safe and effective applications, while minimizing risks to human health and the environment. This study is vital for protecting human and environmental health and promoting sustainability, ensuring that the development and application of nanomaterials do not compromise safety and ecological balance.

In this study, characterizations applied specifically to polymer-based nanocomposites were reviewed. Polymers are macromolecules formed by repeating structural units called monomers. Monomers react with each other and bond via covalent bonds to form long polymer chains. The polymers constituting nanocomposites are highly versatile and can be natural or synthetic, rigid, plastic, or elastomers, and biodegradable or non-biodegradable. During synthesis, their properties can be adjust to meet various industrial and commercial applications. Polymers exhibit a wide range of mechanical properties that categorize them into two main groups: engineering polymers, meticulously designed to offer specific characteristics such as high mechanical strength, chemical resistance, or heat resistance, ideal for applications requiring advanced technical properties; and commodity polymers, widely used in everyday products due to their relatively low cost (MANO; MENDES, 1999; MANO, 2000).

The analysis of polymer nanocomposites is therefore crucial for understanding their properties, potential applications, and developments. This study focuses on the detailed evaluation of these innovative materials using a range of analytical techniques, from conventional to advanced methods. These include Fourier-transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), X-ray diffraction (XRD), nuclear magnetic resonance (NMR) spectroscopy, transmission electron microscopy (TEM), and scanning electron microscopy (SEM). These tools provide profound insights into the structural, thermal, and morphological properties of nanocomposites, which are essential for advancing fields such as composite materials and nanotechnology.

Fourier Transform Infrared Spectroscopy (FTIR) is a widely used technique in chemistry, materials engineering, and other scientific fields to identify and quantify compounds of interest in a sample and analyze its chemical composition. The technique relies on the interaction of infrared radiation with molecules in the sample. Molecules absorb infrared radiation at specific frequencies corresponding to the vibrations of atoms within the molecules, such as stretching and bending of chemical bonds. Each functional group produces a characteristic absorption pattern in different regions of the infrared spectrum, allowing for the identification and quantification of specific compounds. At the core of FTIR is the Fourier Transform, a mathematical tool that converts a signal from the time domain to the frequency domain. This transformation is essential for obtaining the infrared spectrum from the raw data collected during the analysis (CANEVAROLO, 2007; BAUDOT; TAN; KONG, 2010; ISHIDA, 2013; HERMANN et al., 2017).

A typical FTIR spectrometer comprises several key components. An infrared radiation source emits a beam of light directed into an interferometer, which splits the beam into two paths: one directed towards a fixed mirror and the other towards a movable mirror. Upon recombination, they create an interference pattern called an interferogram, containing information about all light frequencies passing through the interferometer. This interference beam then travels towards the sample. As the infrared light passes through the sample, molecules absorb specific frequencies corresponding to their characteristic vibrations. The unabsorbed radiation continues to the detector, which measures the intensity of transmitted light and sends these data to a computer. The computer applies the Fourier Transform to the interferogram, converting it into an infrared absorption spectrum. This spectrum displays peaks at specific frequencies corresponding to the vibrational modes of bonds within the sample molecules. Each molecule or functional group exhibits a distinctive absorption pattern, enabling identification (CANEVAROLO, 2007; HERMANN et al., 2017; LI et al., 2018).

FTIR offers several significant advantages. The technique enables rapid data acquisition due to interferometry and is highly sensitive, capable of detecting signals from small amounts of material. Additionally, FTIR is very versatile, suitable for analyzing solids, liquids, and gases, and provides high-resolution spectra that distinguish between closely spaced peaks. For liquid samples, analysis typically requires a specific sample cell made from infrared-transparent materials. Solid samples can be analyzed directly or prepared as salt pellets or thin films (HERMANN et al., 2017; LI et al., 2018; DA ROCHA et al., 2024).

FTIR was employed across a wide range of applications. It can identify compounds, determine the presence and concentration of components in a sample, monitor chemical reactions over time (such as oxidation aging), and provide insights into molecular structure and bonds. For instance, in the pharmaceutical industry, FTIR ensures the purity and composition of medications. In materials science, it analyzes polymers and other complex substances. However, the technique does have some drawbacks. Analyzing complex substances can produce spectra that are complicated and challenging to interpret. Moreover, FTIR equipment can be costly, and careful sample preparation may be required to achieve accurate results (HERMANN et al., 2017; LI et al., 2018; DA ROCHA et al., 2024).

