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Epoxy/glass fibres composites for civil applications: Comparison between thermal and microwave crosslinking routes

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ABSTRACT

Fiber-reinforced polymers (FRPs) have gained a growing interest for civil applications mainly for their lightness, corrosion resistance and high specific mechanical properties especially in terms of stiffness and strength. In this work, epoxy/glass fibres composite formulations, prepared by thermal and microwave assisted curing routes, are systematically characterized by thermal and mechanical tests. In particular the thermal curing is performed at room temperature and at 100 °C while the microwave conditions are set at 1500 W for 10 min. Calorimetric measurements demonstrate that these processing conditions allow to prepare samples with comparable crosslinking degree. The time and cost-saving microwave approach gives rise to cured FRPs with higher flexural stiffness but, expectedly, characterized by a network with a structure more inhomogeneous with respect to thermal crosslinked ones.

Pull-out strength determination of all investigated epoxy composite systems joined to three different supports: a conventional concrete (CC), an artificial aggregate concrete (AAC) and a geopolymer (G), are performed to highlight their potential use for civil engineering applications. The experimental findings further support the use of microwave cured FRP samples especially with respect to the CC support.

1. Introduction

The development of new materials with improved performances and functions has become a major driver of innovation in recent years.

In this frame, fiber-reinforced polymers (FRP) have gained substantial interest over the last years, mainly due to their very high strength-to-weight and stiffness-to-weight ratios. These peculiarities promote the use and application of composite materials in a lot of fields. However, high operational costs combined with current complex manufacturing techniques often still restrict potentials of such materials. Thus, considerable interest has been also devoted to the development of new processing routes among which the microwave assisted ones are worth to be mentioned.

As well established microwave processing offers several advantages over the conventional thermal processing methods for curing thermosetting polymer based materials. These benefits include rapid, selective and volumetric heating, energy savings, reduced processing time and improved processing control [1–19]. At present, many papers are available about the comparison between thermal cured and microwave cured samples but reported results are often contradictory mainly in terms of kinetics and mechanisms of microwave crosslinking reactions as well as of mechanical properties of products [20–25]. In general, it is established that the “microwave effect”, firstly investigated by Stuerga et al. [26,27], alters the kinetics and the mechanisms of crosslinking reactions but, as already said, an exhaustive interpretation of these phenomena is still not clear. The inability to predict a reliable correlation among processing parameters such to ensure similar levels of crosslinking for the same polymer formulation, has prompt the authors to consider a preliminary parametric study to identify the conditions of a conventional isothermal cure comparable with microwave assisted processes with varying radiation powers and cycle times and, in other words, to assess the influence of microwave processing parameters on the degree of cure of the reference resin.

Regarding the potential applications of FRP, the reinforcement of new eco-sustainable building materials is an important issue to be explored. Specifically, concrete offers a wide range of possibilities to

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use recycled materials with environmental benefits. Also, a wide range of industrial wastes or industrial by-products could be used, either pre- or post-treatment, to become an artificial aggregate or an alternative binder [28–36]. These options reduce the need for raw materials, divert wastes from landfills and decrease the embodied energy in concrete. As a consequence, the interactions between FRP and this new class of concrete have to be evaluated.

Furthermore, in the last decades, a new class of low-energy building materials, characterized by highly desirable chemical and mechanical properties, is emerging and its use are widely studied in several different sectors. These materials are "alkali-activated cements" or "chemically-bonded ceramics" also named "geopolymers" and can be obtained starting from raw materials with a low (or zero) CaO content [37–39]. Also in this case, the compatibility with traditionally or low-energy cured FRP is worth to be studied.

Main aim of this work is to compare mechanical properties of FRPs cured under different conditions: 7 days at room temperature (T_{amb}), 1 h in an oven at 100 °C and 10 min in a microwave oven at a power of 1500 W. Moreover, the potential application of obtained composite materials on different kinds of supports (conventional concrete, artificial aggregate concrete and geopolymer) was also assessed mainly in terms of pull-out strength.

