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Waste Iron Filings to Improve the Mechanical and Electrical Properties of Glass Fiber-Reinforced Epoxy (GFRE) Composites

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Abstract: Several studies have been conducted to improve the mechanical and other value-added properties of glass fiber-reinforced epoxy (GFRE) composites by the addition of different fillers. In this work, waste iron filings (WIFs) obtained from the steel industry were incorporated into GFRE composite samples in varying amounts of up to 50% (%w) to improve their mechanical and electrical properties. The results showed that, with increasing WIF loading from 0 w% to 50 w%, the resultant composite density gradually increased from 1.4 to 2.1 g/cm^3. Surface hardness, Young's modulus, and tensile strength also increased significantly with the addition of up to 9 w% of WIF followed by a significant drop with more WIF addition due to agglomeration. Overall, Young's modulus of the GFRE samples with any WIF content was higher than that of the GFRE composite with no WIF. The elongation at break results showed that the GFRE samples were less ductile upon WIF addition, which decreased from 2% to 0.6% upon loading the composite with 50% WIF. In terms of electrical conductivity, the GFRE samples with WIF content of 15% or more were electrically conductive and their electrical conductivity increased with WIF content. It was clear that more WIF was needed to establish a percolated network in the GFRE composites to render them conductive. The electrical conductivity of the GFRE samples containing 15% WIFs was around 2.9 kS/m and increased to 35 kS/m upon the addition of 50% WIFs. These novel electrically conductive GFRE composites could be promising for structural dynamic monitoring systems in the construction industry. They also support the efforts for the utilization of waste materials towards a circular economy.

Keywords: GFRE; composite; waste iron fillings; mechanical reinforcement; electrical conductivity

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1. Introduction

Composite materials provide combined superior properties of their components compared to traditional monolithic materials. Several combinations of fillers and matrices have been explored in the literature to obtain composites with targeted advantageous performance [1]. The explored matrices included a variety of hydrophobic and hydrophilic, thermoplastic and thermosetting, natural and synthetic materials such as polyethylene, polypropylene, poly(butylene succinate), polyurethanes, urea-formaldehyde resins, polylactic acid, chitosan, lignin, and many others [2–5]. Similarly, a wide range of organic and inorganic fillers and nanofillers have been used to improve the properties of such matrices such as cellulose fibers, nanocellulose, carbon fibers, carbon nanotubes, glass fibers, silica, metal oxides, and others [6–9]. Due to their low cost, high strength-to-weight ratio and tailorable design performance, glass fiber-reinforced epoxy (GFRE) composites have become a substitute for many heavy-duty engineering construction materials and played an important role in many critical technical applications [10,11] such as automobiles, construction, marine industry, and piping fields [12–15]. However, the disadvantages of this type of material cannot be neglected including limited mechanical performance and negligible electrical conductivity. Electrical conductivity can be of great importance for sensing and structural dynamic monitoring systems [16]. With the high-speed industrial development and global transformation trends, the demand to improve GFRE performance in addition to the sustainable use of materials during composite manufacturing have significantly increased and have laid a big challenge for both researchers and manufacturers [17].

The mechanical performance of GFRE composites and their application depend on (a) filler's reinforcement strength (b) matrix strength and (c) the interfacial bonding between the filler and matrix to enable effective stress transfer [10,18]. If the adhesion between the matrix and the filler is not strong enough, it may lead to certain mechanical limitations in the composite's applications. In this situation, many strategies have been proposed in the literature such as the addition of a compatibilizer or through a physical or chemical modification of one or both GFRE components [19]. To enhance the mechanical properties of GFRE, different inorganic materials such as aluminum oxide [20], titanium oxide [21], magnesium hydroxide [22], organic polymers [23], and natural plants [24] have been used as fillers and the influence of fillers' dimensions and content on their enforcement capabilities have been thoroughly studied [25,26]. For instance, the addition of iron (III) oxide as a low-cost material was found to have a significant impact on the mechanical properties of GFRE composites [27,28] and the tensile strength of the composite increased at first and then decreased with higher iron oxide content of more than 10% until a fracture occurred due to a decrease in the effective cross-sectional area of the continuous phase and a sharp increase in the size of the inter-particle interstitial particles, which in turn induced major cracks and therefore reduced the tensile strength. In addition, agglomeration of the dispersed filler particles may have led to a decrease in tensile strength as the strength of the agglomerates was lower [29]. In terms of the electrical properties of GFRE composites, rendering them conductive is of an added value and can increase the range of GFRE composite applications, especially in sensing and dynamic monitoring systems. Similar to mechanical enforcement, the addition of modifiers to the matrix is one of the most popular ways to improve the electrical conductivity of GFRE composites [30]. Several organic modifiers have been used to render GFRE composite conductive such as polyaniline [31] and various carbon fillers including graphite, graphene, conductive carbon black, carbon fibers, and nanotubes [32–35]. The incorporation of metallic fillers such as silver and copper for higher composite conductivity have also been investigated [36,37].

