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).

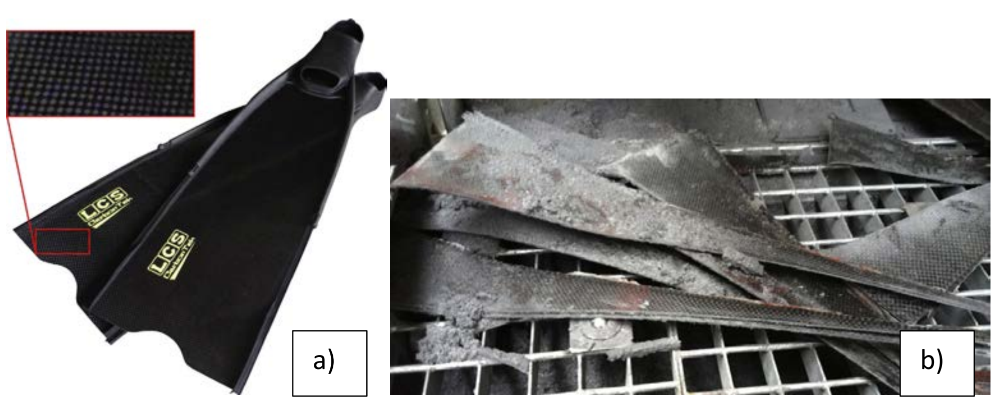

Figure 1 - a) Fins and b) Remains from the cutting process
Source: Thomas *et al*. (2014) [6].

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].

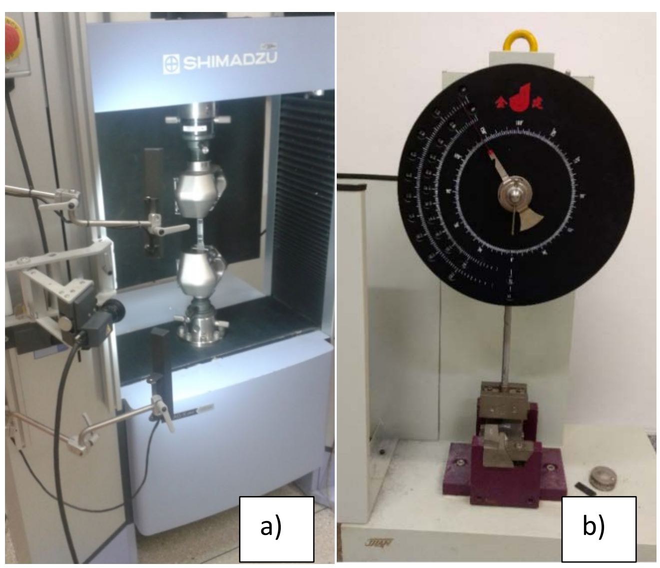

Figure 3 - a) SHIMADZU ® AG-X Plus Universal Testing Machine b) XJJ series impact testing machine

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).

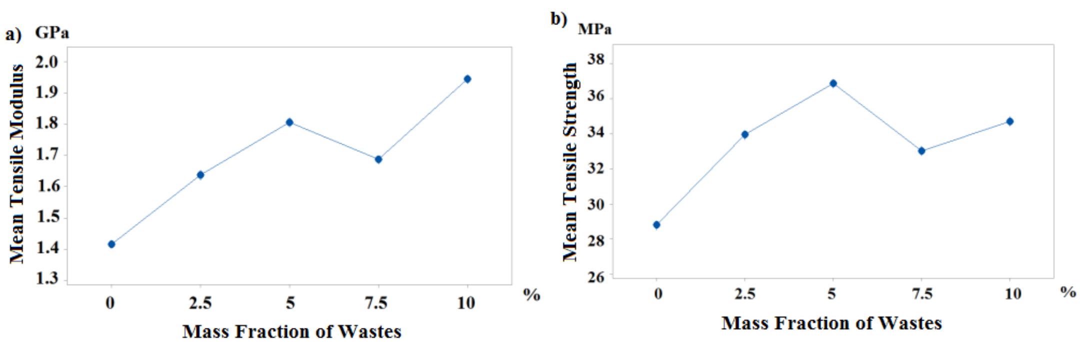

Figure 4 - Main effect plot for mean tensile (a) modulus and (b) strength.

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.