2. Material and methods

2.1. Materials

The formulation considered in this research (manufactured by Mapei S.p.A.) involves an epoxy resin as matrix (Mapewrap 31) and a unidirectional fabric based on glass fibers as reinforcement (Mapewrap UNI-AX G). The resin, in turn, consists of two components:

- *Component A:* hexanediol diglycidyl ether;
- *Component B:* polyethylene amine, *m*-xililendiammina, nonylphenol.

Formulations adopted to obtain prismatic shaped specimen supports of dimensions 300 × 300 × 100 mm^3 are shown in Table 1. Specifically artificial aggregates were prepared as described in previous studies [40], while geopolymers were designed improving mixtures already tested [41,42].

The coal fly ash used for the preparation of the supports was supplied by the Italian electric company (ENEL) and comes from the power plant located in Brindisi (Southern Italy). Its characterization by means of traditional chemical analysis gave the following chemical composition: SiO_2, 44.3%; Al_2O_3, 20.2%; Fe_2O_3, 10.5%; K_2O, 8.1%; CaO, 0.5%; Na_2O, 0.3%. The loss on ignition at 1050 °C amounts at 11.35%.

Alkali metal (Na and K) hydroxides and silica (cristobalite) were reagent grade chemicals supplied by Fluka.

Table 1
Mix proportions and main properties of supports.

Materials	Content [kg/m^3]		
	CC	AAC	G
CEM II 32.5 R	290	290	–
Fly ash	20	35	412
Sodium silicate solution ($SiO_2/Na_2O = 2$)	–	–	102
Sodium hydroxide solution	–	–	41
Fine aggregates	663	670	590
Natural coarse aggregates	1157	–	1248
Artificial coarse aggregates	–	812	–
Superplasticizer (Dynamon SX Mapei)	2.5	2.0	3.0
W/c ratio	0.50	0.45	–
Slump (mm)	255	265	260
Compressive strength (MPa)[a]	34.1	31.5	36.2
Tensile strength (MPa)[b]	3.9	3.2	4.1

[a] Average value of three cubic specimens (100 × 100 × 100 mm^3) tested at 28 days water-curing.
[b] Average value of three cylindrical specimens (h 100 mm x d 200 mm) tested at 28 days water-curing.

2.2. Sample preparation

Plates of the investigated materials were obtained with the aid of a mold consisting of two sheets of Teflon, sized 200 × 200 × 10 mm^3 and separated by a distance of 3.2 mm.

All panels, after curing, were cut to obtain specimens for flexural and dynamic mechanical tests.

FRPs, after applying a suitable primer (Mapewrap Primer 1 by Mapei S.p.A.), have been applied to three different supports (structures): conventional concrete (CC), artificial aggregates concrete (AAC) and geopolymer (G); prepared according to UNI EN 1766.

2.3. Characterization techniques

Microwave curing processes were performed with a closed Microglass s.r.l. chamber oven equipped with a two-dimensional row of wave antennas implemented with a system of slotted waveguides.

In light of the achieved results, microwave conditions were set at 600 s and 1500 W and prepared samples were compared with ones cured for 7 days at ambient temperature and ones crosslinked for 1 h at 100 °C.

In each case, the degree of cure was evaluated by differential scanning calorimetric measurements performed using a DSC Mod. Q20 – TA Instruments at a heating rate of 10°/min. Given that the cure of thermosetting resin is accompanied by an increase of the glass transition temperature (Tg) and the decrease of the heat of cure, non-isothermal tests are commonly used to determine both the total heat of cure of completely uncrosslinked resins (ΔH_T) and the residual heat of cure of samples previously subjected to isothermal stages (ΔH_R). Consequently, the degree of cure (α) can be evaluated as follows:

$$\alpha = \left(\frac{\Delta H_T - \Delta H_R}{\Delta H_T}\right) \cdot 100$$

All cured samples were systematically analyzed by dynamic-mechanical tests performed with a Tritec 2000 DMA (Triton Technology Ltd.) apparatus. In more details, specimens were subjected to oscillating three-point bending loads at the frequency of 1 Hz by heating the same from room temperature up to 100 °C at the rate of 4°/min. Results in terms of storage modulus (G′) and loss factor (Tan δ) were monitored as a function of the temperature for all investigated materials, taking the temperature at which the Tan δ signal is centered as the glass transition temperature (T_g) of the analyzed material.

