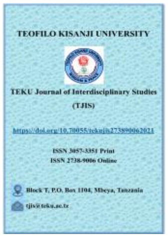
TEOFILO KISANJI UNIVERSITY

TEKU Journal of Interdisciplinary Studies (TJIS)

ISSN 3057-3351 (Print) & ISSN 2738-9006 (Online)

Journal Homepage: https://www.teku.ac.tz/tjis.php



Review Paper

A review on Characterization Methods and Properties of Polymer Nanocomposite Materials

Gatawa Biswalo Gatawa[1] & Suranjana V Mayani[2*]

[1] *Faculty of Science and Technology, Teofilo Kisanji University, P.O. Box 1104, Mbeya, Tanzania*

[2] *Department of Chemistry, Marwadi University, Rajkot-Morbi Road, P.O. Gauridad, Rajkot 360003, Gujarat, India*

**Correspondence: suranjana.mayani@marwadieducation.edu.in*

ARTICLE DETAILS

Received: 12-Oct-24
Revised: 30-Jan-25
Accepted: 20-Feb-25
Published online: 30-Mar-25

DOI: 10.70055/TJISV4I2A01

ABSTRACT

One essential component of polymer nanotechnology is the fabrication of polymer nanocomposite materials. Owing to the increased interest in tiny composite materials, it is crucial to characterize these materials' molecules in order to comprehend their characteristics and create new ones. The creation of polymer nanocomposite materials is quickly becoming a multidisciplinary area of study in materials science, with potential benefits for expanding the uses of polymers across numerous industries. Many techniques are demonstrated in this review. These techniques including X-ray diffraction, Raman spectroscopy, Fourier transform infrared microscopy (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), nuclear magnetic resonance spectroscopy, ultraviolet visible spectrophotometry, X-ray photoelectron spectroscopy, thermogravimetric analysis, fluorescence spectroscopy, as well as energy dispersive X-ray spectroscopy. Furthermore, the characterization of polymer tiny composite materials, a synopsis of the preparation process will be provided. Lastly, this review highlights different properties of PNCs.

Keywords: *Polymer Nanocomposite; Characterization; Spectroscopy; Microscopy; Properties*

1. Introduction

This paper will provide an outline of the characterization of polymer nanocomposite materials. I'll begin by defining nanotechnology, composite materials and polymer tiny composites and giving a brief description of their synthesis. It is expected that the reader is familiar with typical analytical procedures for characterization of PNCs such as thermal analysis, electron microscopy, plus x-ray diffraction and spectroscopic methods (Armstrong, 2015).

1.1 Nanotechnology

Nanotechnology is a term which may be deceptive because it refers to more than one technique or scientific field. Nanotechnology, on the other hand, refers to a set of methodologies, substances, applications, and concepts distinguished by scale (Thostenson et al., 2005). Nanotechnology is engineering at the atomic or molecular scale. It is an umbrella term that encompasses a wide range of discoveries, processing methods, and measurements relating to the smallest sizes of matter manipulation (one to hundred nanometres). Nanotechnology focusses on the creation of particles and materials at the nanoscale. These materials and particles possess unique and mysterious properties that are lacking from normal bulk materials (Khan, Hamadneh, & Khan, n.d.).

1.2 Composite Material

A composite material is defined as a synthetic substance made up of multiple stages. These phases differ chemically, and the material's chemical phases make clear contact. The composite material is comprised of 2 phases. These are, continuous phase known as matrix as well as the reinforcement phase which is scattered inside it, as seen in figure 1. Composite plus tiny composite materials have been made with a variety of matrices, including polymers, carbon, metals, and ceramics, as well as reinforcements including particles, fibres, and layered materials (Armstrong, 2015). Nanoreinforcement is a term which commonly used in nanocomposite materials to define a tiny filler which advances a matrix's mechanical characteristics. Nanofillers can also be used to offer nonconductive polymers electrical properties, boost wear resistance or toughness, and improve thermal resistance or barrier characteristics (Rallini & Kenny, 2017). The term matrix usually refers to the surplus component in the dispersion medium. Filler, on the other hand, is an additional component in the form of the scattered phase (Properties & Materials, 2007). Tiny composites are typically created when fillers are provided ex-situ or synthesized in-situ with particle sizes ranging from 1 to 100 nm along at least one dimension. The discovery of carbon nanotubes in 1991 spawned the study of nanocomposites, which exhibit extraordinary properties (Armstrong, 2015).

Dong et al. (Dong et al., 2017) developed a polymeric tiny composite based on carbon nanotubes and chitosan for drug delivery that is activated by infrared light. Tinny fillers have differing features from their bulk form. When the trait reaches the nanoscale, it may exhibit an entirely opposite behaviour. Polymeric nanocomposites' properties are affected by the synthesis process, the type and distribution of nanofillers, the degree of filler and matrix intermixing, the interfacial bond, the quantity and characteristics of tiny fillers, the nature of the advanced interphase, the size as well as the shape of the tiny material, plus the system morphology (Maron et al., 2018). Nanocomposites can also be found in natural compounds such as proteins, lipids, also carbohydrates. These are examples of nanocomposite materials, which are formed by meshing various phases together to form particles, layers, or threads, with one of the phases measuring in the nanometer range (Puggal et al., 2016).

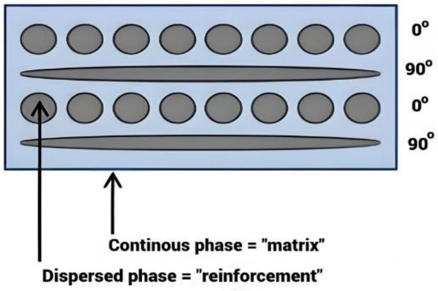


Figure 1. A typical fibre-reinforced composite with continuous phase (matrix) as well as the reinforcement phase which is scattered inside it (Armstrong, 2015).

1.3 Polymer Nanocomposite Materials

Polymer nanocomposite materials are one of the greatest noticeable areas of recent research as well as development in nanotechnology, with the exploration area covering extensive assortment of difficulties. This would take in drug delivery systems using nanocomposite materials, reinforced PNCs, nanoelectronics, and polymeric bionanomaterials. Based on theoretical assumptions, PNCs aim to build a very broad interface between neat polymer macromolecules and nanoscale heterogeneities. The close interaction between macromolecules and tiny particles is expected to provide distinct characteristics when compared to normal microfilled polymer composite materials. Large reinforcement and improvement of other features, such as reduced flammability and increased conductivity, are typically obtained in nanocomposite materials with low tiny filler concentration. These effects, however, are heavily reliant on the hard to attain homogeneous spreading of the nanoadditive inside the polymer medium known as matrix (Pielichowski & Pielichowska, 2018). Over last few decades, the creation of novel PNCs has attracted a growing amount of global scientific attention. PNCs differ from typical composite materials in that they contain fillers smaller than 100 nm. The advantage of polymer tiny composite is that it can improve the intrinsic mechanical capabilities, processability, and light weight of the pure polymer while preserving its inherent properties (Verdejo et al., 2011). The size and properties of the nanofiller, as well as the interface between it and the matrix, are critical components of PNC behaviour and design (Galpaya et al., 2012). It is possible to combine the matrix and nanofillers or perform a chemical reaction between them to alter how they interact. Polymer matrices are extremely efficient because to their large surface area, adaptive in addition appropriate surface chemistry, acceptable mechanical strength, and homogenous pore size. They can also be reused repeatedly with little effort (Ferreira et al., 2018). When these nanocomposites are used for a specific purpose, their qualities improve even more. Nano-sized fillers outperform macro- and micro-sized fillers due to their improved interface performance. To this point, metallic, carbon-based, as well as ceramic tiny fillers have been used as fillers (Shen et al., 2010). However, because tiny fillers agglomerate, PNCs are less stable at low pH levels. Since the tiny materials in nanocomposite materials are not uniformly distributed and have properties that distinguish them from the matrix, they usually display anisotropic effects. One advantage of employing polymeric nanocomposite materials over traditional polymer composite materials is that they use less nanofiller, which performs better than using more micro-fillers. As a result, the manufactured tiny composite will be lighter in weight. PNCs are composed of polymer matrices with uniformly distributed organic and inorganic fillers at the nanoscale (Sharma & Kaith, 2020). Many polymeric materials exhibit consistent physical and chemical properties, as well as excellent dielectric properties. These qualities influence the applications for polymers. Polymeric materials can be treated with various inorganic fillers to alter their electrical, mechanical, and other operational properties. The type, shape, component concentration, and flow technique of the filler's introduction into the matrix all influence how a specific property changes. Nanoscale fillers are used to create PNCs with properties that are not specific to bulk materials (Wang & Herron, 1991). PNCs are used in several industries, including food packaging, electronics, water purification adsorbent materials, scaffolds for antimicrobial wound dressings, as well as biosensors (Sharma & Kaith, 2020).

Different preparation methods including ultrasonication-assisted solution mixing, shear mixing, melt intercalation as well as in-situ polymerization followed by different characterization techniques of polymer nanocomposite materials are described in this work (De La Cruz-Montoya & Rinaldi, 2010). Also, various properties of polymer nano composite materials are described in this review.

2. Fabrication of Polymer Nanocomposite Materials

To date, many approaches have been employed to synthesize polymer nanocomposite materials. The most significant need is the nanomaterials' homogeneous dispersion throughout the medium known as matrix. Because tiny fillers tend to clump together to produce huge particles, it is necessary to make uniform these materials within a medium called matrix to inhibit the combination two particles to form a large-sized particle (Sharma & Kaith, 2020). The methods that are commonly used have been outlined below.