Towards circular economy and environmental protection, the use of waste materials from industrial and agricultural waste streams for the development of functional materials and composites has attracted strong attention in the last decade. Several waste streams have been explored including agro-waste [38], tire waste [39], electronic waste [40], and plastic waste [41]. In addition to these streams of waste, waste iron filings (WIFs) were investigated for a wide range of applications including water treatment [42] and biogas production [43]. WIFs are small particles of iron that are produced as a byproduct of various industrial processes such as machining, grinding, and sanding. They are often found in metalworking shops, factories, and other industrial settings, and can accumulate in large quantities over time. Waste iron filings can pose a risk to the environment if they are not properly disposed of. When WIFs come into contact with water or other liquids, they can leach harmful substances into the soil or water supply, which can have negative impacts on the health of plants, animals, and humans. As such, it is important to properly manage and dispose of WIFs in a way that minimizes their potential environmental impacts. Due to their remarkable durability and mechanical strength, WIFs have been used to reinforce several matrices including thermoplastic starch [44], concrete [45], asphalt [46], polystyrene [47], and others.

This study investigates the use of WIFs to improve the mechanical and electrical properties of GFRE composites. Such electrically conductive GFRE composites have not been reported in the literature and could be interesting material candidates for a wide range of applications including structural dynamic monitoring systems in the construction industry. This work also supports the efforts for the utilization of waste materials in the construction industry towards a circular economy.

2. Materials and Methods

2.1. Materials

In this study, an epoxy resin with a hardener (Clear Tabletop Epoxy Resin, Pro Marine, Kalamazoo, MI, USA) with a mass ratio of 1:1 was used as a matrix for the GFRE composite. The used epoxy resin was a Bisphenol A-epichlorohydrin polymer. It had a density of 1.16 g/cm^3 and a viscosity of 11,000–14,000 cps at 20 °C. The hardener consisted of Nonylphenol, Diaminopolypropylene glycol, and Trimethylolpropane polyoxypropylenetriamine. It had a density of 0.97 g/cm^3 and a viscosity of 1200 cps at 25 °C. Woven S-fiberglass mats (Fiberglass Mat, Evercoat, Cincinnati, OH, USA) with a density of 2.2 g/cm^3 (GSM of 450 g/m^2 and thickness of 1 mm) were used as reinforcement in the manufactured composites. WIF (Fine Iron Filings, Magnetic Iron Powder, Toys Central, Dubai, UAE) with an average particle size of 400 μm were added to the matrix to fabricate the hybrid GFRE composite. All materials were used as received without any pre-or post-treatments.

2.2. Glass Fiber-Reinforced Epoxy Resin (GFRE) Composite Fabrication with and without WIF

All GFRE samples were designed and prepared with dimensions of 250 mm × 150 mm × 2 mm using a fixed mass ratio of 15 w% of glass fibers and 85 w% of the matrix mixture (epoxy resin and hardener with or without WIF particles). At first, the WIF particles were slowly added to the hardener with gentle stirring for 15 min (around 60 rounds per minute) to ultimately prepare GFRE samples containing 0%, 3 %, 6%, 9%, 12%, 15%, 25%, and 50% (w%) of WIF particles. The WIF-containing hardener was then added to the epoxy resin using a mixing mass ratio of 1:1. The mixture was gently stirred for 15 min (around 60 rounds per minute) until the iron particles homogenously distributed in the mixture without generating many bubbles. A hand lay-up method was immediately applied for GFRE composite fabrication, in which the prepared viscous mixture was immediately poured into a wax-coated mold and one layer of glass fiber mats with a size of 250 mm × 150 mm was laid in the mold. The resin mixture was gently spread on the surface of the glass fiber mat using a brush to avoid bubble formation. The curing process took place at room temperature for 48 h. In total, sixteen samples were prepared using eight different WIF addition conditions, and each condition was duplicated. The compositions of GFRE composite samples are presented in Table 1. It is important to mention that the preparation process was critical, and the authors therefore standardized the steps of the process to assure reproducibility and to avoid the formation of bubbles. For the same reason, the samples were prepared at the same time by the same person using the exact same procedure.