Flexural measurements were conducted according to the ASTM D790-03 using a dynamometer Instron model 5566, equipped with a 1 kN load cell. Rectangular specimens of size 127 × 12.7 × 3.2 mm^3 were tested in a three-point flexural configuration by applying a mobile speed of the crosshead equal to 2.35 mm/min. Results, averaged on at least 5 determinations for each sample, are reported in terms of the maximum flexural strength (σ_f), the maximum deflection (ε_f) and the flexural modulus (E_f).

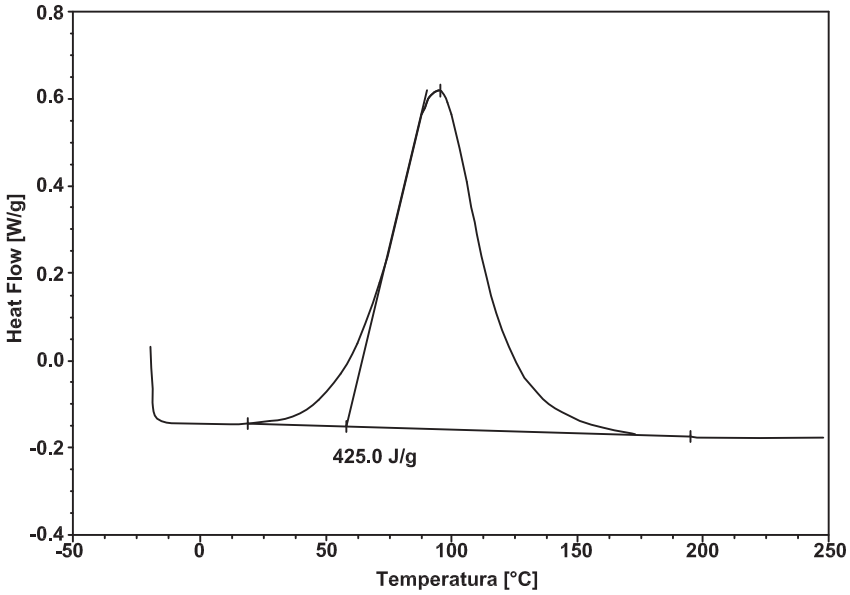

Fig. 1. Thermogram of the resin. Dynamic scanning at 10 °C/min.

Finally, pull-off tests were used to assess the adhesion between the repair materials (FRPs) and 3 concrete substrates. In a concrete patch repair, failures can occur at the substrate concrete, at the bond interface, at the overlay, at the epoxy used to bond the disk to the core, or as a combination of these failures modes. The mode of failure and the pull-off strength provide valuable information about the appropriateness of repair system. The magnitude of the tensile force and the location of the fracture surface give some information of the performance of the repair system (overlay and adhesive). When failure only mobilizes adhesion material, the pull-off test provides a true indication of the bond strength.

According to the UNI-EN 1542 standard [43], a cylindrical disk of 50-mm diameter was joined to the overlay with an epoxy glue. The overlay and concrete substrate were drilled to a depth of 10 mm under the overlay. The tensile load was applied to the steel disk until failure occurred. The pull-off bond strength was calculated by dividing the tensile load at failure (N_{po}) by the area of the test specimen (A_{fs}).

Unreinforced concrete prisms of 300 mm × 300 mm × 100 mm^3 were used as supports. After 28 curing day the casting surface (top surface) was sand-blasted in order to remove grease, dirt and small particles that produce irregular surface.

The adhesion strength was obtained from an average of six pull-off test specimens.

