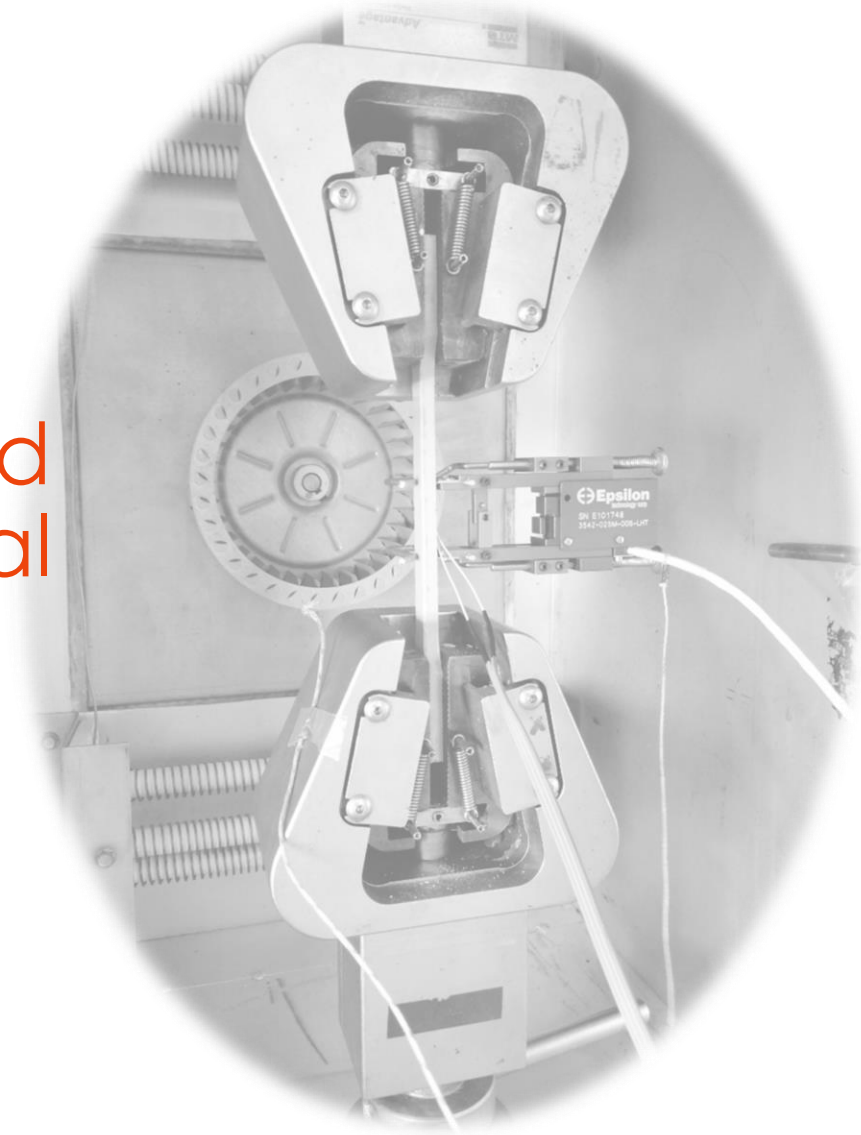


# Influence of curing, post-curing and testing temperatures on mechanical properties of a structural adhesive

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Supervisor: Dr. MARTA BAENA  
Girona, July 2022





