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Microstructural and Chemical Analysis of PBT/Glass Fiber Composites: Influence of Fiber Content and Manufacturing on Composite Performance

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Abstract

This paper provides an in-depth analysis of the microstructural characteristics and the chemical content of Polybutylene Terephthalate (PBT) composites that have different contents of Glass Fiber (GF). Blending of VALOX 420 (30 wt% GF/PBT) with unreinforced VALOX 310 allowed the composites to be prepared, with control of the concentration and distribution of the GF. The GF reinforcement and PBT matrix were characterized by an advanced microstructural spectrum and spatial analysis to show the influence of fiber density, dispersion, and chemical composition on performance. Findings indicate that GF content has a profound effect on microstructural properties and damage processes, especially traction effects in various regions of the specimen. These results highlight the significance of accurate control of GF during fabrication to maximize durability and performance, which can be used to inform the design of superior PBT/GF composites in challenging engineering applications. The implications of these results are relevant to a number of high-performance sectors, especially in automotive, electrical, and consumer electronic industries, where PBT/GF composites are found in extensive use because of their outstanding mechanical strength, dimensional stability, and thermal resistance. The main novelty of the current research is both the microstructural and chemical assessment of PBT/GF composites in different fiber contents, and this aspect is rather insufficiently studied in the literature. Although the mechanical performance or macro-level aging effects have been previously assessed, the Literature usually did not combine elemental spectroscopy or spatial microstructural mapping to correlate the fiber distribution with the damage mechanisms. Further, despite the importance of GF reinforcement in achieving the right balance between mechanical, thermal, and electrical performance, not much has been conducted in detail to describe the correlation between the microstructure and the evolution of damage in short-fiber composites. Conversely, this paper will use the superior spatial elemental analysis to bring out the effects of GF content and dispersion on micro-mechanisms like interfacial traction, cracking of the matrix, and fiber fracture. We, to the best of our knowledge, are the first to systematically combine chemical spectrum analysis with spatial mapping of PBT/GF systems with varied fiber contents—this allows us to give actionable information on material design and optimized manufacturing procedures.

Keywords: microstructural properties; polybutylene terephthalate; glass fiber composites; scanning electron microscopy analysis; chemical composition

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1. Introduction

Fiber-reinforced polymer composites have become more popular because of their exceptional strength-to-weight ratio, durability, and versatility in different engineering fields like aerospace, automotive, biomedical, structural engineering, etc. The behavior of the fibers in the composite matrix is important to optimize the mechanical performance of such a material and to predict the failure mechanism. Some of the recent research studies have highlighted the importance of microstructural and mechanical analysis of fiber-reinforced systems. As an example, Ref. [1] examined the post-cure influence on tensile behavior of hybrid fiber composites, studying the effects with both experimental work and finite element analysis in order to evaluate the structural integrity under different loading regimes. In more recent work, the combination of natural, synthetic, and recycled fibers was investigated to increase the sustainability of the materials whilst maintaining their performance which provides novel information into designing composites that are environmentally friendly [2]. In another study, the pattern of damage in carbon-fiber-reinforced polymers Carbon Fiber-Reinforced Polymer (CFRP) was investigated in detail, with a point being made that proper modeling of the initiation and propagation of cracks is necessary [3]. All these contributions are consistent with more general studies in the area where the synergy between fiber structure, binder chemistry, and processing factors is of the essence in providing optimized composite structures [4–6]. Therefore, this paper leverages this research to develop it further on the microstructural and chemical properties of GF-reinforced PBT composite and in particular the effect of the fiber content and the manufacturing process on their performance and application in harsh conditions.

By analyzing composites made from PBT and GF, this article explores their microstructure to show the relationships between their elements, manufacturing methods, and response patterns. The importance of this study is that it may guide the creation and use of PBT/GF composites in a wide range of engineering applications, since it explains how these components affect performance. Microstructural spectrum analysis and spatial analysis are carried out to finish this study and identify both the GF reinforcement's and PBT matrix's chemical composition. Doing this helps explain how GF content and manufacturing affect a composite's behavior overall as well as their separate parts. All in all, the structured investigation into how GF content, manufacturing, and material behavior are tied together gives valuable support for using PBT/GF composites in many engineering fields. The purpose of this research is to promote new developments in composite material science that lead to stronger, longer-lasting, and more useful materials in engineering. Along with knowing the advantages the reinforced structure gives due to mechanical reinforcement of glass fibers and the present features of PBT as the matrix, a minor amount of metallic elements spotted in the microstructure of the composite is also considered. Even though the primary components of the content of the material system PBT and E-glass fibers are made up predominantly of the elements of carbon, hydrogen, oxygen, and silicon, the minor ones may also include calcium, magnesium, and bromine. These components may come in proprietary additives employed by the manufacturers to create flame retardancy, thermal stability, or processing. Their addition may be intentional or incidental, but it can affect the durability of the composite as well as the response to severe environments. Therefore, the manner of their distribution in space, as well as their interaction with the matrix, becomes the necessary process to achieve a full portrait of the performance of these materials.

A study by [4–7] focused on scrutinizing the influence of GF content on the microstructural properties of the material, elucidating two interlinked micro-mechanisms, compression and traction, which affect the damage incurred by both the matrix and GF in distinct regions of the specimens.