3. Results and discussion

3.1. Thermal analysis

Preliminary calorimetric tests were conducted on a mixture of resin and initiator, with a weight ratio of 4:1, to ascertain the absence of any undesirable volatile substances and to identify the

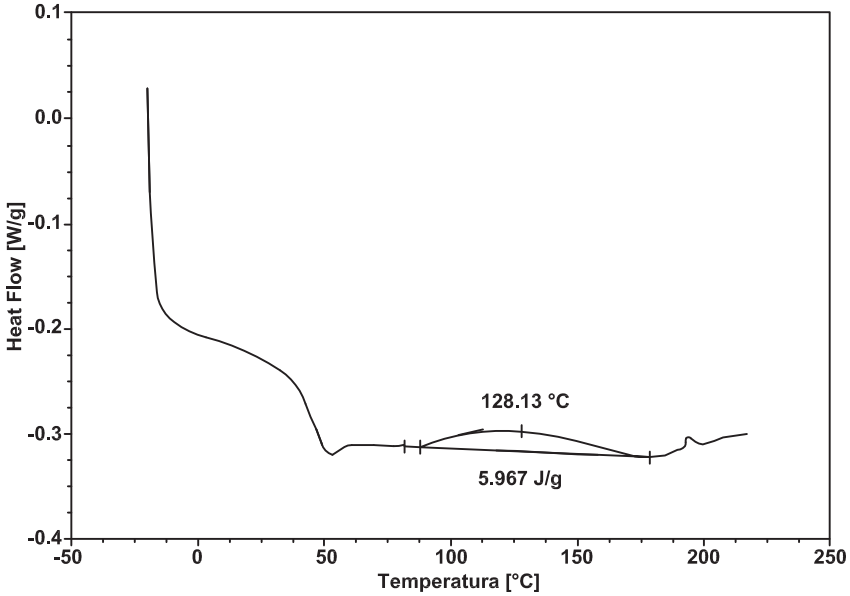

Fig. 2. Residual energy of reticulation of the composite cured at room temperature for 7 days.

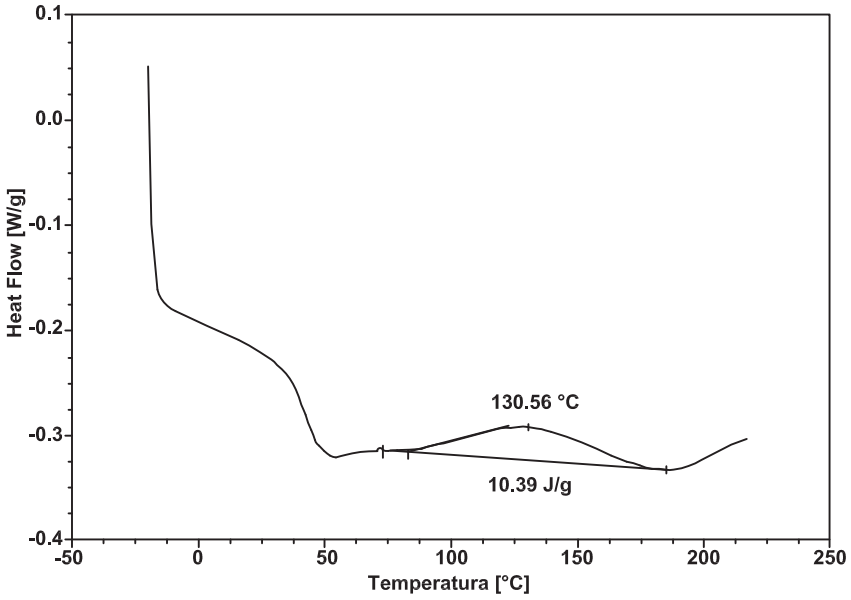

Fig. 3. Residual energy reticulation of the composite cured in an oven at 100 °C for 1 h.

optimal temperature range for its investigation. As clearly indicated by Fig. 1, an endothermic signal is observed on the range of temperatures approximately comprised between 25 °C and 200 °C. The area under this signal, estimated by the TA software and representing the total heat of cure of the reference system, amounts at 425 J/g as highlighted in the same picture.

