RECYCLED GLASS FIBRE/POLYESTER RESIN SYSTEM – INTERFACE CHARACTERIZATION

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Abstract

Polymer composites are finding substantial application in the most diverse engineering fields. Use of polymers and synthetic fibres in large structural composite developments consume several tonnes of material. End-of-life scenarios of composite structures is a major issue in the composite industry due to current norms of environmental policies and impending waste management legislation. Scientist are evaluating new methods and techniques to recycle structural composites. Current article addresses the recycling of glass fibres from a composite structure and regenerating glass fibre functionality through surface treatments. In the present study, bonding between recycled glass fibre and polyester resin are evaluated through experiment approach called micro-droplet test. Results demonstrate strong effect on fibre properties due to various surface treatments used for regenerating recycled glass fibre surface.

Keywords

Glass fibre, Thermal conditioning, Tensile strength, Strength recovery, Interface strength

1. INTRODUCTION

Environmental protection is a growing concern for many industries today, with emphasis on the reduction of carbon dioxide and other poisonous emissions like carbon monoxide -CO, nitrogen oxides- NO_x to mitigate climate change. European Union and major countries in the world demanding solutions for end-of-life scenarios of used materials and structures, hence put more pressure on the industry to address the options available for dealing with FRP waste. Annually around 35% of the total production of FRP are used by transportation and construction industries in Europe. As a raw material, glass fibre accounts for about 90% of the reinforcements used in composite consumption globally. Excluding composites, glass fibres usage is high in other sectors

of product developments targeting properties - good insulation, strong heat resistance, and better in corrosion resistance.

Glass fibre with high strength and modulus can be achieved by adding sizing chemicals modifying fibre surface. Fibre manufacturers develop their own sizing know-how and apply it to the fibre to best suit the needs of targeted composite structures or products. Sizing is essential to glass fiber manufacture and critical to several key fiber characteristics that determine both how fibers will handle during processing and how they perform as part of a composite. Most commonly used polymers for composite structural developments are unsaturated polyester resin and epoxy resin. Interface properties of virgin glass fibre with polyester/epoxy resin are well known in composites community, which are experimentally studied by several scientists using micro-droplet, fibre pull out tests.

From the past decade, composites recycling gained much importance in industry and academic scientists derive methods to recycle thermoplastic polymers and fibre reinforcements (glass and carbon fibres). The academic research slowly moving to industry by knowledge transfer for possible future developments in the areas of end-of-life of composites structures. From the literature [1-4], Pickering [1] demonstrated experimental methods to recycle carbon fibres and reuse the carbon fibre for several product developments. Thomason and co-workers [2-4] developed methods to recreate functionality to recycled glass fibres and generate fibre strength and modulus. Durai Prabhakaran [4] discussed the property enhancements obtained for the recycled glass fibres and studied recycled glass fibre/polyester resin composite performance. Few articles discussed the methods to evaluate interface properties of recycled glass fibre with polymer, articles highlighting the steps of surface treatment which affect of the performance are not many in the literature.

In this article, recycled glass fibres are chosen to study tensile properties and its interface properties with polyester resin. The microdroplet test is carried out under optimal testing conditions, established from our previous work, in order to study debonding and interfacial shear stress analysis of the recycled glass fibre-polyester resin. Glass fibre as received in chopped strand mat (CSM), heat-treated (silane removed), silane coated without prewashed heat treated glass fibres, silane coated after prewashed heat treated glass fibres are chosen to investigate the influence of washing glass fibre surface on the fibre/matrix bond.

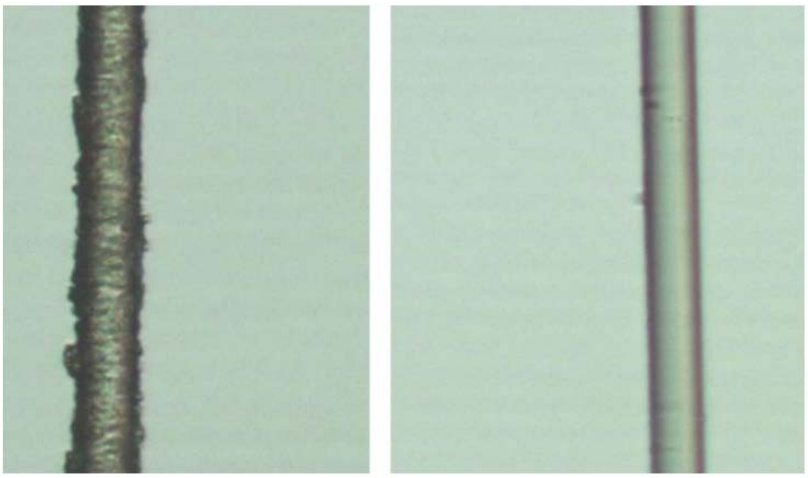

Figure 1: Heat-treated glass fibre surface without wash and with a water wash

2. EXPERIMENTAL WORK

2.1 Materials

CRYSTIC 701 PAX polyester infusion resin and its associated catalyst (methyl ethyl ketone peroxide) were acquired from East Coast Fibreglass Supplier. The chopped strand mat 92 was provided by PPG industries with average fibre diameter of 13μm.