In the evaluation of polymer nanocomposites, FTIR plays a crucial role in detecting functional changes. It primarily aims to identify the successful incorporation of added nanomaterials into the base material compared to its original form. Additionally, FTIR detects potential alterations in the chemical composition of the matrix during processing or after various storage times. Moreover, it is capable of identifying impurities that may have been introduced during the manufacturing process, such as residual solvents (BOKOBZA, 2017). For instance, in a study by

Danielle Mariano et al, FTIR was employed to investigate structural and chemical changes in lamellar zirconium phosphate (ZrP) following modification with Jeffamine and the addition of nano-ZnO. The analysis unveiled the formation of new ionic species and the incorporation of ZnO nanoparticles into the material. The spectral region from 4000 to 3000 cm^{-1} exhibited a significant increase in band area, indicating the formation of ionic species and the presence of OH groups on the surface of nano-ZnO. Absorptions attributed to Jeffamine were observed at 3000 to 2500 cm^{-1} and 1500 to 1300 cm^{-1}. Overlapping bands of P-O and C-O-C appeared at 1200 to 900 cm^{-1}, with a new band emerging at 600 to 700 cm^{-1}. Characteristic bands of ZrP and ZnO were maintain or appear following modification. FTIR also detected additional bands and potential impurities, suggesting chemical interactions and the successful modification, resulting in a multifunctional material with potential for polymer composites (MARIANO et al., 2023).

When a team led by Dan Ciprari analyzed the effects of nano-alumina and magnetite in a nanocomposite system of PMMA and PS, FTIR played a crucial role in investigating the interactions between the PMMA matrix and Al_2O_3 and Fe_3O_4 nanoparticles. Using a Nicolet Instrument Corp. Nexus 870 spectrometer. The spectra were collect revealing the formation of carboxylate groups on the surface of the nanoparticles, resulting from the reaction between esterified groups of PMMA and aluminum atoms in the nanoparticles. The technique demonstrated decreases in various peaks compared to the pure polymer matrix, detecting points of CH_3-O hydrolysis and changes in the ionization gradient of C=O, for example. Other changes in the spectra, such as decreased absorbance at 2950 cm^{-1} and the appearance of peaks at 1687 cm^{-1}, indicated the formation of stable chemical bonds. These data not only confirmed the adsorption mechanism of PMMA on the nanoparticle surfaces but also provided valuable insights into the density and structure of the interface in the nanocomposites, crucial for understanding their mechanical and structural properties (CIPRARI; JACOB; TANNENBAUM, 2006).

Thermogravimetric Analysis (TGA) is a widely used analytical technique in the characterization of materials, particularly in studies involving polymers and nanocomposites. This technique provides precise information regarding the thermal stability and decomposition of materials, essential for understanding their physical and chemical properties. During a TGA experiment, the sample is gradually heat while its mass is continuously monitor. Changes in the sample's mass with temperature variation reveal information about decomposition events, volatilization, oxidation, sublimation, desorption, or other transformations occurring as a function of temperature (CANEVAROLO, 2007; MENCZEL; PRIME, 2009; JESKE; SCHIRP; CORNELIUS, 2012; NG et al., 2018; SAADATKHAH, et al., 2019).

In practice, TGA allows for the determination of the initial and final decomposition temperatures of a material, as well as the identification of intermediate stages of thermal degradation.

This is crucial for assessing the thermal stability of polymers, influenced by factors such as chemical structure, processing type, and additives. Additionally, TGA can be employed to quantify the composition of complex materials, distinguishing between polymers and thermally stable fillers or additives. In polymeric nanocomposites, TGA plays a key role in observing changes in the thermal stability of the polymer matrix due to the presence of nanoparticles. It can reveal how nanoparticles affect the thermal decomposition of the polymer, directly influencing its mechanical and thermal properties. Furthermore, TGA assists in determining the quantity and dispersion of nanoparticles within the polymer matrix, which are essential factors for optimizing the performance of the nanocomposite in specific applications such as engineering materials, advanced coatings, and electronic devices (MENCZEL; PRIME, 2009; JESKE; SCHIRP; LOGANATHAN et al., 2017; NG et al., 2018; SAADATKHAH, et al., 2019).

The application of TGA in polymeric nanocomposites is crucial for understanding the thermal stability and composition of these materials across a wide temperature range. This is essential in developing new materials with enhanced properties, especially for products requiring greater heat resistance compared to the base polymer. Even in applications less focused on thermal resistance, TGA plays a significant role in assessing the purity and quality of the final composite, providing direct comparisons with literature data. Despite being a sample-destructive technique, the detailed insights provided by TGA and the small amount of sample required for analysis compensate for this aspect, allowing for precise and reliable characterization of macro, micro, and nanomaterials (CORCIONE; FRIGIONE, 2012; LOGANATHAN et al., 2017; NG et al., 2018).