# Contents

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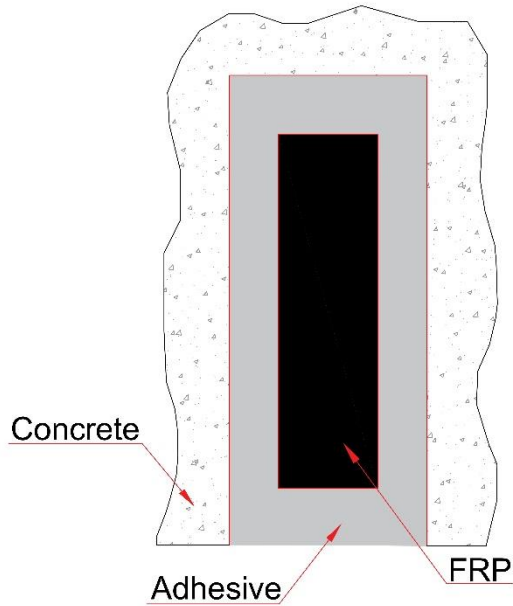
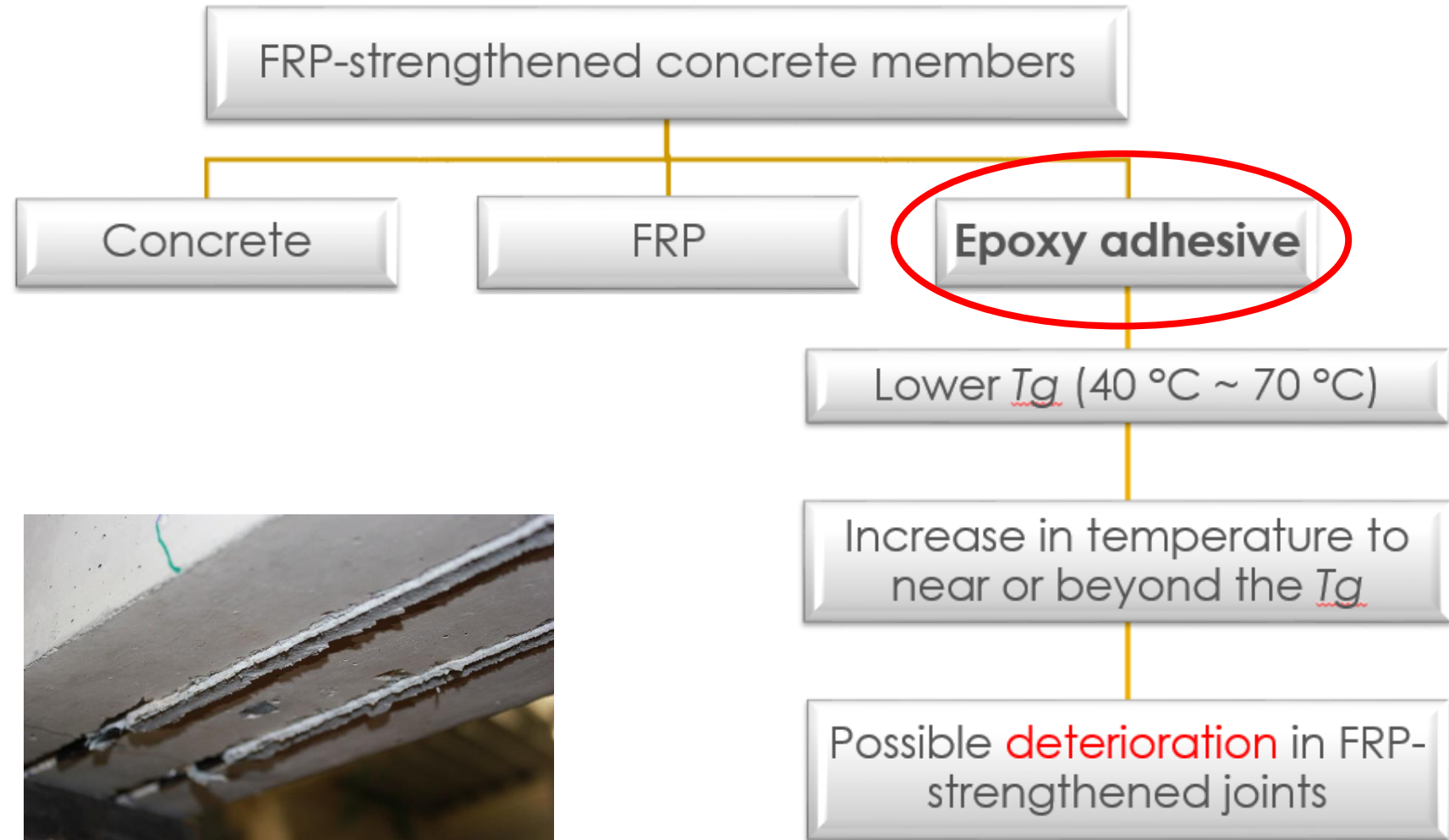
Introduction and motivation.....	3
Experimental program.....	5
Results and discussion.....	11
Conclusion.....	18



# Introduction and motivation

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## Introduction and motivation



NSM FRP-strengthened joint



End-debonding at 70 °C



# Experimental program

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## Test matrix

- **Group 1:** Effect of curing and post-curing temperature on the  $T_g$  of epoxy adhesive (5 specimens cured (post-cured) at 20 °C, 50 °C and 70 °C to measure the  $T_g$ ).
- **Group 2:** Effect of testing temperature on mechanical properties of epoxy adhesive (48 specimens tested at 20 °C, 40 °C, 50 °C, 60 °C, 70 °C and 85 °C).
- **Group 3:** Effect of curing temperature on mechanical properties of epoxy adhesive (30 specimens cured (post-cured) at -15 °C, 50 °C and 70 °C and later tested at 20 °C).
- **Group 4:** Effect of post-curing temperature on mechanical properties of epoxy adhesive (30 specimens post-cured at 40°C, 50 °C, 60°C, 70°C, and 85 °C and later tested at 20 °C).