Non-isothermal measurements provided information about the residual heat of cure of specimens previously cured in isothermal conditions. For example, Fig. 2 shows the thermogram of a sample previously cured at room temperature for 7 days. The variation of the heat flow with the temperature clearly show a first deflection centered approximately at 38 °C followed by an endothermic signal of area about equal to 6 J/g. Attributing the first data, as usual, to the glass transition temperature of the material, the second signal provides the residual heat of cure and consequently, according to the expressions reported within the paragraph 2.2, the crosslinking degree achieved under the applied curing conditions resulted to be approximately equal to 98.6%.

Proceeding similarly for samples subjected to a cure of 1 h in oven at 100 °C and for samples crosslinked under microwave conditions, the thermograms are shown in Figs. 3 and 4 and the achieved degree of crosslinking were estimated to be equal to 98.19% and 94.62%, respectively.

These values, together with the glass transition temperatures, are summarized in Table 2 from which it is clear that the use of microwaves ensures the obtainment of a curing extent comparable with the one achieved by conventional heating routes but with more rapid processes. Moreover, assuming a consumption of

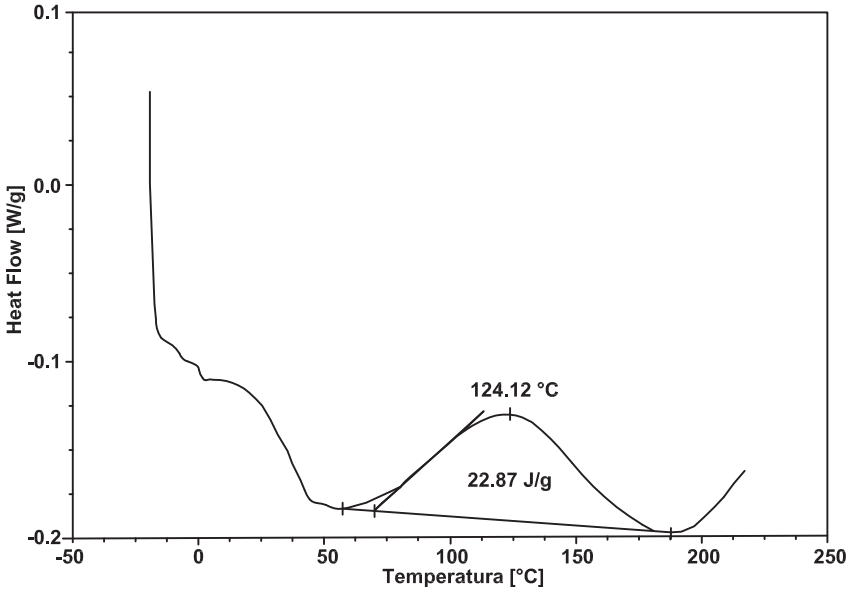

Fig. 4. Residual energy of reticulation of the composite cured in a microwave oven at 1500 W for 10 min.

Table 2
Comparison of time and degree of reticulation.

	T_{amb}	Oven (100 °C)	Microwave (1500 W)
Crosslinking degree	98.60%	98.19%	94.62%
Curing time	7 days	1 h	10 min
T_g	46.87 °C	42.04 °C	34.89 °C

Table 3
Economic saving.

	kW/panel	€/kW	€/panel	€/m^2	Saving
MW (1500 W)	0.25	0.20	0.005	0.33	69%
Oven (100 °C)	0.80	0.20	0.16	1.07	

energy for the oven and for the microwave apparatus equal to 800 W/h and 1500 W/h, respectively, and a cost of 0.20 €/kW, it is easy to appreciate a significant economic saving in favor of the microwave assisted process as evidenced in Table 3.

