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Effect of test conditions on the bending strength of a geopolymer-reinforced composite

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Abstract This paper presents the results obtained for the effects of the loading rate and of the testing temperature on the mechanical properties, particularly on the stiffness and on the ultimate tensile strength, of a geopolymer reinforced with glass or carbon fibres. HIGH-SILICA geopolymer powder from CLUZ- CYECH and two reinforcement fibres (glass fibres—type AR and carbon fibre - HTS 5631) were used. The displacement rate is varied from 0.02 until to 2 mm/s and the testing temperature is increased from the room temperature until the temperature of 300 °C. For the case of geopolymers reinforced with carbon fibres and glass fibres, the increase of the displacement rate from 0.002 to 2 mm/s led to an improvement on the ultimate flexure strength of about 33 and 31%, respectively. The same dependency was observed for the stiffness, with variations of loading rate of 39 and 53%, for carbon fibres and glass fibres, respectively. Increasing the room temperature until the temperature of 300 °C decreases significantly both the ultimate strength and the flexure stiffness for both reinforcements. However, a major drop on both the stiffness and the strength occurred up to 150 °C.

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Introduction

Geopolymer materials were introduced by Davidovits in 1978 [1] and are generally formed by reaction of an aluminosilicate powder with an alkaline silicate solution at rough ambient conditions [1, 2].

In recent years, the geopolymers have emerged as promising materials in various fields as a result of the better properties relatively to ceramics and to cement-based materials. In fact, they present good mechanical properties, inflammability at high temperatures, chemical resistance, long term durability and low permeability [2–4]. On the other hand, the geopolymers require relatively low temperature for their production, and consequently less CO_2 emissions, which result in ecological advantages [4]. These properties make these materials strong candidates to substitute Portland cement in the fields of civil, bridge, pavements, hydraulic, underground and military engineering [5].

Surface deterioration of concrete is becoming one of the major problems for durability of concrete structures. The most efficient way to reduce this deterioration is to prevent the liquid ingress into concrete, thus preventing the ingress of chemicals such as chloride from salts. Works developed by Nazier [6] showed that the inorganic polymer coating is a viable alternative to organic polymer and polymer-modified cement coatings. The primary difference is the compatibility of the new coating with common construction materials such as concrete, concrete bricks, clay bricks, steel and timber. Geopolymers possess good fire resistance and, therefore, concretes produced using geopolymers may possess superior fire resistance compared to conventional concretes produced with ordinary Portland cement, generally considered to provide adequate fire resistance for most applications. However, ordinary Portland cement concrete degenerates at elevated temperatures due to chemical and physical changes [7].

The building sector has signaled a need for reinforcement of geopolymers, specifically through the design and fabrication of new fibre composites and structures. This will permit the increase of their strength, toughness and elastic modulus. The matrix, for example, presents an important contribution for the global properties of the composite. According to the literature [8, 9] an increase of the concentration of alkali activator improves the compressive strength. It has been shown by Van Jaarsveld et al. [10] that geopolymerisation can make a profitable contribution towards recycling and utilisation of previously unused waste materials. This technology is, however, still fairly unknown and predictably viewed with skepticism by most workers in the field of traditional waste processing techniques. Zhang et al. [4], on the other hand, showed that the addition of polyacrylic acid (PAA) and sodium polyacrylate (PAANa) can improve the compressive strength around 29% and crossbending strength around 64.9%.

This paper presents the results of a current study concerned with the effect of test conditions on the bending strength of a geopolymer reinforced composite. The effect of displacement rate on flexural strength and stiffness was studied for the values of 0.02, 0.2 and 2 mm/s at room temperature. The damage mechanisms were studied. Finally temperature's effect was also studied in order to obtain its influence on stiffness and on strength of these composites for the displacement rate of 2 mm/s.