Table 1. The fabrication conditions of the WIF-reinforced GFRE composites.

WIF (w%)	Glass Fibers (w%)	Epoxy (w%)	Hardener (w%)
0	15	42.5	42.5
3	15	41.0	41.0
6	15	39.5	39.5
9	15	38.0	38.0
12	15	36.5	36.5
15	15	35.0	35.0
25	15	30.0	30.0
50	15	17.5	17.5

2.3. Characterization of the Prepared Glass Fiber-Reinforced Epoxy Resin (GFRE) Composites

Specimens with a dimension of 30 mm × 30 mm × 2 mm were cut from the fabricated GFRE composite samples, and their density was determined based on the measured volume and mass. Brinell Hardness Number (BHN) was then estimated to evaluate the surface hardness of the samples. A fixed load was applied to the specimen's surface and the dimensions of the dent were then measured. The following formula was used to determine BHN value:

$$BHN = \frac{2P}{(\pi D)(D - \sqrt{D^2 - d^2})}$$

where BHN was used to characterize the indentation hardness of the samples (kgf/mm^2), P is the force applied (kgf), D is the standard diameter (mm), and d is the dent diameter after applying the force (mm).

Tensile test was conducted at room temperature with a speed of 1 mm/min following the standard ASTM D3039-76 using Instron 8801 (Instron, Norwood, MA, USA) with a maximum force of 100 kN. The samples were 150 mm × 12 mm × 2 mm with a gauge length of 100 mm. An extensometer was used to measure the amount of elongation in the pieces until failure. Three specimens of each sample were tested to obtain the average tensile strength, Young's modulus, and elongation at break. Bluehill software (Version 2, Instron, Norwood, MA, USA) was used for test control, data acquisition, and for analyzing the obtained stress-strain curves.

The microstructure of the GFRE with and without WIF was studied using optical light microscope Leica DM750 (Leica Microsystems, Heerbrugg, Switzerland) using HI PLAN 4x lens. The photos were captured using a Canon EOS 2000 D camera connected to the microscope. Finally, the electrical conductivity of the fabricated GFRE composites was determined following the four-point probe method using two multimeters (UT33C+ Digital Multimeter, UNI-T, Dongguan, China) and two copper as electrodes (Figure 1). The following formula was used to calculate the electrical conductivity:

$$\kappa = \frac{I * L}{V * A}$$

where κ is the electrical conductivity (S/m), L is the length of the sample (m), A is the cross-sectional area of the sample (m^2). V and I represent the voltage and current measured using the two multimeters following the four-point method.

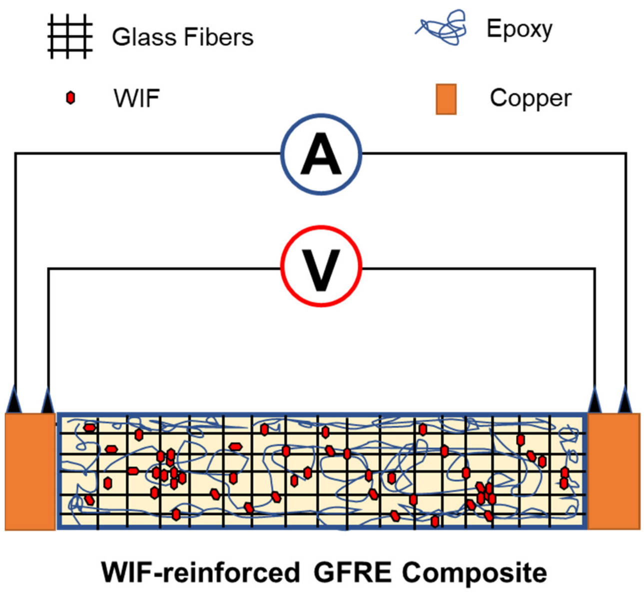

Figure 1. Four-point probe method for measuring the electrical conductivity of the WIF-reinforced GFRE composites.