Lately, new approaches in Structural Health Monitoring (SHM) allow us to put sensors into fiber-reinforced composites. Lin et al. [8] inserted electrospun PVDF (PolyVinylidene Difluoride) sensors with a sandwich structure into Glass Fiber-Reinforced Polymer (GFRP) material, so the sensors can detect stress and cracks in between laminae while not interfering with how strong the material remains. The structure and way the sensor pieces fit together reveal the need for the materials to be fully compatible, as we were studying in our work. Studying how fiber distribution and adhesion between layers change as GF content increases will be valuable for designing better future embedded sensing applications.

Using sustainability in composite engineering is becoming more widespread. El Bitouri et al. [9] research the effects of incorporating GFRP waste in mortars with sand in place. It was proved that post-consumer fibers can handle bending stress well, even though they become less strong when compressed, demonstrating their ability to be used in applications. Even though this study looks at virgin PBT/GF, the integrity revealed through micrographs and Energy-Dispersive X-ray Spectroscopy (EDS) mappings can support efforts to reintegrate fiber waste into PBT matrices.

Karalis et al. [10] introduced a unique way of winding multifunctional GFRP tubes on a cross-scale level. They found that adding nanoscience to GF allows for increases in strength, solidity, and conductivity without losing the easy adaptability of manufacturing. Our findings with respect to how higher GF levels can influence both the structure and element distribution supports their emphasis on consistent distribution of fibers. Knowing the microstructure is key for making larger-scale composites that still contain many useful features.

Sun et al. [11] introduced a convolutional neural network that was able to determine the stress distribution across fiber-reinforced polymer samples from the images of the microstructure that they were given. Now, they can use real-time forecasting instead of long simulations, indicating a move towards using data for design. Input from our high-resolution Scanning Electron Microscopy (SEM) and EDS data may lead to increased accuracy with these models. Elemental examination within the different concentrations of fibers helps discover areas that might be weak which models seek to find and forecast as crucial sites.

Zhao et al. [12] added carbon nanotube-covered GF to GFRP laminates so that Electrical Resistance Tomography (ERT) would allow them to notice damage early on. The research found that their approach of monitoring is both accurate and intrusive, and it showed that it could locate damage areas caused by an impact. Though nanostructures were not included, the research revealed how fiber type and matrix compatibility impact signals which is important for binding smart layers to other components.

Due to the rising need of high-performance composite materials with controlled mechanical behavior and predictable failure modes, this research was spawned by the necessity to find out how microstructural composition, specifically fiber distribution and chemical bonding, affect the performance of the material. In contrast to prior studies that tend to separate mechanical or thermal studies, the approach followed here integrates spectral and spatial microstructural observations using SEM and EDS in order to obtain a two-dimensional outlook. This can be used to provide a detailed analysis of the impact of fiber content throughout the composite. The major difference between this study and the others is that not only is the chemical and spatial analysis combined, but the content of the fibers is varied in a systematic way to identify the points where the performance gains or failure mechanisms become predominant. The findings are likely to be of general interest to industries like the automotive and electronics industries where structural reliability under harsh conditions is critical. By referencing to the latest trends and discussing the drawbacks of them, this project bridges the gap in the connection between microstructure

and functionality and provides new knowledge to consider when optimizing the design and manufacturing procedures in fiber-reinforced thermoplastics.

The established relationship between fiber content, microstructural integrity, and elemental distribution can give engineers a practical guideline to design materials that will perform better, last longer, and be more consistent in manufacturing. By adjusting the proportion of GF and knowing how they influence the spread of damage and the reinforcement-matrix interplay, this study can be used to make stronger, lighter, and less expensive parts like connectors, housings, enclosures, and structural components. Further, the chemical information reveals new opportunities to incorporate recycled fibers or hybrid reinforcements as part of the sustainability agenda in composite engineering. All these contributions have helped to promote the design process and functional dependability of fiber-reinforced thermoplastics in many industrial applications.

The purpose of this study is to examine the microstructure of PBT GF-reinforced composites, supported by up-to-date developments in these technologies and monitoring.

2. Materials and Methods

2.1. Mixing Composition

PBT composites with different GF concentrations were designed by admixtures of two different grades of thermoplastic polymer granules that are produced by Sabic Ltd., Al-Jubail, Riyadh, Saudi Arabia, namely VALOX 420 and VALOX 310. PBT with 30 wt% short GF, VALOX 420, is also designed to provide superior performance in the mechanical load-bearing component. By comparison, VALOX 310 is an unreinforced PBT resin formulation that is used as a base matrix material. These particular grades were selected to be chemically compatible, have similar melt flow characteristics, and because they were less likely to have interfacial defects during processing.

On the one hand, the morphology of VALOX 420 is different because it contains E-glass fibers with a density of 2.55 g/cm^3 and an average fiber diameter of 13.8 µm. Such short fibers are evenly dispersed within the granules and help to increase tensile and flexural strength, stiffness, dimensional stability, and impact resistance of the resulting composite. The PBT matrix has a semi-crystalline structure which offers superior properties against moisture, chemicals, and heat, which is very essential in automotive and electronic housings.