Experimental procedure

Composites manufacture consisted of several steps. First, the geopolymer matrix was produced according the manufacturer recommendations. HIGH-SILICA geopolymer powder, from CLUZ-CYECH, was used with the chemical bond. The maximum diameter of geopolymer's particles is 5 μm. This powder, a dehydroxylated kaolinite, amorphous Al_2O_3 and fine amorphous silica, was associated with a Na_2O reactant and distilled water with the molar rate of 2/1/0.24/3.36 ($SiO_2/Al_2O_3/Na_2O$/distilled water). A mechanical mixture was done during 15 min, at room temperature, in order to obtain a good mixture of the components. At a second step the unidirectional reinforced composites were produced by pultrusion technique as Fig. 1 shows. A filament rolling of tube composite was obtained. Cutting these filaments with the desired length it spreads as a plane layer with the fibres nearly aligned at one direction as shown in Fig. 2. Afterwards six layers of geopolymer composite were put inside a mould with the following dimensions $60 \times 5 \times 5$ mm^3 (Fig. 4a). After, the mould was put in a PE vacuum bags, into an autoclave, at 120 °C and a pressure of 1.2 MPa, during 2 h (as is shown schematically in Fig. 3), for hardening of the specimens. Finally the composite was hardened during 12 h inside of a drying camera at room temperature and having air circulation.

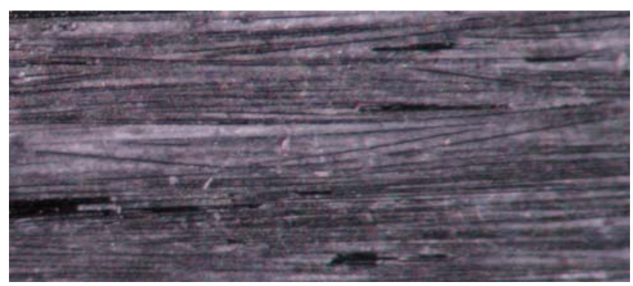

Fig. 2 Fibre distribution along matrix

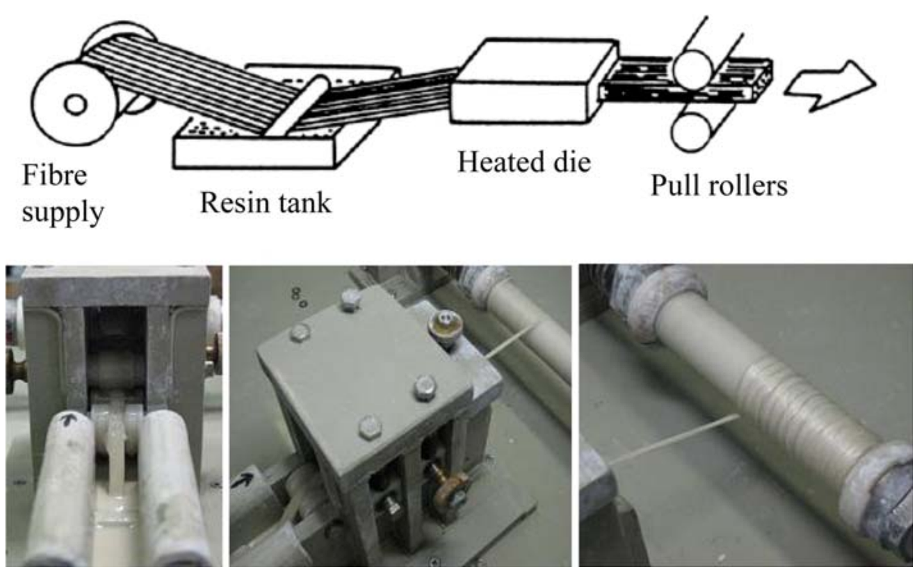

Fig. 1 Wetting mechanism for fibres impregnation and pultrusion process

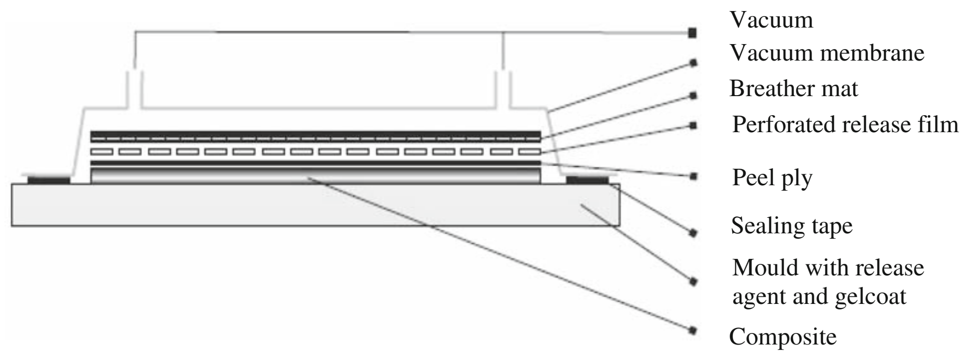

Fig. 3 Schematic view of vacuum bag

Table 1 Properties of the fibres used in the composites

Single fibre	Diameter (μm)	Density (Kg/m^3)	E (GPa)	σ_{UTS} (GPa)	ε_f (%)
Glass (Type AR)	24	2700	70–80	2.5–3.5	4–5
Carbon (Type HTS 5631)	7	1800	290–310	5–6	1.5–2