3. Results and Discussion

One of the most important properties of GFRE composites is their low density (1.4 g/cm^3), which makes them promising light-weight materials for a variety of applications. Therefore, it is of great importance to investigate the impact of WIF addition on their density. The results showed that GFRE composite density significantly increased upon WIF addition because of the high WIF density of around 2.9 g/cm^3 (Figure 2). It gradually increased from around 1.4 g/cm^3 for the composite without WIF to ca. 1.6 g/cm^3 upon the addition of up to 15 w% of WIF. With the addition of 25% WIF, GFRE density increased to around 1.8 g/cm^3, and to 2.1 g/cm^3 upon the addition of 50% WIF. These results are in agreement with calculated theoretical densities of these composites using the densities of the individual GFRE composite and WIF and their mass ratios. This may imply that WIF addition did not induce any major changes to the curing process of the epoxy resin and its structure as a result. Density variation in filler-reinforced composites has been reported and discussed in the literature, which mainly depends on the type and relative proportion of the matrix and reinforcing material [48,49].

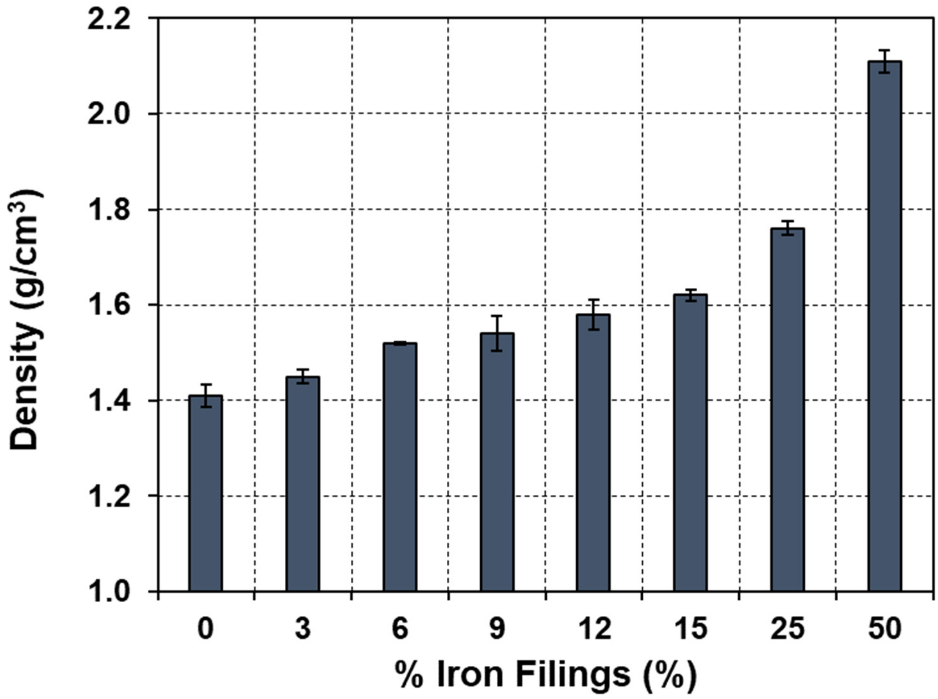

Figure 2. The density of the GFRE composites upon the addition of different WIF amounts.

The surface hardness of the composites was then evaluated based on the Brinell Hardness Number (BHN) (Figure 3). The results showed the BHN value gradually increased from 84 kgf/mm^2 to around 95 kgf/mm^2 upon the addition of up to 9% of WIF. It then gradually but more significantly decreased to 69 kgf/mm^2 for the GFRE containing 25% WIF. It dropped down to 55 kgf/mm^2 upon the addition of 50% WIF. This is in line with the reported filler-reinforced composite behavior, in which the filler initially strengthened the surface hardness of the composite, and then a drop in surface hardness was observed with higher filler content [50]. More WIF content may have negatively impacted the integrity of the GFRE composite by hindering effective entanglement between the epoxy polymeric chains during curing [29].

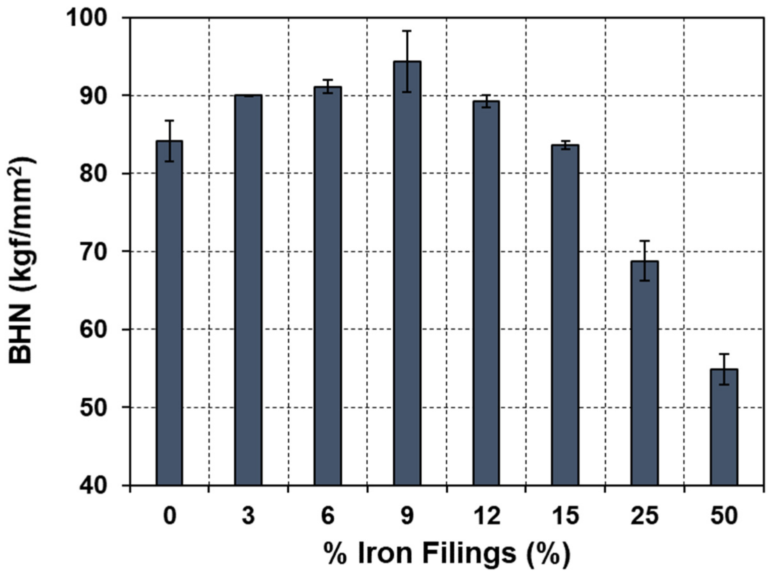

Figure 3. Brinell Hardness Number (BHN) values of the GFRE composites upon the addition of different WIF amounts.

