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REVIEW ARTICLE

Analytical approaches to fiber-reinforced polymer composites: a short review

Marcia Murakoshi Takematsu[1]* and Rita de Cássia Lazzarini Dutra[1]

[1]Departamento de Química, Instituto Tecnológico da Aeronáutica – ITA, São José dos Campos, SP, Brasil
**marciatakematsu@gmail.com*

Abstract

A variety of fiber-reinforced polymer (FRP) has been described in literature, with a considerable subset of studies focused on fiber surface treatment (sizing), performance enhancement of matrix and fibers both synthetic and natural, and development of more ecologically sustainable composites. The present review discusses the different types of fibers and matrices and their applications, depending on the chemical and mechanical properties of their composites. In order to evaluate the performance of FRP composites and explore the characteristics of the involved materials, some analytical techniques are considered paramount, such as thermal analysis, microscopy, Fourier transform-infrared spectroscopy (FT-IR), and others. On this basis, this review addresses the state-of-the-art of material characterization methodologies, provides a comprehensive overview of different types of FRP found in literature, as well as links the analytical techniques with the main applications contributing to future studies and research in this area.

Keywords: *analytical techniques, composite, fiber, polymer.*

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1. Introduction

Fiber-reinforced polymer (FRP) composites are broadly used in technological applications, for example, aerospace, military, automotive, civil, electronic, transport, renewable energy, and biomedical engineering[1-10]. This remarkable material consists of synthetic or natural fibers with specific properties embedded in a polymeric matrix, and the fibers can also have geometry and/or orientation to enhance the performance target of the composite. In aerospace research, Soutis[11] reports that FRP composites have been employed in aircraft structures since 1903 in the Wright Brother's Flyer 1, and their use was expanded to military aircraft, satellites, and space launchers.

Nowadays, FRP composites are a fast-developing field of research and development, given the advances in materials and applications. In this context, many derived classes of FRP have been reported, such as carbon fiber-reinforced plastics (CFRP), natural fiber-reinforced polymer composites (NFRPCs), synthetic fiber-reinforced polymer composites (SFRPCs), glass fiber-reinforced polymer (GFRP), continuous carbon fiber-reinforced polymer composites (CCFRPs), discontinuous carbon fiber-reinforced polymer composites (DCFRPs), and fiber-reinforced soft composites (FRSCs)[1,11,12].

Previous studies by Raju and Shanmugaraja[13], Kerni et al.[14], and Chaudhary and Ahmad[15] highlight FRP composites as an engineering material with sustainability potential; it employs renewable sources, such as natural fibers, sustainable and biodegradable polymers. In a recent review, Mahesh et al.[16] reports the use of natural fibers in combination with different polymeric matrices, focused on mechanical properties. The recent developments in materials science have been focused on alternatives with less impact on the environment and bringing more sustainability to this area of composites[17-21].

Soutis[11] predicted that 50% or more of the structural mass of an aircraft could be made of CFRP composites. According to Hui et al.[1], the CFRP based on epoxy matrix composites makes up to 50% of the wings and fuselages of the Boeing 787 Dreamliner and the Airbus A350 XWB models, due to the mechanical properties of composites with failure by diffuse damage, presenting a different and more adequate fracture resistance than solid materials, such as stainless steel.

This statement highlights how FRP composites are crucial in this area, as well as in other fields. Hui et al.[1] also report a new class of FRP composites known as FRSCs, where the matrix is very soft and resistant. In addition, developments in this class could result in a polymeric matrix with self-healing properties with potential to replace epoxy matrices, which have fundamental roles in aerospace engineering. The matrices that could perform this role are polyampholyte hydrogel, acrylic tapes forming a double network (DN), and self-healing hydrogels[1,11,22].

Alemour et al.[23] also report the use of glass fiber, carbon fiber (CF), FRP and a combination of these materials on aeronautical application that significantly reduce the weight of an aircraft with added resistance when compared to metal alloys, reducing fuel consumption, improving efficiency and operating costs.

Moreover, the aerospace sector is considered one of the most important fields to invest in composites once it is the main responsible for stratospheric pollution, and advances in different fibers or polymers that reduce weight and add resistance to the composite could reflect in energy efficiency, high performance and eco-friendly engineering structures and less climatic footprint[24].

Szabó et al.[25] studied alternatives to synthetic FRP and developed short CF-reinforced polymers derived from cellulosic materials and polyamide 6, monomers that could be obtained from renewable sources, and consider this composite a potential green alternative to FRP composites.

Imre and Pukánszky[26] mention four factors that determine the properties of composites including FRP: component properties, composition, structure, and interaction. Thus, additive manufacturing technologies are being developed to improve the interaction of the mechanical properties of polymeric composites with continuous fibers, with the use of a suitable binder to associate the FRPCs (fiber-reinforced polymer composites) in the interlayer; with the techniques being mainly focused on fiber alignment, significant reduction of porosity, fiber-matrix adhesion and improvement in the bonding of composite layers[27].

Combining a tough hydrogel and a woven fiber fabric, it is possible to provide a synergistic effect that increases the toughness and tensile properties of composites compared to isolated or neat materials. Huang et al.[28] studied the dissipation of energy performed by hydrogel matrix in the final toughness of composites, interfacial bonding, and synergic effects in the mechanical properties of PA-GF (polyampholyte – glass fiber fabric), PAAm-GF (polyacrylamide – glass fiber fabric), hydrogel composites, and PDMS-GF (polydimethylsiloxane – glass fiber fabric) elastomer composite. This study is an example of soft composites with remarkable fracture resistance and provides a good guide to understanding the synergy between hydrogel and fibers. Hydrogels consist of a soft material that could be used in composites that require softness whilst mechanical properties are also required.

Other promising FRSCs applications are 4D printing, biomimetic composites, and embedded sensing/actuation. Spackman et al.[29] studied 3D printing of FRSCs and reported limitations in this type of printing due to a lack of control over the positioning of fibrous structures. One way to mitigate this limitation is to develop laminated FRFCs, which deliver regular composite structure and improvements on properties of fiber alignment when printing, through a combination of an ultraviolet curable polymer, providing better mechanical properties to the soft material.

Illeperuma et al.[30] report another type of matrix used in fiber-reinforced composites: hybrid hydrogels. It is challenging to develop a matrix based on hydrogel and use strong fiber to reinforce this material, but new techniques to improve the toughness of hydrogel are being sought to combine this property and stretchability through networks with covalent and ionic cross-links.

Recently, Ren et al.[31] point out the importance of fiber-reinforced polymer nowadays in new high-technology fields and the opportunity to metal replacement in important areas like the aeronautics-aerospace industry, new energy, and military field.

Basalt fibers (BF) consist of fibers derived from salt rock (volcanic stone) with minerals like plagioclase, pyroxene, and olivine. The features of this fiber are very interesting for FRP application on aerospace, automobile, and navy, as this material is considered more mechanically resistant than GF (glass fiber), eco-friendly, non-toxic to humans, chemically resistant, corrosion-resistant, non-combustible, and stable at high temperatures (above 900 °C). The disadvantage of BF and GF is their high electrical resistance that can interfere in electrostatic discharge, electromagnetic interference shielding, and electric heating. However, CF are being used in composites to complement functions in which GF and BF perform poorly: electrical, thermal, and mechanical properties[10,32,33]. Lopes and D'Almeida[34] studied CF- reinforced ABS (acrylonitrile butadiene styrene) and concluded that the inclusion of CF in the mixture improved thermal stability and mechanical properties in the composite.

Regarding NFRPC (natural fiber-reinforced polymer composite) produced from plant matter, Bledzki et al.[35] reported wood fiber as the lignocellulosic natural fiber most used to reinforce plastic materials. Nevertheless, with the advances in natural fiber treatment, other sources are being studied as reinforcement, such as barley husk, coconut shell, banana, jute, cotton, agave, flax, and others[2,12,13,35,36].

According to Yang et al.[37], aramid fibers present low density, high rigidity, high strength, and high specific modulus. Their main drawback consists in poor interfacial adhesion with common industrial resins; although it could be improved through chemical treatment of the fiber surface with acid solutions, fluorinated compounds, Polyvinyl alcohol (PVA), and dopamine auto-oxidative polymerization with grafting to promote effective chemical bonds and increase adhesion. Plasma and gamma irradiation could also be used.

Thomason[38] recognized the technical importance of characterizing the nature of GF used in FRP production in order to improve quality control, develop new materials and study the prediction of processability influence and composite performance. The study highlights the growing relevance of the analytical methods for polymeric GF sizing in industry and research and synthesizes the main contributions to the field. The set of analyses addressed by Thomason included: X-ray photoelectron spectroscopy (XPS), secondary ion mass spectroscopy (SIMS), electrokinetic analysis (EKA), thermogravimetric analysis (TGA), scanning electron microscopy (SEM), contact angle (CA), dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC), DMA, nuclear magnetic resonance (NMR), FT-IR, ultraviolet spectroscopy (UVS), gel permeation chromatography (GPC), high-performance liquid chromatography (HPLC).