2.1 Ultrasonication-Assisted Solution Mixing Method

Solution mixing is the simplest method for creating polymer nanocomposite materials (Marques et al., 2016). In this procedure, nanofillers are first dissolved in specific solvents before being mixed with polymer solutions. Ultrasonication facilitates the distribution of tiny fillers in polymer media known as matrices by applying ultrasonic drive to tiny filler packs in solvent or polymer solutions. The main premise is this, when high-frequency ultrasonic waves pass through the packs or agglomerates of tiny fillers, they get disturbed. These waves release more energy than the collaboration energy between tiny fillers in agglomerates, causing outer layers towards separation from the agglomerates and form smaller bundles (Konduru et al., 2015). As sonication time increases, these smaller packs of tiny fillers gradually exfoliate into even smaller ones, eventually resulting in a homo-dispersed state in which distinct tiny fillers are totally isolated from one another in polymeric medium. Once adequate distribution commences, the solvent is vaporized to produce tiny composites (Tamayo et al., 2018). Because of its ease of manufacture, this technique is suited for hasty screening of novel tiny materials for tiny composite applications (Stankovich et al., 2006).

2.2 Shear Mixing Method

Though ultrasonication is a versatile method for dispersing tiny fillers in the laboratory, it is not suitable for industrial use as it is invariably causes some damage to the nanofillers (Paton et al., 2014). On the other hand, this method is less labor-intensive also has the prospective to be scaled up for industrial manufacturing. The magnetic stirring is the most popular and basic shear mingling configuration. The agitator revolving within the solution with tiny filler creates cut forces that cause nanofiller agglomerates to separate. Extensive stirring of tiny fillers/polymer solutions can accomplish some level of dispersion, however reagglomerations are frequently observed after some period of dispersal. Low cut force created by a magnetic agitator, which is restricted by its form plus rotational speed causes this. As a result, stronger cut forces are required to create greater shear energies and hence extra efficiently disseminate tiny fillers. Figure 2 depicts a laboratory as well as industrial scale shear mixer (Akpan et al., 2018).

Figure 2. An illustration of industrial scale shear mixer (Akpan et al., 2018).

2.3 Melt Intercalation Method

Melt intercalation is regarded as an environmentally beneficial and far superior substitute for solution fraternization, if permitted. Though, processing circumstances, filler surface adjustment, and filler plus polymer matrix compatibility all have an impact on how successfully the dispersion may be achieved. The entropy loss accompanied with the detention of a polymer melt is recompensed by the entropy gain related with layer parting and the higher conformational energy of aliphatic chains of alkylammonium cations. (Fawaz & Mittal, 2015).

2.4 In-Situ Polymerization Technique

In-situ polymerization has several advantages. First and foremost, thermo-plastic and thermoset-based nanocomposite materials may be synthesized using in-situ polymerization method (Lü et al., 2003). Additionally, it permits the polymers to graft of onto the filler surface that can mend the overall characteristics of the finished composite material. This approach allows for partially exfoliated structures because of good distribution as well as intercalation of fillers in the polymer medium. According to (Abedi & Abdouss, 2014), this method superlative in creating polyolefin/clay nanocomposite materials as it does not have strict thermodynamic requirements as other methods do. The tiny filler must be appropriately disseminated in the monomer solution before the polymerization process begins to ensure that the polymer forms between the nanoparticles. Polymerization can be initiated using a variety of methods such as heat, a suitable initiator, etc. (Fawaz & Mittal, 2015).

3. Characterization of Polymer Nanocomposite Materials

The customarily techniques used in characterizations of PNCs are X-ray diffraction, scanning electron microscopy (SEM), transmission electron microscopy (TEM), (AFM) in long-term atomic force microscopy, Fourier transform infrared microscopy, Raman spectroscopy, nuclear magnetic resonance spectroscopy, ultraviolet visible spectrophotometry, X-ray photoelectron spectroscopy, energy dispersive X-ray spectroscopy, fluorescence spectroscopy as well as thermogravimetric investigation. These techniques are described below.

3.1 X-Ray Diffraction Spectroscopy (XRD)

The XRD apparatus, which consists of a detector, a goniometer for sample positioning, and an X-ray source (usually Cu Kα), is filled with a powdered or thin-film sample. After exposing the sample to X-ray radiation, the diffraction patterns those results is captured when the angle of the X-ray beam is changed. The crystalline structure, phase composition, and crystallinity of the nanocomposite are all revealed by this pattern. The size and distribution of nanoparticles inside the polymer matrix, as well as the identification of crystal phases, may be determined by analyzing the peaks (Abhilash et al., 2016). X-ray diffraction is a most and an effective tool for analyzing nanomaterials. Because X-rays have an atomic wavelength, they are an excellent tool for investigating the structure of nanomaterials. This technique for characterization of PNCs is an essential method for characterizing crystalline materials such as polymers, as illustrated in Figure 3 (R. Sharma et al., 2012). The crystalline as well as amorphous structure of PNCs (polymers, composites, plus fillers) has traditionally stood examined using this technique. X-ray diffraction spectroscopy has grown in popularity due to its easiness, steadfastness, quantitative information, as well as non-destructive nature. This approach has remained applied in investigating the arrangement of bulk also thin film crystalline or poly-crystalline materials at atomic level. Also, elemental content and crystal structure of biological substances such as deoxyribonucleic acid, vitamins, proteins, and drug production are determined using X-ray diffraction. X-ray scattering offers structural evidence at 3 length scales, including one, ten, as well as hundred nm, by conducting scattering studies at large, small, as well as ultra-small angles, one-to-one. Crystalline materials including PNCs have an episodic organization of atoms. When treated using fixed wavelength X-rays, the electrons interact with the radiation then oscillate because of elastic collision. To determine the desired set of crystal characteristics, diffraction peaks are measured, noted, and examined (Abhilash et al., 2016). The wide-angle X-ray diffraction pattern make known the crystallinity as well as lattice structure of the tiny composite. Small-angle X-ray scattering (SAXS) is commonly employed to examine structures 10 Å or larger (Pandey et al., 2017).

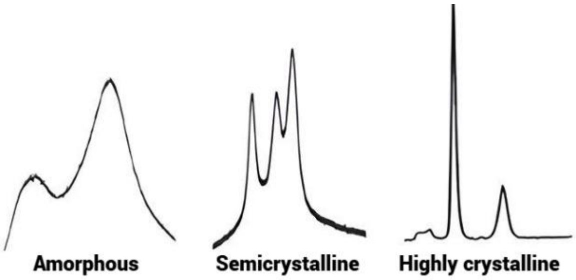


Figure 3. Distinctive peaks for diverse polymer phases a) Amorphous b) Semicrystalline c) Highly crystalline (Abhilash et al., 2016).

3.2 Scanning Electron Microscopy (SEM)

Several essential elements make up the experimental setup for Scanning Electron Microscopy (SEM), which is used to characterize polymer nanocomposites. A conductive coating, usually made of gold, is applied to a sample after it has been chopped or fractured to reveal its underlying structure and stop charging. The SEM chamber, which runs at a high vacuum, is where the sample is put. High-resolution pictures are created by scanning the surface with an electron beam and detecting secondary electrons released by the sample (Dikin et al., 2006). Scanning electron microscopy picture examination of material such as PNC discloses its numerous morphological along with mechanical characteristics (Kundu et al., 2015). The SEM image of composite shows observable morphological structures such as clusters of its essential particles that are immiscible through the material base. These structures are typically described in expressions of their shape as well as size. By properly analyzing scanning electron microscopic images, we can more correctly measure the shape also size of morphological structures. PNC is made up of a polymer medium called matrix and a strong strengthening phase (filler). A PNC is a polymer matrix reinforced with tiny particles. A general conclusion has been obtained that nanocomposite materials have significantly better mechanical characteristics than micro-sized comparable systems (Park & Jana, 2003). Due to their tiny size, tiny particles have a great surface in ratio to volume as well as high energy surfaces (Kundu et al., 2015). PNC theory forecasts that increased bonding between polymer and filler results in better-quality mechanical characteristics (Mittal, 2009). As an example, figure 4 depicts two pictures of a tiny composite produced by SEM (Rallini & Kenny, 2017).

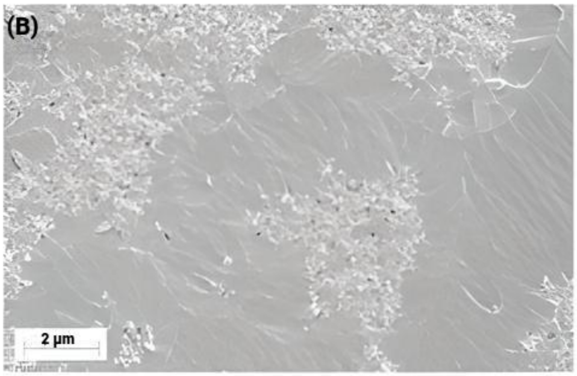


Figure 4. Scanning electron microscopic image of the fracture surface of an epoxy/alumina tiny composite (Rallini & Kenny, 2017).