The BHN value of the GFRE composite provided a first insight about the impact of WIF addition on the composite's surface hardness. However, it is of importance to study the impact of WIF addition on the bulk mechanical properties of the composite. For this goal, tensile testing was conducted on the composite samples and the Young's modulus, tensile strength, and elongation at failure values were determined from the obtained stress-strain curves (Figures 4 and 5). The results showed that the GFRE composite with no WIF had a Young's modulus of around 1.4 GPa and a tensile strength of 21 MPa while the elongation at failure was around 2%, which are in agreement with literature reports. Young's modulus of the composite increased significantly to 4.6 GPa upon the addition of up to 9% of WIF. This is three times the Young's modulus of the GFRE composite without WIF, which is a result of the high Young's modulus of WIF. The modulus then started to decrease down to 1.6 GPa upon the addition of 50% WIF. This is in agreement with the BHN values, which is possibly a result of the negative impact of WIF particles on the integrity of the epoxy resin on the molecular level. The WIF particles may have acted as barriers during the curing of the epoxy resin hindering its moieties from effective crosslinking reducing the overall crosslinking density of the GFRE composite. This behavior is typical in filler-reinforced composites. Moreover, WIF agglomeration in the GFRE composite increased with increasing WIF amount, which can be disadvantageous for obtaining continuous structure, disabling effective stress transfer and deteriorating the mechanical properties as shown in light microscopy images of the GFRE composite in Figure 6. The WIF particles are well-distributed in the GFRE samples with a WIF content of 9% and less while WIF agglomeration is evident in the samples with 15% WIF in addition to more significant agglomeration in the 50% WIF-containing sample.

Similar to the Young's modulus behavior, the tensile strength of the GFRE composite increased from 21 MPa without WIF to 32 MPa upon the addition of 9% of WIF. It then decreased to 10 MPa upon the addition of 50% WIF. In terms of elongation at failure, it gradually decreased from around 2.0% for the composite without WIF to 0.6% upon a WIF addition of 50%. It is important to discuss the stress-strain behavior as a result of WIF addition to the GFRE composite. The stress-strain curve of the GFRE composite without WIF shows a dominant elastic deformation with a barely noticeable plastic deformation. With the addition of up to 9% of WIF, the plastic deformation of the GFRE composite became more significant. It then became less significant with the addition of more WIF. This indicates that the WIF addition of up to 9% not only improved the elastic domains in the GFRE composite but also the plastic fluid-like domains. The elasticity of the composite increased because of the stiff nature of the WIF particles while the plasticity of the composite may be a result of the WIF particles acting as barriers between the epoxy molecules during curing leading to domains of less crosslinking density and therefore more plasticity. With the addition of more WIFs (12% to 50%), the stiffness action of the WIF particles may have become more dominant than their ability to hinder epoxy curing since the stress–strain curves gradually showed more dominant elastic deformation upon further WIF addition.