2.2 Pyrolysis

The recycling was made through burn-off process, in a Carbolite Furnace. A sample was placed in the centre of an aluminium tray and then put into the furnace, at room temperature (25°C). The furnace heats up to 500°C and stays at this temperature until the end of the experiment. The sample stays inside the furnace until the entire polymer is burned-off and only clean fibres remain, this time may vary according to the sample conditions. Glass fibres from pyrolysis are observed under optical microscope to study fibre surface, see Figure 1.

2.3 Single Fibre Tensile Test

Single fibre tensile tests were chosen to evaluate the fibre properties [2]. The fibre strength was determined by using the single fibre tensile test based on ASTM D2256. Gauge length of the test specimen is critical in single fibre tensile test, where single fibre strength was highly dependent on the specimen length. Due to difficulties in separation of single glass fibre from fibre bundle of CSM and specimen preparation issues, the tests are performed for only 20 mm gauge length. Individual fibres were glued onto card tabs with a central cut-out that matched the gauge length chosen for the test as shown in Figure 2. Then the tab ends were gripped by the universal testing machine (Instron® Model 3342) with a 10N load cell. Tests were performed with a gauge length of 20mm and strain rate of 1.5%. All samples were photographed with a microscope lens of 500x, and then the diameter was obtained using ImageJ software with a scale of 113.5 pixel/μm. The mechanical performance of single fibres taken from CSM of well-defined sizing and binder and heat treated E-glass fibre samples, heat treated, heat treated and silane treated, heat treated and washed glass fibre with silane sized were investigated at room temperature after thermal conditioning at temperatures upto 500°C.

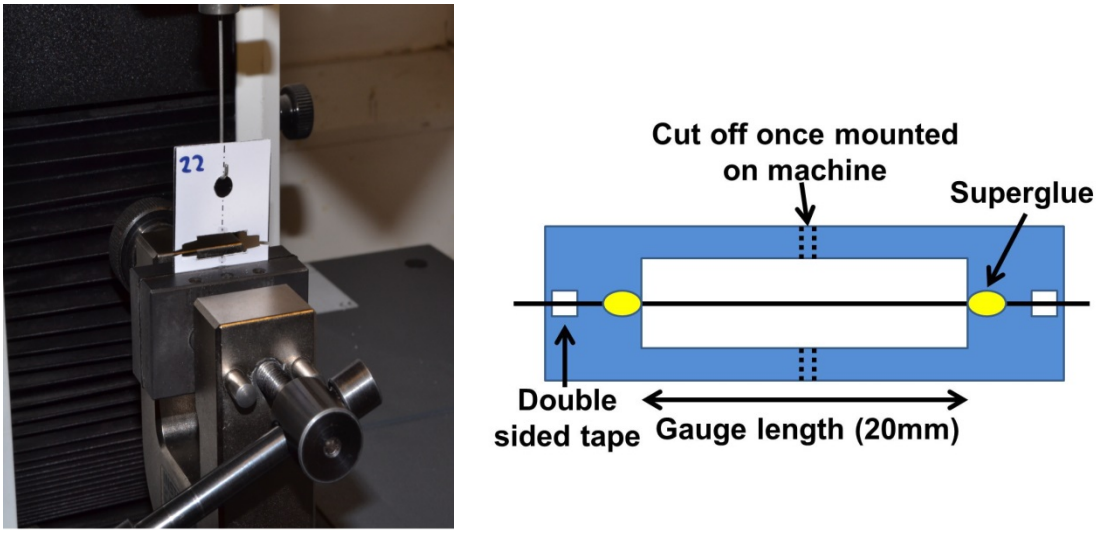

Figure 2: Single fibre tensile test setup: glass fibre glued in a card tabs

2.4 Microdroplet Test

The microdroplet test is a simple experiment technique designed to study interface between fibre and polymer. Where fibre is embedded in a drop of resin and subsequently pulled out while the drop is being supported by two knife-edges, resulting in either debonding of the droplets from the fibres as shown in Figure 3, or breakage of the fibres before debonding can occur. An in-house experimental set-up is designed for the microdroplet test were used to study recycled glass fibre interface properties with polyester resin, refer Figure 4. Specially designed fixture [2-3] with two movable knife edges controlled by a pair of micrometer heads with resolution to 1μm. The microbond tests were conducted with a free distance between fibre and knife edge of 20μm. A stereo-microscope was utilised to aid the positioning of knife edges and monitor the testing process. The same testing machine used in the single fibre tensile test with 10N load cell was employed to carry out the test with the rate of fibre end displacement set to 0.1mm/min. The fibre with bonded resin droplets was mounted in the machine. Some card frame was left taped to the bottom of the fibre to keep it under tension (~0.5mN). The fibre was pulled out of the droplet while the droplet was constrained by the knife edges. Where the load-extension from the sample is recorded and noted the peak load to estimate the Interfacial Shear Strength (IFSS).