As described above, there are several types of FRP to be explored according to fiber surface treatment, advances in the polymeric matrix, and so on. Thus, the main purpose of this review is to address the development, advances, and state-of-the-art on characterization methodologies of FRP composites according to the trends in this class of composites in constant evolution to attend needs in key areas of scientific progress. Furthermore, this review also discusses some applications and the diverse materials used to develop FRP and presents complementary information to recent reviews published on FRP composites[5,13-15,32,39,40].

In addition, different studies on FRP stability are found in the literature and consider many conditions that could affect the material within a reasonable period, and analytical techniques to evaluate the tests. These conditions include the influence of humidity, alkaline and acid solutions, temperature variation, ultraviolet radiation, freeze-thaw and wet-dry cycles, and combined conditions. Considering this, the importance of analytical methodologies in supporting FRP research is clear, and this review will address some techniques widely applied to understand and evaluate different treatments in aging experiments[40].

We also evaluate the most important analytical techniques to develop FRP composites, and the main results on their characterization based on the literature.

2. SEM and SEM/EDS (Scanning electron microscopy/ energy-dispersive X-ray spectroscopy)

Composites with chemical-treated fibers are widely analyzed by SEM in order to visually analyze the interaction of fiber and matrix, indicating the adhesion process, fractures, and presence of gaps between fibers and matrix. El-Shekeil et al.[41] studied the effects of treatment of kenaf (*Hibiscus cannabinus*) fiber-reinforced thermoplastic polyurethane composite and the SEM images suggested that NaOH (sodium hydroxide) + pMDI (polymeric methylene diphenyl diisocyanate) chemical treatment in kenaf fibers presented better results on wetting and adhesion in the studied composite.

On composites produced with CF and ABS, Lopes and D'Almeida[34] performed SEM to observe the expected voids previously reported in the literature, and voids between the CF surface and ABS matrix due to the cooling after the extrusion process. SEM analysis also helped to elucidate the adhesion around the fibers and the mechanism of fractures in neat ABS and reinforced composites.

Furthermore, SEM also contributes to confirming the uniformity of reinforced fibers in the hydrogel matrix. Martin and Youssef[42] used 2wt% (weight %) and 3wt% by weight (relative to swelled hydrogel weight) of E-glass fiber to reinforce alginate/PAAm (polyacrylamide) hydrogel and SEM images showed how the fibers were distributed in the top surface of samples ensuring a degree of uniformity on chopped fiber dispersion. This type of hydrogel reinforced with E-glass fiber could present medical applications.

SEM was also applied to obtain microscopy evidence of some deformation and failure mechanisms such as plastic deformation, crazing, crack arrest, crack deflection, and fiber holes[29]. Images supported Wang et al.'s[43] findings on the durability of epoxy-based GF, basalt fibers, and CF-reinforced polymer composite bars during accelerated stability tests, casting light on fracture morphology.

SEM was also applied to understand the effects of seawater aging on different temperatures, saline concentration, and time on CFRP with epoxy resin, in terms of corrosion damage to the matrix, CF morphology, presence of NaCl (sodium chloride), surface morphology, microcracking, and delamination. These phenomena were evident by images of aging samples[44].

This microscopy can be associated with energy-dispersive X-ray spectroscopy (EDS) to perform elementary analysis[45]. This coupled analysis is widely used to characterize minerals, metallic materials, composites, microplastics, and micro and nanomaterials based on polymers.

3. DSC

DSC is a fundamental thermal analysis applied to evaluate phase transitions (e.g., glass transition, melting, crystallization) and chemical reactions (e.g., curing, oxidation), as a function of temperature, by equipment that consists of a furnace and electronic system able to register the difference in temperature between reference and sample pans according to the heat flow measured in each pan[46,47]. When phase transitions or curing processes are important to be evaluated in FRP composites, DSC analysis is always required.

As some epoxy resins are commercialized in a pre-preg material, where this thermoset matrix is partially cured in the fibers of the composite to facilitate the handling of material, it is needed to evaluate the conditions of the curing process by DSC to characterize the material[47].

Bio-composites are being studied to replace synthetic composites, mainly to promote more sustainable products. Yu et al.[48] reported extensive use of bio-based thermosets in FRP during the last several years. Ferdosian et al.[49] studied the performance of bio-based epoxy composites.

In the bio-polymer field, chicken feathers are being used as reinforcement fibers in the matrix, as these natural sources have interesting chemical (presence of ~90% of keratin protein), physical (low electrical conductivity), and morphological properties; besides the environmental benefits that, associated with synthetic resins, result in applications ranging from electrical insulators to biodegradable plastics. Chicken feathers were studied via DSC to understand the thermal transition temperatures after chemical treatments with sodium dodecyl sulfate and hydrogen peroxide. The authors suggest that chicken feathers have the potential to be used as reinforced fibers in composites due to their properties, and also their light weight[50].

Wang and ElGawady[51] studied the influence of moisture in concrete-filled epoxy-based GF-reinforced polymer tubes, specifically in epoxy-based GF. It was observed that the glass transition temperature (Tg) decreased after contact with humidity due to the plasticizing effect of water when epoxy resin absorbs it.

Mgbemena et al.[24] applied DSC to comprehend the shift of Tg via the design of stability studies of FRP composite, and to verify the cure of the polymeric resin. Therefore, this thermoanalytical technique is essential to characterize the phases of composites, aging, and plasticizing process of the polymeric matrix.

4. TGA

TGA analysis constitutes a well-known thermoanalytical technique that addresses the thermal stability of samples by monitoring the weight of the sample over time and temperature increase, with a controlled flow of gas during the test to ensure the use of inert or oxidizing gas[46,52].

This technique is being used to measure the fiber content in FRP and has the benefit of being faster and requiring less material than digestion methods[53].

Moon et al.[54] applied TGA to determine GF and CF contents of epoxy composites. The different conditions were tested to optimize the fiber content measurement. The advantages of this method relate to being an easy test to carry out, as it is not demanded constant reweighing and requires a small sample to test.

Another contribution of TGA analysis is related to studies of thermal stability of FRP composites. For instance, CF and aramid fibers were studied in a matrix of polybenzoxazine to indicate their function to provide more thermal stability to FRP composites. CF performed better than Kevlar because of higher carbon content, and the presence of graphite in CF structures[55].

Mak and Fan[56] also investigated the influence of wet-dry cycles in NFRP based on flax and epoxy resin by TGA. The resulting peak derivative temperature, representative of cellulose degradation in the samples, was considered evidence of the reduction of thermal stability of flax.

Lopes and D'Almeida[34] studied the thermal stability of CF-reinforced ABS by TGA methodology. Three heating rates (10, 20, and 30 ºC/min) in an inert atmosphere (N_2-Nitrogen gas) and different decomposition levels (2.5 to 20% degradation level) in the samples were applied to investigate the decomposition kinetics using Flynn-Wall calculation that determines the activation energy in each decomposition level per sample. From this calculation, it was inferred that the thermal stability of samples increased or decreased according to the changes in the composition of samples.

5. FT-IR

FT-IR spectroscopy is a well-known analytical technique used in many fields of science and technology to identify and characterize substances or materials that absorb specific infrared radiation bands related to different molecular vibration levels. Through infrared spectroscopy, it is possible to evaluate surface and interfacial phenomena, and complex mixtures, by interpretation of spectra in three different regions of the infrared spectral range[57]:

- NIR (near-infrared): 14000 – 4000 cm^{-1};
- MIR (medium infrared): 4000 – 400 cm^{-1}, where it is found the fingerprint region (the region with main fundamental bands of MIR: 1500 – 400 cm^{-1});
- FIR (far infrared): 500 – 50 cm^{-1}.

FT-IR is also used to characterize the materials used to produce FRP and to evaluate the potential degradation of the polymeric matrix during manufacturing. As studied by Lopes and D'Almeida[34], the CF-reinforced ABS with a variation of fiber concentration and length produced via extrusion, was investigated to understand the degradation process of ABS (180 - 220 °C) and the interaction of this polymer with CF through ATR (attenuated total reflection) mode in the medium infrared region (4000 – 450 cm^{-1}). The results indicated degradation over 200 °C, it was observed absorbance in the region related to the stretching of the carbonyl group (C=O) at 1690 and 1800 cm^{-1}. As ABS does not present oxygen in its molecular structure, this result shows that oxidation is occurring during the extrusion. No influence of the interaction of CF in the matrix in polymer degradation was observed.

Chua et al.[58] applied FT-IR using ATR to observe the surface chemical composition changes along the aging process (37°C for 1, 3, 6, and 12 months) of CFRP for implementable medical devices. CFRP discs with continuous and discontinuous CF, and different matrices based on epoxy resins or vinyl ester. 3D printing fabrication was also tested employing fused filament fabrication technology with a PA (polyamide) thermoplastic matrix. They observed some functional groups like C=O (at 1730 cm^{-1}) and C-O (1240 cm^{-1}) indicating an oxidative process from months 0 to 12. Another band also evaluated by this study was about 3400 cm^{-1}, related to O-H stretching and the absorption of water during the aging process. The authors correlated the FT-IR results with an EDS performed in tandem with SEM. Although EDS accused in all samples a significant increase of oxygen level from 1 to 3 months, the FT-IR evaluation did not show the same tendency[58]. This difference observed between methodologies could be derived from the contact of the sample with the crystal in FT-IR analysis. The ATR is very dependent on good contact between the sample and crystal, as mentioned by Sanches et al.[59].