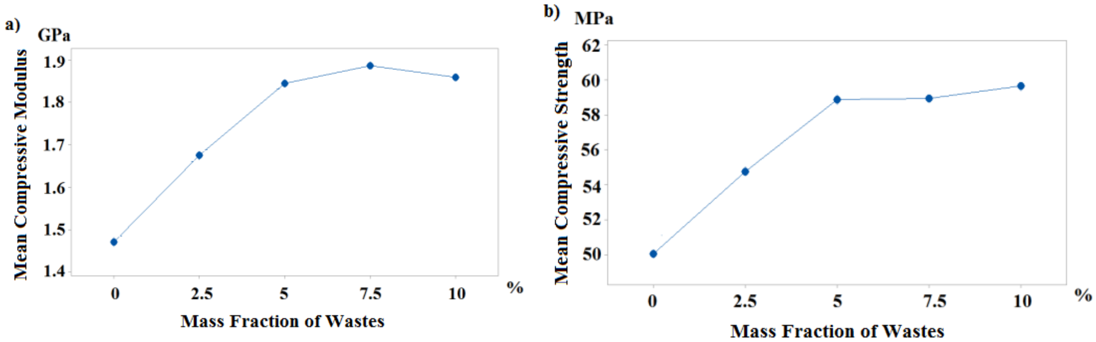

Figure 5 - Main effect plot for mean compressive (a) modulus and (b) strength.

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.

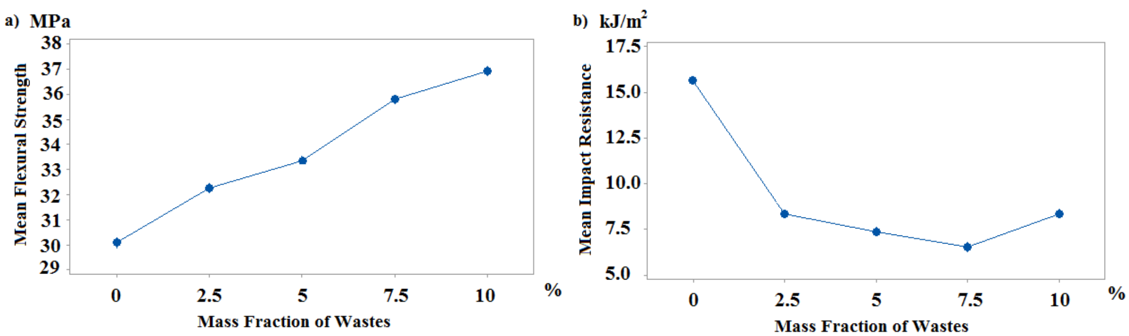

Figure 6 – Main effect plot for (a) mean flexural strength and (b) impact resistance.

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.

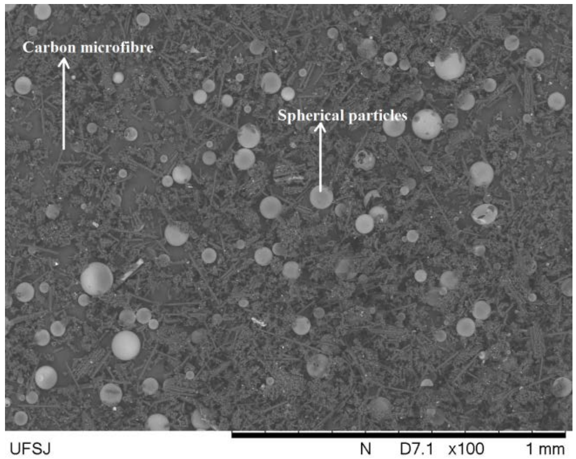

Figure 7 – BSE images of the carbon wastes obtained from the cutting process.

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.

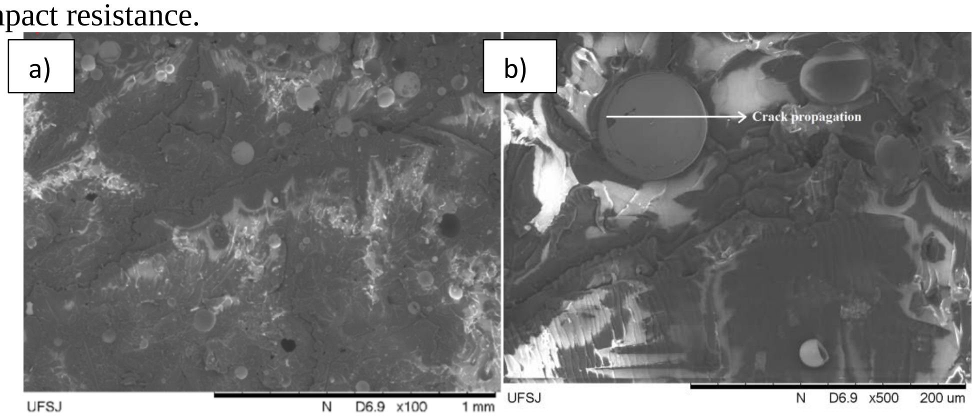

Figure 8 - BSE image: a) Fractured region of tensile test specimen with 10% waste incorporation, magnified 100 times. b) Tensile test fracture surface of a specimen with 10% of waste, magnified 500 times.

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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