Although molecular characteristics are not listed on the material technical datasheets, the literature referenced below suggests that commercial-grade PBT will have an average molecular weight in the region of 20,000–40,000 g/moL and an average molecular weight of 50–80,000 g/moL. The respective polydispersity index (pdi) is normally between 2.0 and 2.5. Because both VALOX 310 and VALOX 420 are based on the same industrial PBT and are produced by the same manufacturer, it can be assumed that in terms of their molecular distribution, these materials also belong to such a standard range. This makes the polymer chains uniform and compatible, making the blending method that is used in this research study appropriate in producing different GF percentages without resulting in a bias in the compositions of the matrix phase. The repeated twin-screw extrusion and fiber dosing equipment required to make sure that there is uniform dispersion of fibers is not available in our lab. Rather, by mixing two existing commercial chemicals, VALOX 420 and VALOX 310, a reliable and reproducible process to obtain a desired fiber concentration was provided such that microstructural consistency could be maintained and no processing defects were introduced. This strategy will ensure that any changes in the mechanical or chemical properties are reflected in the intended GF content, and not in the variation in the quality of the processing or mixing.

In order to prepare controlled-content samples of the composite, VALOX 420 and VALOX 310 were proportionally blended by weight (ranging between 0 wt% and 30 wt%) using a precision digital scale. Table 1 provides the target compositions of each formulation. Mixing was carried out by means of a METER MIX SYSTEMS LTD twin-component mixing machine, specifically the horizontal screw type. The contents were introduced into the chamber at the same time and were left to be exposed to continuous mechanical agitation at ambient temperature over a 30 min period. This time was set by trial and error to be the best time to have a uniform dispersion of the GF without excessive shear which would result in breakage of the fibers.

Care was taken to ensure that during the mixing process there was no static accumulation or moisture absorption as this would have adversely affected the distribution of the fibers and interfacial adhesion. To this effect, all the materials were dried in a dehumidifying temperature of 120 °C for 4 h before mixing to have a residual moisture content of less than 0.02%. The quality of the blend was visually checked to see that it was homogeneous, and the mixture was then transferred to the injection molding machine without delay so as to avoid any form of thermal degradation. Such a careful preparation step was necessary in order to achieve consistency in microstructural characteristics and in order to be certain that any subsequent testing, whether mechanical, chemical, or microscopic, would reveal the effects of GF content and not the variability in processing [13].

Table 1. Mixing percentage for PBT composites with different GF contents [13].

Reinforcement Rate [%]	0	5	10	15	20	25	30
Quantity of Pure PBT [g]	100	500	200	100	50	20	0
Quantity of PBT/30% GF [g]	0	100	100	100	100	100	100

2.2. Specimen Preparation

Before processing, the raw polymer mixture was exposed to a standard drying procedure to remove any remaining moisture that is essential to prevent the occurrence of hydrolytic degradation during molding. Drying has been conducted in a dehumidifier, which is a desiccant type of equipment, at a temperature of 120 °C for 4 h such that the material has a moisture content of less than 0.02%. This is a vital precondition to ensure the structural integrity and uniformity of the composite in the course of thermal processing.

After drying, specimens were produced on a KraussMaffei 50 t injection molding machine that was chosen because of its precision and repeatability in the processing of engineering-grade thermoplastics. The melt temperature was 27 °C, and the injection pressure was 800 bar which were appropriate parameters that were used in the injection molding process of the selected polymer matrix and filler system. The volume of the material feed was 15 cm^3 and was calibrated in order to fill the cavity without overflow and the formation of a cavity.

The parameters of the cycle were optimized precisely in regard to this particular formulation. The time of dosing was adjusted to 2.8 s to ensure homogeneous preparation of melt, and a high-speed injection phase of 0.8 s was aimed to reduce shear-induced degradation. At this step, the clamping pressure was set to 300 bar in order to avoid mold separation and to maintain dimensional stability. Also, 100 bar back pressure was used during plasticizing to achieve better dispersion of the fibers and improve melt homogeneity.

After injection, the parts were cooled down in a controlled cooling step of 60 °C for 10 s under a holding pressure of 400 bar to overcome shrinkage and warp and to achieve homogeneous crystallization. The sprue was manually trimmed off the specimens after demolding, and the specimens were post-processed to ensure dimensional accuracy.

The ultimate shape of the specimens used in the test was 125 mm long, 13 mm wide, and 3 mm thick, which exactly meets the requirement of the test according to the American Society for Testing and Materials (ASTM) D790 standard of flexural testing of reinforced thermoplastics. Such standardization makes the mechanical results comparable to the literature and leads to trustworthy interpretation of flexural modulus and strength results [14].

2.3. Microscopic Analysis

SEM that is fitted with EDS was used to mainly carry out elemental analysis of PBT/GF composites at different fiber contents. The analysis was performed with a Japan Electron Optics Laboratory (JEOL) JSM-6010 PLUS/LA system that is a high-resolution imaging system with integrated spectral capability. The aim of such characterization was not only to visualize the microstructure but more so to ascertain the elemental composition and spatial distribution of important elements within the composite matrix and at the fiber interfaces.

To this end, tensile-fractured specimens were selected with much care; fracture surfaces are considered as zones of mechanical failure. To study structural integrity and the examination of cross-sections, these specimens were embedded in epoxy resin. The samples were then mechanically polished under progressive conditions using silicon carbide papers and diamond suspensions to give a smooth, reflective surface to within an order of 50 nm that is good enough to make precise spectroscopic measurements.