Wang and ElGawady[51] studied concrete filled epoxy-based GF-reinforced polymer tubes to understand the moisture effect in the GFRP, as epoxy can absorb up to 7% moisture by weight, according to the authors, and it will reflect directly in the mechanical properties of the final material. GFRP was analyzed by transmission mode using KBr (potassium bromide) pellets with a ratio of 1:10. The authors considered the carbon-hydrogen bond (-CH) constant in the GFRP and used the OH/CH ratio as an indicator of moisture absorption in the resin. The -OH was assigned a wavenumber of 3421 cm^{-1}, and -CH, 2926 cm^{-1}. The authors also mention the importance of this analysis to understand the reduction of the Tg of resin, as moisture can plasticize the epoxy resin and, consequently, cause changes in Tg.

Thomason[38] in his review of polymeric GF sizing applied diffuse reflectance Fourier transform infrared (DRIFT) mode to analyze silanes and sizing used in the coating of plates and fibers. The author also referred that DRIFT mode was also carried out in a combination of XPS and CA to study the modification of chopped E-glass with long-chain alcohol adsorption.

Magalhães et al.[60] discusses DRIFT mode regarding the sampling depth degree, and that it is not recommended to characterize chemically the surface of Vectran™ fibers, as it is not considered a selective mode for this purpose. According to the authors, other ways of obtaining spectra by reflection, such as ATR or universal attenuated total reflection (UATR), or obtaining spectra by photoacoustic spectroscopy (PAS) detection could be more appropriate to investigate surface treatment in fibers.

Another important type of synthetic fiber used worldwide to reinforce polymer composite is Kevlar, as these fibers are considered chemically inert and present high tenacity[61,62]. However, Kevlar fibers have a smooth surface, and this physical characteristic requires a surface modification, according to the matrix to be used with them. For this reason, Lin[61] studied the grafting of Kevlar fiber surfaces with bromoacetic acid at 50°C/10h, and epichlorohydrin at 25°C/8h. Infrared spectroscopy showed the presence of the carboxyl group (1750 cm^{-1}) when the fiber was treated bay bromoacetic acid and the epoxy group (2990 cm^{-1}) with epichlorohydrin treatment. According to the results presented by the spectra, it seems that the reflection mode was carried out to perform this experiment and this shows the importance of the technique to solve the surface characterization of fibers.

Kondo et al.[63] also applied FT-IR ATR mode to identify the grafting of 3-Acryloxypropyltrimethoxysilane (APTMS) by electron beam irradiation in PET (polyethylene terephthalate) fibers, using C=C in the vinyl group of APTMS as a marker with an absorption peak at 1639 cm^{-1} to accuse the surface presence of APTMS. This was corroborated by SEM/EDS analysis, which provided a mapping of silicon (Si), fundamental to confirm the uniform coating of siloxane linkage by electron beam irradiation.

Especially in natural fiber composites, moisture or the presence of humidity can negatively influence the mechanical properties of the composite, as it can lead to interface degradation. It happens for the natural fibers have hydrophilic properties, and absorb more water than the resin normally used in this type of natural FRP; this condition reflecting in swelling of fibers, micro-cracking in the composite, and loss of interfacial adhesion due to the stress induced by water absorption. This results in a lack of adhesion[41,55]. Another drawback is less durability due to high moisture and chemical absorption[12].

It is recommended to use the reflection techniques, such as UATR or ATR rather than the transmission technique to evaluate the influence humidity, as reflection techniques do not need to prepare a KBr pellet, which could absorb more humidity and influence the final result.

Wang et al.[43], studying bars of basalt, GF, and CF-reinforced polymer composites candidates as replacements for steel, pointed out an accelerated test of these composites in seawater and sea sand concrete, applying FT-IR to assess the degradation mechanism. They used ATR mode, and the region of hydroxyl stretching (O-H) at 3400 cm^{-1} was studied to understand the indicative of water absorption during the wet-dry cycles purposed by the article.

Bansal et al.[64] studied NFRPCs based on bamboo, jute, and coir fibers, with an epoxy resin in random orientations, and discussed the characterization of matrix by FT-IR, applied as a method to differentiate the samples according to the mixture of fibers. Bands related to each type of fiber were detected and they can be used for diagnostic differentiation.

Ramachandran et al.[65] studied bamboo, banana, and linen fibers cut into 2-4 mm of length, with epoxy resin in random orientations. In order to characterize the natural fibers, the authors also carried out FT-IR but did not describe the sample preparation and mode of analysis, as well as the previous study[64]. FT-IR studies have particular conditions and preparations, with different modes available to obtain a spectrum. It is recommended to describe this information to understand the real conditions and achieve the same quality of spectrum as the authors. Any modification of condition, preparation, or mode could impact the result of the spectrum, thus these descriptions contribute significantly to the information of a scientific article.

Furthermore, NFRPCs have used coupling agents or compatibilizers to improve the interface between the polymer and natural fiber fillers. Maleic anhydride is commonly used in NFRPCs, being a component able to bond hygroscopic cellulose with a hydrophobic polymer, due to the reaction of anhydride and hydroxyl groups of cellulose with ester bonds or secondary interactions of H-bond. Bajwa et al.[66] used FT-IR with a photoacoustic detector to analyze biochar and oakwood flour as fillers of PLA (polylactic acid) and HDPE (high-density polyethylene) matrices. FT-IR photoacoustic spectroscopy, a non-destructive and near-surface technique, is normally used to analyze infrared spectra of dark samples, once this type of spectroscopy is based on a physical process combining acoustic signal generation and radiation energy absorption regardless of the IR transmission intensity to the detector[66-69].

Senophiyah-Mary and Loganath[70] used printed circuit border (PCB) to obtain carbonaceous slag to reinforce PVA, as an alternative for a membrane used to treat domestic or industrial wastewater. This is an example of FRP performed by electro-spinning used to synthesize a nanofiber membrane. The FT-IR using transmission mode with potassium bromide pellet associated with Raman spectroscopy allowed to demonstrate that carbonization can transform thermoset polymers derived from PCB into useful activated carbon. In this study, Raman added value to carbonaceous formations as the results of the spectrum showed the presence of bands of carbon at 1336-1604 cm^{-1}.

Ji et al.[71] verified the interference of increasing temperature in the curing of amino silane coupling agent by monitoring the shift of amine functional group N-H (1596 to 1566 cm^{-1}) bands. The absorption in the region of C=O (1660 cm^{-1}) due to the reaction of the amino group with CO_2 (carbon dioxide) and H_2O (water) was observed. Other important regions in the reaction of silanization on the surface were related to Si-OH (3355 cm^{-1}) and Si-O-Si (1000 - 1100 cm^{-1}).

In addition, FT-IR could be coupled to TGA, and the analysis of volatile pyrolysis gases can be performed using a heated transfer line and appropriate cell to receive the gases. Perret et al.[72] carried out experiments with this technique to study flame retardants in CF epoxy resins, and used condensed-product analysis at different phases of thermal decomposition.

A short review of Vectran™ fiber explored some conditions and modes of FT-IR in the fiber field. This study also brings concepts of different modes of acquisition of infrared spectra and discusses the sample depth degree according to the chosen mode, focused on the surface analysis by FT-IR techniques, such as ATR, UATR, DRIFT, NIR, NIRA (near-infrared reflectance analysis), FT-IR microscopy and PAS[60]. In addition, Vectran™ fibers are very important in aerospace and military fields because of their high mechanical performance, and these fibers are applied in FRP composites with epoxy matrix[73].

According to Magalhães et al.[60], it is well known that the studies involving NIR region regularly are associated with chemometrics and algorithm based, increasing the complexity of performing such analysis. However, with the advent of transflectance analysis, such as NIRA, polymer analysis could be performed directly in the equipment without any preparation of the sample, being non-destructive and with the advantage of being considered with high penetration of IR (infrared) beams, and high resolution.

After scrutinizing SEM, SEM/EDS, DSC, TGA, and FT-IR performed in FRP composites, Table 1 presents the principal applications of each instrumental technique addressed in this review.

Given the importance of experimental conditions, different materials performing important roles in the FRP composites, and the array of advanced technologies available to characterize these materials, this review was carried out to study fiber reinforcement interaction with matrix, future trends, and principal characterization techniques involved in FRP studies. Different FRP composite configurations found in the literature were listed in Table 2 with techniques performed in the related composites. All FT-IR analyses were carried out in MIR.

6. Trend

According to the studies reviewed, there is an opportunity related to expanding of the use of FT-IR techniques to characterize polymers, further exploring the NIR region and reflectance techniques (such as NIRA). As the spectrum obtained by NIR brings overtone responses and combination bands, this region is also important to FRP characterization, especially when quantitative studies have to be carried out[60].