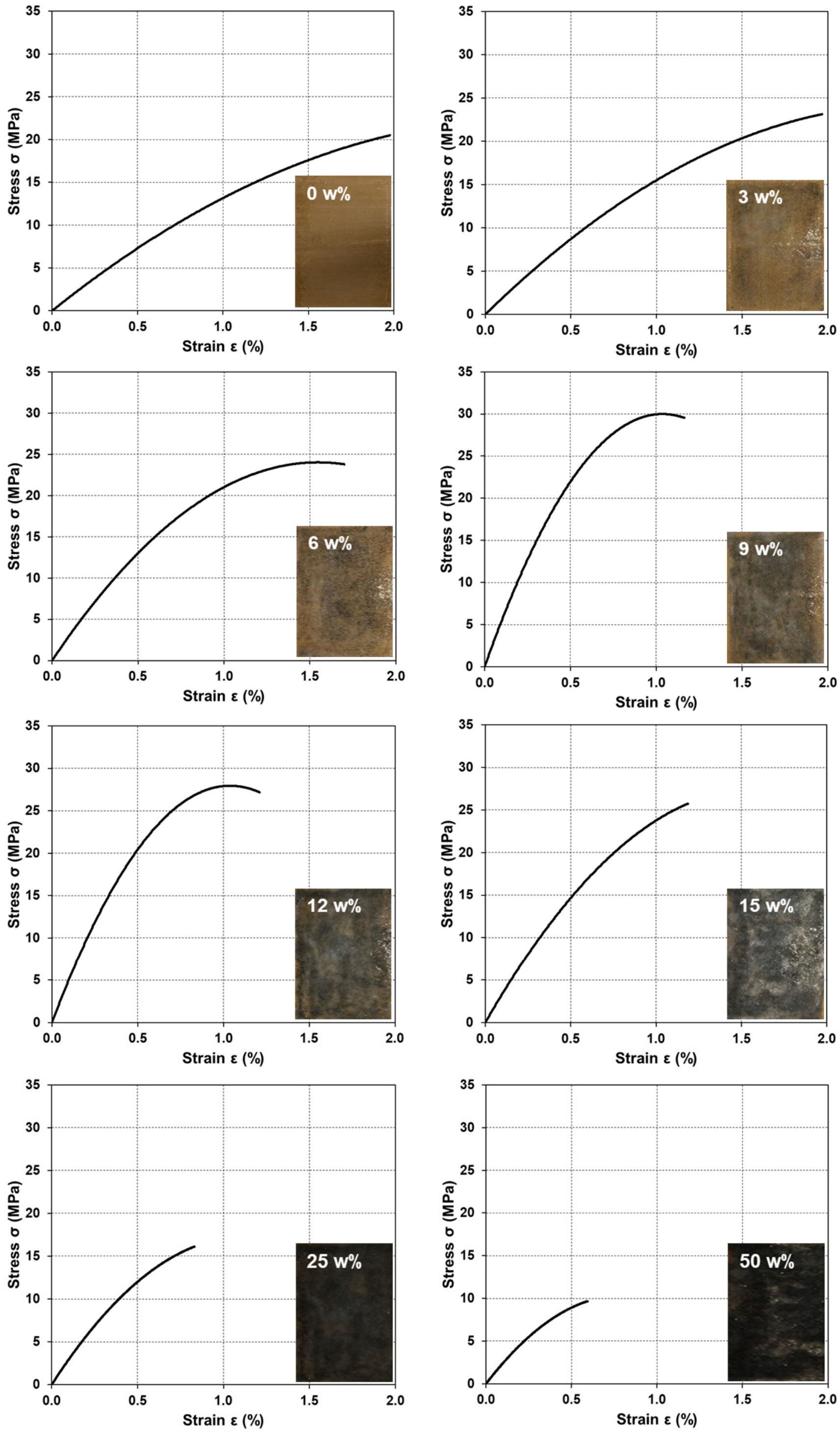

Figure 4. The stress–strain curves obtained from the tensile testing of the GFRE composites upon the addition of different WIF amounts.

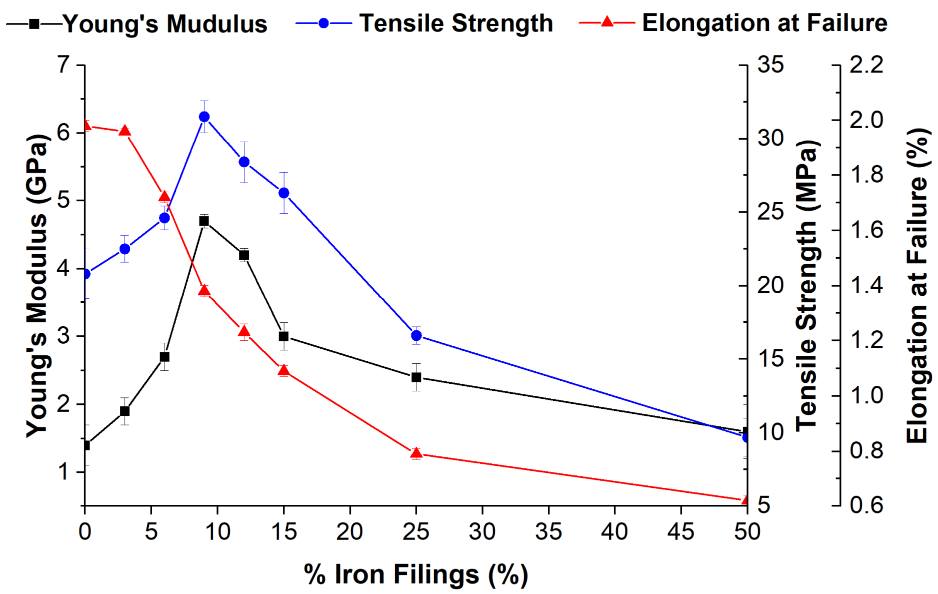

Figure 5. The mechanical properties (Young's modulus, tensile strength, and elongation at failure) of the GFRE composites upon the addition of different WIF amounts.