3.3 Transmission Electron Microscopy (TEM)

TEM has shown to be an extremely strong instrument for examining and exploring the structure of tiny materials (Asadabad & Eskandari, 2015). TEM refers to a microscopy method whereby an electron beam is sent via an ultra-thin specimen and interacts as it moves over the material. The electron beam is fixated on strong magnetic fields, then the electrons follow a coiled route. Electrons conveyed over the material are enlarged as well as fixated on an objective lens to produce the image, which is demonstrated on an imaging screen. The scattering size of tiny particles is a critical as well as major consideration for their use in several industries. The best technique to understand the size of tiny particle materials as well as particle size homogeneity is through transmission electron microscopic pictures. Figure 5 displays a transmission electron microscopic picture of oxide tiny particles. The figure shows a high-quality synthesis method for producing nanoparticle materials. The transmission electron microscopic image clearly confirms the production of tiny particles. A size distribution can be derived for polydisperse particles by measuring their diameters with the eye as well as hand approach. Size spreading can be obtained through the Image Tool software. The size spreading of the tiny particles is an important parameter in production. Image investigation of transmission electron microscopic images can forecast a histogram of size scattering. The histogram of copper (II) oxide tiny particle production is depicted in Figure 6. The histogram for fifty copper (II) oxide tiny particle material is plotted. The average size of the tiny particles is nearly twenty-two point five eight nm, and the majority of the tiny particles are in twenty-two to twenty-four ranges in this histogram. Over time, the transmission electron microscopy has grown into a highly complex device with numerous applications in several scientific areas. Because the transmission electron microscopy has a supreme capability to provide structural as well as chemical evidence over assortment of length scales down to the level of atomic scopes, it has evolved into an essential implement for understanding the characteristics of tiny structured materials also deploying their behaviour (Asadabad & Eskandari, 2015). Transmission electron microscopy provides a qualitative insight of the interior structure, latitudinal dispersal of numerous phases, also views of faulty structures by directly visualizing PNCs, and in certain cases individual atoms (Pandey et al., 2017).

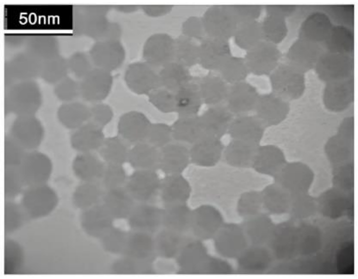


Figure 5. Transmission electron microscopic image, high homogeneity of copper (II) oxide tiny particles in size (Asadabad & Eskandari, 2015).

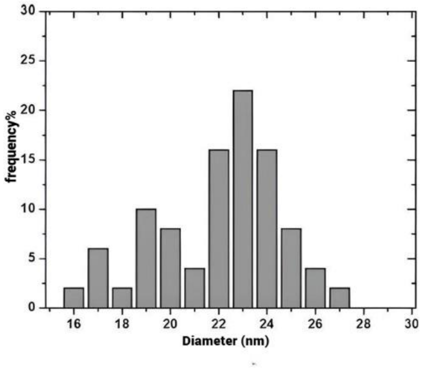


Figure 6. The size distribution of copper (II) oxide tiny particles produced by chemical method (Asadabad & Eskandari, 2015).

3.4 Atomic Force Microscopy (AFM)

Comprehensive study of the interaction concerning various polymer molecules as well as the materials they come into contact with during use has been made possible by recent advancements in our thoughtful of the fundamental mechanisms during atomic force microscopy measurements. The ability to forecast a material's behaviour during use makes this knowledge crucial during the polymeric material development process. The utilization of well-ordered surroundings during measurement allows the modelling of the precise settings one wishes. Atomic force microscopy has been used in the field of polymer sciences to quantify the following: molecular rigidity of hyper-branched large molecules, abrasion of solitary polymers on surfaces, effect of temperature on the steadiness of single chain conformation, surface glass alteration temperature, entropic elasticity of single polymer chains, flexible moduli of tiny wires, single polymer chain elongation (Maver et al., 2013). Additionally, it has been utilized to distinguish between sugar isomers also conduct stretching tests on single carboxy-mehtylated amylase (Maver et al., 2013).

AFM is a scanning probe microscopy method which has been widely used to characterize tiny composite materials. It is capable of characterizing characteristics of surfaces at the nanometre scale (Marrone, 2013). The process of topography imaging normally involves scanning the composite surface with an atomic force microscopy probe (which has a standard radius of curvature of ten nanometer) while keeping an eye on how the probe and sample surface interact. There are two modes of operation (contact as well as intermittent-contact), in addition to three settings which are vacuum, vapour, as well as fluid in which measurements can be performed. The atomic force microscopy analysis tip is skimmed through the surface in the contact mode, feedback is then employed to keep the force between the tip and the sample constant as presented in figure 7. Atomic force microscopy probe is vibrated in the intermittent contact mode, often known as the tapping mode, close to its resonance frequency. The feedback is utilised so as to keep some parameters of the probe's vibration constant. The ability to acquire additional divergence channels, including a phase image, then lower lateral forces than contact mode are two advantages of intermittent-contact mode. Surface roughness of cellulose tiny composite materials has been characterized using atomic force microscopy topography imaging (Aulin et al., 2009). Comparative investigations on the processing of cellulose tiny composite on the resulting surface roughness have been made possible by the sub-nanometer quantifiable height resolution in addition the qualitative nanometre lateral resolution. Investigations on the surface chemistry of tiny composite materials have been conducted using atomic force microscopy methods (Oksman & Moon, 2014).

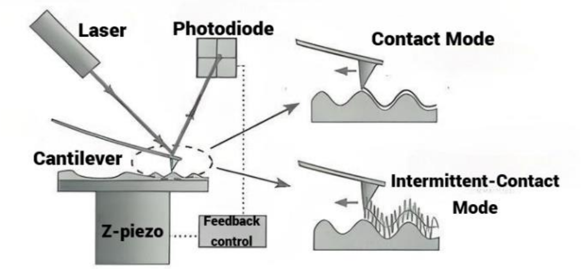


Figure 7. Schematic of atomic force microscopy setup as well as the representative configurations of the AFM cantilever/tip and surface for the different imaging modes; contact as well as intermittent-contact (Oksman & Moon, 2014).

3.5 Fourier Transform infrared Spectroscopy (FTIR)

Several essential elements make up the experimental setup for Fourier Transform Infrared Spectroscopy (FTIR), which is used to characterize polymer nanocomposites. The first and most important step is sample preparation, in which the nanocomposite is either dissolved in an appropriate solvent or compressed into a pellet. A detector, an interferometer, and a beam splitter make up the FTIR spectrometer. The transmitted light is gathered after the infrared light travels through the sample. The identification of functional groups and interactions within the nanocomposite is made possible by the collection of data across a range of wavelengths. The existence of nanoparticles, structural alterations, and chemical bonds may all be ascertained by analyzing the spectra (Jaleh & Fakhri, 2016). FTIR Spectroscopy is a strong optical spectroscopic method which that uses IR light in analyzing the vibrations of a thrilled molecule at a specified wavelength scope, identifying the quivering properties of chemical practical groups in a sample (Ricci et al., 2015). Fourier transform infrared spectroscopy reveals, chemical environments, molecular structures, orientations, as well as conformations of polymer chains. This technique is amongst the most effective ways in qualitatively detecting chemical structures such as functional groups and bonds based on distinctive occurrences. Furthermore, FTIR spectra can quantitatively quantify the sample's component content built on band intensity. Figure 8 depicts the Fourier transform infrared spectra of polyaniline-based nanocomposite materials and formed tiny composites materials (Rangel-Olivares et al., 2021). ATR/FTIR known as attenuated total reflectance/fourier transform infrared spectroscopy is an accompanying approach in exploring molecular structure of tiny composite films. This method is sensitive, fast, and non-destructive (El Hadri et al., 2015).

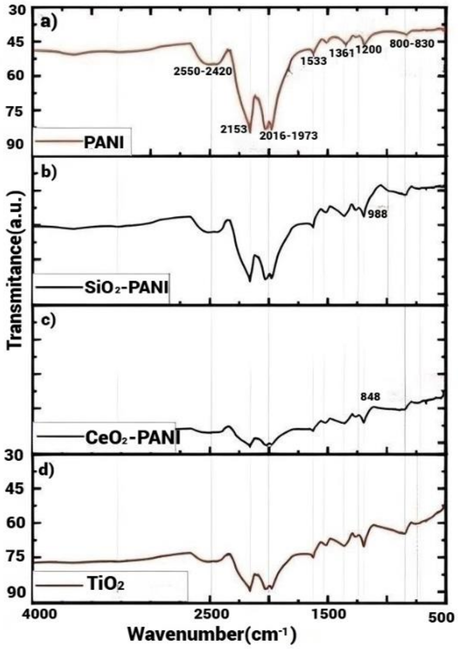


Figure 8. FTIR spectra for (a) PANI and tiny composite materials of (b) SiO_2-PANI, (c) CeO_2-PANI, as well as (d) TiO_2 A-PANI (Rangel-Olivares et al., 2021).

3.6 Raman Spectroscopy

Raman Spectroscopy is a method for studying vibrational spectra that uses laser light to interact with molecular shakings/phonons, shifting the drive of the laser photons active or inactive. Conservative Raman spectroscopy uses energy shift in determining vibrational modes of a system. As described by Gouadec & Colomban, 2007, this method becomes very appropriate in the investigation of tiny materials due to their vibrational spectra permit to recognize the phases also phase transitions in tiny particles, the examination of amorphous domains, and the size identifications of tiny structures (Rallini & Kenny, 2017). Raman spectroscopy uses inelastic light scattering in analyzing the vibrational as well as revolving modes of molecules. The current technique of coherent anti-Stokes Raman scattering is highly sensitive then is employed in vivo spectroscopy as well as imaging (Bokobza, 2018). This technique offers the perfection of saving time in comparison to other traditional techniques. This method is utilized to determine tiny fillers' organization, crossing point interactions, functionalization, physical characteristics, and orientation. In a PNC, the interaction of tiny fillers and polymers is represented in the Raman spectrum as a peak alteration or a conversion in peak intensity or thickness. In other words, band shifts in Raman spectroscopy show mechanical distortion of nanofillers, polymer nanofiller interactions, polymer phase shifts, strain state, and tiny filler Young's modulus (K. Yang et al., 2009).