According to Table 2, some articles report using AFM (atomic force microscopy) to acquire images of FRP composites, which presents some advantages when coupled with IR spectroscopy. Nguyen-Tri et al.[105] described some principles of AFM-IR (atomic force microscopy-based infrared spectroscopy), as well as the correlation between this technique and the chemical characterization of polymers, including crystallization mechanisms, phase separation, and spherulitic structures.

The use of recycled materials, such as CF, has attracted attention as it repurposes waste materials in the end-of-life phase, as well as reduces energy consumption. Fernández et al.[102] studied recycled CF as reinforcement material with PP (polypropylene) by injection process, and suggest that composites made with recycled materials have similar mechanical properties of composites with virgin CF. However, further developments in sizing fiber surfaces could bring more benefits to the use of this eco-friendly material. This study also brings the state-of-the-art on fiber orientation and fiber distribution analysis in composites, using a modern technique of X-ray tomography.

Furthermore, this review reveals a trend in natural sources to develop FRP, as the concerns with sustainability and green alternatives are rising. As an alternative to synthetic material, it has already been reported that bio-based composites are considerably environment-friendly which can reduce the cost, weight of structure, and environmental impacts.

Table 1. Principal instrumental techniques to characterize FRP composites and applications.

Technique	Applications	Ref.
SEM	Morphological assessment; fiber sizing analysis; evaluation of interaction between fibers and matrix; investigation of failures, fractures, adhesion, gaps, corrosion, and deformation.	[29, 34, 41-44, 74-84]
SEM/EDS	Elemental analysis of surfaces after treatment; identification of ratios and chemical composition.	[35, 45, 82, 85-87]
DSC	Determination of phase transition, mainly Tg temperature in the matrix; evaluation of matrix curing process; curing degree.	[24, 36, 46-51, 78-80, 88-90]
TGA	Determination of fiber content; evaluation of thermal stability of composites; assessment of thermal decomposition of materials; characterization of the effects of dehydration and oxidation on material.	[34, 46, 52-56, 78, 80, 84, 91]
FT-IR	Fiber and matrix characterization; assessment of chemical changes, after surface modification of fiber or matrix through specific molecular vibration absorption; relationship between amount of surface treatment of fibers and intensity of infrared absorption ratio in IR spectra; fiber sizing analysis*; degradation studies; aging or stability studies of FRP; water absorption in FRP development; non-destructive analyses: DRIFT, PAS, ATR, microscopy; destructive analysis: KBr pellet analyzed by transmission mode.	[12, 34, 38, 41, 43, 51, 55-73, 82, 84, 92, 93]

* using microscopy features of FT-IR spectrometer coupled with microscopy.

Siglas: SEM: Scanning electron microscopy; SEM/EDS: Scanning electron microscopy/energy-dispersive X-ray spectroscopy; DSC: Differential scanning calorimetry; TGA: Thermogravimetric analysis; FT-IR: Fourier transform-infrared spectroscopy; IR: Infrared; FRP: Fiber-reinforced polymer; DRIFT: Diffuse reflectance Fourier transform infrared; PAS: Photoacoustic spectroscopy; ATR: Attenuated total reflection; KBr: Potassium bromide.

Table 2. FRP composites and characterization techniques with instrumental analysis.

Fiber phase	Matrix phase	Analysis	Ref.
Glass/Kevlar fibers	Epoxy resin	DSC, SEM, TGA	[94]
Coconut fiber	PP	SEM	[95]
Short GF	PBT	DSC, SEM	[96]
GF	PP/EPDM	DSC, SEM, TGA, WAXD	[97]
GF and CF	Epoxy resin	SEM, TGA	[54]
GF	Epoxy resin	AFM, SEM	[98]
Cellulose whisker	PVA	DMA, DSC, SEM	[99]
PET fiber	Natural rubber	FT-IR, SEM/EDS	[63]
GF	Epoxy and polyester resin	SEM	[7]
Natural fibers	Polypropylene	FT-IR, SEM/EDS, TGA, UV-VIS	[35]
CF	Epoxy resin	DMA, FT-IR, TGA	[72]
Kenaf fiber	TPU	FT-IR, SEM	[41]
CF	Phenolic resin	FT-IR, LRS, SEM, XPS	[9]
Natural fibers	Epoxy resin	FT-IR	[65]
GF	Epoxy resin	DSC, FT-IR, GPC, titration	[49]
Nylon-6	UV curable polymer	SEM	[29]
BF, GF, and CF	Epoxy resin	FT-IR, SEM	[43]
Natural fibers	Epoxy resin	FT-IR	[64]
GF fabric	Polyampholyte gel, NaSS, and DMAEA-Q	SEM	[28]
Kevlar fibers and CF	Polybenzoxazine resins	FT-IR, SEM, TGA	[55]
GF	PAAm	DMA, SEM	[42]
UHMWPE	RPU	CA, FT-IR, SEM	[100]
Lignocellulose fiber	Ethylene-norbornene copolymer	AFM, DLS, DMA, DSC, FT-IR, TGA	[19]
GF and flax fibers	Epoxy resin	DSC, FT-IR, SEM, TGA	[36]
GF	Epoxy resin	DSC, FT-IR, SEM/EDS	[51]
GF	PEN-BAPh	DSC, DRA, FT-IR, SEM, TGA, UV-VIS	[31]
MWCNT-coated BF	Epoxy resin	FT-IR, LRS, SEM, XPS	[33]
Wood fiber	HDPE and PLA	DMA, FT-IR, SEM, TGA	[66]
CF	ABS	FT-IR, SEM, TGA	[34]
CF	Epoxy resin	DMA, FT-IR, SEM	[44]
Short CF	Cellulose; PA 6 and PP	DSC, SEM, TGA,XPS	[25]
Sugarcane fibers	PP	SEM	[101]
CF	Epoxy resin	DSC, FT-IR, Rheology, TGA	[89]
CF	PEEK-Ti laminates	FT-IR, SEM, XPS	[71]
GF and flax fibers	Epoxy resin	DSC, FT-IR, SEM, TGA	[56]
Carbonaceus slag from thermoset	PVA	AFM, FT-IR, LRS, TGA	[70]
Natural fiber and GF	Vinyl Ester	DSC, TGA	[88]
Flax fiber	Polyester	^{1}H NMR, FT-IR, SEM/EDS	[82]
CF	PEEK	AFM, FT-IR, SEM/EDS, XPS, WCA	[87]
BF	HDPE	FT-IR, DSC, SEM/EDS, TGA	[92]
Bagasse fiber	Cardanol	FT-IR, SEM, TGA	[84]
Recycled CF	PP and PP-MAH	DSC, SEM, TGA, XCT, XPS	[102]
CF	Epoxy resin	AFM, FT-IR, SEM/EDS	[58]
Flax fiber / PLA woven	Epoxy resin	DMA, DSC, SEM, TGA	[80]
CF	Epoxy resin doped with graphene oxide	DLS, FT-IR, Raman, SEM	[103]
Jute fiber	Bio-based vanillin-derived epoxy	FT-IR, NMR, SEM, tensile test, TGA, WCA	[104]

Siglas: DSC: Differential scanning calorimetry; SEM: Scanning electron microscopy; TGA: Thermogravimetric analysis; PP: Polypropylene; GF: Glass fibers; PBT: Poly(butylene terephthalate); EPDM: Ethylene–propylene–diene terpolymer; WAXD: Wide-angle X-ray diffraction; CF: Carbon fibers; AFM: Atomic force microscopy; PVA: Polyvinyl alcohol; DMA: Dynamic mechanical analysis; PET: Polyethylene terephthalate; FT-IR: Fourier transform-infrared spectroscopy; SEM/EDS: Scanning electron microscopy/energy-dispersive X-ray spectroscopy; UV-VIS: Ultraviolet and visible spectroscopy; TPU: Thermoplastic polyurethane; LRS: Laser Raman scattering; XPS: X-ray photoelectron spectroscopy; GPC: Gel permeation chromatography; UV: Ultraviolet; BF: Basalt fibers; NaSS: Copolymerized from sodium p-styrenesulfonate; DMAEA-Q: Dimethylaminoethylacrylate quaternized ammonium; PAAm: Polyacrylamide; UHMWPE: Ultra-high molecular weight polyethylene; RPU: Rigid polyurethane; CA: Contact angle; DLS: Dynamic light scattering; PEN-BAPh: Phthalonitrile containing aromatic ether nitrile linkage; DRA: Dynamic rheological analysis; MWCNT: Multi-walled carbon nanotube; HDPE: High-density polyethylene; PLA: Polylactic acid; ABS: Acrylonitrile butadiene styrene; PA: Polyamide; PEEK-Ti: Polyetheretherketone-titanium; ^{1}H NMR: Proton nuclear magnetic resonance spectroscopy; PEEK: Polyetheretherketone; WCA: Water contact angle; PP-MAH: Maleic anhydride grafted polypropylene; XCT: X-ray computed assisted tomography; NMR: nuclear magnetic resonance.