**S&P Resin 220 HP**

# Test matrix details

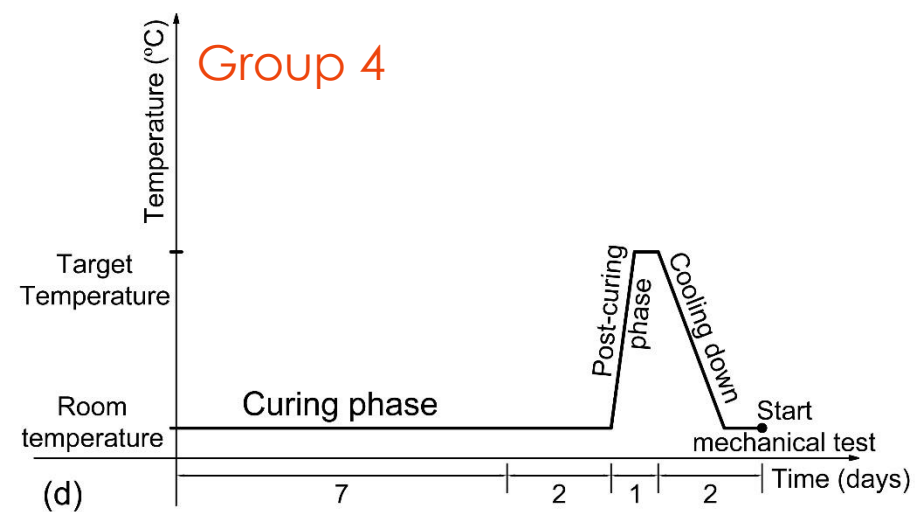
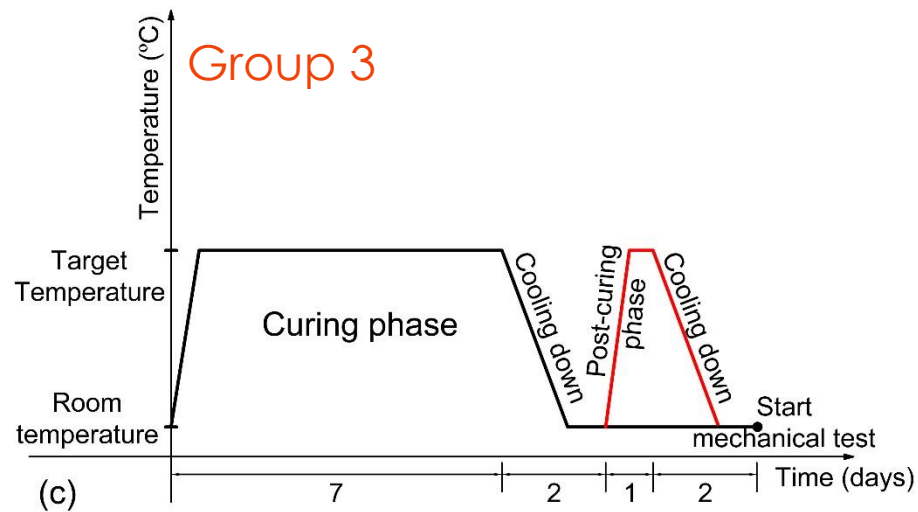
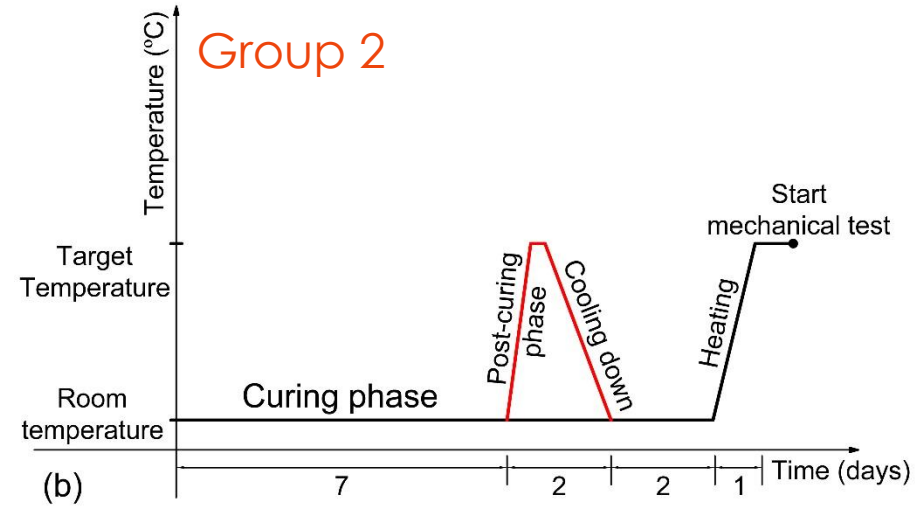
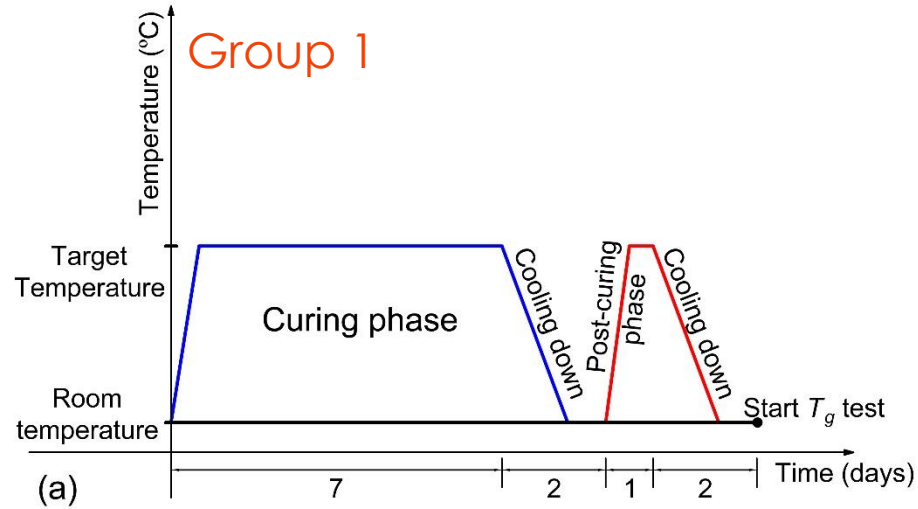
Group	Specimen ID	Number of specimens	Curing process temperature (°C)	Test	Testing temperature (°C)
Group 1	TG-C-20	1	20	Glass transition temperature of epoxy	According to standard
	TG-C-50	1	50		
	TG-C-70	1	70		
	TG-PC-50	1	50		
	TG-PC-70	1	70		
Group 2	TT-TEN-20	3	20	Tensile strength and elastic modulus	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		