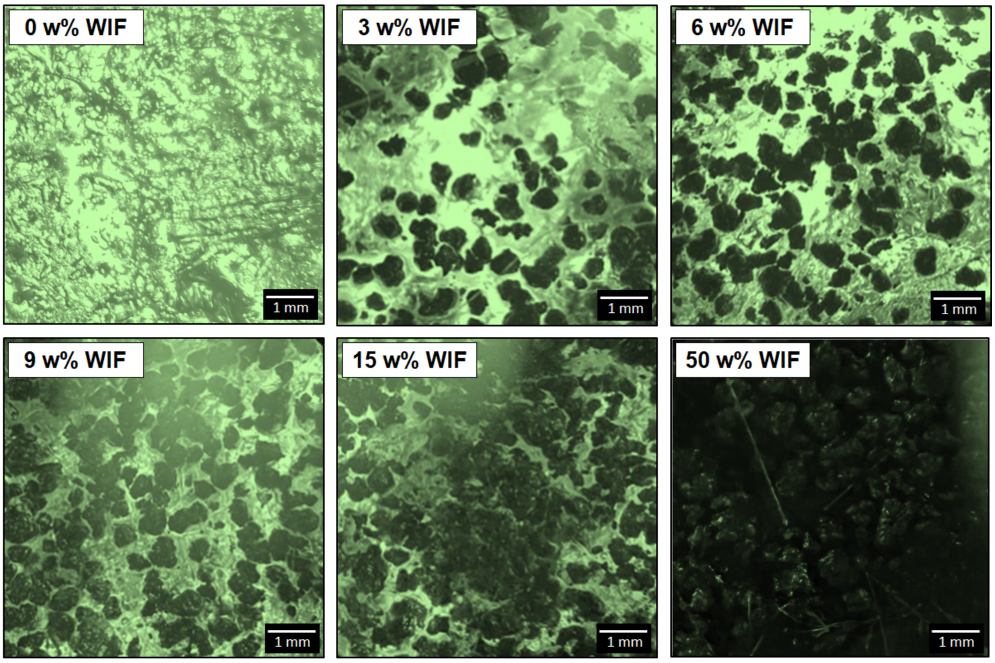

Figure 6. The microstructure of the GFRE composites with and without WIF using optical light microscopy.

Other than improving the mechanical properties of the GFRE composite by the addition of WIF, this work also aims to render it electrically conductive. This will widen the range of applications of GFRE composites. The electrical conductivity of the GFRE composite samples was measured following the four-point probe method as it is considered to be one of the accurate methods for this purpose [51]. A multimeter is used to measure the current flowing in the circuit, which is the same current flowing through the composite, and the voltage drop between its two sides is measured using a second multimeter (Figure 1). The results showed that the samples with WIF content of up to 12 w% had no detectable electrical conductivity, while a conductivity of 2.9 kS/m was measured using a WIF content of 15%, which significantly increased to 13 and 35 kS/m upon the addition of 25% and 50%

of WIF, respectively (Figure 7). These electrical conductivity values are comparable to the reported electrical conductivity of WIF of 200 kS/m [52].