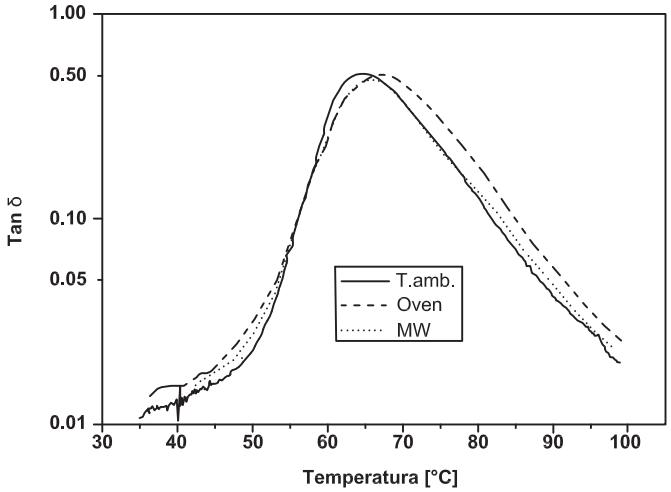

Fig. 6. Tan δ vs. temperature.

3.2. Dynamic mechanical tests

Figs. 5 and 6 compare the trends of storage modulus (G′) and loss factor (tanδ) for the investigated formulation cured in different conditions as a function of the temperature. The use of microwaves moves the storage modulus curve upwards all over the examined temperature range with respect to traces of samples cured by conventional methods. In particular, for these latter it seems that this viscoelastic parameter is not influenced by the temperature of cure.

In each case the storage modulus shows a rapid decrease for temperatures higher than 55 °C, stabilizing again at an almost constant (rubbery) value above 80 °C (glass transition region). This decrement of G′ depends on structural features of the tested material, in turn strongly related to kinetics and mechanisms of crosslinking reactions occurring during the applied cure process. Furthermore, the extent of this G′ reduction between two temperatures, 50 °C and 80 °C, representative of the glassy and the rubbery state of investigated samples, respectively, resulted to be slightly higher for microwave cured materials than for conventionally crosslinked ones as best evidenced by data collected in Table 4.

In terms of the loss factor (tan δ), the curing method does not seem to affect neither the mechanic damping ability of the reference epoxy material nor the position of the tan δ signal always center around 65 °C (see Table 4). The shape of the damping signal, instead, appears to be more broadened for the epoxy resin cured by microwaves. This effect, best highlighted from the values of the full width at half maximum (FWHM) of the tanδ signal also reported in Table 4, may be related to the rapid kinetics of microwave assisted reactions. In fact, reasonably this effect may lead to the formation of networks with a more inhomogeneous structure compared to ones obtained by conventional slow heating procedures.

3.3. Flexural properties

Figs. 7–9 show the flexural modulus (E_f), the flexural strength

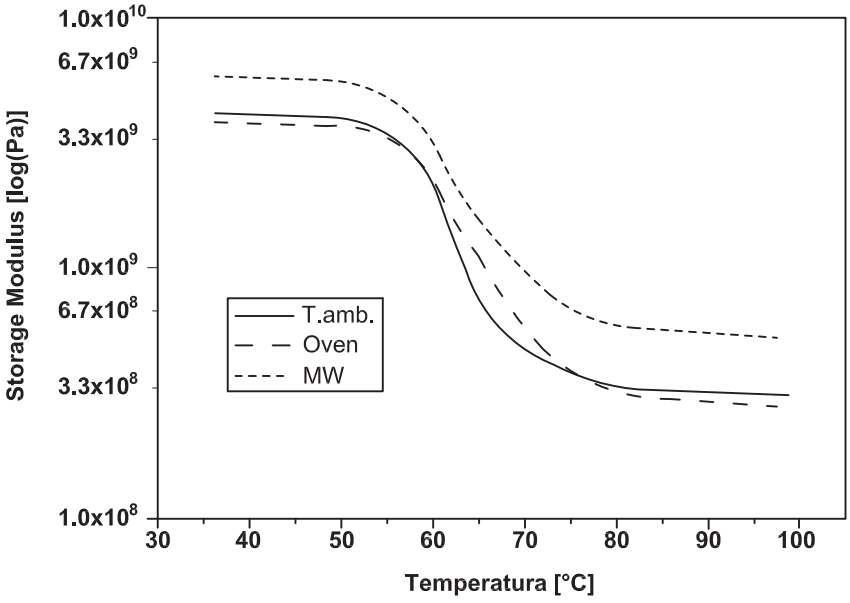

Fig. 5. Elastic modulus vs. temperature.