The SEM-EDS analysis was conducted at a high-vacuum mode, the accelerating voltage was set at 15 kV, and the working distance was set at about 10 to 12 mm. Instead of emphasizing the topographical imaging aspect, the mapping of the spectrums and point-based compositional profile was the highlight of the analysis. Particular areas of interest included the fiber matrix interface regions of the matrix, and bromine (Br), magnesium (Mg), silicon (Si), and calcium (Ca) affected zones were examined using the software to identify and map the existence of carbon (C), oxygen (O), bromine (Br), magnesium (Mg), and silicon (Si). These factors were applied to separate the matrix, the GF, and the additives or degradation products.

Spectral data and elemental maps of EDS generated as a result of this analysis gave an idea about the distribution of chemical in the fiber and how manufacturing conditions influence the chemical distribution in the composite. This is essential information in interfacial behavior, microstructural uniformity, and possible failure sources of materials. Finally, it is the spectroscopy-based methodology that was at the core of the alignment between the chemical properties of the composite and the observed mechanical and thermal properties of the composite.

3. Results

3.1. Spectral Analysis

Spectral analysis was conducted to find out what chemicals are present in each part of the composite, and the findings are shown in Figure 1. No matter how much GF was present, the samples had significant amounts of carbon (C) and oxygen (O) present. The polymer matrix PBT includes these elements, and its main structure is $C_{12}H_{12}O_4$. Many peaks in pure PBT were caused by high levels of carbon (C) and oxygen (O), each one exceeding 30×10^3 electrons. It was also observed that the material contained a large amount of bromine (Br), as shown by multiple peaks and a very high electron content (30×10^3). Unlike the other elements, magnesium (Mg) and silicon (Si) were present in moderate quantities; there were few signals, and their electron content was less than 10×10^3. The same tendencies also applied to PBT strengthened with 5 percent GF (PBT/5%GF), 10 percent GF (PBT/10%GF), and 15 percent GF (PBT/15%GF). For every case, large amounts of carbon (C), bromine (Br), and oxygen (O) were found, along with

many spectral peaks. For magnesium (Mg) and silicon (Si), the electron content never surpassed 40×10^3 in PBT/5% GF, 40×10^3 and 100×10^3 in PBT/10% GF and 50×10^3 in PBT/15% GF. Starting at a 20% GF content, calcium (Ca) could be observed in the samples, as seen in Figure 1. PBT with 20% GF (PBT/20%GF) had abundant carbon (C) and oxygen (O), while its magnesium (Mg) and silicon (Si) levels did not go over 10×10^3 and 40×10^3, respectively. With 25% GF (PBT/25%GF) reinforcement, calcium (Ca) levels went down but could still be detected. The sample contained high levels of carbon (C) and oxygen (O) and average amounts of magnesium (Mg) and silicon (Si) which were 5×10^3 and 20×10^3, respectively. Compared to PBT/20%GF, the calcium (Ca) content in PBT/40% GF increased by a small amount, reaching about 8×10^3. There were still large amounts of carbon (C) and oxygen (O) in PBT/30%GF, and the magnesium (Mg) and silicon (Si) levels were 8×10^3 and 28×10^3, respectively. PBT/30%GF showed a bigger amount of calcium (Ca), with around 10×10^3 units. Chemical analysis found that carbon (C), bromine (Br), and oxygen (O) were always present and at the same level in every type of reinforcement, but calcium (Ca) was only present at 20%GF reinforcement.

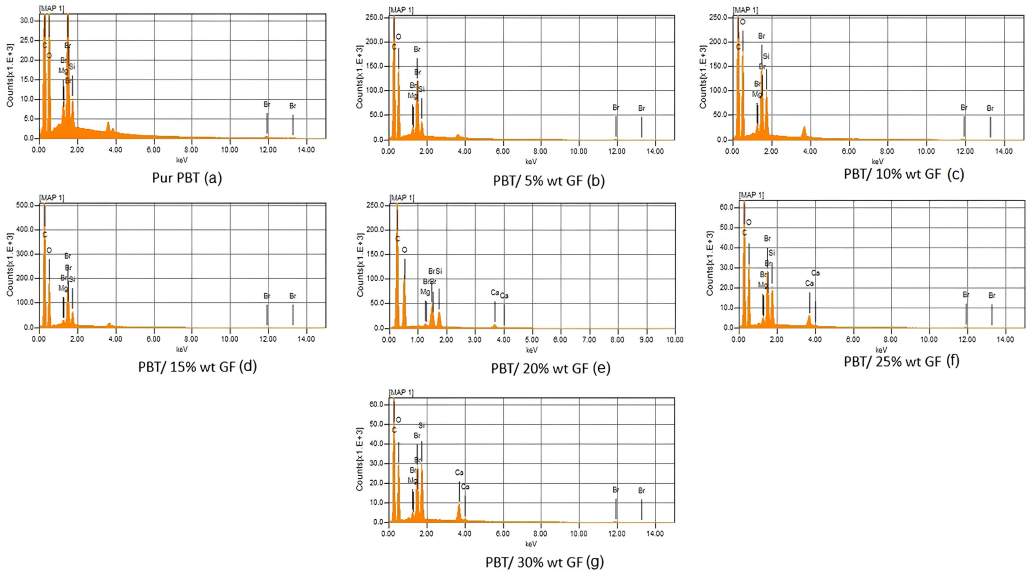


Figure 1. Spectral analysis results showing elemental composition variations with GF content: (**a**) Pur PBT; (**b**) PBT/5% GF; (**c**) PBT/10% GF; (**d**) PBT/15% GF; (**e**) PBT/20% GF; (**f**) PBT/25% GF; (**g**) PBT/30% GF.