In the study of clay dispersed Styrene-co-Glycidal Methacrylate impregnated wood polymer nanocomposite (WPNC), TGA was employed to evaluate thermal properties. The results revealed that the nanocomposite exhibited improved thermal stability at temperatures below 450°C compared to other materials studied, including raw wood. This enhancement in thermal stability suggests that the nano-clay plays a crucial role in the thermal resistance of the composites, directly influencing thermal decomposition and residue formation at high temperatures. Furthermore, the quantitative analysis provided by TGA allowed for a precise comparison of the thermal behavior of materials with and without nano-clay, highlighting the importance of this incorporation for applications requiring high thermal resistance, such as construction and electronics (RAHMAN; HAMDAN; CHANG HUI, 2017).

Analyzing an epoxy resin with hexagonal boron nitride nanoparticles, Ahmed A. Thamer and his team observed a slower mass loss as more filler was added, and a third stage of mass loss was detected (THAMER; YUSR; JUBIER, 2019). However, when comparing the characteristics of epoxy/polyester nanocomposites with graphene platelets, Minh-Tai Le and Shyh-Chour Huang did not detect any variation in the mass loss curves beyond the margins of error. This finding proved that in this case, there were no significant changes in the heat resistance of the system (LE; HUANG, 2015).

Differential Scanning Calorimetry (DSC) is a technique that measures the difference in heat flow between a sample and a reference material as they undergo a controlled temperature program. This allows for the investigation of phase transitions such as melting, crystallization, glass transitions, and endothermic or exothermic chemical reactions. From these measurements, thermophysical and kinetic properties of the sample can be determined (CANEVAROLO, 2007; CORCIONE; FRIGIONE, 2012; ABD-ELGHANY; KLAPÖTKE, 2018; SAXENA; SHUKLA; GAUR, 2020)

In nanocomposites specifically, DSC allows for comparing the phase transition temperatures of the pure matrix and its nanostructured forms, with each providing distinct insights. Moreover, DSC is essential for providing detailed information on important thermal events such as the glass transition temperature (Tg), melting temperature (Tm), crystallization temperature (Tc), and the percentage of crystallinity of the nanocomposites. The Tg indicates the mobility of the polymer chains and the compatibility between the components of the nanosystem. Tm and Tc together reveal how nanoparticles influence the crystallization kinetics and the polymer melting process (CANEVAROLO, 2007; CORCIONE; FRIGION, 2012).

Although DSC provides valuable insights into the molecular structure and interactions within nanocomposites, it does have limitations. These include insufficient thermal resolution for closely spaced transitions, limited sensitivity for minor phase quantities, and challenges in interpreting results due to nanocomposite heterogeneity. Additionally, DSC is a destructive technique, and proper sample preparation is crucial to ensure representative and homogeneous analysis. To overcome these limitations, DSC can be complemented with other analytical techniques such as Thermogravimetry (TGA), X-ray Diffraction (XRD), Nuclear Magnetic Resonance (NMR), and Transmission Electron Microscopy (TEM) for a more comprehensive characterization of the material ((VALENTIM; TAVARES; SILVA, 2014; CORCIONE; FRIGIONE, 2012; ANANDHI; EDWARD; JAISANKAR, 2020). These complementary techniques provide additional insights into thermal stability, crystalline structure, chemical composition, and morphological details, enhancing the overall understanding of nanocomposite properties and behavior.

Valentim, Tavares, and Silva's (2014) study investigated the effects of adding nano TiO_2 to ethylene-vinyl acetate copolymer (EVA) through Differential Scanning Calorimetry (DSC) and Thermogravimetry (TG) analyses. The results indicated that incorporating TiO_2 into EVA/TiO_2 nanocomposites led to a reduction in the material's degree of crystallinity (Xc). Additionally, DSC revealed significant changes in thermal properties, such as alterations in glass transition temperature (Tg) and melting enthalpy (ΔHm), while TGA complemented these findings by assessing the thermal stability of the samples. The combination of these analyses provided crucial insights into how modifications in molecular structure and organization influence the performance of nanocomposites, underscoring the importance of precise control over thermal properties for industrial applications such as packaging and photovoltaic encapsulants.