# Test matrix

Group 3	CT-TEN-(-15)	3	-15	Tensile strength and elastic modulus	20
	CT-TEN-50	3	50		20
	CT*-TEN-50 <sup>1</sup>	3	50		20
	CT-TEN-70	3	70		20
	CT*-TEN-70 <sup>2</sup>	3	70		20
	CT-COM-(-15)	3	-15	Compressive strength	20
	CT-COM-50	3	50		20
	CT*-COM-50 <sup>1</sup>	3	50		20
	CT-COM-70	3	70		20
	CT*-COM-70 <sup>2</sup>	3	70		20
Group 4	PT-TEN-40	3	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

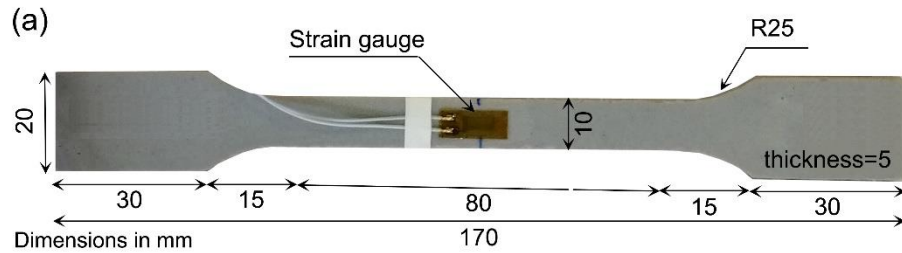


# Curing process

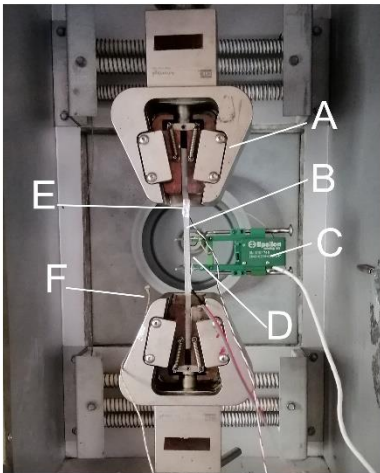


# Test setup

## Tension tests (ISO 527-1)



(b)



Legend:

- A) Tensile test grips
- B) Dog-bone specimen
- C) Axial extensometer
- D) Strain gauge
- E) Thermocouple on the surface of the specimen
- F) Thermocouple for registers of environmental temperature

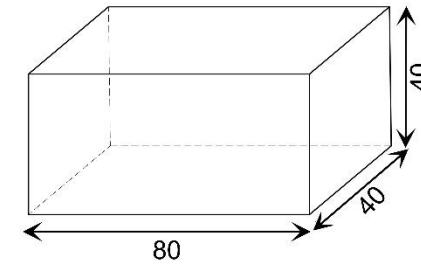
## Compression tests (EN 196-1)

(a)

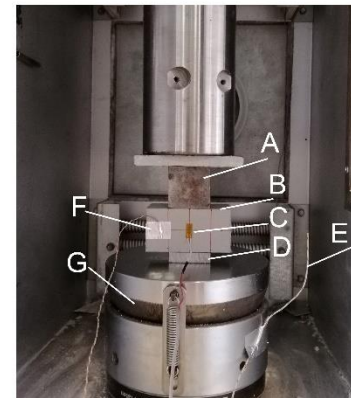


Dimensions in mm

(b)



(c)



Legend:

