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The effect of Portland cement inclusions in hybrid glass fibre reinforced composites based on a full factorial design

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ABSTRACT

The apparent density, flexural modulus and strength of hybrid laminated composites were investigated through a full-factorial Design of Experiment (DoE) approach. Laminates were manufactured by hand lay-up using nine layers of glass fibre cross-ply fabric with an epoxy matrix phase reinforced with Portland cement microparticles. A first experiment investigated the effect of the inclusion site (particles in upper four layers, lower four layers, all layers or none), curing time (7 and 28 days) and compaction method (vacuum or uniaxial pressure). The fibre–matrix volume fraction and the particle mass fraction were fixed at 48.6/51.4% and 10% respectively. A second experiment investigated two distinct fibre–matrix volume fractions (48.6/51.4 and 29.6/70.4%) and five particle mass fractions (0, 2.5, 5.0, 7.5 and 10 wt%). Particle inclusions were restricted to the upper four layers, with 28 days of curing time and uniaxial compaction. The results were analysed via Analysis of Variance (ANOVA). A significant increase in flexural modulus and strength was observed at 28 days of curing time. Enhanced mechanical properties were obtained for laminates with particle inclusions only in the upper half of the structure, manufactured with 48.6/51.4% fibre-matrix volume fraction and uniaxial pressure. Higher flexural strength was achieved for composites manufactured with 51/49% fibre–matrix volume fraction and 2.5% of particle mass fraction. These fibrous-particulate hybrid composite laminates can be considered for future secondary structural parts in lightweight engineering applications.

1. Introduction

Composite materials have been considered as substitutes for metallic materials, owing to their conformability and high specific mechanical properties. Composites have been used in a variety of applications in aerospace, automotive and construction industries owing to their low density and custom-engineered mechanical, thermal and acoustic properties [1,2]. Epoxy polymer is one of the most used thermosetting materials owing to its easy processing and fabrication, simple tooling and excellent adhesive and optically transparent properties [3]. The highly cross-linked polymeric structure of epoxy yields high elastic modulus and strength under tensile and compressive loads, but the tensile modulus and strength may be further increased using fibres.

Amongst all the applications of fibre-reinforced composites, laminated composites are the most common and in which fibres provide *in-plane* reinforcement. No reinforcement, however, is provided by fibres in the *transverse* direction and the resistance to delamination resides on the fracture toughness of the matrix itself. An effective technique to improve interlaminar fracture toughness of laminates is to increase the epoxy matrix toughness by adding micro - or nanosized fillers, such as soft rubber particles or rigid alumina and silica particles [4,5].

Particle inclusions can act as barriers against crack propagation and also enhance compressive modulus and strength of the matrix [6]. Particle size and weight fraction for matrix reinforcement are therefore selected to provide higher specific properties for the composite material [2]. Garg and Mai [7] extensively reviewed the toughening mechanisms of micro fillers in epoxy polymers. The toughening effect of silica nanoparticles on bulk epoxy has been already investigated in previous studies [8,9].

Particles may also increase, under proper conditions, the flexural modulus and strength. Such effect has been, however, much less investigated. Cao and Cameron [10] have reported an increase in flexural

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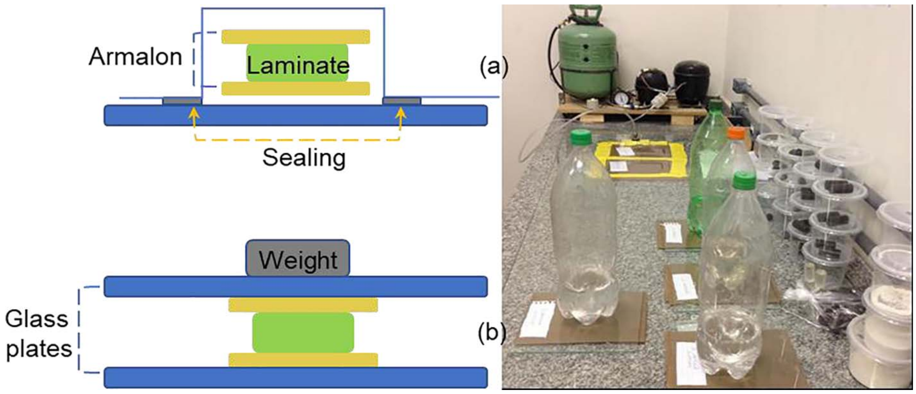

Fig. 1. Illustrations and experimental setup for the compaction methods: (a) vacuum and (b) uniaxial pressure.

Table 1
Experimental conditions studied in experiment A.