In a 2020 article, a group of Indian researchers conducted thermal analysis of poly(methyl methacrylate) (PMMA), polyvinylidene fluoride (PVDF), polysulfone (PSF), PVDF-PSF blends, and PVDF-PMMA bilayer samples using Differential Scanning Calorimetry (DSC). They attempted to calculate the glass transition temperature (Tg) and interpret corresponding changes observed in the PVDF-PSF blend and PVDF-PMMA bilayer formation. The PVDF-PSF blend resulted in a shift in the Tg of PSF, with a single peak confirming adequate miscibility of the two polymers. The DSC curve of the PVDF-PMMA bilayer also confirmed its formation and demonstrated significant changes in its thermal behavior. This was achievable because DSC is one of the most versatile thermal characterization techniques. It can be applied to polymers and their blends, composites, and multilayers to study various thermodynamic processes such as glass transition temperature, specific heat capacity, and other related kinetic properties, as well as to elucidate the structure, morphology, and crystallinity of polymers (SAXENA; SHUKLA; GAUR, 2020).

In a study conducted by researchers from Maria Inês Bruno Tavares' group on PLA nanocomposites with zinc oxide nanoparticles, an increase in glass transition temperature by up to 5°C compared to pure PLA was observed. This result indicates reduced mobility of the amorphous phase of the material, suggesting greater structural rigidity following the addition of zinc oxide nanoparticles. Similar to the previously mentioned DSC research, which revealed a decrease in crystallinity (Xc) and significant changes in thermal properties such as glass transition temperature (Tg) and melting enthalpy (ΔHm), both studies underscore the importance of precise control over thermal properties for various industrial applications (VALENTIM; TAVARES; SILVA, 2014; DA CRUZ FARIA et al., 2021).

Returning to the analysis of epoxy resin with hexagonal boron nitride nanoparticles by Ahmed A. Thamer and his team, the glass transition temperature (Tg) of the composite, analyzed via DSC, increased dramatically (by almost 50°C) with each incremental increase in nanoparticle percentage up to 1%, where it stabilized. This change was attributed to differences in mobility between the systems and was corroborated by TGA data (THAMER; YUSR; JUBIER, 2019).

X-ray Diffraction (XRD) is a technique used to investigate the crystalline structure of various materials. When X-rays interact with a crystalline material, they are diffracted at specific angles due to the ordered arrangement of atoms in the crystal lattice. These diffraction patterns are used to identify the internal structure types of the sample, providing each material with a unique diffractogram, which represents its crystallographic identity (CANEVAROLO, 2007; BOTAN; SABRINA, 2020).

In polymers, X-rays allow for the characterization of crystalline and amorphous phases, determination of crystallinity degree, and evaluation of defects and imperfections within the internal structure. This technique is crucial for understanding the relationship between molecular structure and physical properties of polymers, aiding in the development of materials with specific characteristics such as increased mechanical strength or improved thermal properties. When direct analysis of the polymer material is not feasible, sample preparation for X-ray Diffraction (XRD) may involve methods like film casting, compression into pellets, or powder spraying to ensure uniformity and adequate thickness. The equipment setup typically includes an X-ray source, a goniometer, and a detector. The source generates a focused X-ray beam directed onto the sample, while the goniometer adjusts the incidence angle and the detector records the diffracted X-rays. The sample is positioned on the goniometer's sample holder, and the X-ray beam is directed at different angles, with diffracted X-rays being recorded by the detector. The intensity data of diffraction are collected over a range of angles and plotted on a graph known as a diffractogram, which displays diffraction peaks corresponding to crystalline planes in the polymer (CANEVAROLO, 2007; CAMPBELL; PETHRICK; WHITE, 2017; BOTAN; SABRINA, 2020; MARIANO NETO; BRITO, 2022).

The analysis of the peaks in the diffractogram allows determining structural characteristics of the polymer, such as degree of crystallinity, crystallite size, and presence of crystalline phases. The amorphous halo in a polymer refers to a broad and diffuse region in the X-ray diffractogram, indicating the presence of disordered or amorphous structure, contrasting with the sharp peaks associated with crystalline regions. This halo is characteristic of polymers with a high proportion of amorphous areas, where polymer chains are not organize in a regular and repetitive pattern. The Bragg's law is fundamental in X-ray diffraction and relates the diffraction angle (θ), the wavelength of X-rays (λ), and the distance between the crystalline planes (d), through the formula $n\lambda = 2d \sin\theta$, where n is an integer representing the order of diffraction. This equation is use to determine the distances between atomic or molecular planes in a crystalline sample, enabling detailed analysis of the internal structure of materials. The position of the peaks (2θ values) is compare with known standards to identify phases, and the peak widths are use to calculate the average crystallite size using the Scherrer equation. Therefore, XRD provides detailed characterization of the molecular structure of polymers, essential for understanding and improving their physical and chemical properties (TJONG, 2006; CANEVAROLO, 2007; CAMPBELL; PETHRICK; WHITE, 2017; MARIANO NETO; BRITO, 2022).