- A) Upper plate
- B) Specimen
- C) Strain gauge
- D) Lower plate
- E) Thermocouple for registers of environmental temperature
- F) Thermocouple on the surface of the specimen
- G) Spherical hinge

Tg tests: based on the differential scanning calorimetry (ASTM E1356-08 )

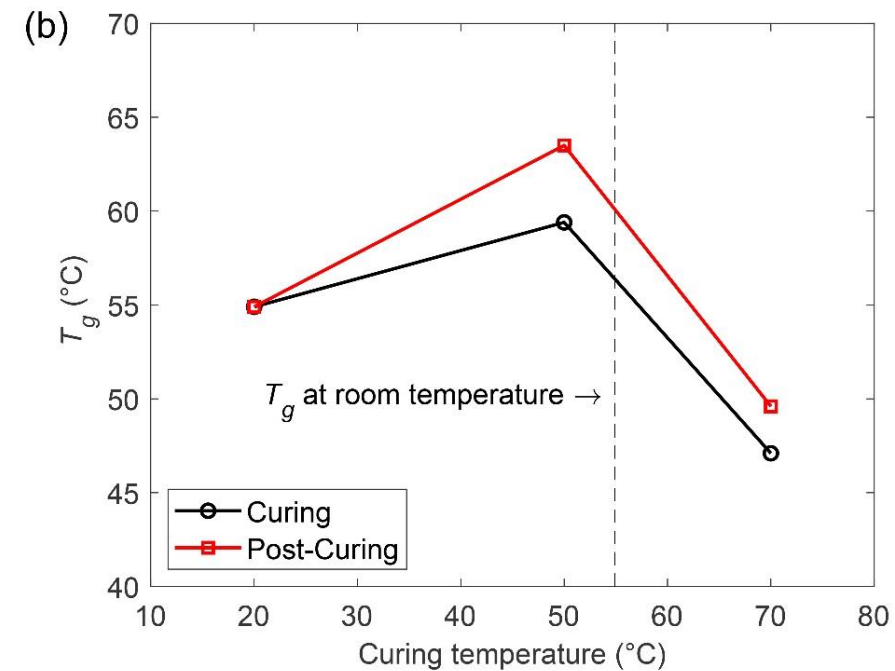
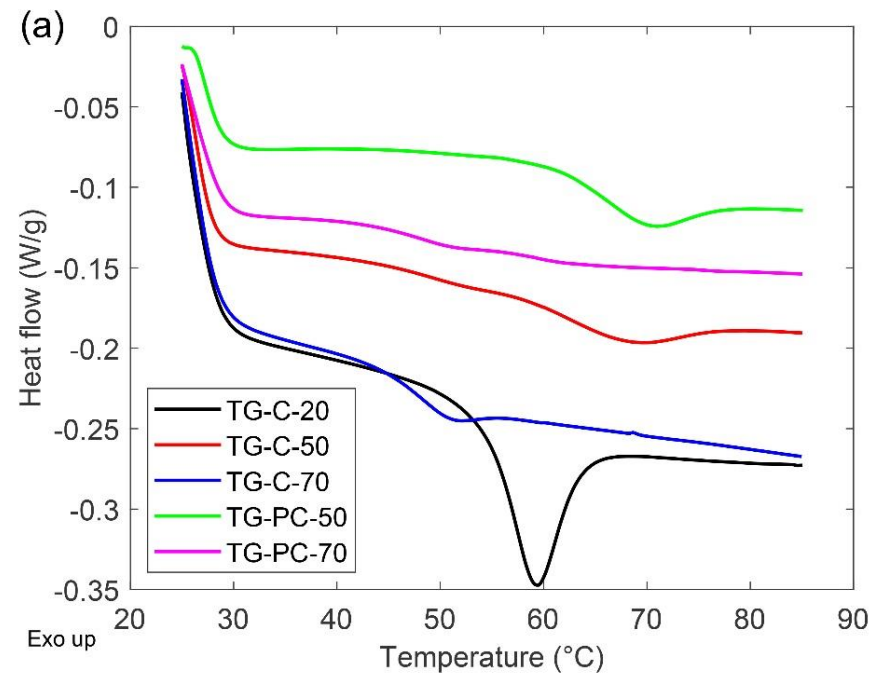