3.2. Spatial Distribution Analysis

This study used SEM to check where the microstructures were found. The analysis was conducted on two main types: pure PBT and PBT reinforced with amounts of 5% to 30%. The way the material is arranged at the microstructural level is displayed in Figures 2–4. The pattern of microstructures was examined to determine how evenly the GF were distributed in the composite. It helps us understand better how the material is deformed and how damage moves within it. The study of surfaces created by fracture in SEM gives us clues about the microstructure's impact on how the material performs mechanically and how damage is distributed. SEM shows where GF reinforcements are placed and provides an understanding of the internal structure and potential stress areas of the composite. This knowledge helps make car and aerospace materials better and more successful. In short, analysis of the SEM images (Figures 2–4) lets us examine GF dispersion, how the composite changes under loading, and the paths of damage. Figure 2 shows the difference between pure PBT and PBT reinforced with 5% GF. It enables us to observe the arrangement of the elements and what they are composed of in the material. It is obvious

after careful observation that the placement of heavy and light atoms in the material is not exactly the same. Knowing the way components are grouped in a composite is easier if the distribution pattern is understood. From the picture, it is evident that bromine (Br) plays a main role in the microstructure.

Bromine is not distributed evenly throughout the examined area. The finding means that bromine (Br) is closely bound to carbon (C) and oxygen (O). The presence of these elements in PBT is explained by the spectral analysis which confirms that they are important components of the polymer matrix. There are small amounts of silicon (Si) and magnesium (Mg) present in the microstructure as well. All this shows that they truly are crucial components of the PBT matrix. Even though they are in smaller amounts than carbon (C), oxygen (O), and bromine (Br), they show that the material is complex. Essentially, Figure 2 gives useful information about the organization of pure PBT and PBT/5% wt GF at a microscopic scale. It makes both the position and partnership between key elements within the material clear. This detail is essential for knowing what the material can do and where it can be used in various industries.

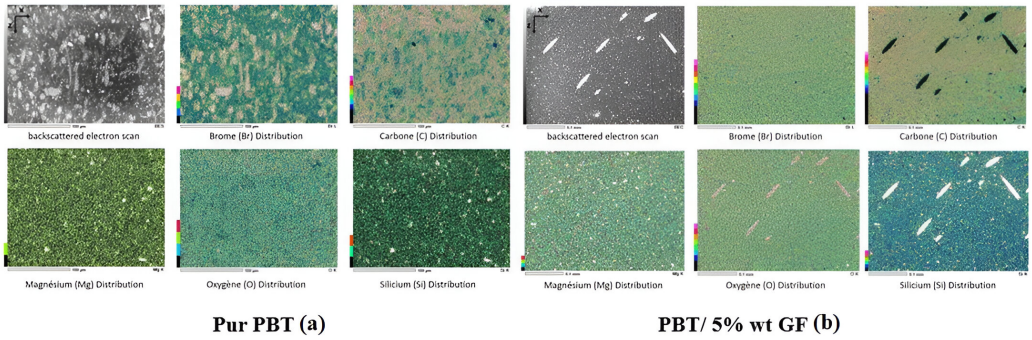


Figure 2. SEM spatial distribution of (**a**) pure PBT and (**b**) PBT with 5% GF.

Figure 3 examines how the GF is distributed at various weight fractions inside PBT-based composites. As we can see in this analysis, the distribution and makeup of elements are quite different from what was seen in Figure 2. Looking closely, it is noticeable that composites made from PBT/10% wt GF and PBT/15% wt GF have more carbon (C), bromine (Br), and oxygen (O) within their structures. Moreover, the distribution density of these elements is significantly elevated throughout the composite structure. This suggests that the addition of GF reinforcements at these weight percentages enhances the presence and distribution of C, Br, and O within the material. An interesting observation is the spatial distribution around the fibers. Here, the distribution of carbon (C) appears to be less prominent, indicating that the GF reinforcements play a role in influencing the distribution of this element. Conversely, bromine (Br) and oxygen (O) show higher concentrations around the fibers, indicating their association with the GF. In contrast, the proportions of magnesium (Mg) and silicon (Si) within the microstructure are less substantial in comparison. However, these elements exhibit a distinctive localization pattern, primarily concentrating around the fibers. This localization is particularly evident in the case of silicon (Si), as indicated by the prominent red coloration in specific areas, signifying a strong presence. The color scale provided on the left of the figure aids in visually interpreting the degree of element localization, with red denoting the highest concentration. In summary, Figure 3 presents a striking contrast to the previous observations, showcasing the impact of varying GF weight percentages on the microstructural distribution within the PBT composite. The higher proportions and distribution density of carbon (C), bromine (Br), and oxygen (O) in these composites reflect the influence of reinforcement on element distribution, while magnesium (Mg) and silicon (Si) exhibit localized concentration patterns primarily around the fibers, underscoring the complex interplay of elements within the composite. Figure 3 examines