AR—Surface treatment of the fibre (*AR* alkali resistant) from Saint-Gobain

Two types of fibres were used: glass fibres (type AR—alkali resistant) and carbon fibres (HTS 5631). The properties of these fibres, according with the manufacturer, are indicated in Table 1. The samples were manufactured with 70% glass fibre or 55% carbon fibre, in weight percentage.

The tests consisted of three point bending with 48 mm span, using an Instron model 1341 servohydraulic machine at a displacement rates of 0.02, 0.2 and 2 mm/s, according to the recommendations of ASTM D 2344 Standard [11]. To study the effect of the temperature this machine was equipped with a furnace SFL, model SF 1089. Four specimens, with the geometry shown in Fig. 4b), were tested for each condition.

Figure 4b shows a schematic view of the three point bending apparatus used in the tests. The nominal bending stress (σ) was calculated using:

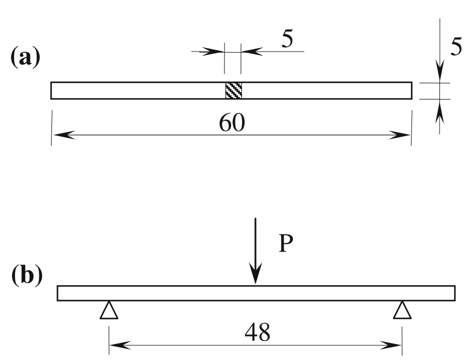

Fig. 4 **a** Specimens geometry (dimensions in mm); **b** schematic view of the three point bending apparatus

$$\sigma = \frac{3\,P\,L}{2\,b\,h^2} \tag{1}$$

being P the load, L the span length, b the width and h the thickness of the specimen.

Bending stiffness modulus was calculated by the linear elastic bending beams theory relationship:

$$E = \frac{\Delta P \cdot L^3}{48 \Delta u \cdot I} \tag{2}$$

where I is the inertia moment of the transverse section and ΔP and Δu are, respectively, the load range and flexural displacement range at middle span for an interval in the linear region of load versus displacement plot.

Results

The flexural properties were obtained from 3 PB static tests. Typical load–displacement curves for geopolymers reinforced carbon fibre are plotted in Fig. 5, showing the effect of displacement rate. These curves are very similar to others obtained for geopolymers reinforced with glass fibres and are practically linear until brittle failure. Both materials showed a nearly fragile behaviour and a sudden drop of the stress after peak stress was reached. According to the literature [12–14] the mechanism of damage consist of fibres fracture, in compression, with quite small delaminations around the broken fibres (Fig. 6). The zigzag aspect of the load–displacement curve results from fibres breakage.

Studies developed by Reis et al. [14] showed that the high compressive stress concentration in the pin load contact region associated with the low compressive strength of the fibres promotes compressive breakage of the longitudinal fibres in this region. Several studies revealed nonlinear-elastic behaviour, in both tension and compression ranges, of the fibres as a result of their specific microstructure [15–20]. However, as the curves show, this material presents linear-elastic behaviour which is result of the matrix behaviour.

