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Influence of curing, post-curing and testing temperatures on mechanical properties of a structural adhesive

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ABSTRACT

Structural cold-curing adhesives are widely used to strengthen Reinforced Concrete (RC) structures with Fibre Reinforced Polymers (FRPs). The performance of these adhesives, and therefore the performance of the strengthening system, may be affected by temperature, as ambient-cured structural adhesives usually have low glass transition temperature (T_g). This paper presents a comprehensive experimental investigation on the influence of temperature on mechanical properties and T_g of a structural epoxy adhesive. The experimental program was divided in four groups of specimens. In Group 1, the effect of curing and post-curing temperature on T_g of the epoxy adhesive was investigated. In Groups 2–4, the effects of testing temperature, curing temperature and post-curing temperature, respectively, on adhesive mechanical properties were studied. Experimental results confirm that curing and post-curing temperature affected T_g differently depending whether the applied temperature was below or beyond the epoxy T_g. Similar behavior was observed in the mechanical properties of the epoxy, as they showed improvements when curing process (curing and post-curing) temperature was below T_g and they were negatively affected when curing process temperature was beyond T_g. Besides, tensile and compressive mechanical properties were negatively affected by testing temperatures beyond 20 °C.

1. Introduction

Structural epoxy adhesives are widely used to bond Fiber Reinforced Polymers (FRPs) to concrete in both Externally Bonded Reinforcement (EBR) and Near-Surface Mounted (NSM) strengthening techniques [1,2]. The performance of the strengthened structure will depend on the bonded joint, whose behavior has been acknowledged to be partially governed by the mechanical properties of the adhesive [3–5].

In civil engineering applications adhesive joint is typically cured at ambient temperature. Accordingly, cold-curing adhesives are the most widely used, as its use is easier and they present good mechanical properties after the required curing time (which depends on the curing agent and temperature). It is a feature of commonly used ambient-cured structural adhesives to have glass transition temperatures (T_g) ranging between 40 °C and 70 °C [6]. Being T_g defined as the temperature range where a thermosetting polymer changes from a glassy to a rubbery state, (i.e. the temperature ranges over which the mobility of the polymer chains increases significantly) [7], these relatively low values of T_g can be a limiting factor because these adhesives can exhibit substantial changes in their mechanical properties if working temperature approaches its T_g [8–14]. As a consequence of the reduction in the adhesion strength of the epoxy as the T_g range is approached, a reduction in the stiffness and ultimate capacity of the FRP-strengthened element can take place and the failure mode can change [8,15–19].

A reduced number of studies have investigated the influence of testing temperature on the mechanical properties of cold-curing epoxy adhesive typically used in civil engineering [9–20]. Firmo et al. [9] presented an experimental work to assess the mechanical properties of a commercial epoxy adhesive typically used in civil engineering at different temperatures (from 20 °C to 120 °C). The results showed that mechanical properties were significantly affected by testing temperatures larger than T_g ($T_g = 47$ °C), as the shear and the tensile strength of epoxy were reduced to around 30% of their corresponding ambient strengths. Additionally, the shear and the tensile modulus were almost negligible for tests at 50 °C. These results are similar to those obtained in other studies on epoxy adhesives with larger T_g values used in other applications, such as aerospace industry [10–12]. As an example, Banea et al. [10,11] investigated the effect of testing temperature on the mechanical properties of an epoxy adhesive having a T_g value equal to 150 °C. In these studies, four different temperatures were applied during

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testing (i.e. room temperature, 100, 150 and 200 °C), and experimental results showed a reduction in the tensile strength and an increase in the ductility when testing temperature was increased. The maximum reduction was observed for testing temperatures near to and beyond T_g, where the adhesive was in rubbery state. Similarly, Wu [12] studied the influence of testing temperature on mechanical properties of an epoxy whose T_g equals 177 °C. According to their experimental results, increasing the testing temperature produced a decrease in both the tensile strength and the elastic modulus. It should be noted that the rate of loss of the mechanical properties was sharply increased for testing temperature beyond T_g. Besides, Reis [13] presented a comparative study on the effect of testing temperature (ranging from 23 to 90 °C) on compressive strength of epoxy polymer mortar and unsaturated polyester mortar (mixture of a quartz foundry sand with the thermoset resin binder). Although both materials experienced a decrease in their mechanical properties when testing temperature was increased, epoxy polymer mortars were more sensitive to temperature changes than unsaturated polyester mortars. This temperature dependency was related to the T_g of the resins used.

In addition to testing temperature, curing and post-curing procedures can also affect the structural performance of an epoxy adhesive, in terms of the value of T_g and the epoxy mechanical properties [21–30]. Michels et al. [21] studied the effect of different curing conditions and mixing processes on mechanical properties of three different epoxy adhesives. Specimens exposed to accelerated curing (consisting in exposing the epoxy specimens to an elevated temperature, 90 °C, for a period of 25 min just after casting) showed higher porosity, which appeared to be the cause of their apparent lower tensile properties. The higher porosity can be attributed to a faster development of strength and stiffness, that finally affects to the cross-linking process, thus showing the influence of the cure-kinetics. Mixing the epoxy under vacuum reduced the porosity of both specimens (with and without accelerated curing) leading to higher tensile properties. Similar results were obtained in Cruz et al. [22]. It has been shown in the literature that longer curing times are needed for lower curing temperatures. This effect, which is of high importance in cold-curing epoxy adhesives, is a consequence of the vitrification of the network that takes place when the resin is cured at a temperature below the ultimate glass transition of the completely cross-linked resin. As a result, the resin is in a glassy but uncured state, so that cross-linking reaction has slowed down dramatically and it may take a long time before the ultimate properties of the adhesives are reached. This dependency was confirmed for cold-curing epoxy adhesives in Lapique and Redford [23] and Moussa et al. [24]. For the case of Lapique and Redford [23], they obtained same mechanical properties with 4 h curing at 64 °C and 28 days curing at room temperature. For the case of Moussa et al. [24], epoxy cured at low temperatures of 5 °C to 10 °C required a 3-days curing period for attaining the full curing, whereas few hours (3.7 h to 1.6 h) were needed for curing temperatures in the range of 35 °C to 60 °C. Lahouar et al [25] investigated the evolution of epoxy T_g under four different curing temperatures (i.e. 20, 50, 82 and 108 °C). According to their results on DSC tests, the T_g increased gradually by increasing the curing temperature. However, this increase in T_g had an upper bound value, as curing beyond 82 °C had no effect on T_g. This upper bound value corresponds to the glass transition temperature of the fully cured network ($T_{g\infty}$), at which the resin reaches its maximum degree of cross-linking. The existence of a T_g upper bound value ($T_{g\infty}$) was also confirmed in a study from Carbas et al. [26], who studied the effect of the curing temperature on the mechanical properties and T_g of three different structural epoxy adhesives. For each adhesive, samples of the bulk adhesive were cured at various temperatures and T_g was measured by a dynamic mechanical analysis using an in-house developed apparatus. It should be mentioned that the main concern of the applied method is related to thermodynamics and the existence of a temperature gradient in the adhesive. Therefore, the speed of the test should be a compromise value that ensures a homogeneous temperature distribution in the specimen and avoids causing a post-curing in the specimen. Once T_g was determined at various curing temperatures, $T_{g\infty}$ could be determined. In addition to the determination of the $T_{g\infty}$ values for the three adhesives, results of their experimental program confirmed that, as far as the curing temperature was below $T_{g\infty}$, any increase in the curing temperature derived in an increase in the mechanical properties and T_g. On the contrary, for curing temperatures above $T_{g\infty}$, the mechanical properties and T_g decreased. Although these findings are in agreement with [27–29], it should be mentioned that T_g is a kinetic parameter that depends on the heating rate and on the measurement conditions [26]. Experimental work presented in [26] was complemented with a second program presented in Carbas et al. [30], where the effect of post-curing was analyzed. Post-curing process can be of high importance for the case of cold-curing epoxy adhesives. It may be the case that an un-complete cure in the epoxy resin exists because of the longer curing time needed, especially if they are cured at ambient temperature. In this case, the reactivation of the cross-linking process of the epoxy can take place if a post-curing process is applied. In this sense, experimental results presented in [30] showed that the effect of post-curing at temperature below $T_{g\infty}$ depended on whether the cross-linking process was complete or not. Finally, post-curing above $T_{g\infty}$ produced thermal degradation that caused a progressive decrease in mechanical properties and T_g. According to the literature [7,21,23,25,26,31,32], cross-linking plays an important role in the mechanical performance of an epoxy. In this sense, results presented in Michel and Ferrier [7] confirmed that cross-linking of an epoxy resin is found to be an irreversible process, so that larger T_g values are obtained when curing at higher temperatures and its value does not change when the polymer is exposed back to a lower temperature. Similar results were obtained in [21].