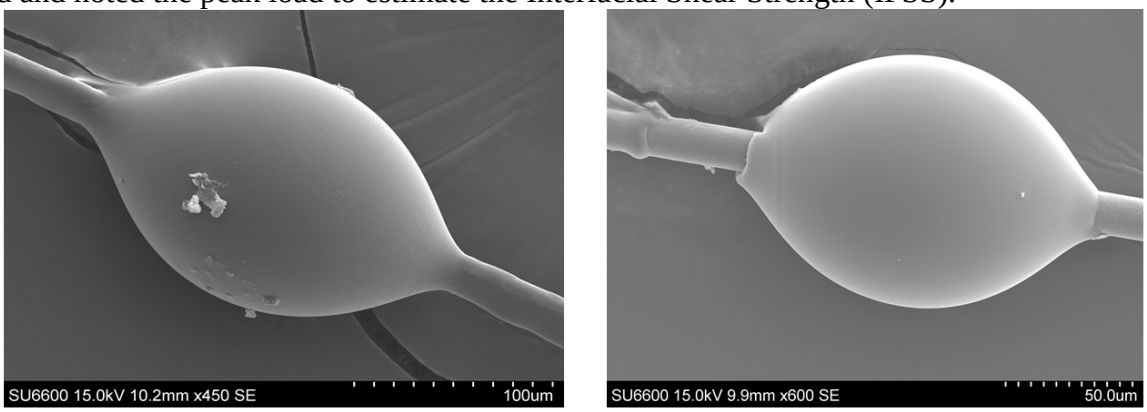

Figure 3: Polyester droplets on glass fibre surface (before and after micro-droplet test)

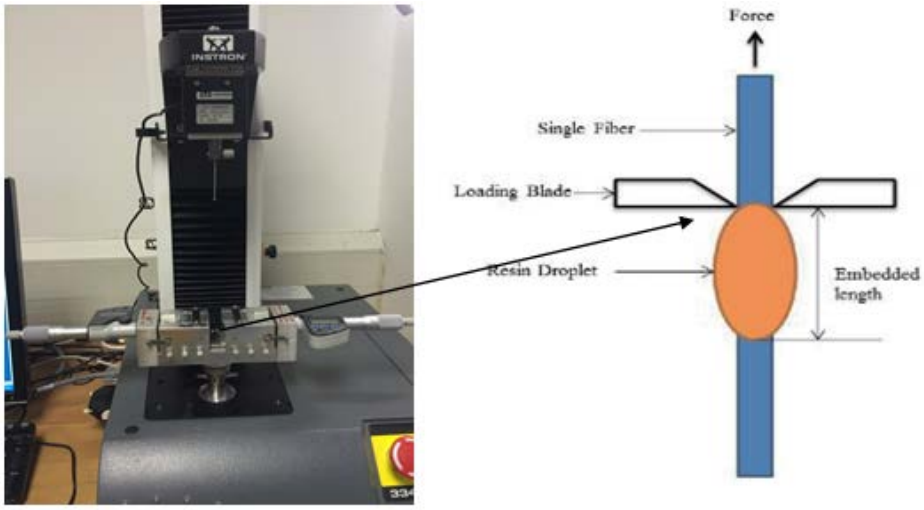

Figure 4: Experimental setup to characterize the interface of recycled glass fibre/polyester