7. Conclusion

FRP composites are very important materials in many fields of science and technology, with several studies being developed to create more resistant, sustainable, and advanced materials. Studies with natural fibers including plants, and mineral fibers or matrices based on cellulose have been developed to contribute to more ecological alternatives for the near future.

A comprehensive review was conducted in FRP composites investigating the principal characterization techniques performed to study new developments on these composites, including aging or stability testing, sizing on fibers, interfacial properties between matrix and fibers, and material treatment.

SEM, SEM/EDS, DSC, TGA, FT-IR, and other analytical techniques were discussed, and many applications to study FRP composites were proposed. Through the reviewed experimental studies, it is possible to conclude that two or more associated techniques provide more support for composite development.

Finally, this review provides a systematic understanding of FRP composite applications for the development and characterization of these materials, as well as bringing the latest advances in this segment of material science.

8. Author's Contribution

- **Conceptualization** – Marcia Murakoshi Takematsu.
- **Data curation** – NA.
- **Formal analysis** – NA.
- **Funding acquisition** - Rita de Cássia Lazzarini Dutra.
- **Investigation** – Marcia Murakoshi Takematsu.
- **Methodology** – Marcia Murakoshi Takematsu.
- **Project administration** – Rita de Cássia Lazzarini Dutra.
- **Resources** – Rita de Cássia Lazzarini Dutra.
- **Software** – NA.
- **Supervision** – Rita de Cássia Lazzarini Dutra.
- **Validation** – NA.
- **Visualization** – Marcia Murakoshi Takematsu.
- **Writing – original draft** – Marcia Murakoshi Takematsu.
- **Writing – review & editing** – Rita de Cássia Lazzarini Dutra.

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10. References

1. Hui, C., Liu, Z., Phoenix, S. L., King, D. R., Cui, W., Huang, Y., & Gong, J. P. (2020). Mechanical behavior of unidirectional fiber reinforced soft composites. *Extreme Mechanics Letters*, *35*, 100642. http://dx.doi.org/10.1016/j.eml.2020.100642.
2. Gouanvé, F., Marais, S., Bessadok, A., Langevin, D., & Métayer, M. (2007). Kinetics of water sorption in flax and PET fibers. *European Polymer Journal*, *43*(2), 586-598. http://dx.doi.org/10.1016/j.eurpolymj.2006.10.023.
3. Balla, V. K., Kate, K. H., Satyavolu, J., Singh, P., & Tadimeti, J. G. D. (2019). Additive manufacturing of natural fiber reinforced polymer composites: processing and prospects. *Composites. Part B, Engineering*, *174*, 106956. http://dx.doi.org/10.1016/j.compositesb.2019.106956.
4. Hofstätter, T., Pedersen, D. B., Tosello, G., & Hansen, H. N. (2017). State-of-the-art of fiber-reinforced polymers in additive manufacturing technologies. *Journal of Reinforced Plastics and Composites*, *36*(15), 1061-1073. http://dx.doi.org/10.1177/0731684417695648.
5. Chavhan, G. R., & Wankhade, L. N. (2020). Improvement of the mechanical properties of hybrid composites prepared by fibers, fiber-metals, and nano-filler particles-A review. *Materials Today: Proceedings*, *27*(Part 1), 72-82. http://dx.doi.org/10.1016/j.matpr.2019.08.240.
6. Suman, J. N., Kathi, J., & Tammishetti, S. (2005). Thermoplastic modification of monomeric and partially polymerized Bisphenol A dicyanate ester. *European Polymer Journal*, *41*(12), 2963-2972. http://dx.doi.org/10.1016/j.eurpolymj.2005.06.006.
7. Pihtili, H. (2009). An experimental investigation of wear of glass fibre-epoxy resin and glass fibre-polyester resin composite materials. *European Polymer Journal*, *45*(1), 149-154. http://dx.doi.org/10.1016/j.eurpolymj.2008.10.006.
8. Suresh, G., & Jayakumari, L. S. (2015). Evaluating the mechanical properties of E-Glass fiber/carbon fiber reinforced interpenetrating polymer networks. *Polimeros: Ciência e Tecnologia*, *25*(1), 49-57. http://dx.doi.org/10.1590/0104-1428.1650.
9. Yuan, H., Wang, C., Zhang, S., & Lin, X. (2012). Effect of surface modification on carbon fiber and its reinforced phenolic matrix composite. *Applied Surface Science*, *259*, 288-293. http://dx.doi.org/10.1016/j.apsusc.2012.07.034.
10. Sanman, S., Manjunath, A., Prashanth, K. P., Shadakshari, R., & Sunil, S. K. (2023). An experimental study on two body abrasive wear behavior of natural fiber reinforced hybrid polymer matrix composites using Taguchi analysis. *Materials Today: Proceedings*, *72*(Part 4), 2021-2026. http://dx.doi.org/10.1016/j.matpr.2022.07.400.
11. Soutis, C. (2005). Fibre reinforced composites in aircraft construction. *Progress in Aerospace Sciences*, *41*(2), 143-151. http://dx.doi.org/10.1016/j.paerosci.2005.02.004.
12. Verma, D., & Senal, I. (2019). *Natural fiber-reinforced polymer composites: feasibiliy study for sustainable automotive industries.* In D. Verma, E. Fortunati, S. Jain, & X. Zhang (Eds.), *Biomass, biopolymer-based materials, and bioenergy: construction, biomedical, and other industrial applications* (pp. 103–122). UK: Woodhead Publishing. http://dx.doi.org/10.1016/B978-0-08-102426-3.00006-0
13. Raju, A., & Shanmugaraja, M. (2021). Recent researches in fiber reinforced composite materials: a review. *Materials Today: Proceedings*, *46*(Part 19), 9291-9296. http://dx.doi.org/10.1016/j.matpr.2020.02.141.
14. Kerni, L., Singh, S., Patnaik, A., & Kumar, N. (2020). A review on natural fiber reinforced composites. *Materials Today: Proceedings*, *28*(Part 3), 1616-1621. http://dx.doi.org/10.1016/j.matpr.2020.04.851.
15. Chaudhary, V., & Ahmad, F. (2020). A review on plant fiber reinforced thermoset polymers for structural and frictional composites. *Polymer Testing*, *91*, 106792. http://dx.doi.org/10.1016/j.polymertesting.2020.106792.
16. Mahesh, V., Joladarashi, S., & Kulkarni, S. M. (2021). A comprehensive review on material selection for polymer matrix composites subjected to impact load. *Defence Technology*, *17*(1), 257-277. http://dx.doi.org/10.1016/j.dt.2020.04.002.