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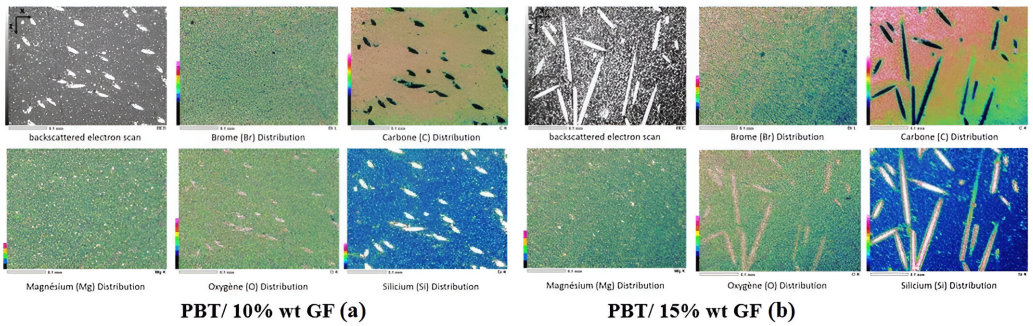


Figure 3. SEM spatial distribution of (**a**) PBT/10% and (**b**) PBT/15%.

In Figure 4, we delve into the microstructural distribution of composite structures representing PBT reinforced with 20% weight GF, 25% weight GF, and 30% weight GF. A noteworthy comparison can be drawn between this observation and the earlier analysis of pure PBT and PBT/5% wt GF in Figure 2, revealing a distinct trend in element presence and distribution. In Figure 4, we observe a noticeable increase in the presence of silicon (Si), calcium (Ca), and magnesium (Mg) within the composite structures. This augmentation is directly attributed to the escalating percentage of GF in the composite. These elements, in conjunction with oxygen (O), are integral chemical structural components of GF. As the percentage of GF reinforcement rises, their presence becomes more pronounced within the microstructure. This observation serves as compelling evidence of the correlation between the GF content and the increased representation of Si, Ca, and Mg within the composite. Importantly, this corroborates and reinforces the findings obtained from the spectral chemical analysis of the various materials at different reinforcement rates. The SEM microstructural analysis aligns with the chemical analysis by visually illustrating how the composition evolves with varying GF percentages. As such, the increase in Si,

Ca, and Mg, in tandem with O, serves as a crucial validation of the material's chemical composition and highlights the structural significance of these elements in GF-reinforced PBT composites. In essence, Figure 4 provides a compelling visual representation of how the microstructural distribution evolves as the GF reinforcement percentage increases. This comparative analysis further emphasizes the interplay between composition and reinforcement, reinforcing the results obtained through both SEM imaging and spectral chemical analysis.

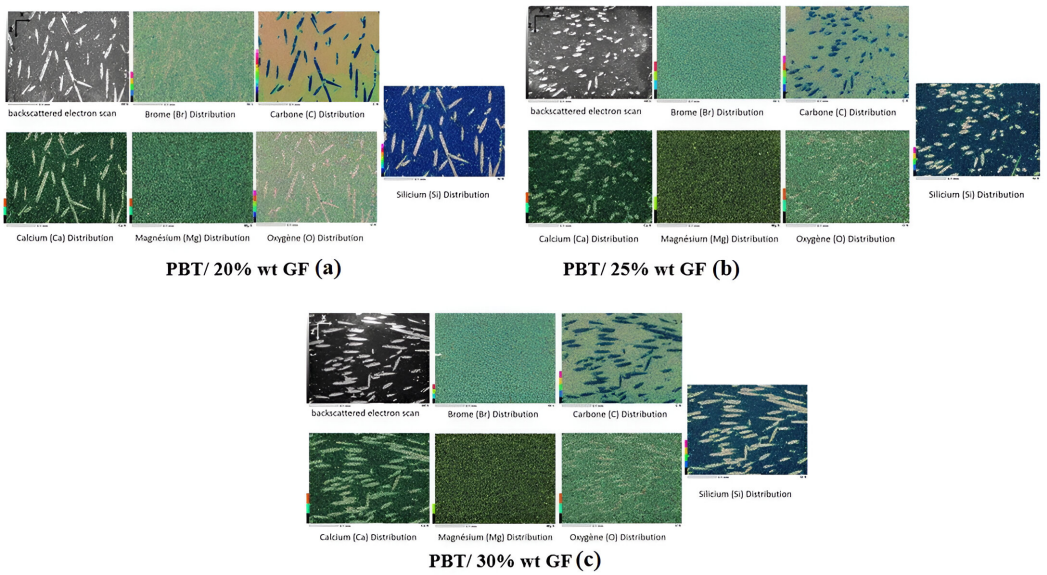


Figure 4. SEM spatial distribution of (**a**) PBT/20%, (**b**) PBT/25%, and (**c**) PBT/30%.