The role of XRD in the characterization of nanocomposites presents multiple valuable aspects. Its advantages include simplicity, reliability, and non-destructive nature, allowing for sample recovery after testing. XRD provides detailed information about the crystalline structure and is versatile, applicable to both bulk materials and thin films. However, it faces limitations when analyzing highly amorphous and semi-crystalline materials, requiring specific preparation and interpreting diffraction patterns with complex halos, which necessitate deconvolution and mathematical treatments (CANEVAROLO, 2007; TJONG, 2006; ABHILASH; RAJENDER; SURESH, 2016; BOTAN; SABRINA, 2020).

A practical example is the use of XRD in the development of polymeric nanocomposites for food packaging, where it aids in enhancing gas barrier properties and optimizing formulation to maximize nanoparticle dispersion and orientation. This results in improved barrier properties and mechanical strength. The dispersion of nanoparticles within the matrix affects the peaks detected in the final nanomaterial. Generally, if peaks attributed to the relatively small quantities of added nanoparticles are not found, it suggests good distribution and dispersion, indicating that nanoparticles have not aggregated. This technique's outcome is also utilized to detect whether amorphous regions were introduced into the matrix during incorporation (TJONG, 2006; ABHILASH; RAJENDER; SURESH, 2016; BOTAN; SABRINA, 2020; DA ROCHA; DA SILVA; TAVARES, 2022; DA ROCHA et al., 2024).

Dillip K. Pradhan et al. (2018) investigated polymer-clay electrolyte nanocomposites composed of polyethylene oxide (PEO) and potassium iodide, with varying concentrations of organically modified sodium montmorillonite clay (DMMT). X-ray diffraction (XRD) analysis was use to examine the formation of the nanocomposites and changes in the structural properties of the materials. The XRD patterns of the nanocomposites closely resembled the characteristics of the clay particles as more DMMT was add, confirming proper formation of the nanocomposites with this modified form.

In another recent study in the field, BARAMA et al. (2023) employed XRD technique to investigate the structural properties of aluminum nanocomposites reinforced with 1.5% multi-walled carbon nanotubes (MWCNTs). Through diffraction analysis, it was observed that the interaction between aluminum and MWCNTs resulted in the formation of carbides and carbon dissolution into the crystalline lattice of the matrix. These reactions also contributed to grain refinement in the matrix structure and a more homogeneous distribution of residual MWCNTs within the composite material. Furthermore, XRD revealed changes in the crystalline structure of the material, indicating alterations in the crystalline phase and orientation of aluminum crystals due to the presence of nanotubes. These observations suggest that the incorporation of nanotubes altered the structure and crystalline organization of the aluminum matrix, which is crucial for understanding how nanocomposites can enhance mechanical and thermal properties compared to pure aluminum. Thus, XRD played a critical role in elucidating the structural changes induced by the addition of MWCNTs in aluminum nanocomposites, providing valuable insights into the formation of new phases, grain refinement, and distribution of nanotubes within the matrix.

Nuclear Magnetic Resonance (NMR) exploits the magnetic properties of atomic nuclei in a magnetic field, allowing for the investigation of molecular structure and interactions in both solid and liquid materials. Radiofrequency pulses cause atomic nuclei to absorb energy and emit detectable signals, providing information about molecular structure, chemical connectivity, and molecular dynamics. In solids such as polymers and nanocomposites, high-field NMR offers high spectral resolution and significant sensitivity, enabling detailed atomic-level analyses. Low-field NMR, while offering lower resolution, is useful for preliminary studies and screening due to less complex equipment and reduced operational costs. NMR is also crucial in nanomaterials, examining changes in dynamics and physical interactions following nanoparticle addition. It stands out as a rapid, non-destructive, and informative method for various scientific and industrial applications (AVOLIO et al., 2010; ALAM; JENKINS, 2011; DOS SANTOS ALMEIDA et al., 2012; BATOOL; MUHAMMAD; JAVED, 2022).

In polymeric materials, high-field NMR can characterize morphology, chemical composition, and molecular mobility. For instance, it can distinguish between crystalline and amorphous phases, identify functional groups, and analyze the distribution of polymer segments within the matrix. The technique provides insights into atomic connectivity and local organization of the polymer chain, which are crucial for understanding properties such as chain flexibility and formation of structural domains. On the other hand, low-field NMR is commonly used for liquid samples, offering dynamic analysis of molecular interactions in solutions and dispersions. In liquid polymers or nanocomposite solutions, for example, low-field NMR can elucidate solvent structure, charge distribution, and kinetics of polymer-polymer or polymer-nanoparticle interactions. It serves as a valuable tool for studying the dynamics and behavior of polymers in solution phases, providing essential information for various scientific and industrial applications (ALVES et al., 2020; BALTHAZAR et al., 2021; MERAT et al., 2023; RAMOS et al., 2023).