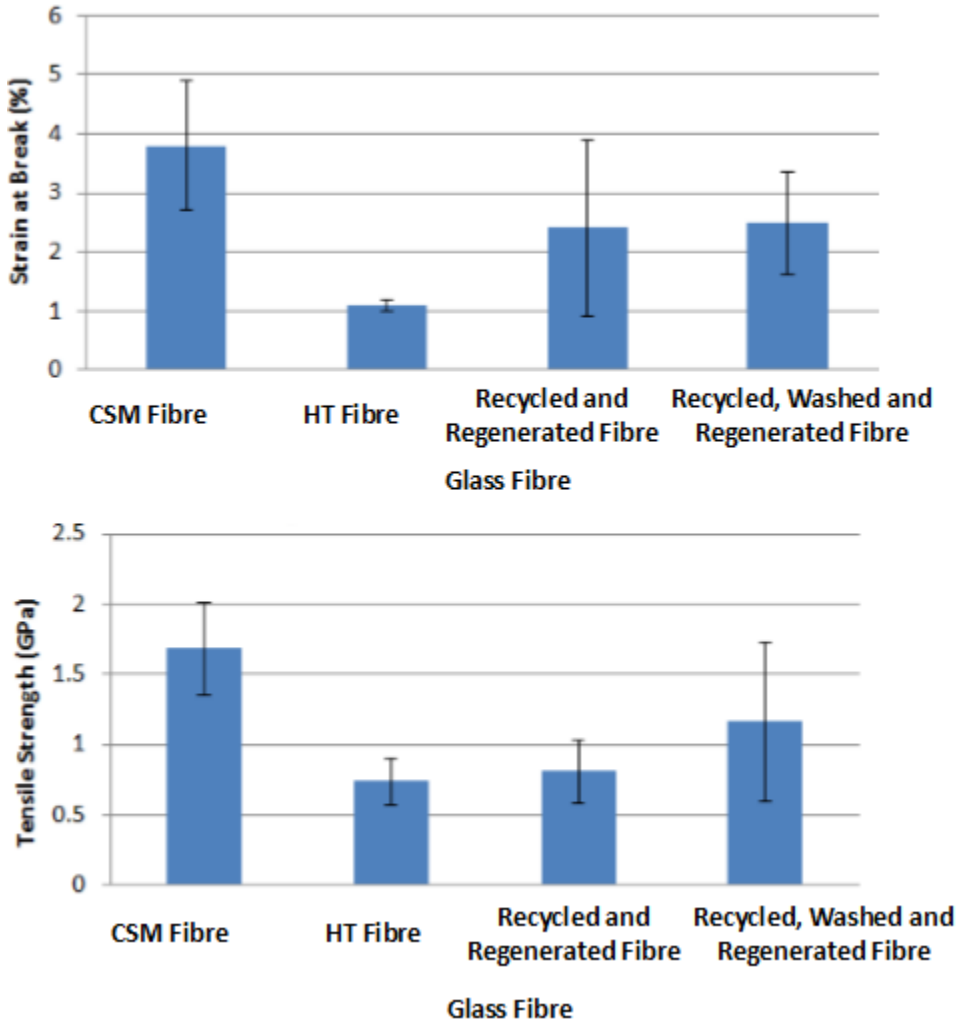

Figure 5: Single Glass Fibre Tensile Properties Before and After Heat Treatment

Table 1. Tensile Properties – Recycled Glass Fibres

Fibre Type	Diameter (μm)	Force Peak (N)	Strain Peak (%)	Stress at fibre break (GPa)	Youngs Modulus (GPa)
CSM Fibre	14.0 ± 0.9	0.26 ± 0.03	3.8 ± 1.1	1.687 ± 0.330	59.3 ± 8.30
HT fibre (500deg C @30min)	13.6 ± 1.4	0.11 ± 0.02	1.1 ± 0.1	0.739 ± 0.162	63.3 ± 17.2
Recycled and Regenerated Fibre	12.6 ± 0.8	0.11 ± 0.03	2.4 ± 1.5	0.810 ± 0.226	57.5 ± 11.3
Recycled, Washed @70deg C and Regenerated Fibre	11.6 ± 0.9	0.15 ± 0.05	2.5 ± 0.9	1.376 ± 0.401	67.6 ± 10.0

4. RESULTS AND DISCUSSIONS

4.1 Fibre Strength

Recreating fibre functionality and regenerating fibre strength is a major task in composites recycling. This involves sizing chemistry and requires series of experiments to study sizing chemical compatibility with fibre surface and with the polymer. The results for the average single fibre strength (at 20mm testing gauge length) of CSM fibre with standard sizing's, CSM fibres after heat treatment, and heat treated fibres and regenerated with silane sized, heat treated fibres and regenerated with a hot water wash and silane sized are shown in Figure 5. The results indicate that thermal conditioning can cause a considerable strength reduction for fibre samples after heat treatment, with a loss of over half of the original strength in the case of 30 minutes at 500°C. It can be seen that heat-treated glass fibre types reduce in strength, with the silane-sized glass falling by a greater percentage of its original strength. In general glass fibres can be extracted after thermal conditioning 500°C and above. Above this threshold temperature the average fibre strength is seen to decrease rapidly.

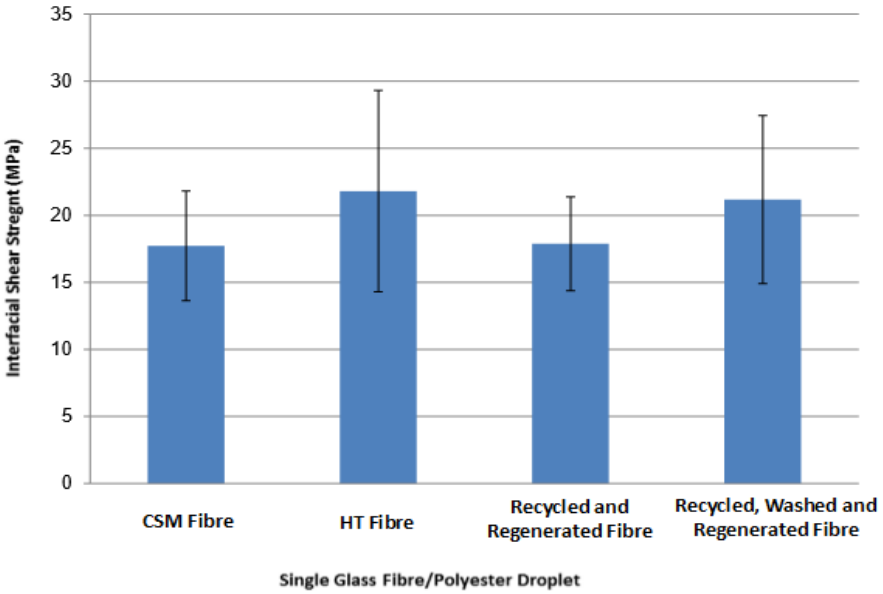

Figure 6: Single Glass Fibre/Polyester Resin Interface Properties

Comparison with the results in Figure 5 indicates that the single fibre tensile strength results of recycled washed and regenerated/sized fibres recover its strength around 83% compared to fibre strengths of CSM glass fibres. Diameter of fibres used in the study are given in Table 1 shows fibre after heat treatment have polyester particles resulting diameter similar to CSM. After hot water washing of heat treated fibres and silane treated the fibre diameter reaches to 11.6 μm forming better coating surface for glass fibres. The CSM fibres and fibres taken after heat treatment or during sizing treatments are having greater potential for damaging due to bundle-bundle interactions while separating from the fabrics (also damaging due to fibre-fibre interactions within the fibre bundles) taken from CSM (fibres separating from bundles are extremely difficult). In some cases the fibres after separating from the fibre bundles seems extremely difficult to handle or fixing single fibres on card frame which can also damage the fibres. The results shown in Figure 5, indicates recycled-washed-and-regenerated fibres can recover strain-to-failure around 66%

compared to CSM fibres, which is a good sign of tensile property improvement. Whereas the modulus measured for the four fibre types show different trend. Fibres taken from CSM gave 60GPa, whereas for the heat-treated fibres the modulus is a bit higher than the modulus of CSM (63GPa). The recycled and regenerated fibres shown better performance and the modulus recorded is 57.5GPa lesser than the recycled-washed-regenerated fibres (68GPa). Therefore, fibre washing is an important step in regenerating fibre strength and modulus. In addition, the results demonstrate the strength of glass fibres is strongly influence by thermal conditioning at temperatures and times, which may commonly be experience in the processing of such fibres in engineering composite materials.