The observed and seeming difference in the size of the fiber in all SEM images including Figure 4 are largely attributed to the random orientation of the glass fibers in the PBT matrix but cannot be said to be the change in diameter of the fiber. The composite fabrication process used a blend of short E glass fibers with random orientation, so one will see some of the fibers in a cross-section which are shorter and circular, and others will be caught longitudinally and appear longer and more elongated depending upon their intersections with the polished surface. This difference in the viewing angle results in the difference in the size of perceived fibers between the images. Also, increased fiber loadings lead to more overlaps, clustering, or pull-outs of fibers, and this may also affect the visual contrast and sharpness under SEM imaging. The observed features are in line with the randomness of fiber placement in injection-molded short-fiber composites and do not reflect the inconsistency in the fiber material or preparation method.

4. Discussion

Based on the results from the spectral analysis, the predominant presence of carbon (C) and oxygen (O) in the sample can be attributed to the intrinsic composition of the polymer matrix, specifically PBT. Comprising carbon, hydrogen, and oxygen atoms, PBTT's fundamental formula, $C_{12}H_{12}O_4$, underscores the substantial presence of C and O. The incorporation of increasing weight percentages of GF reinforcements can modify the composite's elemental composition, introducing additional C and O. This is due to the GF being embedded within the polymer matrix, thereby affecting the composite's overall elemental distribution. Both PBT and its GF-reinforced types have elevated bromine (Br) which is due to the intentional use of brominated flame retardants to increase the fire resistance. Magnesium (Mg) and silicon (Si) are found in PBT and its composites which can occur due to impurities in the raw materials or due to special additives used. They could be utilized

to boost certain traits in the material or to make handling the material easier. Since calcium (Ca) is seen at 20% GF reinforcement, it could mean the GF have calcium compounds or it may be introduced during the manufacturing process. How uniform GF is in all the pores of the porous matrix can have an impact on spectral readings, where unevenness may lead to biased results. Instrument features and the lab's selected methods can change how accurately the elements are found. If sample preparation includes mixing, the method can affect where the elements are distributed which might cause the results to differ. Varying the temperature and pressure during processing might affect the way the matrix interacts with the reinforcements which can alter the visible elemental composition. Additional additives or fillers could be mixed within the plastic during production to improve specific aspects. Paying attention to these variables is necessary when you study spectral data. Additional detailed studies could be needed to understand where and why some elemental concentrations occur and what they mean.

The elements found in EDS analysis, which are mostly not expected to be seen in pure PBT or standard glass fibers, including metallic elements (Ca, Mg, and Br), are consistent with industrial additives. These additives are typically added during formulation in order to add properties such as flame resistance (e.g., brominated compounds), mechanical stabilization, or enhancement in adhesion at the fiber–matrix interface. They are spatially correlated with areas of damage or fiber clustering as observed by EDS mapping, which implies that they may control the local mechanical response and damage behavior. The awareness of these trace constituents does not only help to give a clearer picture of the material constitution but also serves to give an idea of how this slight chemical variation could affect the working of the material in some extreme use.

Studies by [15] reported on the same spectral analyses and confirmed that there are notable concentrations of carbon (C), silicon (Si) and calcium (Ca) in GF, among other elements. A recent study by Sundaram R.K. et al. [16] investigated the effects of adding a compatibilizer on the properties of polymer blend composites (PBCs). Both PBT and polytrimethylene terephthalate were used, with and without including GF reinforcement when creating the PBC. It was noted that the compatibilizer helps the immiscible polymer phases stick together more successfully. The research pointed out that the crosslinked polybutadiene improved tensile, flexural, and impact strength. Having GF in the PBT increased all of the examined mechanical and thermal properties. The distribution and relationship between the composite components were made clear by conducting morphological analysis on the composite using field emission scanning electron microscopy. Additives, reinforcements, and compatibilizers play an important part in polymer composites, so their role should always be included in the interpretation of spectral information and overall understanding [16].

Spectral analysis and element analysis reveal that a major part of PBT reinforced with GF is composed of carbon (C) and oxygen (O). This happens because PBT's major atoms are all carbon, hydrogen, and oxygen. The simple formula of $C_{12}H_{12}O_4$ demonstrates that carbon (C) and oxygen (O) are the main elements in PBT. Bringing in additional GF, especially with higher percentages, causes the amount of C and O to increase in the composite. Because GF are added to the polymer matrix in manufacturing, the substance and elemental distribution of the composite are affected. Also, the increased bromine (Br) in both plain and GF-filled PBT is believed to be the result of adding brominated flame retardants to improve the flame resistance of PBT [17]. You can find magnesium (Mg) and silicon (Si) in PBT and its composites because they may have come from impurities in the raw materials or from special additives used to upgrade the material or process it [18]. The fact that calcium (Ca) appears as GF reinforcement levels increase means the GF could contain calcium or it may be added by the manufacturing process [19]. Noticeably, the level

of consistency in GF distribution inside the matrix can also affect the spectral pattern. How populations are distributed can make the findings about them unbalanced. Apart from the main materials, additional ingredients may be included during production to support possible functions [20].

More attention is now being given to the mechanical properties of GFRP composites, thanks to a drive for better and more environmentally friendly materials. El Bitouri and colleagues [9] investigated using GFRP waste to replace sand in cement mortar, finding that at lower rates (e.g., 3%) the strength of the mortar remains unchanged, but as the proportion of GFRP waste increases, the strength decreases. This agrees with our belief that the right GF content ensures that the composite has both stiffness and toughness.