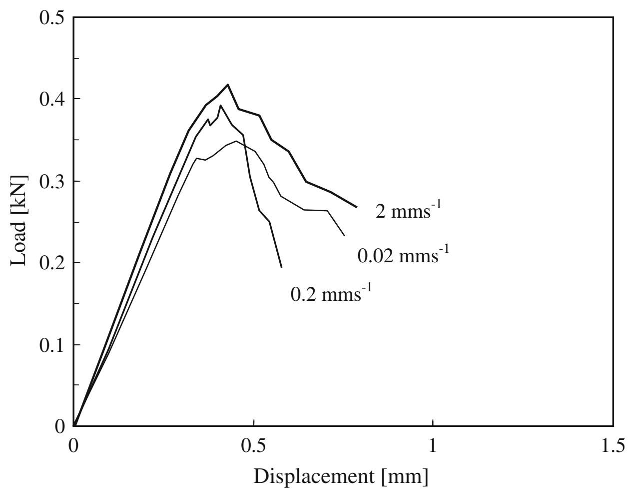

Fig. 5 Load–displacement curves for geopolymers reinforced carbon fibres

Fig. 6 Typical damage observed during the flexural tests

Table 2 presents the results obtained in 3 PB static tests, of flexural strength and stiffness versus displacement rate. The average values and the standard deviation are indicated in the table. Ultimate stresses, σ_{US}, were obtained using Eq. 1 with the peak load values. Once the load displacement curves are nearly linear, the stiffness modulus was obtained by linear regression of the stress–strain curves considering a loading segment ranging from zero to a defined strain value. It is possible to observe that the average value of the ultimate stress is higher for geopolymers reinforced with carbon fibres by about 9.8% in comparison with geopolymer reinforced with glass fibres, for a displacement rate of 2 mm/s. These results are expected, for example using the rule of mixtures, since the carbon fibres have a tensile stiffness much higher than the glass fibres. For both materials the increasing of the displacement rate increases the flexural strength. The difference observed between displacement rates of 0.002 and 2 mm/s is around 33% for the geopolymers reinforced with carbon fibres while for geopolymers reinforced with glass fibres this difference is of 31%. These results are consistent with reported in literature, for other composite materials, that are sensitive to the strain rate due to the viscoelastic behaviour of the matrix [20, 21]. The same tendency is observed for the stiffness. For geopolymers reinforced with carbon fibres and for geopolymers reinforced with glass fibres it is possible to observe that the increase of the displacement rate promotes increases on the stiffness values, reaching values around 46.7 and 34.3 GPa for displacement rate of 2 mm/s, respectively. This difference of 26.6% is much higher than observed for the flexural strength (9.8%). When the effect of displacement rate is analysed for these materials, in terms of stiffness, it is possible to observe that increasing the displacement rate from 0.002 to 2 mm/s promotes the increase of the stiffness around 39% for geopolymers reinforced with carbon fibres while for geopolymers reinforced with glass fibres the stiffness increases about 53%. For flexural strength the increase of the displacement rate promotes an increase of the strength in a very similar way, around 31–33%. In terms of the stiffness this effect is major for the geopolymers reinforced glass fibres with increases of 53%. This phenomenon can be explained by the different stiffness that occurs between reinforced fibres.

Figure 7 shows the effect of the temperature on the flexural strength. The marks represent the average values and the bands indicate the maximum and minimum values obtained from the tests. It is possible to observe that ultimate tensile strength decreases with increasing temperature. At 300 °C, for example, the flexural strength for geopolymers reinforced with carbon fibres is 213.5 MPa and for geopolymers reinforced with glass fibres is about 135.9 MPa. These values are, respectively, 24.6 and 46.7% lower than those observed at room temperature. However, when compared with the flexural strength obtained at 150 °C these differences are only 6.4% for geopolymers reinforced with carbon fibres and 13.2% for geopolymers reinforced with glass fibres. Therefore, the major drop of the ultimate tensile stress occurs between the room temperature and 150 °C.

Figure 8 presents the results observed for the stiffness and once again the marks represent the average values and the bands the maximum and minimum values of stiffness. It is observed the same tendency presented in Fig. 7, in which the stiffness decreases with increasing temperature. At 300 °C the stiffness is about 25.5 GPa for geopolymers reinforced with carbon fibres and about 10.4 GPa for geopolymers reinforced with glass fibres. The major decreasing of the stiffness, for both materials, occurs once again for the gap between room temperature and 150 °C.

Table 2 Influence of the loading rate on flexural strength at room temperature

Composites	Displacement rate (mm s^{-1})	σ_{US} (MPa)	Average σ_{US} (MPa)	Standard deviation (MPa)	E (GPa)	Average E (GPa)	Standard deviation (GPa)
Geopolymers + Carbon fibres	0.02	192.2	190.1	20.4	34.7	28.3	5.8
		190.6			21.8		
		164.0			31.4		
		213.7			25.4		
	0.2	240.4	239.4	23.9	21.3	33.6	8.6
		266.7			39.2		
		208.4			33.9		
		241.9			39.8		
	2	278.6	283.0	9.5	45.9	46.7	8.5
		288.8			58.9		
		292.8			42.3		
		272.0			39.7		
Geopolymers + Glass fibres	0.02	169.3	175.9	6.6	13.6	16.1	2.4
		171.6			19.2		
		183.2			16.4		
		179.6			15.1		
	0.2	216.3	209.7	15.3	18.7	23.9	5.3
		221.3			24.9		
		187.1			30.9		
		213.9			21.2		
	2	252.2	255.2	16.0	28.6	34.3	5.9
		277.5			31.3		
		251.8			42.3		
		239.3			34.9		