17. Pukánszky, B. (2005). Interfaces and interphases in multicomponent materials: Past, present, future. *European Polymer Journal*, *41*(4), 645-662. http://dx.doi.org/10.1016/j.eurpolymj.2004.10.035.
18. Krouit, M., Bras, J., & Belgacem, M. N. (2008). Cellulose surface grafting with polycaprolactone by heterogeneous click-chemistry. *European Polymer Journal*, *44*(12), 4074-4081. http://dx.doi.org/10.1016/j.eurpolymj.2008.09.016.
19. Wolski, K., Cichosz, S., & Masek, A. (2019). Surface hydrophobisation of lignocellulosic waste for the preparation of biothermoelastoplastic composites. *European Polymer Journal*, *118*, 481-491. http://dx.doi.org/10.1016/j.eurpolymj.2019.06.026.
20. Tribot, A., Amer, G., Alio, M. A., Baynast, H., Delattre, C., Pons, A., Mathias, J.-D., Callois, J.-M., Vial, C., Michaud, P., & Dussap, C.-G. (2019). Wood-lignin: supply, extraction processes and use as bio-based material. *European Polymer Journal*, *112*, 228-240. http://dx.doi.org/10.1016/j.eurpolymj.2019.01.007.
21. Rajak, D. K., Pagar, D. D., Menezes, P. L., & Linul, E. (2019). Fiber-reinforced polymer composites: Manufacturing, properties, and applications. *Polymers*, *11*(10), 1667. http://dx.doi.org/10.3390/polym11101667. PMid:31614875.
22. Picha, K., & Samuel, J. (2018). A model-based prediction of droplet shape evolution during additive manufacturing of aligned fiber-reinforced soft composites. *Journal of Manufacturing Processes*, *32*, 816-827. http://dx.doi.org/10.1016/j.jmapro.2018.03.012.
23. Alemour, B., Badran, O., & Hassan, M. R. (2019). A review of using conductive composite materials in solving lightening strike and ice accumulation problems in aviation. *Journal of Aerospace Technology and Management*, *11*, e1919. http://dx.doi.org/10.5028/jatm.v11.1022.
24. Mgbemena, C. O., Li, D., Lin, M.-F., Liddel, P. D., Katnam, K. B., Kumar, V. T., & Nezhad, H. Y. (2018). Accelerated microwave curing of fibre-reinforced thermoset polymer composites for structural applications: a review of scientific challenges. *Composites. Part A, Applied Science and Manufacturing*, *115*, 88-103. http://dx.doi.org/10.1016/j.compositesa.2018.09.012.
25. Szabó, L., Milotskyi, R., Fujie, T., Tsukegi, T., Wada, N., Ninomiya, K., & Takahashi, K. (2019). Short carbon fiber reinforced polymers: utilizing lignin to engineer potentially sustainable resource-based biocomposites. *Frontiers in Chemistry*, *7*, 757. http://dx.doi.org/10.3389/fchem.2019.00757. PMid:31781540.
26. Imre, B., & Pukánszky, B. (2013). Compatibilization in bio-based and biodegradable polymer blends. *European Polymer Journal*, *49*(6), 1215-1233. http://dx.doi.org/10.1016/j.eurpolymj.2013.01.019.
27. Zindani, D., & Kumar, K. (2019). An insight into additive manufacturing of fiber reinforced polymer composite. *International Journal of Lightweight Materials and Manufacture*, *2*(4), 267-278. http://dx.doi.org/10.1016/j.ijlmm.2019.08.004.
28. Huang, Y., King, D. R., Sun, T. L., Nonoyama, T., Kurokawa, T., Nakajima, T., & Gong, J. P. (2017). Energy-dissipative matrices enable synergistic toughening in fiber reinforced soft composites. *Advanced Functional Materials*, *27*(9), 1605350. http://dx.doi.org/10.1002/adfm.201605350.
29. Spackman, C. C., Frank, C. R., Picha, K. C., & Samuel, J. (2016). 3D printing of fiber-reinforced soft composites: process study and material characterization. *Journal of Manufacturing Processes*, *23*, 296-305. http://dx.doi.org/10.1016/j.jmapro.2016.04.006.
30. Illeperuma, W. R. K., Sun, J.-Y., Suo, Z., & Vlassak, J. J. (2014). Fiber-reinforced tough hydrogels. *Extreme Mechanics Letters*, *1*, 90-96. http://dx.doi.org/10.1016/j.eml.2014.11.001.
31. Ren, D., Li, K., Chen, L., Chen, S., Han, M., Xu, M., & Liu, X. (2019). Modification on glass fiber surface and their improved properties of fiber-reinforced composites via enhanced interfacial properties. *Composites. Part B, Engineering*, *177*, 107419. http://dx.doi.org/10.1016/j.compositesb.2019.107419.
32. Prasanna, S. M., Yogesha, K. B., Mruthunjaya, M., Shivakumar, B. P., Siddappa, P. N., & Raju, B. R. (2019). Mechanical and tribological characterization of hybrid composites: a review. *Materials Today: Proceedings*, *22*(Part 4), 2351-2358.
33. Kim, M., Lee, T.-W., Park, S.-M., & Jeong, Y. G. (2019). Structures, electrical and mechanical properties of epoxy composites reinforced with MWCNT-coated basalt fibers. *Composites. Part A, Applied Science and Manufacturing*, *123*, 123-131. http://dx.doi.org/10.1016/j.compositesa.2019.05.011.
34. Lopes, B. J., & D'Almeida, J. R. M. (2019). Initial development and characterization of carbon fiber reinforced ABS for future Additive Manufacturing applications. *Materials Today: Proceedings*, *8*(Part 3), 719-730. http://dx.doi.org/10.1016/j.matpr.2019.02.013.
35. Bledzki, A. K., Mamun, A. A., & Volk, J. (2010). Barley husk and coconut shell reinforced polypropylene composites: the effect of fibre physical, chemical and surface properties. *Composites Science and Technology*, *70*(5), 840-846. http://dx.doi.org/10.1016/j.compscitech.2010.01.022.
36. Mak, K., & Fam, A. (2019). Freeze-thaw cycling effect on tensile properties of unidirectional flax fiber reinforced polymers. *Composites. Part B, Engineering*, *174*, 106960. http://dx.doi.org/10.1016/j.compositesb.2019.106960.
37. Yang, G., Park, M., & Park, S.-J. (2019). Recent progresses of fabrication and characterization of fibers-reinforced composites: A review. *Composites Communications*, *14*, 34-42. http://dx.doi.org/10.1016/j.coco.2019.05.004.
38. Thomason, J. (2020). A review of the analysis and characterisation of polymeric glass fibre sizings. *Polymer Testing*, *85*, 106421. http://dx.doi.org/10.1016/j.polymertesting.2020.106421.
39. Benin, S. R., Kannan, S., Bright, R. J., & Moses, A. J. (2020). A review on mechanical characterization of polymer matrix composites & its effects reinforced with various natural fibres. *Materials Today: Proceedings*, *33*(Part 1), 798-805. http://dx.doi.org/10.1016/j.matpr.2020.06.259.
40. Liu, T. Q., Liu, X., & Feng, P. (2020). A comprehensive review on mechanical properties of pultruded FRP composites subjected to long-term environmental effects. *Composites. Part B, Engineering*, *191*, 1079. http://dx.doi.org/10.1016/j.compositesb.2020.107958.
41. El-Shekeil, Y. A., Sapuan, S. M., Khalina, A., Zainudin, E. S., & Al-Shuja'a, O. M. (2012). Influence of chemical treatment on the tensile properties of kenaf fiber reinforced thermoplastic polyurethane composite. *Express Polymer Letters*, *6*(12), 1032-1040. http://dx.doi.org/10.3144/expresspolymlett.2012.108.
42. Martin, N., & Youssef, G. (2018). Journal of the Mechanical Behavior of Biomedical Materials Dynamic properties of hydrogels and fi ber-reinforced hydrogels. *Journal of the Mechanical Behavior of Biomedical Materials*, *85*, 194-200. http://dx.doi.org/10.1016/j.jmbbm.2018.06.008. PMid:29908487.
43. Wang, Z., Zhao, X.-L., Xian, G., Wu, G., Raman, R. K. S., & Al-Saadi, S. (2017). Durability study on interlaminar shear behaviour of basalt-, glass- and carbon-fibre reinforced polymer (B/G/CFRP) bars in seawater sea sand concrete environment. *Construction & Building Materials*, *156*, 985-1004. http://dx.doi.org/10.1016/j.conbuildmat.2017.09.045.
44. Li, H., Zhang, K., Fan, X., Cheng, H., Xu, G., & Suo, H. (2019). Effect of seawater ageing with different temperatures and concentrations on static/dynamic mechanical properties of carbon fiber reinforced polymer composites. *Composites. Part B, Engineering*, *173*, 106910. http://dx.doi.org/10.1016/j.compositesb.2019.106910.
45. Newbury, D. E., & Ritchie, N. W. M. (2015). Performing elemental microanalysis with high accuracy and high precision by scanning electron microscopy/silicon drift detector energy-dispersive X-ray spectrometry (SEM/SDD-EDS). *Journal of Materials Science*, *50*(2), 493-518. http://dx.doi.org/10.1007/s10853-014-8685-2. PMid:26346887.