In the time domain, NMR provides insights into molecular dynamics by analyzing the relaxation times of atomic nuclei. Longitudinal (T1) and transverse (T2) relaxation times reflect molecule mobility and intermolecular interactions. In polymers, these measures can indicate chain flexibility, the presence of defects, and the influence of additives or nanoparticles on molecular mobility. Time domain analysis complements information obtained in the frequency domain, providing a comprehensive view of the dynamic properties of the materials under study (AVOLIO et al., 2010; ALAM; JENKINS, 2011; ALVES et al., 2020; BATOOL; MUHAMMAD; JAVED, 2022).

Typical NMR equipment includes a magnetic magnet and a detection system. The sample is placed in the magnetic field of the magnet, where atomic nuclei align themselves with the magnetic field. Application of radiofrequency pulses induces resonance in the nuclei, which emit signals that are detected and analyzed to generate spectra. In polymers, NMR can distinguish different chemical regions and provide information on molecular dynamics such as rotational movements and molecular diffusion. NMR also has its limitations, such as the requirement for relatively pure samples and dependence on the sensitivity of the specific nucleus (e.g., ^{1}H, ^{13}C) (AVOLIO et al., 2010; ALAM; JENKINS, 2011; ALVES et al., 2020; BATOOL; MUHAMMAD; JAVED, 2022).

Proper sample preparation and equipment programming are crucial to avoid artifacts and ensure accurate results in the characterization of polymers and their nanocomposites. The choice of NMR pulse sequence depends on the desired information about the sample and the properties of the observed nuclei. Firstly, the type of nucleus is considered, such as ^{1}H or ^{13}C, which require different frequencies and parameters. Next, the type of information needed is defined: 1D spectroscopy for basic chemical structure information, 2D for spin coupling details, relaxometry for studying molecular dynamics, or diffusion for investigating molecular mobility. The sample state also influences the choice: for solids, techniques like Cross Polarization/Magic Angle Spinning Nuclear Magnetic Resonance (CP/MAS) are use to enhance sensitivity and resolution; in liquids, standard sequences such as Single Pulse (SP) or Inversion Recovery (IR) are applied. Depending on the objectives, sequences like Heteronuclear Single Quantum Coherence (HSQC) or Heteronuclear Multiple Bond Correlation (HMBC) are chosen for detailed atomic connectivities, or CPMG for studying molecular dynamics. Parameters such as pulse duration and intensity are adjust to optimize sensitivity and resolution. Examples include Single Pulse for basic spectra, Inversion Recovery for measuring T_1 relaxation times, and CP/MAS in solids for improved resolution. The final choice involves knowledge of the sample, desired information, and the capabilities of the NMR equipment (ALVES et al., 2020; AVOLIO et al., 2010; ALAM; JENKINS, 2011; BALTHAZAR et al., 2021; MUHAMMAD; JAVED, 2022; MERAT et al., 2023; RAMOS et al., 2023).

Transmission Electron Microscopy (TEM) is a powerful technique employed across various scientific fields, including physics, chemistry, and biology, to obtain high-resolution images and analyze the internal structure of materials at the atomic scale. The technique relies on transmitting a beam of electrons through an ultra-thin sample. Electrons interact with the sample, producing high-resolution images that reveal the internal structure with subnanometer detail. TEM is particularly useful for studying the morphology, size, and distribution of nanoparticles, as well as the crystalline structure, defects, interfaces, and chemical composition of materials in general (WILLIAMS; CARTER, 2009; REIMER, 2013; EGERTON, 2016).

When applied to solid materials such as polymers, TEM provides high-resolution images that reveal molecular organization, the presence of crystalline and amorphous phases, as well as the dispersion and orientation of nanoparticles within the polymer matrix. The technique can distinguish between different phases within a material and examine defects in detail, such as clusters or disordered areas, which can influence the mechanical and thermal properties of the material. For liquid samples or solutions, TEM can be adapted to cryo-TEM (cryogenic TEM), allowing visualization under low-temperature conditions while preserving the native structure of the samples. This adaptation is particularly useful for studying biological samples or materials sensitive to conventional TEM preparation techniques (GOLDSTEIN et al., 2003; SUGA et al., 2014).