This paper presents part of a larger experimental program on the flexural behavior of NSM FRP-strengthened Reinforced Concrete (RC) beams tested at different temperatures. Specifically, the main objective of this paper is the assessment of a commercial structural epoxy adhesive commonly used for RC strengthening and retrofitting with FRP materials. To this end, a comprehensive experimental program was performed to evaluate the influence of the temperature on mechanical properties (namely the uniaxial tensile strength (σ_{tu}), the tensile elastic modulus (E), the uniaxial ultimate compressive load (F_{cu}), the uniaxial compressive strength (σ_{cu})) and T_g of a structural epoxy adhesive. Different curing temperatures, post-curing temperatures and testing temperatures have been considered. The experimental program is described and the main results are presented and discussed.

2. Experimental program

2.1. Material

The adhesive used in this study is a high performance, solvent-free, thixotropic, and grey two-component epoxy adhesive specially developed for bonding Carbon Fiber Reinforced Polymers (CFRP) on concrete, that is traded under the commercial name of *S&P 220 HP*. According to the manufacturer's product data sheet [33], the components A (Bisphenol A and Bisphenol F based resin) and component B (hardener, with a mixture of amines) should be mixed at a ratio of 2:1 by weight, and the suggested curing duration is 7 days. Furthermore, the nominal elastic modulus, compressive strength and T_g of epoxy declared by the manufacturer are 7.1 GPa, 81 MPa and 58.2 °C, respectively. The mixture of epoxy adhesive is shown in Fig. 1.

2.2. Specimens curing process and testing configuration

In this experimental program, 4 groups of specimens were considered. Group 1 comprised those specimens used for determining the effect of curing and post-curing temperature on T_g of the epoxy adhesive. Groups 2, 3 and 4 comprised those specimens used for evaluating the effect of testing temperature (Group 2), curing temperature (Group 3)

Fig. 1. Epoxy adhesive mixture.

and post-curing temperature (Group 4) on mechanical properties of the epoxy adhesive. The specimens were designated as X-Y-Z. In this designation X indicates the testing group, so that TG refers to specimens tested to determine the T_g (i.e. Group 1), TT refers to specimens tested to study the influence of testing temperature on epoxy mechanical properties (i.e. Group 2), CT refers to specimens tested to analyze the influence of curing temperature on epoxy mechanical properties (i.e. Group 3) and PT refers to specimens tested to determine the influence of post-curing temperature on epoxy mechanical properties (i.e. Group 4). In the proposed specimens' designation, Y indicates the curing process

Table 1
Details of the experimental program.

Group	Specimen ID	Number of specimens	Curing process	Curing process temperature (°C)	Test	Testing temperature (°C)
Group 1	TG-C-20	1	See Fig. 2a	20	Glass transition temperature of epoxy	According to standard
	TG-C-50	1		50		
	TG-C-70	1		70		
	TG-PC-50	1		50		
	TG-PC-70	1		70		
Group 2	TT-TEN-20	3	See Fig. 2b	20	Tensile strength and elastic modulus	20
	TT-TEN-40	3		20		40
	TT-TEN-50	3		20		50
	TT*-TEN-50	3		50		50
	TT-TEN-60	3		20		60
	TT-TEN-70	3		20		70
	TT*-TEN-70	3		70		70
	TT-TEN-85	3		20		85
	TT-COM-20	3		20	Compressive strength	20
	TT-COM-40	3		20		40
	TT-COM-50	3		20		50
	TT*-COM-50	3		50		50
	TT-COM-60	3		20		60
	TT-COM-70	3		20		70
	TT*-COM-70	3		70		70
	TT-COM-85	3		20		85
Group 3	CT-TEN-(−15)	3	See Fig. 2c	−15	Tensile strength and elastic modulus	20
	CT-TEN-50	3		50		20
	CT*-TEN-50[1]	3		50		20
	CT-TEN-70	3		70		20
	CT*-TEN-70[2]	3		70		20
	CT-COM-(−15)	3		−15	Compressive strength	20
	CT-COM-50	3		50		20
	CT*-COM-50[1]	3		50		20
	CT-COM-70	3		70		20
	CT*-COM-70[2]	3		70		20
Group 4	PT-TEN-40	3	See Fig. 2d	40	Tensile strength and elastic modulus	20
	PT-TEN-50	3		50		20
	PT-TEN-60	3		60		20
	PT-TEN-70	3		70		20
	PT-TEN-85	3		85		20
	PT-COM-40	3		40	Compressive strength	20
	PT-COM-50	3		50		20
	PT-COM-60	3		60		20
	PT-COM-70	3		70		20
	PT-COM-85	3		85		20

[1] Curing at 50 °C for 7 days and later exposed to a one-day post-curing at 50 °C.
[2] Curing at 70 °C for 7 days and later exposed to a one-day post-curing at 70 °C.

type (C for curing and PC for post-curing) for specimens of Group 1 and refers to mechanical test type (TEN for tension test and COM for compression test) for specimens of Groups 2–4. Finally, Z denotes the temperature. For example, TG-C-50, refers to a specimen that was cured at 50 °C and tested in order to determine the value of T_g (Group 1). Similarly, PT-TEN-70 refers to a specimen that was post-cured at 70 °C and tested in order to analyze the effect of post-curing temperature (Group 4) on tensile properties of the epoxy adhesive. It should be mentioned that the asterisk symbol appearing in some specimens' designation shows one extra day of post-curing. All specimens were tested at the age of 12 days. Curing and post-curing periods with target temperature different from 20 °C lasted 7 days and 1 day, respectively. Details on the curing process and testing configuration of each group are presented below and summarized in Table 1:

- *Group 1:* effect of curing and post-curing temperature on T_g

This group includes a total of 5 specimens, whose curing and post-curing temperatures varied with the aim at analyzing its effect on the T_g of the epoxy adhesive. One specimen was cured at room temperature (20 °C) for 12 days as a reference sample (TG-C-20) (see black line in Fig. 2a. Two other specimens were cured for 7 days under the target temperature (50 °C and 70 °C) to be later left 5 more days at 20 °C before testing the T_g (TG-C-50 and TG-C-70) (see blue line in Fig. 2a. To conclude, two specimens were initially cured for 9 days at 20 °C, after which a post-curing process of one-day under the target temperature (50 °C and 70 °C) was applied, that was finally followed by a cooling-down process for 2 days at 20 °C prior to T_g testing (TG-PC-50 and TG-PC-70) (see red line in Fig. 2a.

- *Group 2:* effect of testing temperature on mechanical properties

This group includes a total of 48 specimens whose testing temperatures varied to evaluate its effect on epoxy adhesive mechanical properties. The specimens were cured at 20 °C for 11 days (see black line in Fig. 2b. After this curing process, specimens were heated to the target temperature for one-day (20, 40, 50, 60, 70 and 85 °C) to be finally tested under tension and compression. It should be mentioned that the one-day heating process previous to testing should be considered as a one-day post-curing process. Furthermore, two additional configurations (marked with asterisk symbol) were considered where an additional one-day post-curing at the target temperature (50 and 70 °C) was applied (see red line in Fig. 2b.

- *Group 3:* effect of curing temperature on mechanical properties

This group includes a total of 30 specimens whose curing temperature varied with the aim at evaluating its effects on epoxy adhesive mechanical properties. Immediately after being cast, specimens were moved to the chamber to be cured at different target temperatures (-15, 50 and 70 °C) for a period of 7 days. After this curing phase, specimens were left at 20 °C for 5 days before testing (see black line in Fig. 2c. As in previous group, two additional configurations (marked with asterisk symbol) were exposed to a one-day post-curing at target temperature (50 and 70 °C) and cooled-down again at 20 °C (see red line in Fig. 2c.

- *Group 4:* effect of post-curing temperature on mechanical properties

This group includes a total of 30 specimens that were post-cured at different temperatures to evaluate its effect on epoxy adhesive mechanical properties. In this last group, the specimens were cured at 20 °C for 9 days, to be later exposed to a one-day post-curing at different target temperatures (40, 50, 60, 70 and 85 °C) and a final cooling-down at 20 °C before testing (see Fig. 2d.