46. Canevarolo, S. V., Jr (2003). *Técnicas de caracterização de polímeros.* São Paulo: Artliber Editora.
47. Vijaya Kumar, K., Shailesh, P., Ranga Babu, J. A., & Kumar Puli, R. (2017). Preparation and characterization of GFRPC material. *Materials Today: Proceedings*, *4*(2), 3053-3061. http://dx.doi.org/10.1016/j.matpr.2017.02.188.
48. Yu, A. Z., Rahimi, A. R., & Webster, D. C. (2018). High performance bio-based thermosets from dimethacrylated epoxidized sucrose soyate (DMESS). *European Polymer Journal*, *99*, 202-211. http://dx.doi.org/10.1016/j.eurpolymj.2017.12.023.
49. Ferdosian, F., Zhang, Y., Yuan, Z., Anderson, M., & Xu, C. (2016). Curing kinetics and mechanical properties of bio-based epoxy composites comprising lignin-based epoxy resins. *European Polymer Journal*, *82*, 153-165. http://dx.doi.org/10.1016/j.eurpolymj.2016.07.014.
50. Tesfaye, T., Sithole, B., Ramjugernath, D., & Mokhothu, T. (2018). Valorisation of chicken feathers: characterisation of thermal, mechanical and electrical properties. *Sustainable Chemistry and Pharmacy*, *9*, 27-34. http://dx.doi.org/10.1016/j.scp.2018.05.003.
51. Wang, S., & ElGawady, M. A. (2019). Effects of hybrid water Immersion, environmental exposures, and axial load on the mechanical properties of concrete filled epoxy-based glass fiber reinforced polymer tubes. *Construction & Building Materials*, *194*, 311-321. http://dx.doi.org/10.1016/j.conbuildmat.2018.10.232.
52. Shubham, S. K., Purohit, R., Yadav, P. S., & Rana, R. S. (2019). Study of nano-fillers embedded in polymer matrix composites to enhance its properties: a review. *Materials Today: Proceedings*, *26*(Part 2), 3024-3029.
53. Yee, R. Y., & Stephens, T. S. (1996). A TGA technique for determining graphite fiber content in epoxy composites. *Thermochimica Acta*, *272*, 191-199. http://dx.doi.org/10.1016/0040-6031(95)02606-1.
54. Moon, C., Bang, B., Choi, W., Kang, G., & Park, S. (2005). A technique for determining fiber content in FRP by thermogravimetric analyzer. *Polymer Testing*, *24*(3), 376-380. http://dx.doi.org/10.1016/j.polymertesting.2004.10.002.
55. Ghouti, H. A., Zegaoui, A., Derradji, M., Cai, W.-A., Wang, J., Liu, W.-B., & Dayo, A. Q. (2018). Multifunctional hybrid composites with enhanced mechanical and thermal properties based on polybenzoxazine and chopped Kevlar/carbon hybrid fibers. *Polymers*, *10*(12), 1308. http://dx.doi.org/10.3390/polym10121308. PMid:30961233.
56. Mak, K., & Fam, A. (2020). The effect of wet-dry cycles on tensile properties of unidirectional flax fiber reinforced polymers. *Composites. Part B, Engineering*, *183*, 107645. http://dx.doi.org/10.1016/j.compositesb.2019.107645.
57. Rohman, A., Windarsih, A., Hossain, M. A. M., Johan, M. R., Ali, M. E., & Fadzilah, N. A. (2019). Application of near- and mid-infrared spectroscopy combined with chemometrics for discrimination and authentication of herbal products: a review. *Journal of Applied Pharmaceutical Science*, *9*(3), 137-147. http://dx.doi.org/10.7324/JAPS.2019.90319.
58. Chua, C. Y. X., Liu, H.-C., Di Trani, N., Susnjar, A., Ho, J., Scorrano, G., Rhudy, J., Sizovs, A., Lolli, G., Hernandez, N., Nucci, M. C., Cicalo, R., Ferrari, M., & Grattoni, A. (2021). Carbon fiber reinforced polymers for implantable medical devices. *Biomaterials*, *271*, 120719. http://dx.doi.org/10.1016/j.biomaterials.2021.120719. PMid:33652266.
59. Sanches, N. B., Pedro, R., Diniz, M. F., Mattos, E. C., Cassu, S. N., & Dutra, R. C. L. (2013). Infrared spectroscopy applied to materials used as thermal insulation and coatings. *Journal of Aerospace Technology and Management*, *5*(4), 421-430. http://dx.doi.org/10.5028/jatm.v5i4.265.
60. Magalhães, R. F., Barros, A. H., Takematsu, M. M., Sanches, N. B., Amado Quagliano, J. C., & Dutra, R. C. L. (2020). FT-IR surface analysis of poly [(4-hydroxybenzoic)-ran-(2-hydroxy-6-naphthoic acid)] fiber: a short review. *Polymer Testing*, *90*, 106750. http://dx.doi.org/10.1016/j.polymertesting.2020.106750.
61. Lin, J. (2002). Effect of surface modification by bromination and metalation on Kevlar fibre-epoxy adhesion. *European Polymer Journal*, *38*(1), 79-86. http://dx.doi.org/10.1016/S0014-3057(01)00176-8.
62. Dai, Y., Meng, C., Tang, S., Qin, J., & Liu, X. (2019). Construction of dendritic structure by nano-SiO2 derivate grafted with hyperbranched polyamide in aramid fiber to simultaneously improve its mechanical and compressive properties. *European Polymer Journal*, *119*, 367-375. http://dx.doi.org/10.1016/j.eurpolymj.2019.08.011.
63. Kondo, Y., Miyazaki, K., Takayanagi, K., & Sakurai, K. (2008). Surface treatment of PET fiber by EB-irradiation-induced graft polymerization and its effect on adhesion in natural rubber matrix. *European Polymer Journal*, *44*(5), 1567-1576. http://dx.doi.org/10.1016/j.eurpolymj.2008.02.020.
64. Bansal, S., Ramachandran, M., & Raichurkar, P. (2017). Comparative analysis of bamboo using jute and coir fiber reinforced polymeric composites. *Materials Today: Proceedings*, *4*(2), 3182-3187. http://dx.doi.org/10.1016/j.matpr.2017.02.203.
65. Ramachandran, M., Bansal, S., & Raichurkar, P. (2016). Experimental study of bamboo using banana and linen fibre reinforced polymeric composites. *Perspectives in Science*, *8*, 313-316. http://dx.doi.org/10.1016/j.pisc.2016.04.063.
66. Bajwa, D. S., Adhikari, S., Shojaeiarani, J., Bajwa, S. G., Pandey, P., & Shanmugam, S. R. (2019). Characterization of bio-carbon and ligno-cellulosic fiber reinforced bio-composites with compatibilizer. *Construction & Building Materials*, *204*, 193-202. http://dx.doi.org/10.1016/j.conbuildmat.2019.01.068.
67. Yu, Z., Assif, J., Magoon, G., Kebabian, P., Brown, W., Rundgren, W., Peck, J., Miake-Lye, R., Liscinsky, D., & True, B. (2017). Differential photoacoustic spectroscopic (DPAS)-based technique for PM optical absorption measurements in the presence of light absorbing gaseous species. *Aerosol Science and Technology*, *51*(12), 1438-1447. http://dx.doi.org/10.1080/02786826.2017.1363866.
68. Badawi, A., Al-Gurashi, W. O., Al-Baradi, A. M., & Abdel-Wahab, F. (2020). Photoacoustic spectroscopy as a non-destructive technique for optical properties measurements of nanostructures. *Optik (Stuttgart)*, *201*, 163386. http://dx.doi.org/10.1016/j.ijleo.2019.163389.
69. Takematsu, M. M., Diniz, M. F., Mattos, E. C., & Dutra, R. C. L. (2018). Sheath-core bicomponent fiber characterization by FT-IR and other analytical methodologies. *Polimeros: Ciência e Tecnologia*, *28*(4), 339-347. http://dx.doi.org/10.1590/0104-1428.09016.
70. Senophiyah-Mary, J., & Loganath, R. (2020). A novel method of utilizing waste Printed Circuit Board for the preparation of Fibre Reinforced Polymer. *Journal of Cleaner Production*, *246*, 119063. http://dx.doi.org/10.1016/j.jclepro.2019.119063.
71. Ji, C., Wang, B., Hu, J., Zhang, C., & Sun, Y. (2020). Effect of different preparation methods on mechanical behaviors of carbon fiber-reinforced PEEK-Titanium hybrid laminates. *Polymer Testing*, *85*, 106462. http://dx.doi.org/10.1016/j.polymertesting.2020.106462.
72. Perret, B., Schartel, B., Stöß, K., Ciesielski, M., Diederichs, J., Döring, M., Krämer, J., & Altstädt, V. (2011). Novel DOPO-based flame retardants in high-performance carbon fibre epoxy composites for aviation. *European Polymer Journal*, *47*(5), 1081-1089. http://dx.doi.org/10.1016/j.eurpolymj.2011.02.008.