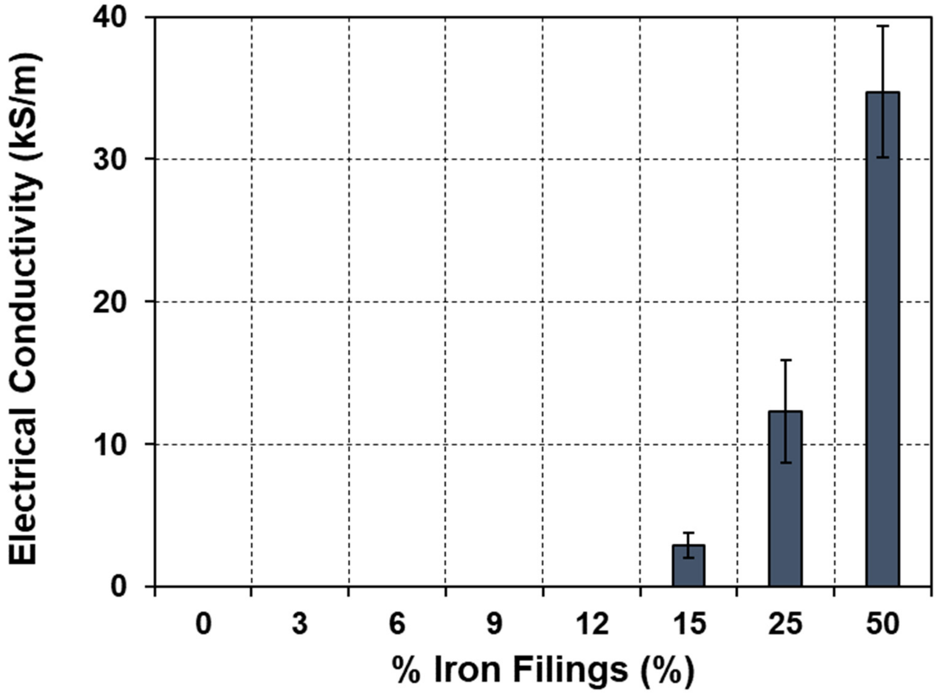

Figure 7. The electrical conductivity of the GFRE upon the addition of different WIF amounts.

The literature reports on the electrical conductivity of binary composites composed of conductive metallic filler and insulative polymer matrix are numerous [53–55]. The leading mechanism that has been discussed to drive the transition from insulation to conduction is based on percolation theory [55–57]. It has been observed that the electrical conductivity of these two-phase composites grows abruptly as the fillers' concentration increases. The concentration value where this behavior occurs is related to the percolation threshold, which is dependent on the filler species, dimensions, and aspect ratio [54]. By increasing the number of metallic particles in the composites, interconnection between the particles will be formed by aggregates, followed by the formation of effective networks and paths facilitating electron hopping within these binary-system composites, leading to integral electrical conductivity. However, when a third phase is involved in the composite system formation, such as glass fiber reinforcement in this study, the scenario seems to be more complicated. A higher number of conductive fillers are required in order to achieve sufficient connectivity for effective conduction. As observed in this work, 15 w% and more of WIF microparticle are needed to obtain electrically conductive GFRE composites. Small aggregates might be generated and randomly distributed among different phases with the existence of only small number of conductive particles in the composites. Lack of successive conduction networks and useful connectivity could be the major reason for non-detectable electrical conductivity when WIF content was less than 15 w% as shown Figure 6. The WIF particles in the GFRE samples with less than 15% WIF content are clearly far away from each other while they are closer and start to connect to each other using 15 % WIF content and more.

In summary, the GFRE composite with 15% WIF content is worth exploring in applications, in which light-weight composites with advantageous electrical and mechanical properties are needed. This composite has a density of around 1.6 g/cm^3, Young's modulus of 3 GPa, tensile strength of 25 MPa, and excellent electrical conductivity of 2.9 kS/m. For instance, such material can be a potential candidate for dynamic structural monitoring in the construction industry and would be in line with the strong efforts in this industry to reduce its environmental impact through the utilizing of waste streams such as WIF for the fabrication of construction materials.

4. Conclusions

In this work, waste iron filings (WIFs) were used to develop novel electrically conductive glass fiber-reinforced epoxy (GFRE) composites with improved mechanical properties. The impact of WIF addition on the structural, physical, mechanical, and electrical properties of the composites was thoroughly investigated. The results showed that with increasing the amount of WIF content from 0 w% to 50 w%, the surface hardness, Young's modulus, and tensile strength of the GFRE composite significantly increased until 9% of WIF content followed by a gradual drop with more WIFs introduced to the composite due to WIF agglomeration, which was evident in the microscopic images. The GFRE composites were found to be electrically conductive upon WIF addition of 15% w% or more. The electrical conductivity of the GFRE composite was 2.9 kS/m upon the addition of 15% WIF and increased to 35 kS/m upon the addition of 50% WIF. Overall, the reuse of WIFs as a waste material was shown to be promising for producing GFRE composites with enhanced mechanical and electrical performance. Such an electrically conductive GFRE composite has not been reported in the literature and could be an interesting material candidate for a wide range of applications including structural dynamic monitoring systems in the construction industry. It also supports the efforts for the utilization of waste materials towards a circular economy. Further testing and characterization of this composite should be conducted to investigate its feasibility in different applications.

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