# Results and discussion

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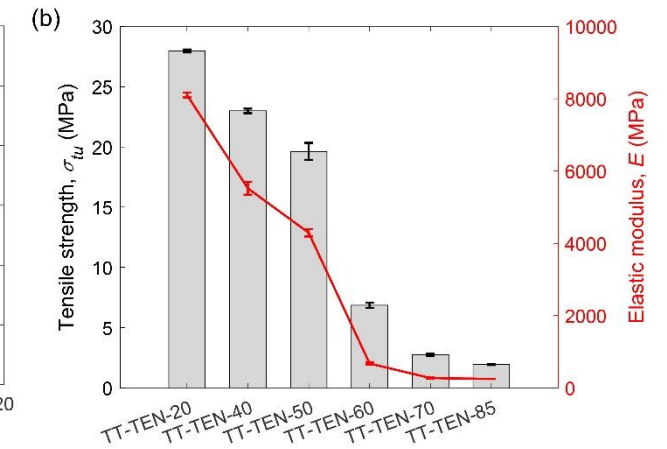
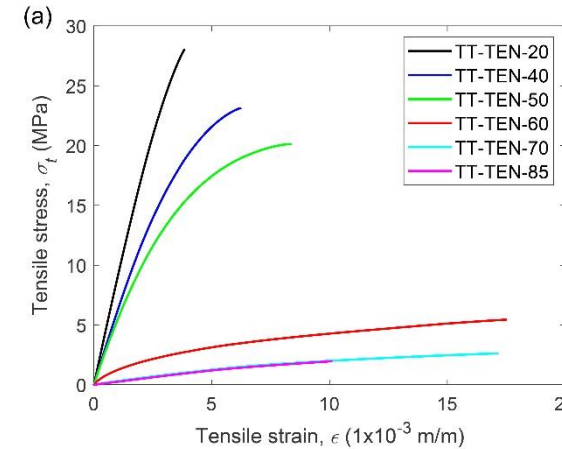
## Effect of curing and post-curing temperature on $T_g$

- ❑ Curing or post-curing temperature (i.e. 50 °C) <  $T_g=54.9$  °C →  $T_g$  increased.
- ❑ Curing or post-curing temperature (i.e. 70 °C) >  $T_g=54.9$  °C →  $T_g$  decreased.
- ❑ There is an upper bound value corresponds to the glass transition temperature of the fully cured network ( $T_g^\infty$ ).
- ❑  $50$  °C <  $T_g^\infty$  <  $70$  °C

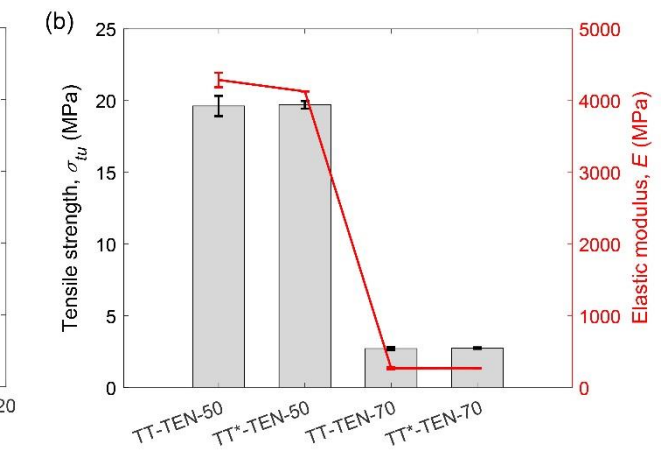
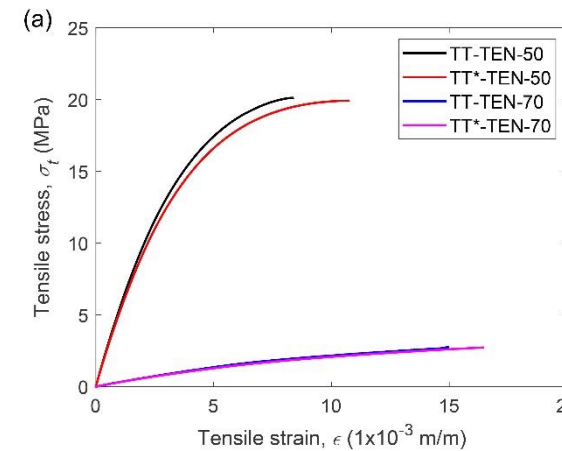


# Effect of testing temperature on tensile mechanical properties

- ▣ Increase in testing temperature → Tensile strength and elastic modulus decrease
- ▣ The large reduction observed at the temperature beyond the  $T_g$  (60 °C).