73. Donadon, B. F., Mascia, N. T., Vilela, R., & Trautwein, L. M. (2020). Experimental investigation of glued-laminated timber beams with Vectran-FRP reinforcement. *Engineering Structures*, *202*, 109818. http://dx.doi.org/10.1016/j.engstruct.2019.109818.
74. Jayakrishna, K., Rajiyalakshmi, G., & Deepa, A. (2019) *Structural health monitoring of fiber polymer composites.* In M. Jawaid, M. Thariq, & N. Saba (Eds.), *Structural health monitoring of biocomposites, fibre-reinforced composites and hybrid composites* (pp.75-91). UK:Woodhead Publishing. http://dx.doi.org/10.1016/B978-0-08-102291-7.00005-8.
75. Essabir, H., Bouhfid, R., & Qaiss, A. (2019) *Fracture surface morphologies in understanding of composite structural behavior.* In M. Jawaid, M. Thariq, & N. Saba (Eds.), *Structural health monitoring of biocomposites, fibre-reinforced composites and hybrid composites* (pp. 277-293). UK: Woodhead Publishing. http://dx.doi.org/10.1016/B978-0-08-102291-7.00014-9.
76. Prabhu, L., Krishnaraj, V., Sathish, S., Gokulkumar, S., & Karthi, N. (2019). Study of mechanical and morphological properties of jute-tea leaf fiber reinforced hybrid composites: effect of glass fiber hybridization. *Materials Today: Proceedings*, *27*(Part 3), 2372-2375.
77. Idicula, M., Malhotra, S. K., Joseph, K., & Thomas, S. (2005). Dynamic mechanical analysis of randomly oriented intimately mixed short banana/sisal hybrid fibre reinforced polyester composites. *Composites Science and Technology*, *65*(7–8), 1077-1087. http://dx.doi.org/10.1016/j.compscitech.2004.10.023.
78. Chandekar, H., Chaudhari, V., & Waigaonkar, S. (2020). A review of jute fiber reinforced polymer composites. *Materials Today: Proceedings*, *26*(Part 2), 2079-2082. http://dx.doi.org/10.1016/j.matpr.2020.02.449.
79. Liu, X., He, Y., Qiu, D., & Yu, Z. (2019). Numerical optimizing and experimental evaluation of stepwise rapid high-pressure microwave curing carbon fiber/epoxy composite repair patch. *Composite Structures*, *230*, 111529. http://dx.doi.org/10.1016/j.compstruct.2019.111529.
80. Di Mauro, C., Genua, A., Rymarczyk, M., Dobbels, C., Malburet, S., Graillot, A., & Mija, A. (2021). Chemical and mechanical reprocessed resins and bio-composites based on five epoxidized vegetable oils thermosets reinforced with flax fibers or PLA woven. *Composites Science and Technology*, *205*, 108678. http://dx.doi.org/10.1016/j.compscitech.2021.108678.
81. Fu, Y., Zhou, H., & Zhou, L. (2021). Phase-microstructure-mechanical properties relationship of carbon fiber reinforced ionic liquid epoxy composites. *Composites Science and Technology*, *207*, 108711. http://dx.doi.org/10.1016/j.compscitech.2021.108711.
82. Xu, Y., Adekunle, K., Ramamoorthy, S. K., Skrifvars, M., & Hakkarainen, M. (2020). Methacrylated lignosulfonate as compatibilizer for flax fiber reinforced biocomposites with soybean-derived polyester matrix. *Composites Communications*, *22*, 100536. http://dx.doi.org/10.1016/j.coco.2020.100536.
83. Miao, Y., Chen, H., Cui, G., & Qi, Y. (2021). Preparation of new conductive organic coating for the fiber reinforced polymer composite oil pipe. *Surface and Coatings Technology*, *412*, 127017. http://dx.doi.org/10.1016/j.surfcoat.2021.127017.
84. Balaji, A., Udhayasankar, R., Karthikeyan, B., Swaminathan, J., & Purushothaman, R. (2020). Mechanical and thermal characterization of bagasse fiber/coconut shell particle hybrid biocomposites reinforced with cardanol resin. *Results in Chemistry*, *2*, 100056. http://dx.doi.org/10.1016/j.rechem.2020.100056.
85. Louwsma, J., Carvalho, A., Lutz, J.-F., Joly, S., & Chan-Seng, D. (2021). Adsorption of phenylalanine-rich sequence-defined oligomers onto Kevlar fibers for fiber-reinforced polyolefin composite materials. *Polymer*, *217*, 123465. http://dx.doi.org/10.1016/j.polymer.2021.123465.
86. Sánchez, M. L., Patiño, W., & Cárdenas, J. (2020). Physical-mechanical properties of bamboo fibers-reinforced biocomposites: influence of surface treatment of fibers. *Journal of Building Engineering*, *28*, 101058. http://dx.doi.org/10.1016/j.jobe.2019.101058.
87. Yu, W., Zhang, H., Lan, A., Yang, S., Zhang, J., Wang, H., Zhou, Z., Zhou, Y., Zhao, J., & Jiang, Z. (2020). Enhanced bioactivity and osteogenic property of carbon fiber reinforced polyetheretherketone composites modified with amino groups. *Colloids and Surfaces. B, Biointerfaces*, *193*, 111098. http://dx.doi.org/10.1016/j.colsurfb.2020.111098. PMid:32498001.
88. Revanth, J. S., Madhav, V. S., Sai, Y. K., Krishna, D. V., Srividya, K., & Sumanth, C. H. M. (2019). TGA and DSC analysis of vinyl ester reinforced by Vetiveria zizanioides, jute and glass fiber. *Materials Today: Proceedings*, *26*(Part 2), 460-465.
89. Mphahlele, K., Ray, S. S., & Kolesnikov, A. (2019). Cure kinetics, morphology development, and rheology of a high-performance carbon-fiber-reinforced epoxy composite. *Composites. Part B, Engineering*, *176*, 107300. http://dx.doi.org/10.1016/j.compositesb.2019.107300.
90. Rahmani, N., Willard, B., Lease, K., Legesse, E. T., Soltani, S. A., & Keshavanarayana, S. (2015). The effect of post cure temperature on fiber/matrix adhesion of T650/Cycom 5320-1 using the micro-droplet technique. *Polymer Testing*, *46*, 14-20. http://dx.doi.org/10.1016/j.polymertesting.2015.05.012.
91. Zin, M. H., Abdan, K., & Norizan, M. N. (2018). *The effect of different fiber loading on flexural and thermal properties of banana/pineapple leaf (PALF)/glass hybrid composite.* In M. Jawaid, M. Thariq, & N. Saba (Eds.), *Structural health monitoring of biocomposites, fibre-reinforced composites and hybrid composites* (pp. 1–17). UK: Woodhead Publishing.
92. Adole, O., Anguilano, L., Minton, T., Campbell, J., Sean, L., Valisios, S., & Tarverdi, K. (2020). Basalt fibre-reinforced high density polyethylene composite development using the twin screw extrusion process. *Polymer Testing*, *91*, 106467. http://dx.doi.org/10.1016/j.polymertesting.2020.106467.
93. Shelly, D., Nanda, T., & Mehta, R. (2021). Addition of compatibilized nanoclay and UHMWPE fibers to epoxy based GFRPs for improved mechanical properties. *Composites. Part A, Applied Science and Manufacturing*, *145*, 106371. http://dx.doi.org/10.1016/j.compositesa.2021.106371.
94. Alagar, M., Kumar, A. A., Mahesh, K. P. O., & Dinakaran, K. (2000). Studies on thermal and morphological characteristics of E-glass/Kevlar 49 reinforced siliconized epoxy composites. *European Polymer Journal*, *36*(11), 2449-2454. http://dx.doi.org/10.1016/S0014-3057(00)00038-0.
95. Rozman, H. D., Tan, K. W., Kumar, R. N., Abubakar, A., Ishak, Z. A. M., & Ismail, H. (2000). Effect of lignin as a compatibilizer on the physical properties of coconut fiber-polypropylene composites. *European Polymer Journal*, *36*(7), 1483-1494. http://dx.doi.org/10.1016/S0014-3057(99)00200-1.
96. Ishak, Z. A. M., Ariffin, A., & Senawi, R. (2001). Effects of hygrothermal aging and a silane coupling agent on the tensile properties of injection molded short glass fiber reinforced poly(butylene terephthalate) composites. *European Polymer Journal*, *37*(8), 1635-1647. http://dx.doi.org/10.1016/S0014-3057(01)00033-7.
97. Wang, W., Tang, L., & Qu, B. (2003). Mechanical properties and morphological structures of short glass fiber reinforced PP/EPDM composite. *European Polymer Journal*, *39*(11), 2129-2134. http://dx.doi.org/10.1016/S0014-3057(03)00157-5.
98. Olmos, D., & González-Benito, J. (2007). Visualization of the morphology at the interphase of glass fibre reinforced epoxy-thermoplastic polymer composites. *European Polymer Journal*, *43*(4), 1487-1500. http://dx.doi.org/10.1016/j.eurpolymj.2007.01.004.

99. Roohani, M., Habibi, Y., Belgacem, N. M., Ebrahim, G., Karimi, A. N., & Dufresne, A. (2008). Cellulose whiskers reinforced polyvinyl alcohol copolymers nanocomposites. *European Polymer Journal*, *44*(8), 2489-2498. http://dx.doi.org/10.1016/j.eurpolymj.2008.05.024.

100. Meng, L., Li, W., Ma, R., Huang, M., Wang, J., Luo, Y., Wang, J., & Xia, K. (2018). Long UHMWPE fibers reinforced rigid polyurethane composites: an investigation in mechanical properties. *European Polymer Journal*, *105*, 55-60. http://dx.doi.org/10.1016/j.eurpolymj.2018.05.021.

101. Anggono, J., Farkas, Á. E., Bartos, A., Móczó, J., Antoni, Purwaningsih, H., & Pukánszky, B. (2019). Deformation and failure of sugarcane bagasse reinforced PP. *European Polymer Journal*, *112*, 153-160. http://dx.doi.org/10.1016/j.eurpolymj.2018.12.033.

102. Fernández, A., Santangelo-Muro, M., Fernández-Blázquez, J. P., Lopes, C. S., & Molina-Aldareguia, J. M. (2021). Processing and properties of long recycled-carbon-fibre reinforced polypropylene. *Composites. Part B, Engineering*, *211*, 108653. http://dx.doi.org/10.1016/j.compositesb.2021.108653.

103. Florek, P., Król, M., Jeleń, P., & Mozgawa, W. (2021). Carbon fiber reinforced polymer composites doped with graphene oxide in light of spectroscopic studies. *Materials (Basel)*, *14*(8), 1835. http://dx.doi.org/10.3390/ma14081835. PMid:33917218.

104. Kumar, B., Roy, S., Agumba, D. O., Pham, D. H., & Kim, J. (2022). Effect of bio-based derived epoxy resin on interfacial adhesion of cellulose film and applicability towards natural jute fiber-reinforced composites. *International Journal of Biological Macromolecules*, *222*(Pt A), 1304-1313. http://dx.doi.org/10.1016/j.ijbiomac.2022.09.237 PMid:36198365.

105. Nguyen-Tri, P., Ghassemi, P., Carriere, P., Nanda, S., Assadi, A. A., & Nguyen, D. D. (2020). Recent applications of advanced atomic force microscopy in polymer science: a review. *Polymers*, *12*(5), 1142. http://dx.doi.org/10.3390/polym12051142. PMid:32429499.

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