2.3. Test procedures

2.3.1. Differential scanning calorimetry test

The differential scanning calorimetry (DSC) technique [34] was used to calculate the T_g of the epoxy adhesive after different curing and post-curing conditions (Group 1 of the experimental campaign). For each curing condition, one specimen was tested. The DSC technique provides information about changes in physical, chemical, and heat capacity of the adhesive which may lead to important information about their

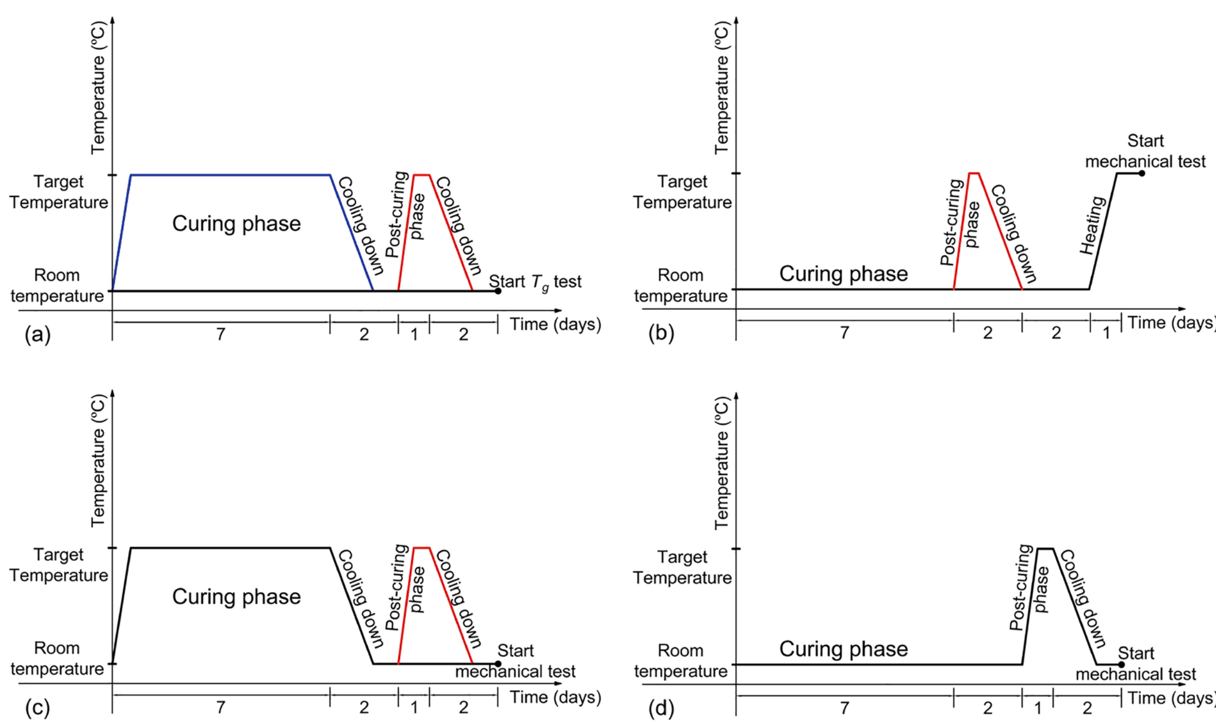

Fig. 2. Diagram of the curing processes. (a) Group 1; (b) Group 2; (c) Group 3; and (d) Group 4.

thermal history (i.e. the temperature that epoxy undergoes), stability, processing conditions, progress of chemical reactions and possible changes in mechanical properties [34]. The dynamic DSC tests were carried out using the DSC Q2000 machine. A heating rate of 10 °C/min was applied using nitrogen as the purge gas at 50 mL/min. The temperature range was between 20 and 80 °C. To determine T_g, and according to ASTM E1356-08 [34], the extrapolated onset temperature (T_f) and the extrapolated end temperature (T_e) were measured. The midpoint temperature (T_m), computed as the mean between T_f and T_e, is the most commonly used as the glass transition temperature [34].

2.3.1.1. Tension test. Tensile properties of the epoxy adhesive were determined following ISO-527–1 [35] specifications. To this end, dog-bone specimens were manufactured following the geometry and dimensions shown in Fig. 3a and tested under displacement-control at a speed of 1 mm/min. For each curing condition, three specimens were tested. The test setup is shown in Fig. 3b. As mentioned in section 2.2, specimens of Group 2 were heated to the target testing temperature for a duration of 24 h prior to testing and, once temperature was stabilized, the test was performed. To allow the curing process, a thermal chamber was mounted on the testing machine (see Fig. 3b). Two strain gauges located at the back and front sides of the specimens and one axial extensometer with a 25 mm gauge length were used to measure the strain during the tension tests. Furthermore, two thermocouples (one to measure the environmental temperature inside the thermal chamber and the other glued on the surface of specimen) were used to measure the temperature before and during the test. In each specimen, registers of loads were transformed to stresses by using the mean cross-section area from three different measurements. The tensile elastic modulus was obtained from the slope of axial stress–strain curve between 0.05% and 0.25% of the maximum tensile strain [35]. Details of the specimens and test setup are shown in Fig. 3.

2.3.1.2. Compression test. The compressive strength of the epoxy adhesive was determined according to EN 196-1 [36], with the aim at following the same standard used by the epoxy manufacturer. According to EN 196–1 [36], 80 mm length prism having a 40x40 mm^2 cross-section should be used for determining the epoxy compressive strength (see Fig. 4a and 4b. During the compression test, the load should be increased smoothly, under force-control, at a ratio of 2400 ± 200 N/s until fracture. Similar to tension tests, also for compression specimens, three specimens were considered for each curing condition. The test setup is shown in Fig. 4c. Two centralized steel plates, with dimensions of 40x40 mm^2, were placed at the top and the bottom of the specimen to transfer the load. Moreover, a spherical hinge was located at one loading side to avoid the effect of any possible eccentricity during loading. Similar to tension tests, a thermal chamber was mounted on the testing machine for the curing process (see Fig. 4c. One strain gauge was installed to measure the evolution of specimen's longitudinal strain. However, data obtained from the strain gauge were not reliable due to the earlier detachment and peeling in the surface that took place during the loading. Therefore, only the ultimate compressive strength of specimens was finally evaluated. Similar to tension tests, two thermocouples (one to measure the environmental temperature and the other glued on the surface of the specimen) were used to record the temperature before and during the test. The ultimate compressive strength of epoxy was calculated by dividing the ultimate load by initial contacted surface area. Due to possible unavoidable variations in dimensions of the specimens, measurements of the area were taken for each specimen prior to testing. Details of the specimen and test setup are shown in Fig. 4.

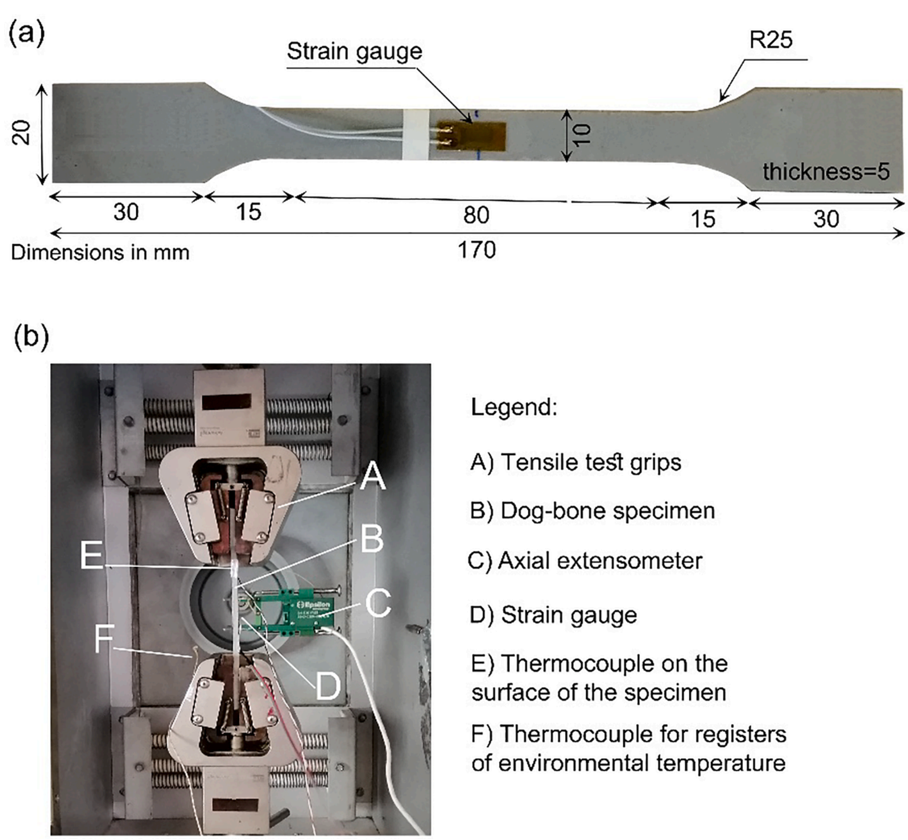

Fig. 3. Details of the tension test. (a) specimen dimensions; (b) Test setup inside the thermal chamber.