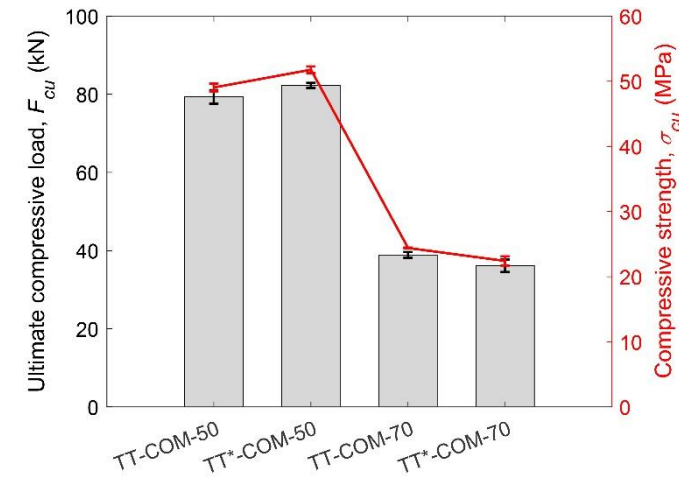
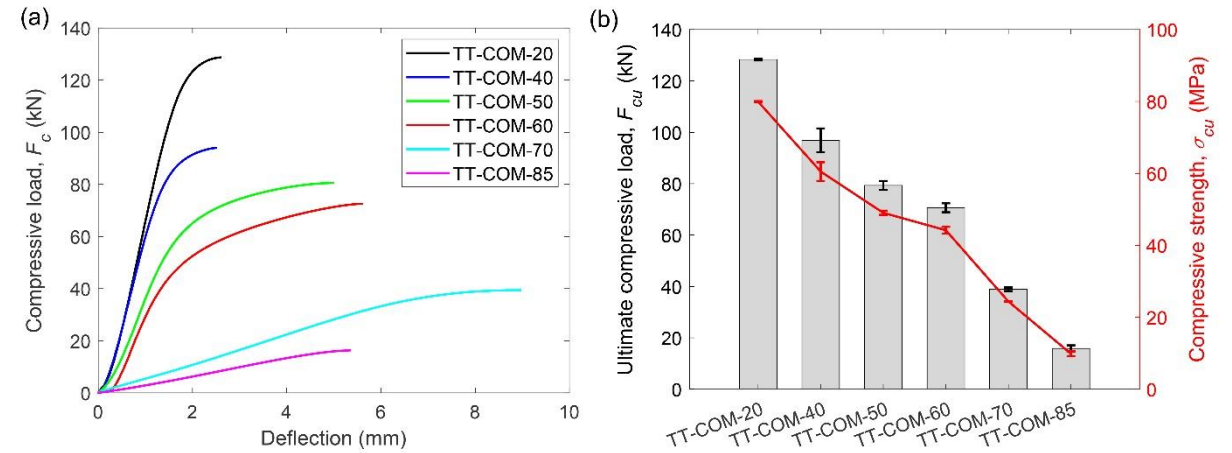


- ▣ Second cycle of post-curing (at the same testing temperature) did not have any effect on the tensile mechanical properties.



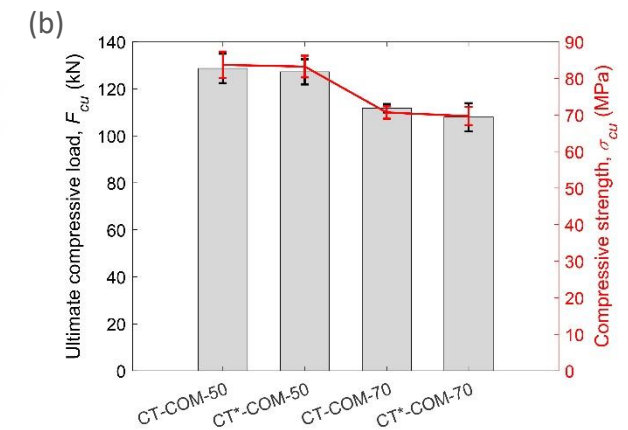
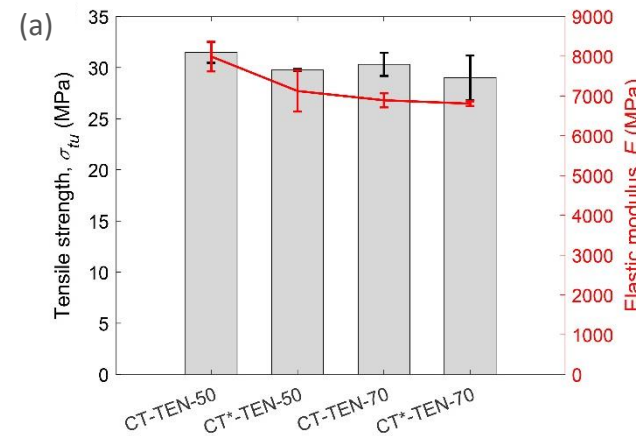
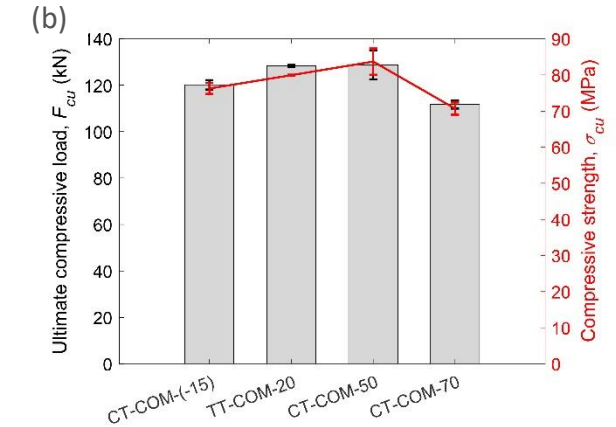
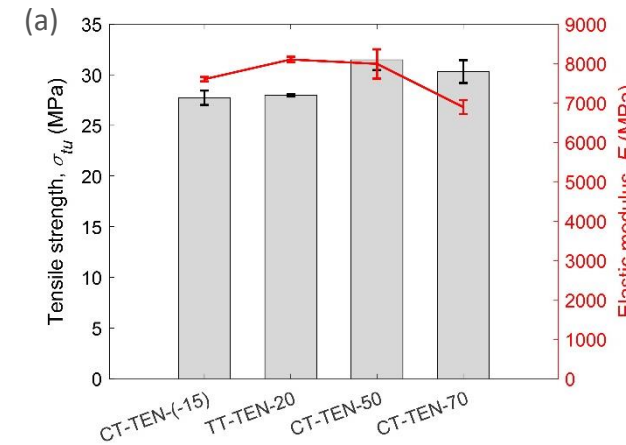
# Effect of testing temperature on compressive mechanical properties