4.2 Interfacial Shear Strength (IFSS)

Interface strength for single glass fibre/polyester can been determined by using fibre pull-out and microbond methods. In the current study, microbond test is chosen to evaluate the interfacial properties. The apparent IFSS is an adequate quantitative parameter which can characterise the mechanism of interfacial failure in any fibre reinforced polymer composites. Sample preparation is one difficult task to use single fibres from CSM fabrics. To replicate the composite processing technique, microbond test specimens are prepared exactly similar to vacuum infusion technique used for laminate trials (i.e. droplets are formed under vacuum for 24 hours and then dried over an oven at 80°C for 4 hours). The average fibre tensile strength of CSM fabrics is 1.69±0.33GPa at 20 mm gauge length was obtained by the single fibre tensile test. The tests are conducted for 30 specimens each, and the average interfacial shear strength for each case is shown in Figure 6.

Different failure modes are observed during the microdroplet test: (a) fully debonded droplet, (b) partially debonded (broken) droplet and (c) mixed mode of the previous two modes. SEM pictures shown in Figure 3 demonstrates fully debonded droplet for a glass fibre (CSM) having silane sizing interface with polyester. Fibres taken from CSM and recycled-washed-regenerated fibres were able to perform good interfacial tests i.e. shown constant interfacial friction after debonding. Whereas the heat-treated fibres, as the fibre surface is not coated and the surface is damaged due to thermal conditioning to 500°C, where the droplets formed on the fibre surface could not show good bond strength with polyester and debonding similar to previous case. This indicates the specimens fail before droplet able to initiate debond and also observed specimen to encounter fibre breakage rather than fibre pull-out from the polyester droplet.

The results shown in Figure 6 demonstrate washing of glass fibre surface after heat treatment plays major role. Hot water with 70°C were used to clean the glass fibre surface after heat treatment. Figure 1 shows the polyester particles after burn off still stick to the fibre surface resulting into partial bonding with polyester resin. Washed glass fibre after heat treatment and then silane sized showed better bonding and hence better IFSS value compared as received CSM case (refer Figure 6). In general the reference case (as received CSM) have sizing on glass fibre surface and binders to keep the fibre bundles to form a fabric structure. Therefore the IFSS values recorded in the microbond test for reference case shown less values compared to heat treated glass fibre cases. The IFSS values recorded are in the range of 18-22 MPa, the values agrees well with the IFSS-range published in the literature for glass fibre-thermoset polymer interface (evaluated by the single fibre pullout technique).

5. CONCLUSIONS

In this paper, experimental work is conducted to evaluate recycled glass fibre performance under tensile and microdroplet test. Single fibre tensile properties and single fibre/polyester interface properties are determined experimentally. Summary of the results are as follows :

1. Single fibre testing presented here clearly show that the thermal conditioning (heat treatment at 500°C) likely cause damage to glass fibres and can potentially show decrease in the fibre properties compared to the room temperature fibre strength (i.e. CSM glass fibres).
2. The washed fibre after heat treatment and coated with silane sizings show nearly full recovery of interfacial shear strength as shown in Figure 6.
3. SEM micrographs reveal poor interface bonding can cause damage to microdroplet under loading and can move down after debonding occurs.
4. Hot water washing for a heat treated glass fibres before silane treatment, play key role in regenerating recycled glass fibre strength and modulus.

Theoretical and numerical models will be developed in the future work to find reasonable correlation between measured data and the predictions of interface properties through finite element models.

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EPOXY POLYMERS REINFORCED WITH CARBON POWDER WASTES

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Abstract

This paper investigates the incorporation of recycled carbon micro fibres obtained from the cutting process of laminate composites into epoxy polymers at different mass fractions (0, 2.5, 5, 7.5 and 10%). The elastic modulus and strength under tensile, compressive, flexural and impact loadings were investigated via Analysis of Variance (ANOVA). The tensile (compressive) modulus progressively increases up to 36.6% (28.6%) with the inclusion of carbon powder wastes. The inclusion of 5% mass fraction waste resulted in an increase of 27% (19%) in tensile (compressive) strength. The flexural strength also increased 28.6% when 10wt% carbon powder wastes were added. Carbon powder waste led, however, to a dramatic decrease (approx. 50%) in impact resistance attributed to the increase in stiffness.

Keywords: recycled, epoxy polymers, carbon powder waste, mechanical tests.

1. INTRODUCTION

The demand for composite materials has progressively increased in various technological applications due to their low density and improved mechanical performance compared to conventional materials. However, the fabrication of such materials generates waste that may lead to environmental damage if improperly disposed of. Landfills are the most common strategy, but several countries have already limited or banned the disposal of composites in landfills due to environmental issues [1,2,3]. Polymeric composites, especially thermosets, dominate the global market and, owing to their long-life cycle, alternatives to landfills need to be sought and boosted. Mechanical, thermal and chemical recycling have been developed in order to address this problem.