3.7 Nuclear Magnetic Resonance Spectroscopy (NMR)

A sample is placed inside a powerful magnetic field produced by an NMR magnet as part of the experimental setup for Nuclear Magnetic Resonance (NMR) spectroscopy, which is used to characterize polymer nanocomposites. The sample is exposed to radiofrequency pulses after being prepared as a solid or dissolved in an appropriate solvent. The nuclear spins of particular atoms in the sample are excited by these pulses. The signals that are released are picked up and transformed into a spectrum that provides details on the dynamics, interactions, and structure of molecules. The chemical environment, chain mobility, and nanoparticle distribution inside the polymer matrix are all revealed by NMR (Sadasivuni et al., 2016). Nuclear magnetic resonance encompasses a wide spectrum of phenomena involving the contact of electromagnetic energy with materials. NMR spectroscopy is the process of exposing a molecule to a magnetic field to detect the captivation of radio waves by 1 proton, 13 carbon, 19F, 31phosposous, or other nuclei. NMR spectroscopy empowers the discovery of a unique quantum mechanical magnetic behaviour of the atomic nucleus, particularly in systems of large molecular composite (Mäntylahti, 2014). NMR spectroscopy is a suitable method for studying polymers, including end groups, branching, functionalization, and other characteristics. Nuclear magnetic resonance spectroscopy is used to characterize tiny composites regardless of the composition of the polymer or filler (Mallakpour & Azimi, 2020). The type of the polymer or filler has no bearing on how NMR spectroscopy is used to characterize tiny composite. Nevertheless, in most composite instances, including elastomers, thermo-plastics, as well as biopolymers, nuclear magnetic resonance spectroscopy elucidates the interactions of the polymeric filler interfacial, nature of distribution, interspacial distances of filler particles, attachment, existence of cavities, and so on in a comparable manner (Sadasivuni et al., 2016).

Gutowsky et al. (Gutowsky & Pake, 1950) produced the first report of Hindered Rotation 1 proton NMR spectroscopy on solids and the technique has since been widely utilized to investigate the polymer and its tiny composite characterization (Renard & Jarvis, 1999). T_2 relaxometry is utilised to study polymer mobilities in nanocomposite materials, as well as the immobilized polymer chains that surround the filler particles (Thomas et al., 2016). Figure 9(a) and 9(b) show the 1proton NMR spectra of PLLA/ZnO tiny composites (two percent), in ppm, and the 13 carbon NMR spectrum of PLLA/ZnO (two percent) respectively (Mittal, 2009).

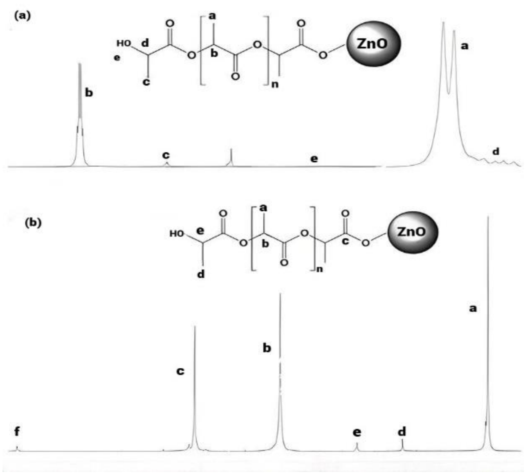


Figure 9. (a) 1 proton NMR spectrum of PLLA/ZnO tiny composites (two percent), in ppm; (b) 13 carbon NMR spectrum of PLLA/ZnO (two percent) tiny composites (Sadasivuni et al., 2016).

3.8 Ultraviolet Visible Spectrophotometry

In order to characterize polymer nanocomposites, UV-Visible (UV-VIS) spectroscopy requires an experimental setup that includes a spectrophotometer with a light source (usually a tungsten lamp for visible light and a deuterium lamp for UV radiation). Samples can be put in a quartz cuvette or container after being produced as thin films or solutions. The transmitted light at different wavelengths is measured after it passes through the sample. By recording the transmittance and absorbance spectra, information about optical characteristics and electronic transitions is obtained. This technique aids in figuring out the composite's bandgap, absorption properties, and possible interactions between the nanoparticles and polymer matrix (Venkatachalam, 2016). The absorbance of substances in a specific wavelength can be investigated by ultraviolet visible spectroscopy technique. The method is centered on the idea of electronic shift in molecules or atoms, that is generated by captivation of light in the visible part of the electromagnetic spectrum (four hundred to eight hundred nanometer) when the electron is excited. Ultraviolet visible spectroscopy is amongst of the utmost imperative methods for characterizing the optical characteristics exhibited by PNCs (Mallakpour & Azimi, 2020). It contributes to a better understanding of the interaction between the medium known as matrix with the tiny filler, as well as the role of tiny fillers in refining the characteristics of nanocomposite materials. When combined by other characterisation methods, Ultraviolet visible spectroscopy is a crucial instrument for determining the appropriate optical characteristics of tiny fillers in a polymer medium called matrix. Built on the absorption characteristics of PNCs, added fine alteration of the required optical characteristics may be accomplished by carefully selecting a kind as well as composition of tiny filler in a polymer medium (Venkatachalam, 2016). In addition, the Ultraviolet visible spectroscopy technique displays the transparency, dispersal zones, refractive index, as well as optical gang gap (Sharma et al., 2011). Anisotropic particles exhibit 2 peaks. These peaks are, transverse peak, irrespective of location, peak intensity, and peak width, provides enormous as well as valued gen of metal tiny particles. When metal tiny particles are utilized as tiny fillers in polymer medium, Ultraviolet visible spectroscopy can be one of the greatest significant characterisation techniques for understanding of tiny filler's size as well as distribution in the medium known as matrix, the steadiness of the tiny composite, also the interaction between the medium with the filler. The durability of the produced tiny composite colloidal solution is investigated using Ultraviolet visible spectroscopy, which follows the nano tiny particles' SPR absorption peak (Venkatachalam, 2016).

3.9 X-ray Photoelectron Spectroscopy

A number of crucial elements make up the experimental setup for X-ray Photoelectron Spectroscopy (XPS), which is used to characterize polymer nanocomposites. A vacuum chamber is employed to prevent contamination, and an X-ray source (usually Al Kα) creates X-rays that irradiate the sample surface. An electron analyzer gathers the released photoelectrons and calculates their kinetic energy. To improve the accuracy of the analysis, sample preparation may entail surface cleaning or thin film deposition (Son et al., 2020). XPS is a surface investigation method that involves irradiating a material with an X-ray beam while also gaging the kinematic energy as well as quantity of electrons which discharged from the top one to ten nanometers of the substance under study. This examination analyses a limited depth of the sample also offers elemental as well as chemical state evidence for the elements present in a material (Sardela, 2014). XPS examines the surface adjustment of natural as well as man-made polymers, carbon tiny fillers, the chemical composition (elements) of a surface, also the bonding states of those elements, also is used to analyze modified catalysts, engineered polymer coatings, as well as small electronic heterostructures (Yang et al., 2010). XPS is a well-known and commonly used technique for analyzing solid-state materials. Centered on Einstein's photoelectric effect (Son et al., 2020), it quantifies the active energy of released electrons using photoelectric effects caused by X-ray radiance. One of the distinguishing characteristic of the elements is the energy of liberated core electrons, which may be used in determining the number of elements as well as chemical bonds in the scheme (Powell, 1988). X-ray photoelectron spectroscopy is better known as electron spectroscopy for chemical examination (ESCA). Because the photoelectron created in a solid sample might be dispersed through interactions with extra electrons in the system, its cruel free path is only a few nanometers (Chromatogr et al., 1982). Therefore, XPS is commonly used for both qualitative also quantitative investigation of the chemical structure as well as content of the sample's surface. Its spectroscopic principle allows for non-destructive analysis without causing damage to the samples. As a result, the usage of XPS is not restricted providing the sample of concern is solid (Son et al., 2020).

3.10 Energy-dispersive X-ray spectroscopy (EDX)

When characterizing polymer nanocomposites, the experimental setup for Energy Dispersive X-ray Spectroscopy (EDX or EDS) usually works in conjunction with a Scanning Electron Microscope (SEM). The sample is put into the SEM chamber after being prepared similarly to the SEM (usually coated for conductivity). X-rays that are distinctive of the elements present are produced when the electron beam scans the surface. These X-rays are collected by the EDX detector, which then produces a spectrum by analyzing their energy. The elemental makeup and relative abundance of the components in the nanocomposite are determined by this spectrum. When evaluating the concentration and dispersion of nanoparticles inside the polymer matrix, EDX is very helpful (Son et al., 2020). Energy-dispersive X-ray spectroscopy is a strong technology exploited for elemental analysis or else chemical definition of a single specimen. Since this is a form of spectroscopy, it investigates a sample using interactions between electromagnetic radiation with the substance. The Energy-dispersive X-ray spectroscopy method characterizes the chemical composition of micro- as well as tiny materials by identifying the ray spectra generated by a sample attacked with an electron ray and displaying distinct spreading of the components by X-ray mapping (Mishra et al., 2017).