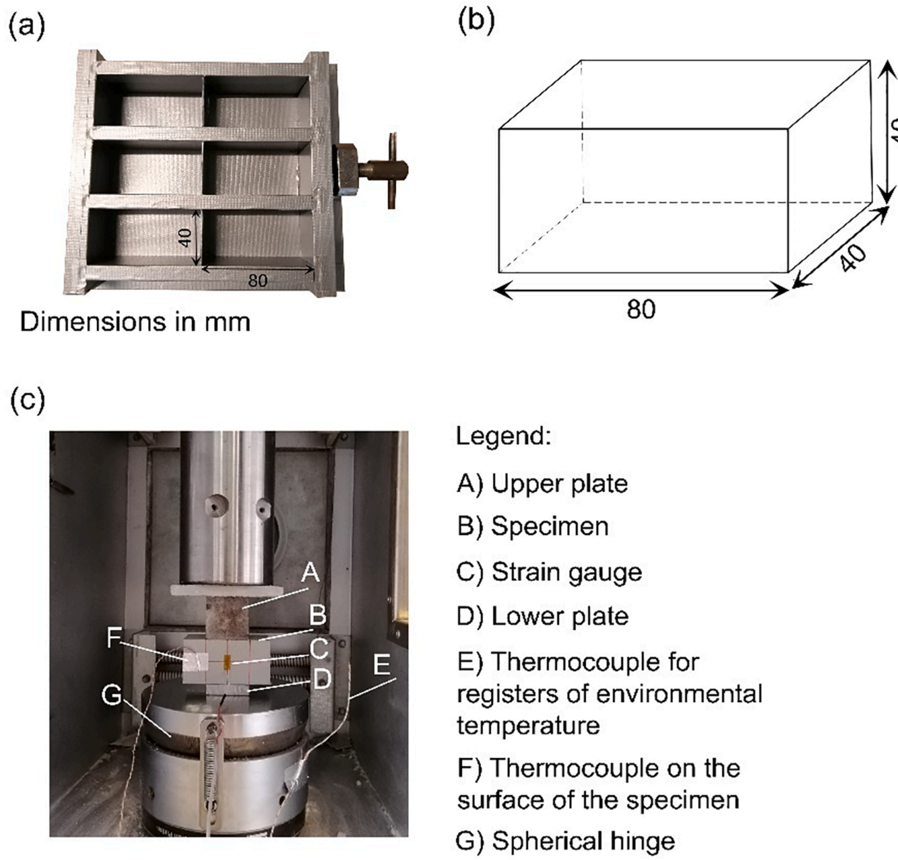

Fig. 4. Details of the compression test. (a) Typical mould; (b) specimen dimensions; and (c) Test setup inside the thermal chamber.

3. Results and discussions

3.1. Effect of curing and post-curing temperature on T_g

Results of the DSC tests performed on specimens of Group 1 are presented in Fig. 5a in the form of curves showing the variation of the heat flow versus temperature. It is acknowledged that the change of the heat flow versus temperature curve is related to an absorption of energy and can, therefore, be an indicator of a possible change in the physical state of the resin. Considering that the T_g is defined as the temperature at which the physical state of the resin changes from the glassy state into the rubbery state, it has been accepted that T_g is in the range of the temperatures limiting the change in the baseline of the curve. It should be mentioned that in some of the curves presented in Fig. 5a, an enthalpy relaxation peak can be observed immediately after the T_g range. This is an indication that physical aging has occurred [37]. It is worth noting that physical aging occurs in an amorphous polymer held below its T_g.

Table 2
Experimental values of T_g from DSC tests.

Specimens ID	TG-C-20	TG-C-50	TG-C-70	TG-PC-50	TG-PC-70
T_g (°C)	54.9	59.4	47.1	63.5	49.6
Difference with T_g at 20 °C (%)	–	8	−14	16	−10

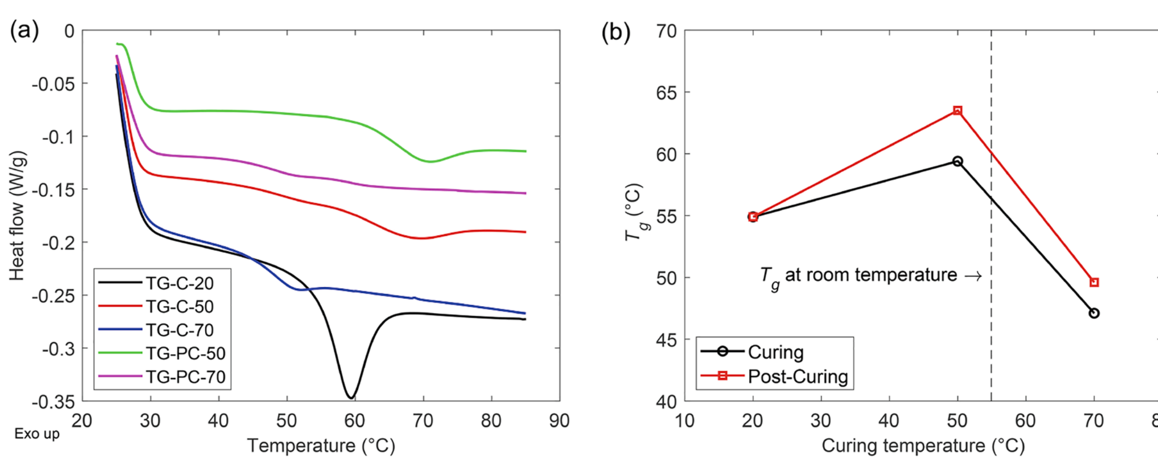

Fig. 5. (a) Results from DSC tests (b) Evolution of T_g as a function of the curing and post-curing temperatures.

According to experimental results presented in Table 2, the T_g value of the epoxy cured at room temperature (specimen TG-C-20) was 54.9 °C. Based on this, two different behaviors can be observed in Fig. 5a: curing or post-curing the specimens at a temperature below epoxy T_g (i.e. specimens cured (and post-cured) at 50 °C of the present experimental program) resulted in a shift to the right of the peak in the heat flow versus temperature curve; contrarily, curing or post-curing the specimens at a temperature beyond epoxy T_g (i.e. specimens cured (and post-cured) at 70 °C of the present experimental program) resulted in a shift to the left of the peak in the heat flow versus temperature curve. This shifts to the right and left are therefore indicating an increase and reduction of the T_g value, respectively. These two different trends can also be observed in Fig. 5b, where the dashed line represents the T_g value of the specimen cured at room temperature (TG-C-20), and the black and red solid lines represent the evolution of T_g for specimens that were cured and post-cured, respectively. When curing or post-curing of an adhesive takes place at a temperature below $T_{g\infty}$, T_g increases as the curing temperature increases. On the contrary, when curing or post-curing of an adhesive takes place at a temperature above $T_{g\infty}$, T_g decreases as the curing temperature increases [24,26,28]. This change in epoxy T_g is due to possible variations in the degree of cross-linking (i.e. possible thermal degradation or oxidative cross-linking) in the epoxy adhesive [7,12,30–32]. Based on this, and according to experimental results, for the epoxy adhesive studied in this paper, the curing temperature that leads to the fully cured network ($T_{g\infty}$) should be between 50 °C and 70 °C. Table 2 summarizes the experimental values of the T_g and its percentages of variation with respect to reference specimen (specimen TG-C-20).

3.2. Effect of testing temperature on mechanical properties

In this section, results from tension and compression tests on specimens of Group 2 are presented and analyzed to study the effect of testing temperature on mechanical properties of the epoxy adhesive.

Regarding to the tension tests, Fig. 6a shows the axial tensile stress versus strain curves, for one representative specimen of each testing temperature. From Fig. 6a it can be observed that specimen tested at room temperature (TT-TEN-20) showed an almost perfect linear behavior up to failure. With the application of higher testing temperature (but still below T_g), the initial linear behavior was followed by a non-linear one before specimen's failure. Lately, when testing temperature was beyond T_g, the stress–strain curves became highly non-linear and properties were greatly affected, as a result of the rubbery behavior of the epoxy. Same behavior was observed in [9–13].