Mechanical recycling involves shredding and grinding and the subsequent separation of the fibre-rich fractions for reuse [4]. Mechanically recycled composites are usually reincorporated into new composites as fillers or reinforcement [1]. Thermal processes have also been developed for

energy recovery (combustion of the composite waste) or selective material recovery for reuse (fluidized beds and pyrolysis). In pyrolysis, for example, a large amount of thermal energy is required in order to remove the matrix phase. Chemical recycling involves the decomposition of the polymeric structure and the high-quality end products (monomers, hydrocarbon molecules, gases and chemical intermediates for polymerization) are reused to produce new components [5].

The literature suggests that wastes from the manufacture of composites can also be reused in recycled composites. The CarbonTek S.L.® company (Spain) has recently used powder waste resulting from the cutting process of carbon fibre-based products as polymeric matrix reinforcements for the fabrication of carbon fins, as well as to reduce the volume of waste generated [6]. The residue is composed of carbon microfibres coated with epoxy polymer, sometimes with pigment particles used to produce fins. Thomas *et. al.* [6] evaluated the effect of micro carbon fibres wastes inclusions at three different mass fractions (0, 10 and 20 wt%) on the thermal and mechanical properties of epoxy composite materials. These authors report an increase in compressive and flexural strength and impact resistance proportional to the mass fraction of residues. Although less significantly, hardness and erosion resistance also increased with waste inclusion. The use of waste at 10 wt% (20 wt%) increased the compressive strength by 6% (20%) relative to the pure polymer, being attributed to the additional energy expended by the cracks to overcome the micro fibres and particles.

Compared to most recycling processes, the methodology proposed by Thomas *et. al.* [6] is economically more feasible. Therefore, this work further investigates the effect of different mass fractions of carbon powder residues on the mechanical properties of the materials, extending the analysis to tensile modulus and strength.

2. MATERIALS AND METHODS

Carbon powder, supplied by CarbonTek S.L. ®, Spain, was obtained from the cutting process of laminated composites used in the manufacture of fins. These wastes were incorporated into an epoxy matrix phase. Figure 1a shows a pair of carbon fins from CarbonTek S.L., after the cutting and assembly processes. Figure 1b presents the cutting process leftovers. The fin-cutting process generates a powder that can be considered as carbon microfibres enveloped by a polymer matrix (Figure 2).

Figure 1 - a) Fins and b) Remains from the cutting process
Source: Thomas *et al*. (2014) [6].

Figure 2 - Powder collected after the cutting process

The particle size distribution of the carbon microfibres was performed by sieving at the range of 100-200 US TYLER. These particles were incorporated into the epoxy polymeric matrix (Resin MX-14 and hardener ARADUR HY 951, resin/hardener proportion of 10:1). The wastes were mixed to the epoxy matrix phase in the following mass fractions: 0; 2.5; 5; 7.5; 10%. For each condition 5 specimens were fabricated for each type of test (tensile, compression, flexural and impact) and later replicated. The components were mixed for 5 minutes and left for a curing period of 2 weeks to finally undergo mechanical testing.

2.1. Mechanical tests

Tensile, compressive and flexural tests were performed in a SHIMADZU AG-X Plus testing machine (Figure 3a) equipped with a 100 kN load cell, at a crosshead speed of 2 mm/min, according to ASTM D638-14 [7], ASTM D695 [8], ASTM D790 [9] standards. The elongation of the specimens was measured using a digital video-extensometer. Impact tests were performed in an XJJ series impact testing machine with a 15 J hammer (Figure 3b) according to ASTM D6110 [10].

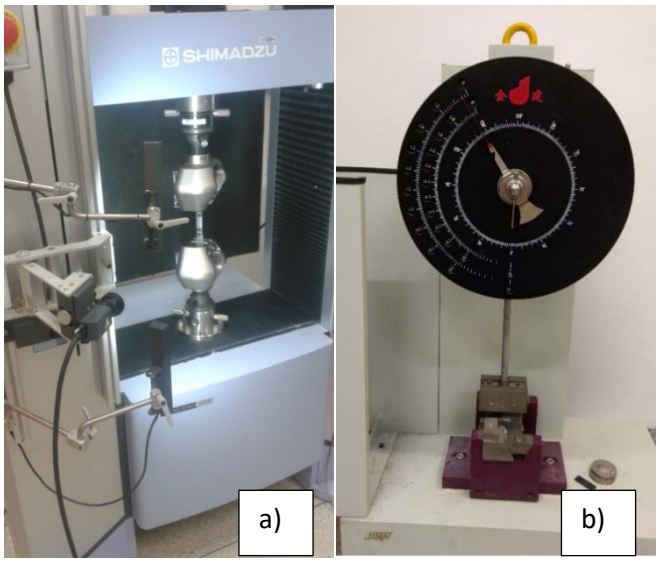

Figure 3 - a) SHIMADZU ® AG-X Plus Universal Testing Machine b) XJJ series impact testing machine

2.2. Scanning Electron Microscopy

A TM3000 Hitachi Analytical Microscope apparatus was used to investigate the morphological aspects of the carbon powder waste as well as the surface of the fractured samples. The images were obtained in secondary electron mode at 15kV.

2.3. Statistical Analysis

The experimental data was analysed via Analysis of Variance (ANOVA) and Tukey's test using Minitab 17, within a 95% confidence interval.