Experimental Conditions	Curing time (days)	Compaction method	Particle inclusion site
A1	7	Uniaxial	None
A2	7	Uniaxial	Upper half
A3	7	Uniaxial	Lower half
A4	7	Uniaxial	All layers
A5	7	Vacuum	None
A6	7	Vacuum	Upper half
A7	7	Vacuum	Lower half
A8	7	Vacuum	All layers
A9	28	Uniaxial	None
A10	28	Uniaxial	Upper half
A11	28	Uniaxial	Lower half
A12	28	Uniaxial	All layers
A13	28	Vacuum	None
A14	28	Vacuum	Upper half
A15	28	Vacuum	Lower half
A16	28	Vacuum	All layers

Table 2
Experimental conditions studied in experiment B.

Experimental Conditions	Fibre-matrix volume fraction (%)	Particle mass fraction (%)
B1	48.6/51.4	0.0
B2	48.6/51.4	2.5
B3	48.6/51.4	5.0
B4	48.6/51.4	7.5
B5	48.6/51.4	10.0
B6	29.6/70.4	0.0
B7	29.6/70.4	2.5
B8	29.6/70.4	5.0
B9	29.6/70.4	7.5
B10	29.6/70.4	10.0

modulus (approx. 10%) and strength (approx. 30%) of unidirectional glass fibre reinforced epoxy composites manufactured with silica microparticle-coated fibres relatively to uncoated fibres. Such improvement was associated to fibre-matrix interlocking effects.

Jeyakumar et al. [11] have also reported that nanoclay efficiently improved the flexural modulus and strength of glass fibre epoxy composites up to 5 wt% of nanoclay mass fraction, with a slight decrease in the flexural properties for higher particle mass fractions.

Detomi et al. [12] have investigated the effects of ceramic microparticle inclusions on the apparent density, flexural modulus and strength of glass fibre reinforced epoxy polymer composites under three-point flexural test. Silica or silicon carbide microparticles were added on the upper half or in the whole structure of five-layered laminates resulting in an increase of 112% in specific flexural strength when silica microparticles were incorporated at a 10 wt% level on the upper half of the laminate, relatively to non-particulate glass fibre laminates. The specific flexural modulus was however 26% lower.

Santos et al. [13] have reported that carbon fibre composites with silica nanoparticles presented higher flexural modulus (47%) and strength (15%) relatively to non-particulate composites, mainly when a 2 wt% particle mass fraction was used. These authors observed a good correlation between tensile and flexural modulus, which corroborates the observations that the increase in flexural modulus can be attributed to the stiffness increase of the reinforced matrix under tensile and compressive loadings [14,15]. Considering that particulate composites exhibit, in general, higher *compressive* stiffness and strength while fibrous materials exhibit higher *tensile* stiffness and strength, Torres et al. [16] have investigated the effects of silica and cement particle inclusions on hybrid glass fibre reinforced epoxy composites. Particle inclusions at the upper (under compression) beam side provided a significant increase in flexural modulus (19.60% for silica and 28.70% for cement particles). The highest flexural strength was achieved for composites fabricated with 5 wt% of cement particles. Cement particles were considered by these authors owing to experiment results reported in the literature concerning the hydration of cement particles in the presence of epoxy polymers.

A preliminary work, carried out in 1977 [17], on the use of Portland cement and other inorganic materials as fillers for different polymers, such as epoxy and polyester, considered the probabilities for chemical interaction between Portland cement and epoxides to be very low. The authors remarked that, up to that time, no evidence had been found for a real chemical interaction between epoxy polymer and Portland cement. However, Panzera et al. [18], based on infrared spectroscopy analysis, have reported compelling evidence of hydration of cement particles embedded in epoxy polymer, implying the formation of epoxy-portlandite hydrogen bonds. Soles and Yee [19] have in fact observed that water molecules penetrate the structure of the epoxy resin through a network of nanopores of 5.0 to 6.1 Å in diameter, which occupies 3 to 7% of the total volume of the cured polymeric structure. Water molecules can easily penetrate the structure, since the diameter of water molecules is much smaller (around 3.0 Å).

In this work, the potential enhancement of the flexural properties of hybrid glass fibre epoxy composites is investigated using cross-ply glass fibre fabrics in an effort to take advantage from the interlocking effect promoted by particles at the interlaminar region.

2. Material and methods

Hybrid composite laminates were manufactured by hand lay-up using nine layers of glass fibre cross-ply fabrics (Owens-Corning, 200 g/m^2) and Portland cement microparticles (diameter $<$ 44 μm, supplied by Lafarge-Holcim, Brazil). The matrix phase consisted of the epoxy polymer RenLam-M with the HY 951 catalyser (both supplied by Huntsman) at the proportion 5:1 (according to the manufacturer), mixed with cement microparticles according to the experimental