In a typical transmission electron microscope, an electron gun generates a beam of electrons that is accelerated to a high voltage. The electron beam passes through a series of electromagnetic lenses that focus it onto an ultra-thin sample (approximately 100 nm thick) mounted on a specialized sample holder. The electrons are accelerate by an electromagnetic field within the sample holder. Electrons that transmit through the sample are scattered according to its atomic structure and are collect by a detector, enabling the formation of a detailed image on a high-resolution electron detector. In addition to revealing crystalline structure, defects, and interfaces in the material, TEM can be equipped with an Energy Dispersive X-ray Spectrometer (EDS) and an Electron Energy Loss Spectrometer (EELS). These additions provide detailed information about the elemental composition and chemistry of materials. EDS detects X-rays emitted from the sample when it is bombard by electrons, allowing for qualitative and quantitative elemental analysis. EELS measures the energy loss of electrons as they interact with the sample, providing information about the chemical bonding and electronic structure of the material. Together, these capabilities make TEM a versatile tool for studying materials at the atomic level, offering insights into their structural and chemical properties with exceptional resolution and detail (WILLIAMS; CARTER, 2009; REIMER, 2013; EGERTON, 2016).

TEM offers several significant advantages. The technique allows for atomic resolution imaging, providing a detailed view of the internal structure of materials. Moreover, TEM can provide information about the chemical composition and atomic-level crystalline structure. However, sample preparation is a critical and challenging aspect. Samples must be extremely thin, typically ranging from tens to hundreds of nanometers, to allow electron transmission. Careful sample preparation is essential to avoid damage and artifacts that can compromise image quality. Additionally, TEM requires a high vacuum environment and complex equipment, which may limit its use in some applications (WILLIAMS; CARTER, 2009; REIMER, 2013; EGERTON, 2016).

In the evaluation of polymeric nanocomposites, TEM is utilize to investigate the size, distribution, and orientation of nanoparticles embedded within the polymer matrix, as well as to analyze the crystalline structure and defects at interfaces. For instance, a study by Li et al. employed TEM to probe the internal structure of poly(lactic acid) nanocomposites reinforced with carbon nanotubes. TEM images revealed that the carbon nanotubes were well-disperse in the polymer matrix, forming an interconnected network that significantly enhanced the mechanical and thermal properties of the nanocomposite. Detailed analysis of the interfaces between the carbon nanotubes and the polymer matrix unveiled strong adhesion, which contributed to the material's improved properties. (LI et al., 2021).

Revisiting the experiment on the characterization of alumina and magnetite nanocomposites in PMMA/PS led by Dan Ciprari (2006), the obtained results differed significantly from literature and supplier data, indicating a high normal distribution of sizes and a strong flocculation phenomenon. Analyzing this distribution, a mathematical formula was employ to estimate other relevant data of the nanocomposite such as cluster size and anchor number.

In an epoxy matrix with titanium nanotubes used for anti-corrosion coating, Muddasir Nawaz et al. (2019) employed TEM to analyze the morphology of these nanotubes, confirming their hollow structure and validating their loading with dodecylamine, a stabilizer.

In another example, Huang et al. used TEM to investigate the morphology and crystalline structure of polyethylene nanocomposites reinforced with silica nanoparticles. TEM images revealed that the silica nanoparticles were uniformly distribute within the polyethylene matrix, forming a homogeneous structure. Detailed analysis of the crystalline structure showed that the silica nanoparticles induced the formation of polyethylene crystals with specific orientation, thereby enhancing the mechanical and thermal properties of the nanocomposite (HUANG et al., 2023).

Scanning Electron Microscopy (SEM) is a widely used technique in materials science, biology, and engineering to investigate the morphology and composition of surfaces at the nanoscale. Based on the interaction of an electron beam with the sample surface, SEM generates and collects various signals, including secondary electrons, backscattered electrons, and characteristic X-rays, to form detailed images of the surface by scanning it. These high-resolution images reveal the topography, composition, and electrical properties of the analyzed surfaces. Particularly useful for analyzing solid surfaces such as polymers and nanocomposites, SEM provides detailed three-dimensional images showing surface structure, particle size, porosity, and the distribution and orientation of dispersed materials. When studying polymers, SEM allows direct visualization of the morphology of crystalline and amorphous phases, the distribution of additives or nanoparticles, and the presence of surface defects. The technique is essential for examining surface roughness, texture, or bulk fractures, providing crucial information to understand properties such as adhesion, wear resistance, and moisture behavior of materials (GOLDSTEIN et al., 2003; REIMER, 2013; WILLIAMS; CARTER, 2009).