Experimental results in terms of tensile strength and elastic modulus are presented in Table 3 and Fig. 6b. It can be observed that, the increase of the testing temperature was followed by a monotonic decrease in tensile strength and elastic modulus, with the most remarkable reduction taking place when testing temperature changed from 50 °C to 60 °C (epoxy T_g being within this range of temperatures). From this testing temperature on, the elastic modulus was almost negligible. These results are in agreement with previous studies on cold-curing epoxy adhesives [9,20].

According to the test matrix (Table 1, two additional configurations were considered whose curing included a one-day post-curing (specimens TT*-TEN-50 and TT*-TEN-70 in Table 1 and red line in Fig. 1b. As mentioned before, the process of heating prior to testing should be considered as a post-curing and therefore, in specimens TT*-TEN-50 and TT*-TEN-70 two cycles of post-curing were applied. According to the experimental results, no significant effect of an extra cycle of post-curing was observed in the tensile strength and elastic modulus of the epoxy resin (see Fig. 7 and Table 3. This might be a sign of the resin achieving a stable situation after the first post-curing, so that no additional evolution of the mechanical properties was possible. A representative typical failure mode under tension test (rupture near to the mid-section of the specimens) is shown in Fig. 8a.

In addition to tensile properties, behavior under compression was also evaluated under different testing temperatures. Fig. 9a shows the compressive load versus deflection curves for one representative specimen of each testing temperature. Although one strain gauge was installed in every specimen, the peeling of specimen external surface invalidated the registered data. Therefore, deflections presented in Fig. 9a correspond to the movement between grips of testing machine. In general, as observed in tension tests, the increase in the testing temperature was followed by a more flexible response and a decrease of the

Table 3
Tensile strength and elastic modulus for specimens tested at different temperatures (average ± standard deviation).

Specimens ID	Tensile strength, σ_{tu} (MPa)	$\sigma_{tu}/\sigma_{tu\_20}$ [1]	Elastic modulus, E (MPa)	$E / E_{\_20}$ [1]
TT-TEN-20	28.0 ± 0.1	–	8102.4 ± 67.0	–
TT-TEN-40	23.0 ± 0.2	0.82	5520.8 ± 179.0	0.68
TT-TEN-50	19.6 ± 0.7	0.70	4289.6 ± 100.7	0.53
TT*-TEN-50	19.7 ± 0.3	0.70	4128.7 ± 0.1	0.51
TT-TEN-60	6.9 ± 0.2	0.25	673.2 ± 24.5	0.08
TT-TEN-70	2.7 ± 0.1	0.10	271.5 ± 12.9	0.03
TT*-TEN-70	2.8 ± 0.0	0.10	273.9 ± 2.1	0.03
TT-TEN-85	1.9 ± 0.0	0.07	247.5 ± 1.8	0.03

[1] Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-TEN-20 tested at 20 °C)

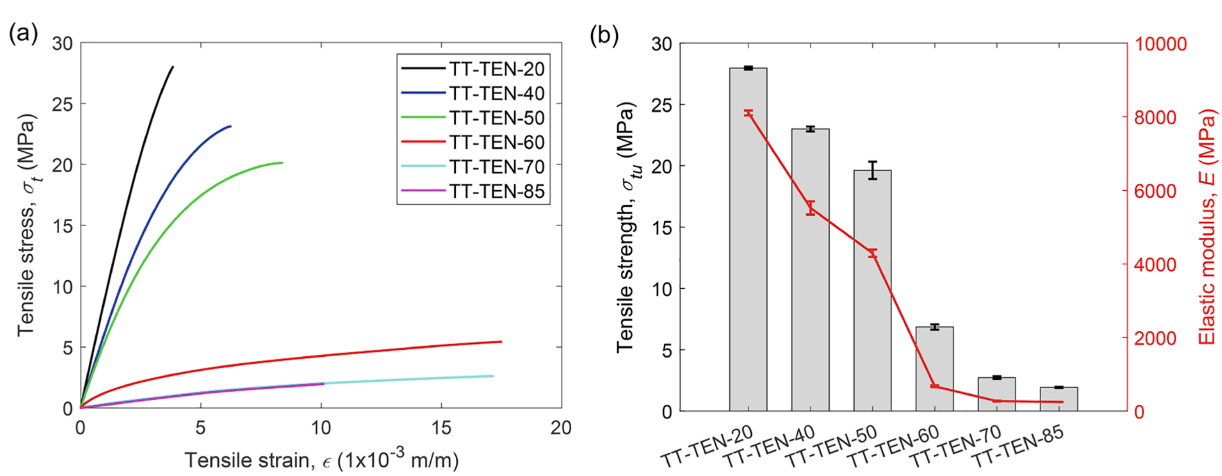

Fig. 6. Effect of testing temperature on tensile mechanical properties of the epoxy resin. (a) Representative stress–strain curves; (b) Evolution of tensile strength and elastic modulus.

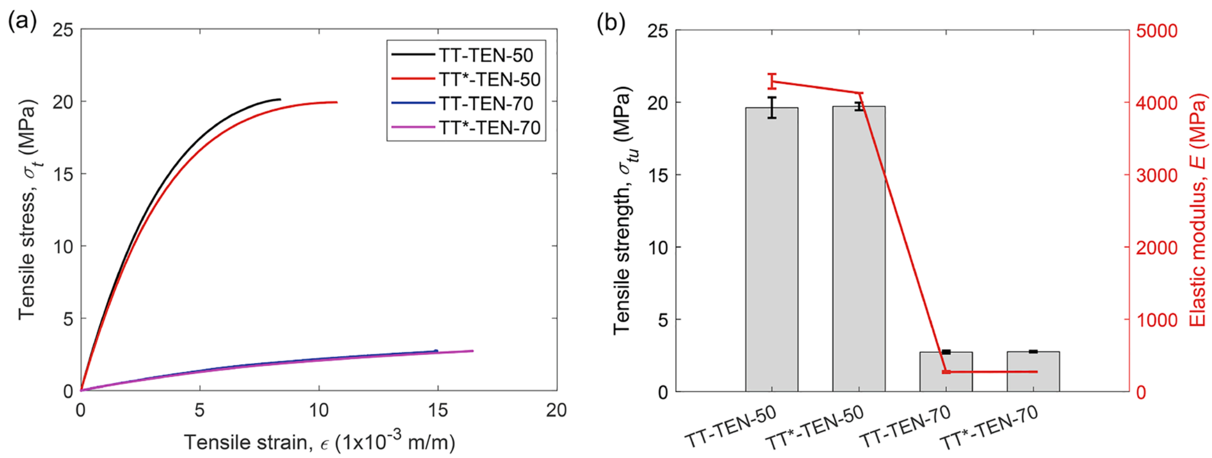

Fig. 7. Effect of additional post-curing on tensile mechanical properties of specimens in Group 2. (a) Representative stress–strain curves; (b) Evolution of tensile strength and elastic modulus.

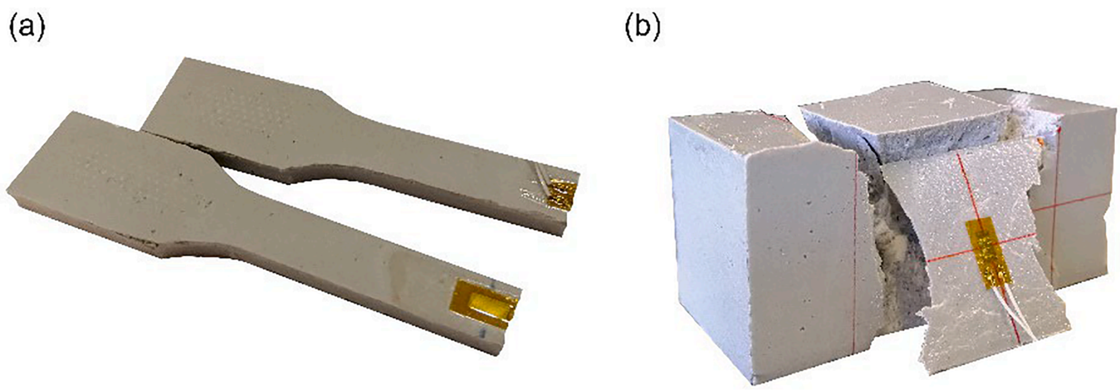

Fig. 8. Typical failure modes (a) Tension test; (b) Compression test.