3. RESULTS AND DISCUSSIONS

Table 1: P-value ANOVA

Test	P-value
Mean Elasticity Modulus in Tensile	0.000
Mean Tensile Strengh	0.002
Mean Elasticity Modulus in Compression	0.000
Mean Compressive Strengh	0.000
Mean Flexural Strengh	0.000
Mean Impact Resistance	0.000

There are P-values of ANOVA in Table 1. The mean values of tensile modulus varied from 1.42 to 1.94 GPa (Figure 4a). Tukey's test indicates that carbon powder waste inclusions increased the stiffness at all levels. In particular, for 10 wt%, the tensile modulus was 36.6% higher relative to the reference level (non-particulate samples). Tensile strength ranged from 28.83 to 36.87 MPa (Figure 4b). Based on Tukey's test, waste inclusions increase the tensile strength at all levels, especially at 5 wt% (30% above the reference level).

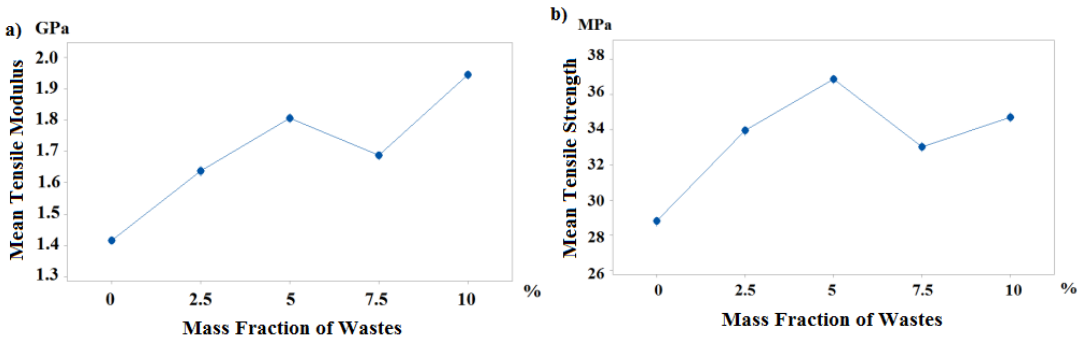

Figure 4 - Main effect plot for mean tensile (a) modulus and (b) strength.

The mean compressive modulus varied from 1.47 to 1.89 GPa (Figure 5a). According to Tukey's test, the incorporation of 7.5 wt% and 10 wt% resulted similar, exhibiting a significant increase of 28.6% relative to the reference. The mean compressive strength ranged from 50.06 to 59.64 MPa (Figure 5b), with an enhancement of approximately 19% when 5 wt% wastes are added. Such behaviour can be attributed to the microfibres, which inhibit crack proliferation by the bridging effect. Behind the crack front, bridging fibres stretch freely along the separating crack faces and in analogy to Hook's law, absorb energy that will otherwise be available at the crack tip.

In addition, as discussed below (microscopical analyses), particles are present in the carbon powder waste and also prevent the propagation of cracks.

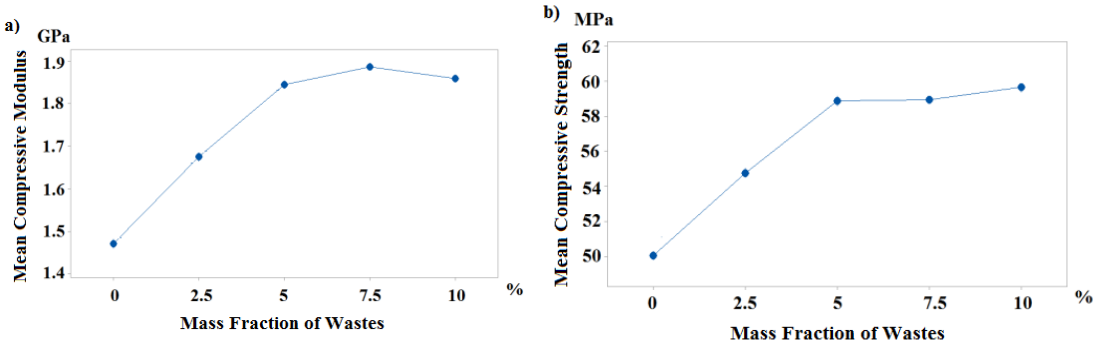

Figure 5 - Main effect plot for mean compressive (a) modulus and (b) strength.

The flexural strength ranged from 30.11 to 36.94 MPa (Figure 6a). According to Tukey's test, the effect of 7.5 and 10 wt% waste inclusions was similar, with an increase of approximately 23% relative to the reference. The impact resistance varied from 6.53 to 15.64 kJ /m^2 (Figure 6b). All levels of particle inclusions led to a dramatic reduction of the impact resistance (approx. 55%). This behaviour may be attributed to the increased stiffness of the reinforced composites, which makes the material more brittle and consequently reduces the impact resistance.