3.11 Fluorescence spectroscopy

Fluorescence is a two-step process in which light is absorbed at a specified wavelength (excitation) and then emitted at a greater wave length. The emanation of light is known as fluorescence. This technique detects the energy shift of photons when they are bare to great-energy photons, resulting in the discharge of photons with low energy through the sample. In biological and medical disciplines, it is a well-known technology for confocal microscopy, fluorescence resonance energy transference, as well as fluorescence lifetime imaging (Albrecht, 2008)**.** Fluorescence spectroscopy is broadly used amongst polymer chemists as well as nanotechnology researchers. Their investigation can offer extensive information about molecular processes such as solvent mixtures in tiny composite, rotating diffusion of molecules, tiny filler distances, conformational alterations, as well as tiny filler interaction with the polymer medium (Goesmann & Feldmann, 2010).

3.12 Thermal Gravimetric Analysis (TGA)

Thermogravimetric analysis is a well-established and commonly utilized technique for determining change in weight features of studied samples throughout heating, as well as the kinetics of associated reactions (Singh et al., 2012). In polymers, it is mostly applied to examine degradation behaviour, nevertheless it can also be used to determine the number of contaminants, additives, or solvent residues. Thermogravimetry is commonly utilized to study the effect of nanoadditives on the thermal steadiness of PNCs (Pielichowski & Pielichowska, 2018). Thermogravimetric examination is a well-established technology for determining weight loss characteristics also reaction kinetics. Pyrolysis thermogravimetric investigation includes thermal deterioration of the sample (usually five to twenty mg sample weight) in an inert atmosphere. The loss in weight is recorded as the temperature is raised uniformly. The study yields net loss in weight, in addition to the computation of kinematic parameters is centered on streamlining assumptions that do not always correlate to the complicated chemical reactions involved in the thermal deterioration of the waste sample. Though, the informations allow for suitable comparisons of reaction parameters including temperature as well as the rate of heating (Singh et al., 2012). This review delves into the various methods employed to assess the thermal properties of polymer nanocomposites. It discusses Thermogravimetric Analysis (TGA), Differential Scanning Calorimetry (DSC), and other relevant techniques, highlighting their applications in characterizing nanocellulose-reinforced polymer nanocomposites.

3.12.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a fundamental technique for assessing the thermal stability and decomposition kinetics of materials. In TGA, a sample is heated at a controlled rate in a controlled atmosphere (typically inert or oxidizing), and its weight change is monitored as a function of temperature or time. This weight loss provides information on the thermal degradation processes occurring within the material. TGA is particularly valuable in investigating the thermal behavior of polymer nanocomposites reinforced with nanocellulose. Studies have shown that the incorporation of nanocellulose can either enhance or reduce the thermal stability of the polymer matrix, depending on the type of nanocellulose, its surface modification, and the processing conditions. For instance, the replacement of surface sulfonate groups in cellulose nanocrystals (CNCs) with carboxyl groups through TEMPO oxidation has been shown to significantly improve their thermal stability (Voronova et al., 2022).

3.12.2 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is another crucial technique for characterizing the thermal properties of materials. In DSC, a sample and a reference material are heated at a controlled rate, and the difference in heat flow between the sample and the reference is measured as a function of temperature. This difference in heat flow provides information on the thermal transitions occurring within the sample, such as glass transitions, melting transitions, and crystallization transitions. DSC is widely used to study the phase behavior of polymer nanocomposites reinforced with nanocellulose. DSC can also provide information on the thermal stability of the nanocomposite, revealing any changes in the decomposition behavior compared to the neat polymer (Zielińska et al., 2022).

3.12.3 Thermomechanical Analysis (TMA)

Thermomechanical analysis (TMA) measures the dimensional changes of a material as a function of temperature or time under a constant load. TMA provides information on the thermal expansion coefficient, the glass transition temperature, and the softening point of the material. It is particularly useful for characterizing materials that undergo significant dimensional changes upon heating, such as polymers and polymer nanocomposites. In the context of polymer nanocomposites, TMA can be used to study the effects of nanocellulose on the thermal expansion and softening behavior of the polymer matrix (Nassif et al., 2024).

4. Properties of Polymer Nanocomposite Materials

Numerous factors, including surface energy, the type of tiny-filler substrates, medium known as matrix interface type, surface adhesion, surface development, specific surface area, tiny filler morphology as well as size, also the dissemination of tiny particles in the polymer medium, influence the different properties of PNCs. Due to the increased interfacial area between the polymers and the tiny-fillers, polymer nanocomposite materials exhibit improved mechanical as well as physical properties compared to the host pure polymers. PNCs exhibit distinct mechanical and physical properties not exhibited by micro-composites (Khan et al., 2022). The following is a description of polymer tiny composites' qualities.

4.1 Magnetic Properties

The potential uses of magnetic tiny particles as well as tiny composites in the disciplines of biomedicine, information technology, catalysis, telecommunication, also environmental remediation make them intriguing materials. A coating or graft of organic species, such as polymers or surfactants, can stabilize the bare magnetic tiny particles also prevent them from aggregating. The dual nature of their organic in addition inorganic counterparts makes hybrid magnetic tiny composite materials intriguing materials, and several techniques are being employed in their synthesis. Applications for magnetic PNCs in the biological also environmental domains include the elimination of heavy metals, hazardous effluents, oil, targeted drug delivery, magnetic resonance imaging, as well as cancer treatment (Kalia et al., 2014). It has been demonstrated that the degree of aggregation, size distribution, plus interaction of the tiny particles play a crucial role in the magnetic behaviour of these materials. Dipole interaction can cause in the formation of microscopic structures made up of several small particles that behave as a single magnetic unit in more concentrated samples. In diluted samples, the magnetic properties can be explained in terms of non-interacting or weakly interacting tiny particles (Barrera et al., 2019).

4.2 Flame retardancy

Condensed flame retardant research for polymers typically advances already developed technologies. These are materials based on phosphorus or metal hydroxides. But while these materials increase resistance to flammability, they often deteriorate mechanical integrity. In this field, no significant advancement in flame retardant technology has occurred in a while. The enhanced mechanical also thermal properties of polymer-clay tiny composite materials have attracted a lot of attention. An important benefit over current condensed phase flame retardants is their enhanced flammability resistance while retaining high mechanical qualities. Two varieties of polymer clay tiny composite materials were used by Morgan et al. (Camargo et al., 2009) to investigate the flammability of polymer tiny composite materials. Polycaprolactum (PA-6) as well as polyethelene-co-vinyl acetate (EVA) were combined to create nanocomposite materials. In general, flame retardants increase flammability while decreasing the mechanical qualities of polymers. Effective flame retardancy is provided by polymer layered silicate (PLS) tiny composite materials, which don't harm the environment during combustion, recycling, or end-product disposal (Thomas et al., 2006).

4.3 Mechanical Properties

When tiny particles are added to the polymer medium called matrix, the hardness also scratch resistance increase (S. P. Thomas et al., 2006). According to research by Kojima et al. (Kojima et al., 1993), the ductile strength plus modulus of the hybrid are enhanced to more than twice that of the virgin polymer by nanoscale silicate intercalation into nylon-6. Once more, Kojima et al. (Kojima et al., 1993) demonstrated that filler loading plus compatibilization affect stretchable strength also elongation at break. It has been discovered that exfoliated polymer silicate systems offer better mechanical characteristics than traditionally filled systems. With a tensile strength of 60 MPa, polymer tiny composite materials demonstrate remarkable mechanical characteristics and exceptional flexibility (Khan et al., 2022). The mechanical properties of polymer nanocomposites are profoundly influenced by the type of nanocellulose utilized and any surface modifications applied. For instance, studies using epoxy resin reinforced with hemp nanocellulose have shown significant improvements in tensile strength, flexural strength, impact strength, and hardness with increasing filler loading up to 2 wt.%, beyond which the improvements were less pronounced (Voronova et al., 2022). Mechanical properties are explained below

4.3.1Tensile Strength

Tensile strength measures a material's resistance to breaking under tensile stress. The high aspect ratio and stiffness of nanocellulose fibrils contribute to improved load transfer from the polymer matrix to the nanocellulose, enhancing the material's overall strength. Studies have demonstrated significant improvements in tensile strength upon the addition of nanocellulose to various polymer matrices, such as polyacrylonitrile (PAN) (Jiang et al., 2019) and epoxy resin (Rana & Gupta, 2021).

4.3.2Young's Modulus

Young's modulus, or the modulus of elasticity, is a measure of a material's stiffness, representing its resistance to elastic deformation under stress. The high stiffness of nanocellulose, particularly CNCs, contributes significantly to increase Young's modulus in the nanocomposite. The nanocellulose fibrils restrict the movement of polymer chains, increasing the overall stiffness of the nanocomposite. This enhancement is particularly important for applications requiring high rigidity and dimensional stability (Rana & Gupta, 2021).

4.3.3Toughness

Toughness measures a material's ability to absorb energy before fracture. Nanocellulose enhances toughness by several mechanisms, including crack deflection, crack bridging, and energy dissipation. The high aspect ratio and flexibility of CNFs are particularly beneficial for enhancing toughness. The nanofibrils can deflect propagating cracks, preventing catastrophic failure, and can bridge cracks, further hindering their propagation. The nanocellulose network also dissipates energy through various mechanisms, including frictional interactions between the fibrils and the polymer matrix. Studies have shown that the addition of nanocellulose can significantly improve the toughness of polymer nanocomposites, making them more resistant to impact and fracture (Aigaje & Riofrio, 2023).