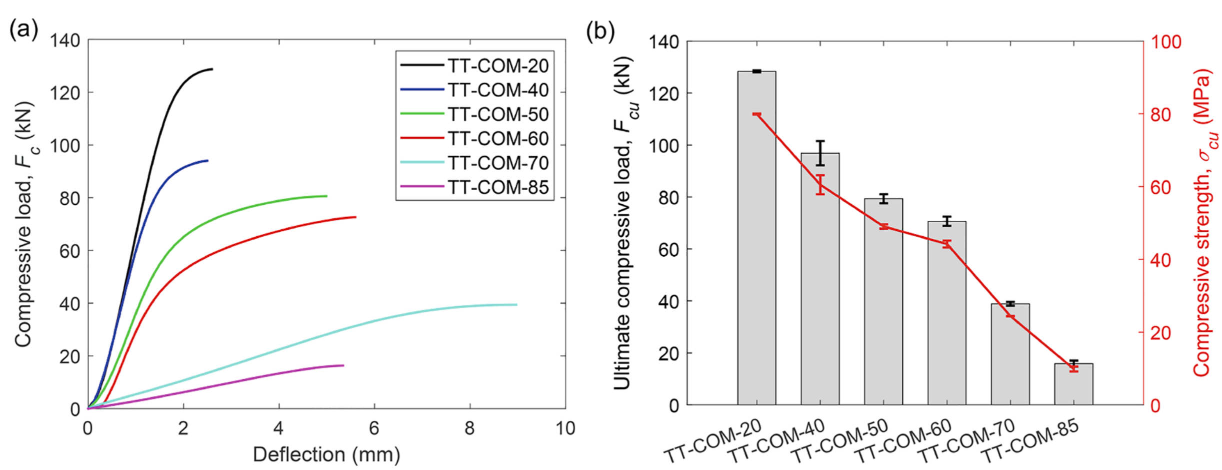

Fig. 9. Effect of testing temperature on compressive mechanical properties of the epoxy resin. (a) Representative load–deflection curves; (b) Compressive strength and ultimate compressive load.

ultimate compressive load.

Averaging the experimental value of ultimate compressive load over the initial area of contact allowed determining the compressive strength of each specimen. Experimental results of compressive properties (ultimate compressive load and compressive strength) are presented in Table 4 and Fig. 9b. According to these results, compressive strength was almost halved for testing temperatures equal to 60 °C, and the reduction rate highly increased beyond 60 °C.

With the aim at analyzing the possible effect of the inclusion of an extra cycle of post-curing phase in the curing process of the specimens, and similar to what was done for tension tests, two additional configurations were considered (specimens TT*-COM-50 and TT*-COM-70 in Table 1 and red line in Fig. 1b. According to experimental results presented in Table 4 and Fig. 10, the extra cycle of post-curing resulted in a

Table 4
Ultimate compressive load and compressive strength for specimens tested at different temperatures (average ± standard deviation).

Specimens ID	Ultimate compressive load, F_{cu} (kN)	F_{cu} /F_{cu_20} [1]	Ultimate compressive strength, σ_{cu} (MPa)	σ_{cu} /σ_{cu_20} [1]
TT-COM-20	128.4 ± 0.4	–	79.9 ± 0.1	–
TT-COM-40	96.9 ± 4.7	0.75	60.5 ± 2.6	0.76
TT-COM-50	79.3 ± 1.7	0.62	49.0 ± 0.6	0.61
TT*-COM-50	82.2 ± 0.7	0.64	51.8 ± 0.5	0.65
TT-COM-60	70.6 ± 1.8	0.55	44.2 ± 1.0	0.55
TT-COM-70	38.9 ± 0.7	0.30	24.4 ± 0.1	0.31
TT*-COM-70	36.1 ± 1.6	0.28	22.4 ± 0.7	0.28
TT-COM-85	15.9 ± 1.2	0.12	9.9 ± 0.7	0.12

[1]Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-COM-20 tested at 20 °C)

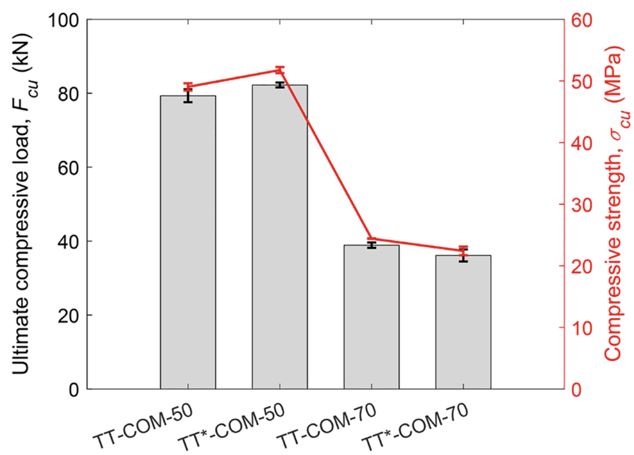

Fig. 10. Effect of additional post-curing on compressive mechanical properties of specimens in Group 2.

small variation in the ultimate compressive load and compressive strength, which can be attributed to typical scatter in experimental results.

A representative typical failure mode of the compressive specimens is shown in Fig. 8b.

3.3. Effect of curing temperature on mechanical properties

With the aim at analyzing the effect of curing temperature on the mechanical properties of the epoxy resin, results on specimens of Group 3 are presented and discussed in this section.

Experimental results in terms of tensile strength and elastic modulus of epoxy cured at different temperatures are presented in Fig. 11 and Table 5, and compared to results on specimens TT-TEN-20, belonging to Group 2, which were cured and tested at 20 °C. From this comparison it can be observed that curing the specimens at −15 °C (CT-TEN-(−15)) had no significant effect on epoxy tensile strength and had some detrimental effects on the elastic modulus. The application of −15 °C during curing, in fact, postponed the initiation of curing, as 7 days after being casted, epoxy resin was still soft. Same observations were reported in [7,38]. These observations suggest that, due to cold temperature postponing the initiation of curing, suitable considerations should be taken into account in the cold weather regions to facilitate the adhesive curing process.

According to experimental results on tensile strength and elastic modulus, the increase of curing temperature below $T_{g\infty}$ promotes the increase of cross-linking, which results in the increase of the performance of the adhesive (specimen CT-TEN-50). On the contrary, curing at temperatures above $T_{g\infty}$ possibly produces thermal degradation on the adhesive, which results in a decrease of its mechanical properties (specimen CT-TEN-70). These results confirm that mechanical properties and the T_g properties have similar behaviors.

In addition to previous specimens, two more configurations were also tested whose curing process was followed by a one-day post-curing (specimens CT*-TEN-50 and CT*-TEN-70). Experimental results on tension tests presented in Table 5 and Fig. 12 revealed that the post-curing process did not highly affect the mechanical properties of the

Table 5
Tensile strength and elastic modulus for specimens cured at different temperatures (average ± standard deviation).

Specimens ID	Tensile strength, σ_{tu} (MPa)	σ_{tu} /σ_{tu_20} [1]	Elastic modulus, E (MPa)	E / $E_{\_20}$ [1]
CT-TEN-(−15)	27.7 ± 0.7	0.99	7601.9 ± 56.3	0.94
TT-TEN-20	28.0 ± 0.1	–	8102.4 ± 67.0	–
CT-TEN-50	31.5 ± 1.0	1.13	7992.0 ± 372.8	0.99
CT*-TEN-50	29.8 ± 0.1	1.06	7124.9 ± 516.1	0.88
CT-TEN-70	30.3 ± 1.1	1.08	6892.5 ± 176.4	0.85
CT*-TEN-70	29.0 ± 2.2	1.04	6808.0 ± 60.2	0.84

[1]Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-TEN-20 tested at 20 °C)

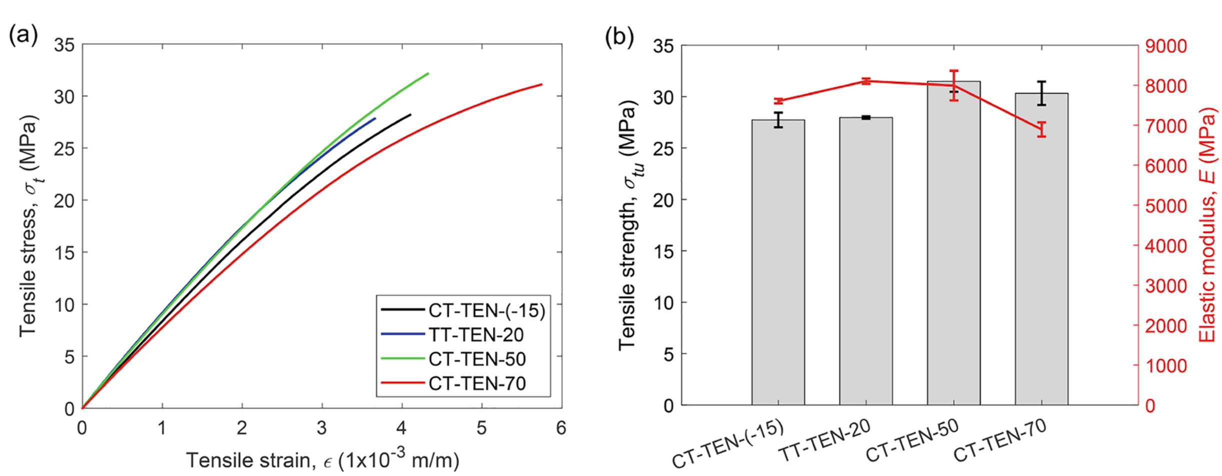

Fig. 11. Effect of curing temperature on tensile mechanical properties of the epoxy resin. (a) Representative stress–strain curves; (b) Evolution of tensile strength and elastic modulus.