Table 4
Comparison of Tg, tan δ, G′ and G″, for the three types of cure.

	Tg [°C]	FWHM (°C)	G′ [Pa]	G″ [Pa]
T_{amb}	64.6		50 °C - 3.997×10^9	50 °C - 8.926×10^7
			80 °C - 3.379×10^8	80 °C - 4.163×10^8
Oven (100°C)	66.9		50 °C - 3.704×10^9	50 °C - 1.135×10^8
			80 °C - 3.257×10^8	80 °C - 5.978×10^7
MW (1500W)	65.2		50 °C - 5.559×10^9	50 °C - 1.561×10^8
			80 °C - 6.256×10^8	80 °C - 8.501×10^7

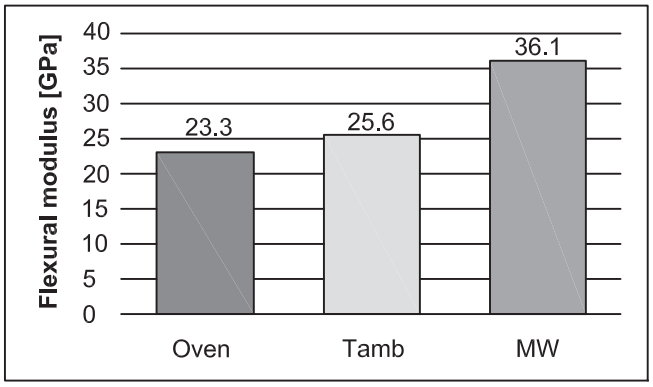

Fig. 7. Flexural modulus (E_f).

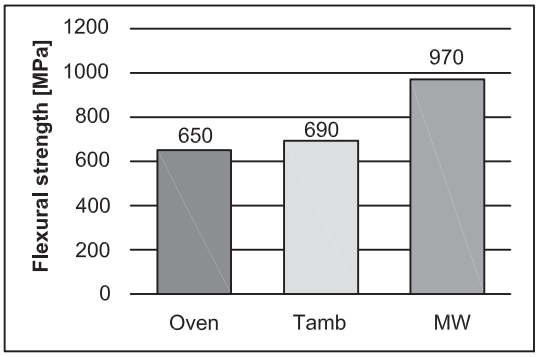

Fig. 8. Maximum flexural strength (σ_f).

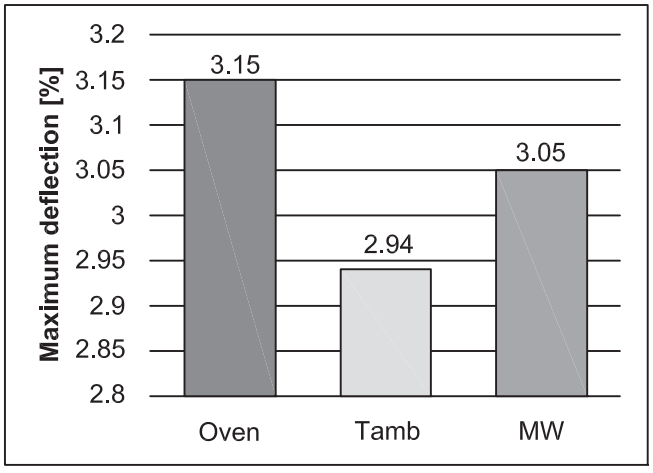

Fig. 9. Maximum deflection (ε_f).

(σ_f) and the maximum deflection (ε_f) for the composites cured for: (a) 7 days at room temperature, (b) 1 h in an oven at 100 °C and (c) 10 min in the microwave at 1500 W.