4.4 Barrier Properties

It is anticipated that the barrier qualities of the nanocomposite materials will be significantly improved in comparison to the corresponding polymer due to the scattering of the ultra-thin inorganic strata over the polymer medium. The water vapour transmission rates in PVA-Na+ montmorillonite tiny composites were examined for both the pure polymer and a few of its low montmorillonite tiny composites. For silicate loadings of only 4-6 weight percent, the permeability dropped to roughly 40% of the pure water vapour transmission values. The improved elasticity of the polymer matrix as well as the increased route tortuosity of the penetrant molecules are credited for this decrease (Thomas et al., 2006).

4.5 Electrical Properties

The electrical properties of polymer nanocomposites are enhanced by the addition of conductive nanoparticles, resulting in improved conductivity and reduced dielectric constants. These modifications enable applications in flexible electronics, sensors, and electromagnetic shielding, making them valuable in modern technological advancements (Mutiso & Winey, 2015). Researchers are very interested in PNCs with carbon nanotube fillers because they improve mechanical in addition electrical conductivity. Highly sensitive strain sensors, solar cells, organic field emission displays, and materials that interfere with electromagnetic waves are a few possible uses for tiny composites as functional materials. Because of the abundant electrical conductivity as well as aspect ratio of carbon tiny tubes, compared to conventional electronic composite materials comprising fillers like short carbon fibres or carbon flakes, exceptional electrical characteristic can be attained with carbon nanotube tiny composites containing far less filler material (Hu et al., 2008). CNTs, carbon nanobers, as well as graphene-based reinforced thermoplastic tiny composites have good electrical properties (Hu et al., 2008).

4.6 Rheological Properties

Rheological properties of polymer nanocomposites reflect their flow behavior and deformation under stress. These properties are crucial for processing and application, influencing viscosity, elasticity, and thermal stability. Nanoparticles can enhance mechanical strength and modify flow characteristics, leading to improved performance in applications like coatings and films (Guo et al., 2017). Giannelis et al. (Giannelis et al., 1999) provided the first description of the rheological characteristics of in-situ polymerized tiny composite materials with end-tied polymer chains. It was discovered that the flow behaviour of poly(∑-caprolactone) plus polyamide-6 tiny composite materials was significantly different from that of the pristine matrices. On the other hand, the behaviour of the matrix governed the thermo-rheological parameters of the composite materials. However, these in-situ polymerization-based tiny composites show a rather wide molar mass distribution of the polymer medium, making it difficult to interpret the data because it obscures important structural information (Thomas et al., 2006).

4.7 Thermal Properties

The thermal stability of the PNCs is ascertained by differential scanning calorimetry (DSC) as well as thermogravimetric investigation (TGA). Thermolysis of GMA-f-NGP by Mazumdar et al. (Mazumdar et al., 2018) demonstrates that the PNCs are thermally stable because the primary mass loss occurs at around 209°C, continues quickly. When it reaches about 400°C, the PNCs start to degrade. Using molecular dynamics simulations, Wang et al. have investigated the issues of improving the thermal transport across the interface between graphene and epoxy tiny composites (Yu Wang et al., 2016). The thermal properties of polymer nanocomposites are significantly influenced by the type of nanocellulose used and the processing methods employed (Rana & Gupta, 2021). The crystallinity and surface chemistry of nanocellulose play a significant role in determining the thermal behavior of the nanocomposite. Cellulose nanocrystals (CNCs) produced by acid hydrolysis have a higher crystallinity index than cellulose nanofibrils (CNFs) produced by TEMPO-mediated oxidation. This difference in crystallinity can affect the thermal stability and decomposition behavior of the nanocomposite. The surface chemistry of the nanocellulose also plays a crucial role, with surface functional groups influencing the interactions with the polymer matrix and affecting the thermal properties. For example, the presence of sulfate groups in acid-hydrolyzed CNCs can catalyze thermal degradation while the carboxyl groups in TEMPO-oxidized CNFs can enhance thermal stability (Voronova et al., 2022).

5. Conclusion

Researchers are particularly interested in PNCs because of the unexpected synergistic effects resulting from the two components. This article provides some useful examples of different methodologies used to characterize polymer nanocomposite materials. It was discovered that XRD is used to analyze the crystallographic nature of PNCs, scanning electron microscopic images show observable morphological structures of PNCs, TEM images are the best technique for understanding tiny particle size and particle size homogeneity, and AFM is utilized to straight characterize essential mechanical features including elastic modulus as well as adhesion energy on the surfaces of PNCs. The unique quantum mechanical magnetic behaviour of the atomic nucleus can be discovered using Raman Spectroscopy, which is especially useful in macromolecular composite systems. FTIR spectroscopy is a powerful optical spectroscopic technique that is utilized in identifying the vibration properties of chemical functional groups in PNCs. The absorbance of PNCs at a particular wavelength can be measured using ultraviolet visible spectroscopy; the chemical composition of PNCs can be characterized using the energy dispersive X-ray spectroscopy method, X-ray photoelectron spectroscopy analyses the elemental chemical composition of a surface also the bonding states of those elements, thermal stability of PNCs can be identified by TGA, a widely recognized technique that measures the energy change of photons when they are subjected to high-energy photons is Fluorescence spectroscopy. These methods allow for the investigation of various polymer tiny composites' characteristics. Because of the nanofillers, polymer nanocomposites have unique properties when compared to a clean polymer matrix. Enhancing the polymers' electrical, thermal, magnetic, mechanical, flame-retardant, as well as gas-barrier qualities is the aim of the tiny fillers. Due to their larger precise interfacial area, further controlled interfacial interactions, greater attainable loads, and higher general compliance, nanofillers are superior to conventional fillers. Polymer nanocomposites possess several benefits over conventional composites plus neat polymers. These include enhanced tensile and thermal properties, reduced weight, better barrier qualities, greater flammability, and increased biodegradability.

Acknowledgements

We extend our sincere gratitude to all individuals who contributed to the development of this review article. In particular, we wish to acknowledge Dr. Musa Mpelwa, Head of Department at Teofilo Kisanji University (Tanzania), for his invaluable insights and constructive feedback throughout the preparation of this manuscript. We also express our heartfelt appreciation to our families for their unwavering support and encouragement during the course of this work.

Funding

None

Conflict of Interest

The author declares that there are no conflicts of interest regarding the publication of this article because no funding agent was involved.

References

Abedi, S. & Abdouss, M. (2014). A review of clay-supported Ziegler-Natta catalysts for production of polyolefin/clay nanocomposites through in situ polymerization. *Applied Catalysis A: General*, *475*, 386–409. https://doi.org/10.1016/j.apcata.2014.01.028

Abhilash, V., Rajender, N. & Suresh, K. (2016). X-ray diffraction spectroscopy of polymer nanocomposites. In *Spectroscopy of Polymer Nanocomposites.* Elsevier Inc. https://doi.org/10.1016/B978-0-323-40183-8.00014-8

Aigaje, E. & Riofrio, A. (2023). Impact of Nanocellulose / Biopolymer Composites : A Review. *Polymers*, *15*, 1219.

Akpan, E. I., Shen, X., Wetzel, B. & Friedrich, K. (2018). Design and Synthesis of Polymer Nanocomposites. In *Polymer Composites with Functionalized Nanoparticles: Synthesis, Properties, and Applications* (pp. 47–83). Elsevier. https://doi.org/10.1016/B978-0-12-814064-2.00002-0

Albrecht, C. (2008). Joseph R. Lakowicz: Principles of fluorescence spectroscopy, 3rd Edition. *Analytical and Bioanalytical Chemistry*, *390*(5), 1223–1224. https://doi.org/10.1007/s00216-007-1822-x

Armstrong, G. (2015). An introduction to polymer nanocomposites. *European Journal of Physics*, *36*(6), 63001. https://doi.org/10.1088/0143-0807/36/6/063001

Asadabad, M. A. & Eskandari, M. J. (2015). Transmission electron microscopy as best technique for characterization in nanotechnology. *Synthesis and Reactivity in Inorganic, Metal-Organic and Nano-Metal Chemistry*, *45*(3), 323–326. https://doi.org/10.1080/15533174.2013.831901

Aulin, C., Ahok, S., Josefsson, P., Nishino, T., Hirose, Y., Österberg, M. & Wågberg, L. (2009). Nanoscale cellulose films with different crystallinities and mesostructures - Their surface properties and interaction with water. *Langmuir*, *25*(13), 7675–7685. https://doi.org/10.1021/la900323n

Barrera, G., Tiberto, P., Allia, P., Bonelli, B., Esposito, S., Marocco, A., Pansini, M. & Leterrier, Y. (2019). Magnetic properties of nanocomposites. *Applied Sciences (Switzerland)*, *9*(2). https://doi.org/10.3390/app9020212

Bokobza, L. (2018). Spectroscopic techniques for the characterization of polymer nanocomposites: A review. *Polymers*, *10*(1). https://doi.org/10.3390/polym10010007

Camargo, P. H. C., Satyanarayana, K. G. & Wypych, F. (2009). Nanocomposites: Synthesis, structure, properties and new application opportunities. *Materials Research*, *12*(1), 1–39. https://doi.org/10.1590/S1516-14392009000100002

De La Cruz-Montoya, E. & Rinaldi, C. (2010). Synthesis and characterization of polymer nanocomposites containing magnetic nanoparticles. *Journal of Applied Physics*, *107*(9), 9–12. https://doi.org/10.1063/1.3358017

Dikin, D. A., Kohlhaas, K. M., Dommett, G. H. B., Stankovich, S. & Ruoff, R. S. (2006). Scanning electron microscopy methods for analysis of polymer nanocomposites. *Microscopy and Microanalysis*, *12*(SUPPL. 2), 674–675. https://doi.org/10.1017/S1431927606067080