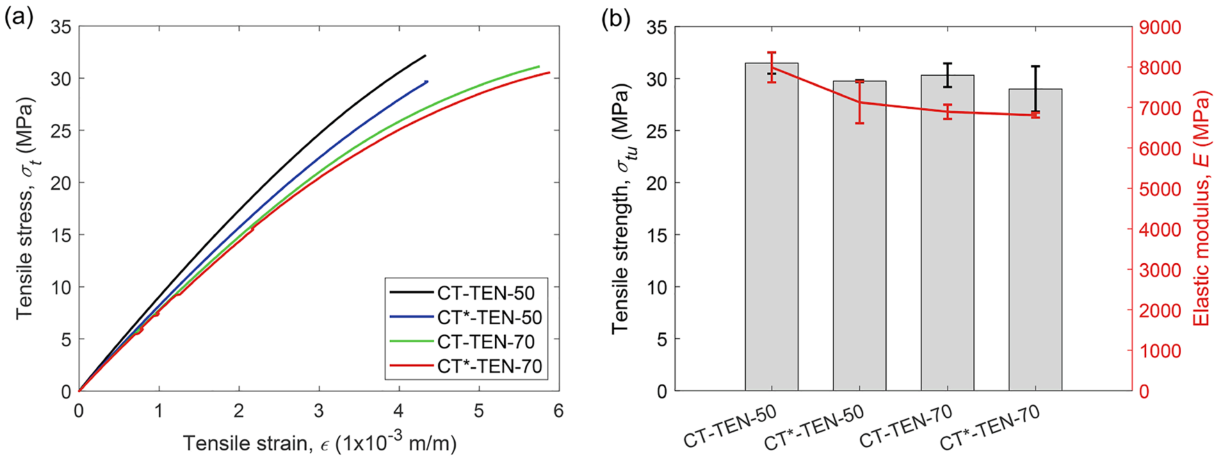

Fig. 12. Effect of additional post-curing on tensile mechanical properties of specimens in Group 3. (a) Representative stress–strain curves; (b) Evolution of tensile strength and elastic modulus.

adhesive used in this experimental program. It should be mentioned that a large scatter in experimental results on elastic modulus can be observed for the specimen with an additional post-curing (specimens CT*-TEN-50 and CT*-TEN-70). These results, along with experimental results on specimens of Group 2 having an additional post-curing process (TT*-TEN-50 and TT*-TEN-70), indicate that the application of a post-curing process at a temperature equal to that of the curing process produces no evolution in mechanical properties, because of the stability in the epoxy resin.

Compression tests were also performed to study the possible influence of curing temperature on the compressive mechanical properties of the epoxy resin. Results of these tests are presented in Table 6 and Fig. 13a. The increase in the curing temperature to values below epoxy T_g (i.e. up to 50 °C of the present experimental program) resulted in a monotonic increase of epoxy ultimate compressive load and compressive strength. On the other hand, when curing temperature was beyond epoxy T_g (i.e. 70 °C), a decrease in the compressive mechanical properties was observed. Besides, and according to results on additional specimens with one-day post-curing (specimens CT*-COM-50 and CT*-COM-70), no significant differences were observed between compressive mechanical properties of specimens with and without the post-curing process (see Fig. 13b.

3.4. Effect of post-curing temperature on mechanical properties

In this section, results on mechanical properties of specimens of Group 4 are presented and discussed, with the aim at analyzing the effect of a post-curing process at different temperatures (ranging from 20 °C to 85 °C) on mechanical properties of specimens that were previously cured at room temperature (i.e. 20 °C). Therefore, specimens TT-TEN-20 and TT-COM-20, from Group 2, are considered as the reference ones.

Experimental results on tensile properties of specimens in Group 4 are presented in Fig. 14 and Table 7, where the mean value and standard deviation are also included. As highlighted in the literature [12,29,30,39], epoxy adhesives cured at ambient temperature may suffer from a not complete cross-linking, that can be reactivated with a post-curing process. This was confirmed by experimental results of Group 4, as tensile strength monotonically increased with the increase in the post-curing temperature, and elastic modulus tended to a constant value. However, for post-curing temperatures larger than 60 °C, an abrupt decay in tensile strength and a more sharped reduction on elastic modulus were observed in specimens post-cured at 70 °C, thus indicating that $T_{g\infty}$ of the adhesive might be in the range of 60 °C to 70 °C. Finally, results on elastic modulus for specimens post-cured at 85 °C continued their downward trend, whilst a groundless increase in tensile strength was observed.

The effect of post-curing on compressive mechanical properties of the epoxy adhesive is shown in Fig. 15 and Table 8. From Fig. 15 it can be observed that the increase in the post-curing temperature from 20 °C to 40 °C resulted in an increase in the ultimate compressive load and compressive strength. On the other hand, for post-curing temperatures beyond 40 °C, a decrease in the compressive mechanical properties took place, with no meaningful changes and results falling within the same range of reference specimen (TT-COM-20).

Table 6
Ultimate compressive load and compressive strength for specimens cured at different temperatures (average ± standard deviation).

Specimens ID	Ultimate compressive load, F_{cu} (kN)	F_{cu} /F_{cu_20} [1]	Ultimate compressive strength, σ_{cu} (MPa)	σ_{cu} /σ_{cu_20} [1]
CT-COM-(−15)	120.2 ± 2.0	0.94	76.2 ± 1.5	0.95
TT-COM-20	128.4 ± 0.4	–	79.9 ± 0.1	–
CT-COM-50	128.7 ± 6.2	1.00	83.7 ± 3.6	1.05
CT*-COM-50	127.2 ± 5.4	0.99	83.2 ± 3.0	1.04
CT-COM-70	111.8 ± 1.8	0.87	70.7 ± 1.7	0.88
CT*-COM-70	107.9 ± 6.0	0.84	69.7 ± 2.5	0.87

[1]Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-COM-20 tested at 20 °C)

4. Conclusion

In the present work, a comprehensive experimental program was performed to evaluate the effect of temperature (i.e. testing temperature, curing temperature and post-curing temperature) on the mechanical properties and the T_g of an epoxy adhesive.

From the analysis of the effect of temperature on the measured T_g of the epoxy adhesive (specimens in Group 1), it can be concluded that curing and post-curing temperature affected T_g differently depending whether the applied temperature was below or beyond the epoxy $T_{g\infty}$.