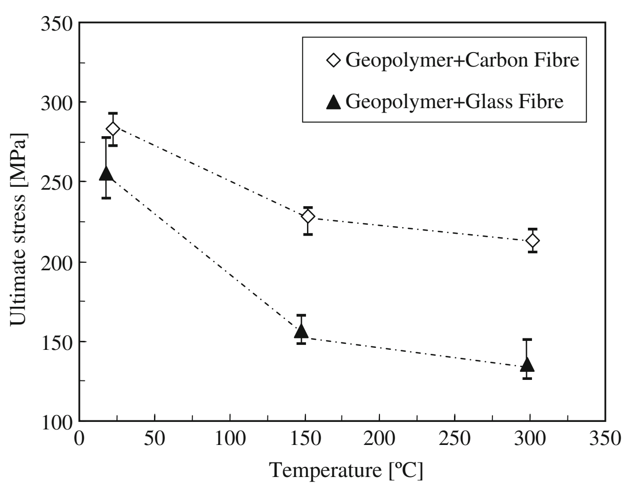

Fig. 7 Influence of the temperature on flexural strength

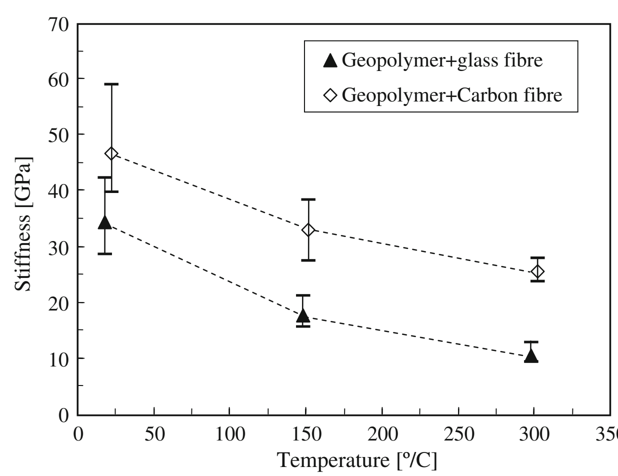

Fig. 8 Influence of the temperature on stiffness

These differences present values around 29.8% for geopolymers reinforced with carbon fibres and 48.7% for geopolymers reinforced with glass fibres. When the values obtained at room temperature and at the temperature of 300 °C are compared, the difference of values is of 45.4 and of 69.7%, respectively. These values result from the different behaviour of the fibres at higher temperatures. On the other hand, according to Kong and Sanjayan [22], this is attributed to the increase in a combination of polymerization reaction and sintering at elevated temperatures. The studies developed by these authors show that the strength of the fly ash-based geopolymer declined with the inclusion of aggregates, i.e. geopolymer/aggregate composites. Evidence presented in this paper suggests that the decline in strength was caused by the differential on the thermal expansion coefficient between the geopolymer and aggregates. However, TGA results obtained by Kong et al. [23] showed that metakaolin-based geopolymers had significantly higher moisture loss than the fly ash-based geopolymers. The smaller amount of moisture having to escape from fly ash matrix may be the reason why there is less damage to the matrix. By contrast, the plate-like structures of residual metakaolin particles do not provide similar moisture escape routes at elevated temperatures causing damage to the matrix. According with these conclusions the metakaolin geopolymer studied in this paper shows significant bending strength loss when exposed to temperatures up to 300 °C.

Conclusions

This paper reports the results of the effects of loading rate and testing temperature on the mechanical properties, particularly the stiffness and ultimate strength, of a geopolymer reinforced with glass or carbon fibres. For both reinforcements ultimate flexure strength improves about 30% when the displacement rate increases from 0.002 to 2 mm/s. The stiffness follows the same trend, but in this case the effect of loading rate is significantly more pronounced.

An important decreasing on ultimate strength and flexure stiffness with the increasing of the temperature from room temperature to 300 °C was obtained for both reinforcements. However, the major drop on both the stiffness and the strength occurs up to 150 °C.

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