Dong, X., Wei, C., Liang, J., Liu, T., Kong, D. & Lv, F. (2017). Thermosensitive hydrogel loaded with chitosan-carbon nanotubes for near infrared light triggered drug delivery. *Colloids and Surfaces B: Biointerfaces*, *154*, 253–262. https://doi.org/10.1016/j.colsurfb.2017.03.036

El Hadri, M., Achahbar, A., El Khamkhami, J., Khelifa, B., Tran Le Tuyet, C., Faivre, V., Abbas, O., El Marssi, M., Bougrioua, F. & Bresson, S. (2015). Vibrational behavior of Gelucire 50/13 by Raman and IR spectroscopies: A focus on the 1800-1000 cm-1 spectral range according to temperature and degree of hydration. *Journal of Molecular Structure*, *1083*, 441–449. https://doi.org/10.1016/j.molstruc.2014.10.062

Fawaz, J. & Mittal, V. (2015). Synthesis of Polymer Nanocomposites : nanocomposite method. *Synthesis Techniques For Polymer Nanocomposite*, 1–30. https://application.wiley-vch.de/books/sample/3527334556_c01.pdf

Ferreira, F. V., Pinheiro, I. F., Gouveia, R. F., Thim, G. P. & Lona, L. M. F. (2018). Functionalized cellulose nanocrystals as reinforcement in biodegradable polymer nanocomposites. *Polymer Composites*, *39*, E9–E29. https://doi.org/10.1002/pc.24583

Galpaya, D., Wang, M., Liu, M., Motta, N., Waclawik, E. & Yan, C. (2012). Recent Advances in Fabrication and Characterization of Graphene-Polymer Nanocomposites. *Graphene*, *01*(02), 30–49. https://doi.org/10.4236/graphene.2012.12005

Giannelis, E. P., Krishnamoorti, R. & Manias, E. (1999). Polymer-silicate nanocomposites: Model systems for confined polymers and polymer brushes. *Advances in Polymer Science*, *138*, 108–147. https://doi.org/10.1007/3-540-69711-x_3

Goesmann, H. & Feldmann, C. (2010). Nanoparticulate functional materials. *Angewandte Chemie - International Edition*, *49*(8), 1362–1395. https://doi.org/10.1002/anie.200903053

Gouadec, G. & Colomban, P. (2007). Raman Spectroscopy of nanomaterials: How spectra relate to disorder, particle size and mechanical properties. *Progress in Crystal Growth and Characterization of Materials*, *53*(1), 1–56. https://doi.org/10.1016/j.pcrysgrow.2007.01.001

Guo, Q., Zhu, P., Li, G., Wen, J., Wang, T., Lu, D. (Daniel), Sun, R. & Wong, C. (2017). Study on the effects of interfacial interaction on the rheological and thermal performance of silica nanoparticles reinforced epoxy nanocomposites. *Composites Part B: Engineering*, *116*, 388–397. https://doi.org/10.1016/j.compositesb.2016.10.081

Gutowsky, H. S. & Pake, G. E. (1950). *Structural Investigations by Means of Nuclear Magnetism . II . Hindered Rotation in Solids Structural Investigations by Means of Nuclear Magnetism . 162*. https://doi.org/10.1063/1.1747580

Hu, N., Masuda, Z., Yan, C., Yamamoto, G., Fukunaga, H. & Hashida, T. (2008). The electrical properties of polymer nanocomposites with carbon nanotube fillers. *Nanotechnology*, *19*(21). https://doi.org/10.1088/0957-4484/19/21/215701

J Chromatogr, R. A., Appl Spectrosc, J. D., Turner, N. H. & Colton, R. J. (1982). Analyst (London) 1981, 706. 921. (15H)-’ktareiii. *C. A.; Aikemade, C. Th. J. J. Quant. Spectrosc. Radiat. Transfer*, *5*(4), 488.

Jaleh, B. & Fakhri, P. (2016). Infrared and Fourier transform infrared spectroscopy for nanofillers and their nanocomposites. In *Spectroscopy of Polymer Nanocomposites*. Elsevier Inc. https://doi.org/10.1016/B978-0-323-40183-8.00005-7

Jiang, E., Maghe, M., Zohdi, N., Amiralian, N., Naebe, M., Laycock, B., Fox, B. L., Martin, D. J. & Annamalai, P. K. (2019). Influence of Different Nanocellulose Additives on Processing and Performance of PAN-Based Carbon Fibers. *ACS Omega*, *4*(6), 9720–9730. https://doi.org/10.1021/acsomega.9b00266

Kalia, S., Kango, S., Kumar, A., Haldorai, Y., Kumari, B. & Kumar, R. (2014). Magnetic polymer nanocomposites for environmental and biomedical applications. *Colloid and Polymer Science*, *292*(9), 2025–2052. https://doi.org/10.1007/s00396-014-3357-y

Khan, I., Khan, I., Saeed, K., Ali, N., Zada, N., Khan, A., Ali, F., Bilal, M. & Akhter, M. S. (2022). Polymer nanocomposites: an overview. *Smart Polymer Nanocomposites: Design, Synthesis, Functionalization, Properties, and Applications*, *November*, 167–184. https://doi.org/10.1016/B978-0-323-91611-0.00017-7

Khan, W. S. W. A., Hamadneh, N. N. & Khan, W. S. W. A. (n.d.). *Science and applications of Tailored Nanostructures 50 4 Polymer nanocomposites-synthesis techniques, classification and properties Outline*.

Kojima, Y., Usuki, A., Kawasumi, M., Okada, A., Fukushima, Y., Kurauchi, T. & Kamigaito, O. (1993). Mechanical properties of nylon 6-clay hybrid. *Journal of Materials Research*, *8*(5), 1185–1189. https://doi.org/10.1557/JMR.1993.1185

Konduru, N. V., Jimenez, R. J., Swami, A., Friend, S., Castranova, V., Demokritou, P., Brain, J. D. & Molina, R. M. (2015). Silica coating influences the corona and biokinetics of cerium oxide nanoparticles. *Particle and Fibre Toxicology*, *12*(1), 1–15. https://doi.org/10.1186/s12989-015-0106-4

Kundu, S., Jana, P., De, D. & Roy, M. (2015). SEM image processing of polymer nanocomposites to estimate filler content. *Proceedings of 2015 IEEE International Conference on Electrical, Computer and Communication Technologies, ICECCT 2015*, *March*, 1–5. https://doi.org/10.1109/ICECCT.2015.7226104

Lü, C., Cui, Z., Wang, Y., Li, Z., Guan, C., Yang, B. & Shen, J. (2003). Preparation and characterization of ZnS-polymer nanocomposite films with high refractive index. *Journal of Materials Chemistry*, *13*(9), 2189–2195. https://doi.org/10.1039/b304154a

Mallakpour, S. & Azimi, F. (2020). Spectroscopic characterization techniques for layered double hydroxide polymer nanocomposites. In *Layered Double Hydroxide Polymer Nanocomposites*. Elsevier Ltd. https://doi.org/10.1016/b978-0-08-101903-0.00006-4

Mäntylahti, S. (2014). *Development of NMR methods to study disordered proteins* (Issue June).

Maron, G. K., Noremberg, B. S., Alano, J. H., Pereira, F. R., Deon, V. G., Santos, R. C. R., Freire, V. N., Valentini, A. & Carreno, N. L. V. (2018). Carbon fiber/epoxy composites: Effect of zinc sulphide coated carbon nanotube on thermal and mechanical properties. *Polymer Bulletin*, *75*(4), 1619–1633. https://doi.org/10.1007/s00289-017-2115-y

Marques, C. F., Matos, A. C., Ribeiro, I. A. C., Gonçalves, L. M., Bettencourt, A. & Ferreira, J. M. F. (2016). Insights on the properties of levofloxacin-adsorbed Sr- and Mg-doped calcium phosphate powders. *Journal of Materials Science: Materials in Medicine*, *27*(7). https://doi.org/10.1007/s10856-016-5733-2

Marrone, P. (2013). Chambers, RT. *Etica e Politica*, *15*(1), 583–605. https://doi.org/10.1093/acprof

Maver, U., Maver, T., Persin, Z., Mozetic, M., Vesel, A., Gaberscek, M. & Stana-Kleinschek, K. (2013). Polymer Characterization with the Atomic Force Microscope. *Polymer Science*. https://doi.org/10.5772/51060

Mazumdar, P., Chockalingam, S., Rattan, S. & Gupta, B. K. (2018). Tunable mechanical, electrical, and thermal properties of polymer nanocomposites through GMA bridging at interface. *ACS Omega*, *3*(4), 3675–3687. https://doi.org/10.1021/acsomega.8b00194

Mishra, R. K., Thomas, S. & Zachariah, A. K. (2017). Energy-Dispersive X-ray Spectroscopy Techniques for Nanomaterial. In *Microscopy Methods in Nanomaterials Characterization*. Elsevier Inc. https://doi.org/10.1016/B978-0-323-46141-2.00012-2

Mittal, V. (2009). Polymer layered silicate nanocomposites: A review. *Materials*, *2*(3), 992–1057. https://doi.org/10.3390/ma2030992

Mutiso, R. M. & Winey, K. I. (2015). Electrical properties of polymer nanocomposites containing rod-like nanofillers. *Progress in Polymer Science*, *40*(1), 63–84. https://doi.org/10.1016/j.progpolymsci.2014.06.002

Nassif, R. A., Hilal, R. H. & Khalef, W. K. (2024). *Enhancing Anticorrosive Characteristics Nanocellulose Reinforcement of Epoxy through*. *11*(3), 45–54.