A typical scanning electron microscope consists of components such as an electron gun (source), electromagnetic lens system, column, scanning system, sample chamber, secondary electron detectors (SE), backscattered electron detectors (BSE), energy dispersive X-ray detector

(EDS), vacuum control system, computer and control software, and cooling system. The electron gun generates a beam that is accelerated towards the sample. The electron beam is focused and directed onto the sample surface by a series of electromagnetic lenses. During interaction with the surface, electrons generate various signals. Secondary electrons are emitted from the surface and collected by a secondary electron detector, producing a high-resolution image of the surface topography. Backscattered electrons are reflected from the surface and collected by a backscattered electron detector, providing information about the sample's composition. Additionally, electron-sample interactions can generate characteristic X-rays, collected by an energy dispersive detector, allowing elemental analysis of the sample composition via EDS (GOLDSTEIN et al., 2003; REIMER, 2013; WILLIAMS; CARTER, 2009).

SEM offers several significant advantages, such as obtaining high-resolution images with notable depth of field, providing a detailed view of surface morphology. Additionally, it is capable of analyzing a wide range of materials, including polymers, metals, ceramics, and biological materials. Elemental analysis with EDS complements surface imaging by allowing identification and quantification of elements present in the sample. However, sample preparation is crucial for obtaining good results. Conductive samples can be analyzed directly, whereas non-conductive samples often need to be coated with a thin layer of conductive material, such as gold or carbon, to prevent surface charging. Furthermore, SEM requires a high vacuum environment, which may limit analysis of materials that cannot withstand these conditions (HAQUE, 2002; GOLDSTEIN *et al*., 2003; REIMER, 2013).

In the evaluation of polymeric nanocomposites, SEM is used to analyze the dispersion and distribution of nanoparticles within the polymer matrix, to investigate the morphology of the matrix-nanoparticle interfaces, identify agglomerates, and verify the integrity of the surface layer. The technique also allows observation of morphological changes induced by manufacturing processes or thermal treatments, providing insights into the impact of these processes on the structure and properties of the material (HAQUE; SAIF, 2002; SUGA *et al*., 2014).

A study conducted by Silva et al. (2022) utilized SEM to investigate the dispersion of clay nanoparticles in a polypropylene matrix. SEM images revealed that the nanoparticles were well dispersed within the matrix, forming a homogeneous structure. Furthermore, EDS analysis confirmed the presence of characteristic elements from the clay nanoparticles in the polymer matrix, providing a detailed understanding of the composition and morphology of the nanocomposite.

Returning to the epoxy matrix with titanium nanotubes studied by Muddasir N. et al., the tubular shape of the nanotubes and surface porosity of the nanostructure as a whole were confirmed through SEM analysis. Additionally, an analysis of the quality and presence (or absence, in this case) of major defects in the morphology of the matrix at a larger scale (NAWAZ et al., 2019).

In another example, Zhang et al. (2017) utilized SEM to investigate the morphology of polyurethane nanocomposites reinforced with graphene oxide nanoparticles. The micrographs showed that the graphene oxide nanoparticles were well distribute within the polyurethane matrix, forming an interconnected network that significantly enhanced the mechanical properties of the nanocomposite. Detailed analysis of the morphology of the interfaces between the nanoparticles and the polymer matrix revealed strong adhesion, contributing to the material's improved properties.

FINAL CONSIDERATIONS

The multimethod review of analytical techniques has provided a comprehensive and detailed understanding of polymeric nanocomposites, yielding fundamental insights and conclusions regarding methodologies that describe their properties and practical applications. This study underscores the importance of an integrated approach in assessing complex materials like nanocomposites and emphasizes the ongoing need for advancements in this field to meet growing demands for high-performance materials.

Specifically highlighted in the literature is the use of Fourier Transform Infrared Spectroscopy (FTIR) for identifying functional groups and qualitatively analyzing the chemical composition of nanocomposites. Thermogravimetric Analysis (TGA) enables the determination of thermal stability and composition, along with thermal degradation analysis of nanocomposites. Differential Scanning Calorimetry (DSC) is widely used to study thermal transitions such as melting, crystallization, and glass transitions, as well as to determine heat capacity. X-ray Diffraction (XRD) can be employ to analyze crystalline structure, degree of crystallinity, and crystal orientation in nanocomposites. Nuclear Magnetic Resonance (NMR) allows for investigation of molecular structures and dynamics of polymer chains, including intermolecular interactions. Lastly, scanning electron microscopy (SEM) is frequently use in various studies to analyze surface morphology and microstructure of nanocomposites, providing high-resolution images of surface topography, while transmission electron microscopy (TEM) handles detailed observation of morphology and internal structure, including charge distribution.

The different combinations of these techniques reveal how the chemical, thermal, structural, and morphological properties of nanocomposites interact and influence the final material performance. This is crucial for the development and optimization of nanocomposites with specific desired properties for diverse applications.

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