Static tests not only confirmed the higher flexural stiffness of the samples cured by microwaves but also showed an improved flexural strength of the same, although having a lower degree of crosslinking, compared to those cured by conventional heating approaches. This behavior does not seem to significantly influence the maximum tolerable deflection that seems to be even slightly higher than the value characteristic of samples cured at ambient temperature.

3.4. Pull-out results

Fig. 10 shows the pull-off bond strength of the investigated epoxy formulation, cured according to the 3 considered methodologies, with respect to 3 different substrates: conventional concrete (CC), artificial aggregates concrete (AAC) and geopolymer (G).

Different trend can be observed from this experiment since they were considered throughout the test campaign and appear to be relevant with regard to standard deviations.

Even if all the support were designed to have similar compressive strength, both AAC and G slabs showed a support failure while the CC had a mixed failure mode.

In particular, no significant evolution of S_{po} was detected for AAC and G. In fact, for this kind of failure, S_{po} is linked especially to the tensile strength of the support. For CC slabs, instead, an increase of S_{po} was detected for FRPs cured at 100 °C, further enhanced for ones crosslinked under microwave conditions.

4. Conclusions

Fiber-reinforced polymers based on epoxy/glass fibers and prepared by three different curing processes: 7 days at room temperature, 1 h at 100 °C and 10 min in a microwave oven at a power of 1500 W; were compared in terms of thermal and mechanical properties. Results demonstrated that the applied conditions allow to obtained samples with comparable degree of cure. With regard to the mechanical performance of cured samples, flexural tests carried out in static and dynamic mode demonstrated microwaves give rise to more stiff and structurally inhomogeneous epoxy networks with respect to ones obtained by conventional heating approach. However, the stiffening of the former does not compromise their maximum tolerable flexural strain at break and, above all, the microwave route confirms its time and cost-saving peculiarities.

Finally, pull-out strength measurements of cured FRPs from three different supports have demonstrated that apparently the curing conditions do not significantly affect this mechanical parameter in case of artificial aggregates concrete and geopolymer based support.

With respect to conventional concrete, instead the pull-out strength seems to be improved with the curing temperature in case of conventional processes but best results are achieved in presence of microwave crosslinked FRPs.

In light of results obtained so far by employing a closed microwave chamber, the research will also be extended to the use of an open chamber oven for in situ applications, however paying particular attention to the dangers arising from the greater likelihood of exposition of users to the electromagnetic fields.

We address the numerical simulation of the pull-out phenomenon in the examined materials, via penalty-techniques [44,45] and/or variational fracture mechanics [46], to future work.

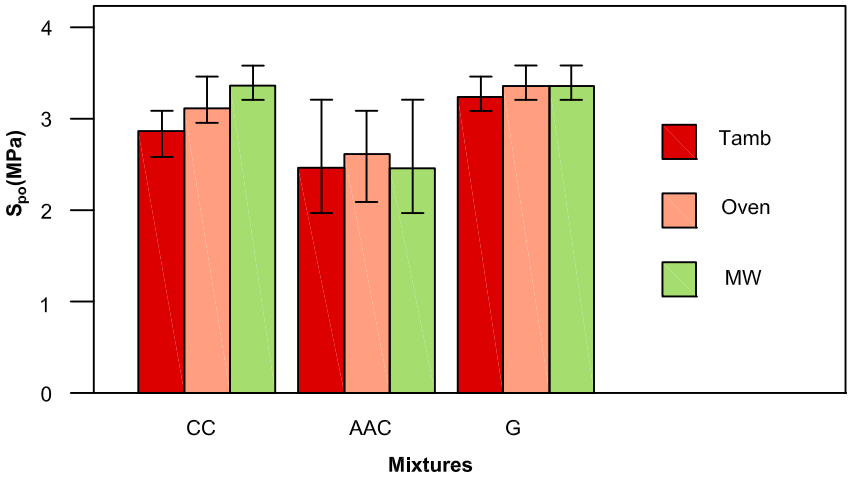

Fig. 10. Pull-out strength of cured FRPs on different supports.

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