From the analysis of the effect of testing temperature on mechanical properties of the epoxy adhesive (specimens in Group 2), the following conclusions can be drawn:

- The falling trend in tensile mechanical properties experienced the largest decrease when testing temperature exceeded the T_g of the epoxy adhesive. Besides, for testing temperatures beyond 60° C, due to complete rubbery state of the epoxy, the elastic modulus was

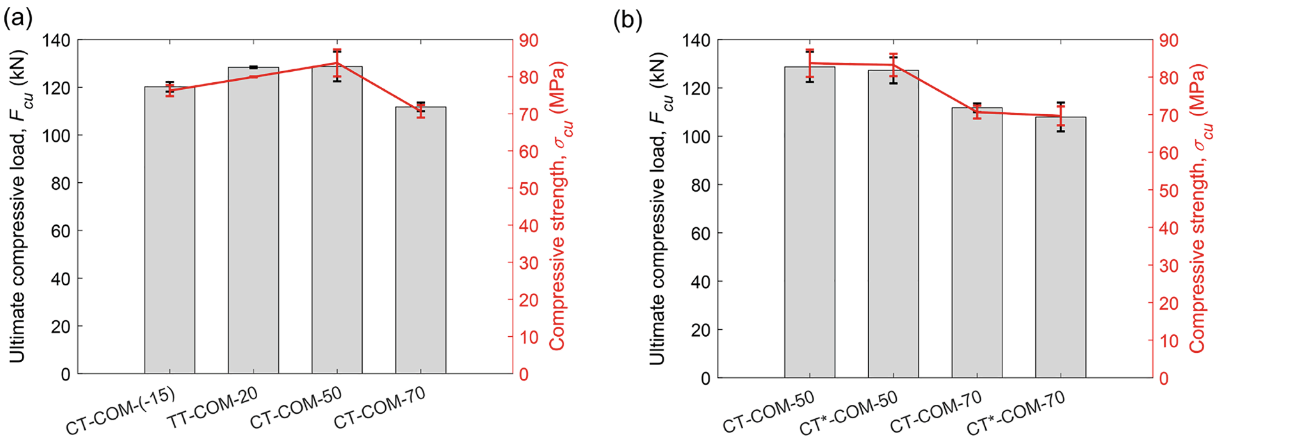

Fig. 13. (a) Effect of curing temperature on compressive mechanical properties of the epoxy resin; (b) Effect of additional post-curing on compressive mechanical properties of specimens in Group 3.

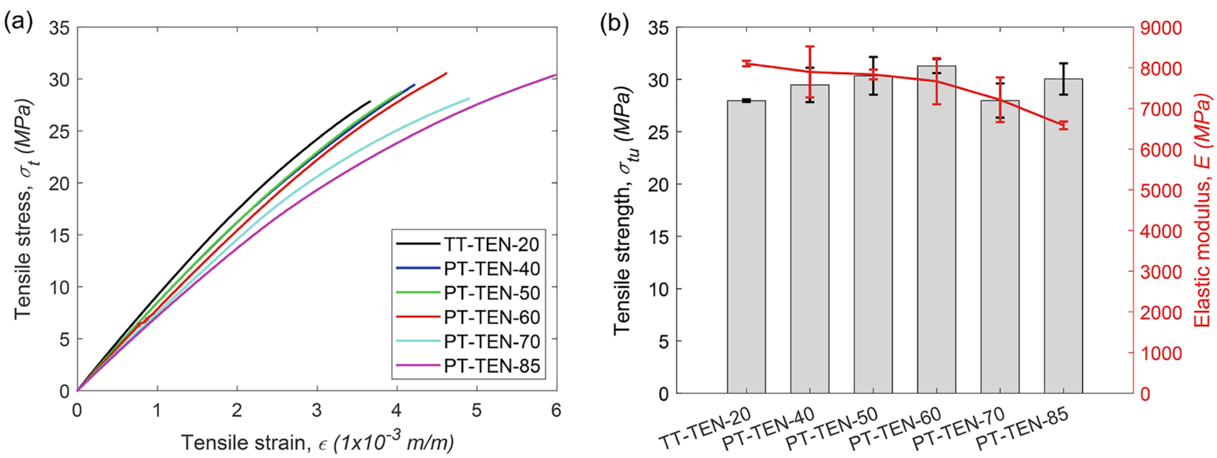

Fig. 14. Effect of post-curing temperature on tensile mechanical properties of the epoxy resin. (a) Representative stress–strain curves; (b) Evolution of tensile strength and elastic modulus.

Table 7
Tensile strength and elastic modulus for specimens post-cured at different temperatures (average ± standard deviation).

Specimens ID	Tensile strength, σ_{tu} (MPa)	$\sigma_{tu}/\sigma_{tu\_20}$ [1]	Elastic modulus, E (MPa)	$E/E_{\_20}$ [1]
TT-TEN-20	28.0 ± 0.1	–	8102.4 ± 67.0	–
PT-TEN-40	29.5 ± 1.6	1.05	7896.9 ± 625.5	0.97
PT-TEN-50	30.3 ± 1.8	1.08	7834.9 ± 119.8	0.97
PT-TEN-60	31.3 ± 0.7	1.12	7668.1 ± 566.6	0.95
PT-TEN-70	28.0 ± 1.6	1.00	7212.0 ± 548.1	0.89
PT-TEN-85	30.0 ± 1.5	1.07	6584.8 ± 95.3	0.81

[1]Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-TEN-20 tested at 20 °C)

almost negligible. For the compression tests, this large reduction took place at the temperature equal 70 °C.

- The inclusion of an extra cycle of post-curing barely affected the mechanical properties of epoxy specimens.

From the analysis of the effect of curing temperature on mechanical properties of the epoxy adhesive (specimens in Group 3), the following conclusions can be drawn:

- Curing temperatures below $T_{g\infty}$ resulted in larger mechanical properties because of the cross-linking promotion. On the other hand, curing temperatures beyond $T_{g\infty}$ produced a possible thermal degradation on the adhesive that resulted in a decrease of its mechanical properties.
- No significant effect of an additional post-curing process was observed. This may be due to stabilization of epoxy because of post-curing at the same curing temperature.

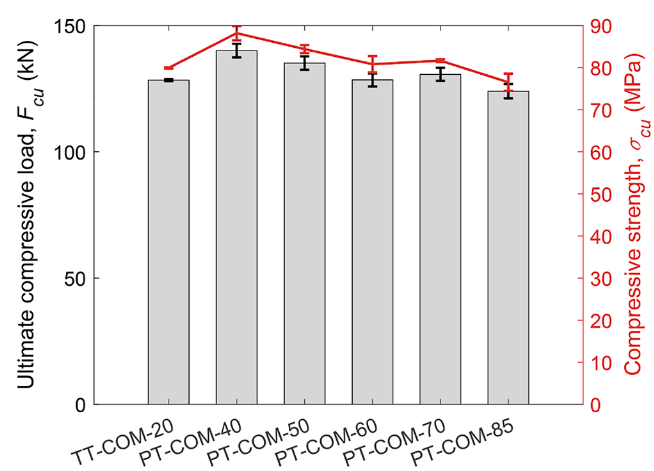

Fig. 15. Effect of post-curing temperature on compressive mechanical properties of the epoxy.

Table 8
Ultimate compressive load and compressive strength for specimens post-cured at different temperatures (average ± standard deviation).

Specimens ID	Ultimate compressive load, F_{cu} (kN)	F_{cu} $/F_{cu\_20}$ [1]	Ultimate compressive strength, σ_{cu} (MPa)	σ_{cu} $/\sigma_{cu\_20}$ [1]
TT-COM-20	128.4 ± 0.4	–	79.9 ± 0.1	–
PT-COM-40	140.1 ± 2.7	1.09	88.2 ± 1.7	1.10
PT-COM-50	135.1 ± 2.6	1.05	84.2 ± 1.0	1.05
PT-COM-60	128.4 ± 2.6	1.00	80.8 ± 1.9	1.01
PT-COM-70	130.6 ± 2.6	1.02	81.7 ± 0.3	1.02
PT-COM-85	124.0 ± 2.8	0.97	76.5 ± 2.1	0.96

[1]Defined as the ratio between the property value under the specific curing process and the reference property value (i.e. TT-COM-20 tested at 20 °C)

From the analysis of the effect of post-curing temperature on mechanical properties of the epoxy adhesive (specimens in Group 4), the following conclusions can be drawn:

- Post-curing the epoxy at a temperature below the temperature that leads to a fully cured network (near to $T_{g\infty}$) enhanced the mechanical properties because of the reactivation of cross-linking of the non-complete cured epoxy adhesive.
- The largest benefit of post-curing on epoxy compressive strength took place for post-curing temperature equal to 40 °C.

The increase in the mechanical properties of epoxy at curing or post-curing temperatures below or near to the T_g of epoxy can be attributed to the fact that the degrees of cross-linking between the epoxy's molecules has increased, thus having the consequence of an increase in T_g.

CRediT authorship contribution statement

Younes Jahani: Conceptualization, Methodology, Validation, Investigation, Data curation, Writing – original draft. **Marta Baena:** Conceptualization, Validation, Formal analysis, Writing – review & editing, Supervision, Project administration, Funding acquisition. **Cristina Barris:** Formal analysis, Writing – review & editing, Project administration, Funding acquisition. **Ricardo Perera:** Formal analysis, Writing – review & editing, Project administration, Funding acquisition. **Lluís Torres:** Validation, Formal analysis, Writing – review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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