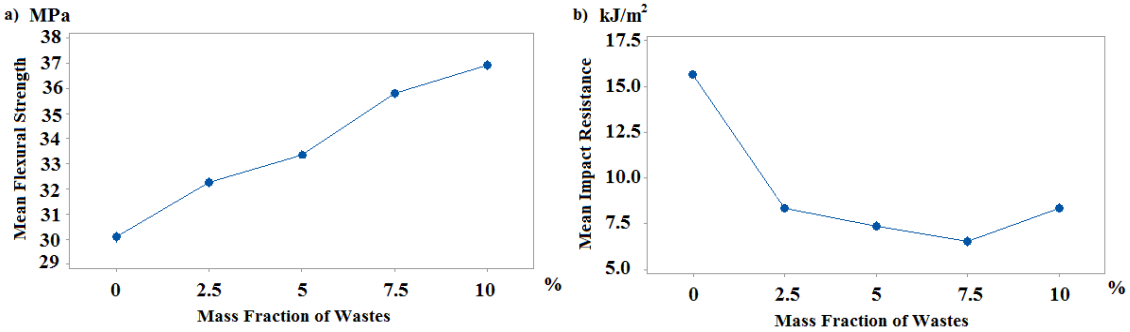

Figure 6 – Main effect plot for (a) mean flexural strength and (b) impact resistance.

The scanning electron microscopic analysis of the carbon particle waste (Figure 7) also reveals spherical particles among carbon microfibre residues. Such particles are metal oxides used as pigments during the fabrication of fins. Figure 8a presents the fractured surface of a specimen after the tensile test with 10 wt% waste inclusions, magnified 100 times.

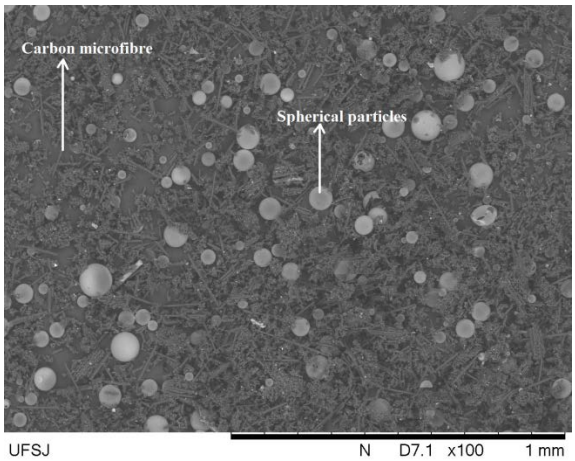

Figure 7 – BSE images of the carbon wastes obtained from the cutting process.

Figure 8b shows the same region with 500-fold magnification, where it is possible to observe the crack propagation along a spherical particle. During crack propagation, such rigid particles may function as barriers along the interface due to their high strength, inhibiting crack growth with subsequent enhancement of mechanical properties [9,15,16]. Thomas *et al.* in fact reported that the inclusion of these wastes generally improved the stiffness and strength of the epoxy polymer composites, including under impact loadings [6]. However, a substantial reduction in impact resistance was observed in this study, as discussed above. According to Dassios [16] fibre pull-out is the most important mechanism of impact energy dissipation in fibre-reinforced composites. It is worth noting that no evidence of fibre pull-out was observed here. However, such mechanism is present in the fractographic analysis presented by Thomas *et al.* [6] and may therefore explain the increase in impact resistance.

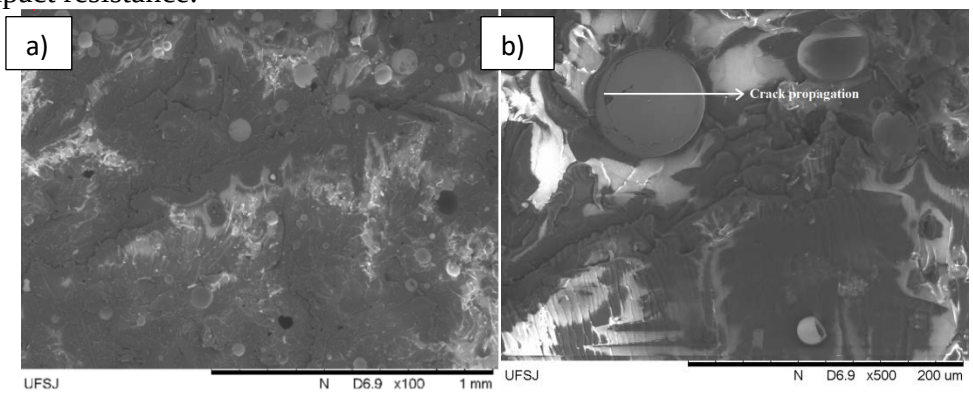

Figure 8 - BSE image: a) Fractured region of tensile test specimen with 10% waste incorporation, magnified 100 times. b) Tensile test fracture surface of a specimen with 10% of waste, magnified 500 times.

4. CONCLUSIONS

The incorporation of carbon powder waste into epoxy polymer promotes an increase in tensile modulus and strength, which increases with the waste mass fraction within the range considered. Results indicate an increase in tensile modulus (strength) up to 36.6% (30%) for 10 wt% (5 wt%) waste inclusions. Similar results were observed for compressive modulus and strength and for flexural strength. The compressive modulus (strength) increased up to 28.6% (19%) for 7.5 wt%

or more (5 wt%) of waste inclusions. The flexural strength increased up to 22% for 7.5 wt% or more of waste inclusions. In contrast, the increased stiffness renders the material more brittle and therefore dramatically reduces the impact strength in 55% for all waste mass fraction levels considered. Carbon fibre wastes derived from the cutting process of laminated composites can therefore be employed as epoxy polymeric matrix reinforcement so as to promote significant enhancements of stiffness and strength. In addition, this low-cost recycling process prevents improper waste disposal with environmental and economic benefits.

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