Oksman, K. & Moon, R. J. (2014). *Characterization of Nanocomposites Structure*. 89–105. https://doi.org/10.1142/9789814566469_0021

Pandey, N., Shukla, S. K. & Singh, N. B. (2017). Water purification by polymer nanocomposites: an overview. *Nanocomposites*, *3*(2), 47–66. https://doi.org/10.1080/20550324.2017.1329983

Park, J. H. & Jana, S. C. (2003). The relationship between nano- and micro-structures and mechanical properties in PMMA-epoxy-nanoclay composites. *Polymer*, *44*(7), 2091–2100. https://doi.org/10.1016/S0032-3861(03)00075-2

Paton, K. R., Varrla, E., Backes, C., Smith, R. J., Khan, U., O’Neill, A., Boland, C., Lotya, M., Istrate, O. M., King, P., Higgins, T., Barwich, S., May, P., Puczkarski, P., Ahmed, I., Moebius, M., Pettersson, H., Long, E., Coelho, J., … Coleman, J. N. (2014). Scalable production of large quantities of defect-free few-layer graphene by shear exfoliation in liquids. *Nature Materials*, *13*(6), 624–630. https://doi.org/10.1038/nmat3944

Pielichowski, K. & Pielichowska, K. (2018). Polymer Nanocomposites. In *Handbook of Thermal Analysis and Calorimetry* (2nd ed., Vol. 6). Elsevier B.V. https://doi.org/10.1016/B978-0-444-64062-8.00003-6

Powell, C. J. (1988). The quest for universal curves to describe the surface sensitivity of electron spectroscopies. *Journal of Electron Spectroscopy and Related Phenomena*, *47*(C), 197–214. https://doi.org/10.1016/0368-2048(88)85012-6

Properties, F. & Materials, C. (2007). Thermal Decomposition of Composites in Fire. In *Fire Properties of Polymer Composite Materials*. https://doi.org/10.1007/978-1-4020-5356-6_2

Puggal, S., Dhall, N., Singh, N. & Litt, M. S. (2016). A Review on Polymer Nanocomposites: Synthesis, Characterization and Mechanical Prop. *Indian Journal of Science and Technology*, *9*(4). https://doi.org/10.17485/ijst/2016/v9i4/81100

Rallini, M. & Kenny, J. M. (2017). Nanofillers in Polymers. In *Modification of Polymer Properties* (pp. 47–86). Elsevier Inc. https://doi.org/10.1016/B978-0-323-44353-1.00003-8

Rana, S. S. & Gupta, M. K. (2021). Fabrication of bionanocomposites reinforced with hemp nanocellulose and evaluation of their mechanical, thermal and dynamic mechanical properties. *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications*, *235*(11), 2470–2481. https://doi.org/10.1177/14644207211004640

Rangel-Olivares, F. R., Arce-Estrada, E. M. & Cabrera-Sierra, R. (2021). Synthesis and characterization of polyaniline-based polymer nanocomposites as anti-corrosion coatings. *Coatings*, *11*(6). https://doi.org/10.3390/coatings11060653

Renard, C. M. G. C. & Jarvis, M. C. (1999). A cross-polarization, magic-angle-spinning, 13C-nuclear-magnetic-resonance study of polysaccharides in sugar beet cell walls. *Plant Physiology*, *119*(4), 1315–1322. https://doi.org/10.1104/pp.119.4.1315

Ricci, A., Olejar, K. J., Parpinello, G. P., Kilmartin, P. A. & Versari, A. (2015). Application of Fourier transform infrared (FTIR) spectroscopy in the characterization of tannins. *Applied Spectroscopy Reviews*, *50*(5), 407–442. https://doi.org/10.1080/05704928.2014.1000461

Sadasivuni, K. K., Cabibihan, J. J. & Al-Maadeed, M. A. S. (2016). NMR spectroscopy of polymer nanocomposites. In *Spectroscopy of Polymer Nanocomposites*. Elsevier Inc. https://doi.org/10.1016/B978-0-323-40183-8.00008-2

Sardela, M. (2014). Practical materials characterization. In *Practical Materials Characterization*. https://doi.org/10.1007/978-1-4614-9281-8

Sharma, A. K. & Kaith, B. S. (2020). Handbook of Polymer and Ceramic Nanotechnology. *Handbook of Polymer and Ceramic Nanotechnology*, *October*, 0–26. https://doi.org/10.1007/978-3-030-10614-0

Sharma, P. K., Kumar, M. & Pandey, A. C. (2011). Green luminescent ZnO:Cu2+ nanoparticles for their applications in white-light generation from UV LEDs. *Journal of Nanoparticle Research*, *13*(4), 1629–1637. https://doi.org/10.1007/s11051-010-9916-3

Sharma, R., Bisen, D. P., Shukla, U. & Sharma, B. G. (2012). X-ray diffraction: a powerful method of characterizing nanomaterials. *Recent Research in Science and Technology*, *4*(8), 77–79. http://recent-science.com/

Shen, S., Henry, A., Tong, J., Zheng, R. & Chen, G. (2010). Polyethylene nanofibres with very high thermal conductivities. *Nature Nanotechnology*, *5*(4), 251–255. https://doi.org/10.1038/nnano.2010.27

Singh, S., Wu, C. & Williams, P. T. (2012). Pyrolysis of waste materials using TGA-MS and TGA-FTIR as complementary characterisation techniques. *Journal of Analytical and Applied Pyrolysis*, *94*, 99–107. https://doi.org/10.1016/j.jaap.2011.11.011

Son, D., Cho, S., Nam, J., Lee, H. & Kim, M. (2020). X-ray-based spectroscopic techniques for characterization of polymer nanocomposite materials at a molecular level. *Polymers*, *12*(5). https://doi.org/10.3390/POLYM12051053

Stankovich, S., Dikin, D. A., Dommett, G. H. B., Kohlhaas, K. M., Zimney, E. J., Stach, E. A., Piner, R. D., Nguyen, S. B. T. & Ruoff, R. S. (2006). Graphene-based composite materials. *Nature*, *442*(7100), 282–286. https://doi.org/10.1038/nature04969

Tamayo, L., Palza, H., Bejarano, J. & Zapata, P. A. (2018). Polymer Composites With Metal Nanoparticles: Synthesis, Properties, and Applications. Synthesis, Properties, and Applications. In *Polymer Composites with Functionalized Nanoparticles: Synthesis, Properties, and Applications* (Issue May 2019). https://doi.org/10.1016/B978-0-12-814064-2.00008-1

Thomas, S. P., Stephen, R., Bandyopadhyay, S. & Thomas, S. (2006). Polymer nanocomposites: Preparation, properties and applications | Polymere nanoverbundwerkstoffe: Pr?paration, eigenschaften und anwendungen. *Gummi, Fasern, Kunststoffe*, *59*(9).

Thomas, S., Rouxel, D. & Ponnamma, D. (2016). Spectroscopy of Polymer Nanocomposites. In *Spectroscopy of Polymer Nanocomposites*. https://doi.org/10.1016/C2014-0-03751-3

Thostenson, E. T., Li, C. & Chou, T. W. (2005). Nanocomposites in context. *Composites Science and Technology*, *65*(3–4), 491–516. https://doi.org/10.1016/j.compscitech.2004.11.003

Venkatachalam, S. (2016). Ultraviolet and visible spectroscopy studies of nanofillers and their polymer nanocomposites. In *Spectroscopy of Polymer Nanocomposites*. Elsevier Inc. https://doi.org/10.1016/B978-0-323-40183-8.00006-9

Verdejo, R., Bernal, M. M., Romasanta, L. J. & Lopez-Manchado, M. A. (2011). Graphene filled polymer nanocomposites. *Journal of Materials Chemistry*, *21*(10), 3301–3310. https://doi.org/10.1039/c0jm02708a

Voronova, M. I., Surov, O. V., Kuziyeva, M. K. & Atakhanov, A. A. (2022). Thermal and Mechanical Properties of Polymer Composites Reinforced By Sulfuric Acid-Hydrolyzed and Tempo-Oxidized Nanocellulose: a Comparative Study. *ChemChemTech*, *65*(10), 95–105. https://doi.org/10.6060/ivkkt.20226510.6596

Wang, Y. & Herron, N. (1991). Nanometer-sized semiconductor clusters: Materials synthesis, quantum size effects, and photophysical properties. *Journal of Physical Chemistry*, *95*(2), 525–532. https://doi.org/10.1021/j100155a009

Wang, Yu, Yang, C., Pei, Q. X. & Zhang, Y. (2016). Some Aspects of Thermal Transport across the Interface between Graphene and Epoxy in Nanocomposites. *ACS Applied Materials and Interfaces*, *8*(12), 8272–8279. https://doi.org/10.1021/acsami.6b00325

Yang, K., Gu, M., Guo, Y., Pan, X. & Mu, G. (2009). Effects of carbon nanotube functionalization on the mechanical and thermal properties of epoxy composites. *Carbon*, *47*(7), 1723–1737. https://doi.org/10.1016/j.carbon.2009.02.029

Yang, X., Tu, Y., Li, L., Shang, S. & Tao, X. M. (2010). Well-dispersed chitosan/graphene oxide nanocomposites. *ACS Applied Materials and Interfaces*, *2*(6), 1707–1713. https://doi.org/10.1021/am100222m

Zielińska, D., Skrzypczak, A., Peplińska, B. & Borysiak, S. (2022). Nanocellulose-Based Polymer Composites Functionalized with New Gemini Ionic Liquids. *International Journal of Molecular Sciences*, *23*(24). https://doi.org/10.3390/ijms232415807