Advances in SHM of GFRP composites have led to the use of innovative sensors. By using GF coated with Multi-Walled Carbon NanoTubes (MWCNTs), Zhao et al. [12] were able to detect damage almost immediately without reducing the mechanical performance. As expected, having uniformly distributed fibers and strong microstructure were important for keeping high strength at high GF levels.

In addition, Lin et al. [8] came up with a specially designed PVDF sensor able to sense stress and crack damage within composite layers. The design of these structures which keeps moving parts reliable and allows for sensing with piezoelectric materials could be applied to injection-molded GFRP pieces. Setting conditions in the study shows that bonding between sensor and matrix layers is still a problem, underlining the significance of matching these materials.

It was found in a 2024 study that High-Voltage Fragmentation (HVF) leads to better preservation of fiber length than mechanical shredding [21]. This research backs up ideas for recycling or reusing our composite materials at the end of their useful lives. Preserving the original length of reinforcing fibers in the composites means less loss of strength when they are reprocessed.

Dong and Davies [22] looked into using material extrusion to make continuous GF composites. It was found that the strength of the samples in tension and flexure was greatly affected by how the fibers were arranged and stacked which agreed with our findings showing that more GF leads to increased orientation and improved network structure. Achieving the same results from both GF/PBT methods show that our use of pre-compounded GF/PBT satisfies the reinforcement needs.

Last, piezoelectric sensors added to GFRP laminates have given promising outcomes for monitoring during use. A study in 2023 [23] found that these sensors are useful for monitoring damage as materials are bent repeatedly. As a result, intelligent composites may have practical uses in industry because of what we learned through testing with tensile loads.

The comparison with other recent studies is in-depth, which also reinforces the relevance of our findings. Ref. [24] examined the fatigue-induced modification of dynamic moduli of glass-fiber-reinforced PBT and stated that the degradation of stiffness is strongly associated with the GF content and the viscoelastic phenomena, which is also supported by our SEM/EDS data, as similar localized damage areas are observed that relate to the higher concentration of fibers and the transformation of elements. Another study by [25] investigated the effects of thermal aging on GF/PBT composites and revealed significant losses of tensile and flexural strength at higher temperatures; our study is related to the fact that we also found that chemical alterations such as the presence of Si, Ca, and Mg are stable markers of degradation, as evidenced by microstructure analysis. Ref. [26] studied the influence of different fiber concentration and strain rate on injection-molded PBT and found that the increase in GF content increases the tensile strength but also contributes to the stress concentration—which is reflected in our discovery of the traction-based damage

mechanisms occurring in dense fiber areas. Ref. [27] increased the interfacial bonding in GFRP composites through the use of matrix modification and were able to improve mechanical properties significantly; we found the same to be true of better fiber dispersion correlating with better elemental interfacial bonding. Finally, Ref. [28] have designed a multiscale model to characterize the evolution of stiffness in short-fiber reinforced PBT under cyclic fatigue with a special focus on viscoelastic damage; our experimental data support the validity of the model by indicating the correlation between the distribution of fibers, the elemental microstructure, and the macro-mechanical response.

5. Conclusions

This study deeply examines the changes in microstructure and elements in PBT composites reinforced with different portions of GF. Through mixing VALOX 420 (30 wt% GF) with VALOX 310 (only PBT) and using injection molding, we produced a range of composites that helped us analyze the links between structure and properties.

Evaluating the microstructure with SEM showed that including more GFs led to more fiber concentration and made the reinforcing elements appear more clustered in the matrix. This analysis consistently revealed that all the samples had carbon (C), oxygen (O), and bromine (Br), and higher GF content brought about traces of silicon (Si), magnesium (Mg), and calcium (Ca).

The results prove that GF reinforcement not only influences the shape of the composite but also its chemical structure, therefore having direct effects on its strength and possibility for damage propagation. GF in a composite are very important for performance, most of all in structural uses because they make the materials more durable and maintain their strength if damaged.

They help us see the impact microstructures have on the works of composites. Laboratory experiments show what parameters matter when shaping PBT-based composites in ways best suited for engineering uses requiring sturdiness, resistance to damage, and heat tolerance.

The specified goals of this study have been successfully addressed, as a thorough examination of the effect of GF content on microstructural and chemical properties of PBT-based composites has been conducted. The study gave a profound explanation on the spatial distribution of the reinforcing fibers and elemental composition, interfacial bonding behavior in the composite by use of sophisticated analytical tools like SEM and EDS. The noted tendencies such as higher fiber clustering, better silicon and calcium traces, and better interfacial traction, are highly consistent with the objective to determine how the GF content and production methods influence the overall performance of the composite. Such discoveries not only have scholarly importance but also can be practically applied in the design and manufacturing of fiber-reinforced thermoplastics with high-performance applications. This study can provide a basis of optimizing composite formulations in areas such as automotive, electrical, and consumer electronics where the structural reliability, thermal resistance, and dimensional accuracy are of utmost importance. The correlating between microstructure and mechanical functionality opens new perspectives for the development of the next generation of composite materials, which are lighter, more durable, and better suited to operate under harsh conditions.

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