- ▣ Increase in testing temperature → Compressive strength decreases
- ▣ The large reduction observed at the temperature beyond the  $T_g$  (70 °C).
- ▣ Second cycle of post-curing (at the same testing temperature) had a slight effect on the compressive mechanical properties.



# Effect of curing temperature on mechanical properties

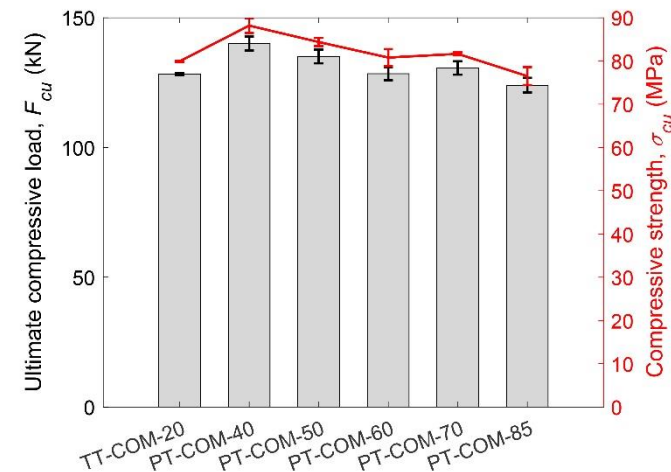
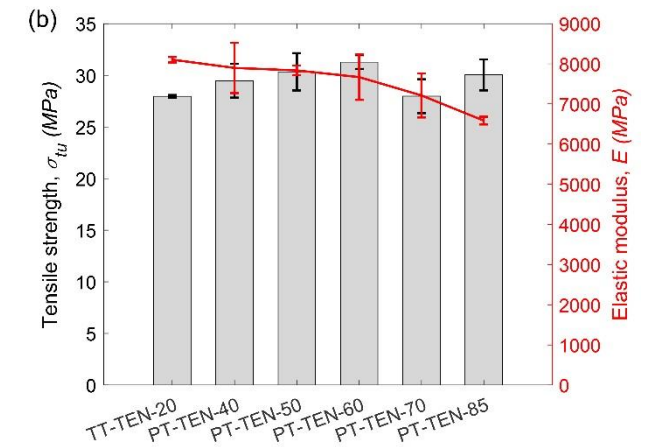
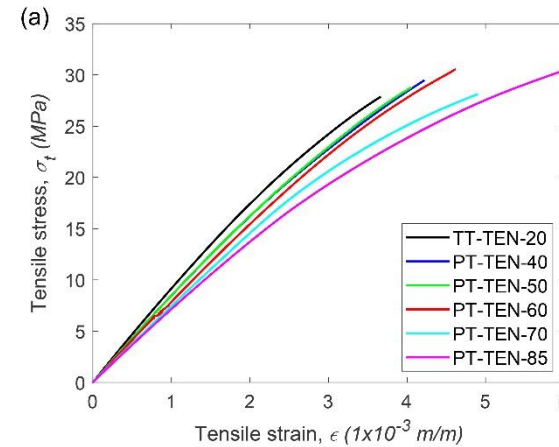
- 📌 Curing at  $-15\text{ }^{\circ}\text{C}$   $\rightarrow$  no considerable effect on mechanical properties
- 📌 Curing at  $-15\text{ }^{\circ}\text{C}$   $\rightarrow$  curing did not initiated (epoxy was soft)  $\rightarrow$  Suitable considerations should be taken into account in the cold weather regions
- 📌 Curing temperature (i.e.  $50\text{ }^{\circ}\text{C}$ )  $< T_g^{\infty}$   $\rightarrow$  cross linking promotion  $\rightarrow$  mechanical properties increase.
- 📌 Curing temperature (i.e.  $70\text{ }^{\circ}\text{C}$ )  $> T_g^{\infty}$   $\rightarrow$  possible thermal degradation  $\rightarrow$  mechanical properties decrease.
- 📌 Post-curing process (at same curing temperature) did not highly affect the mechanical properties of the adhesive.





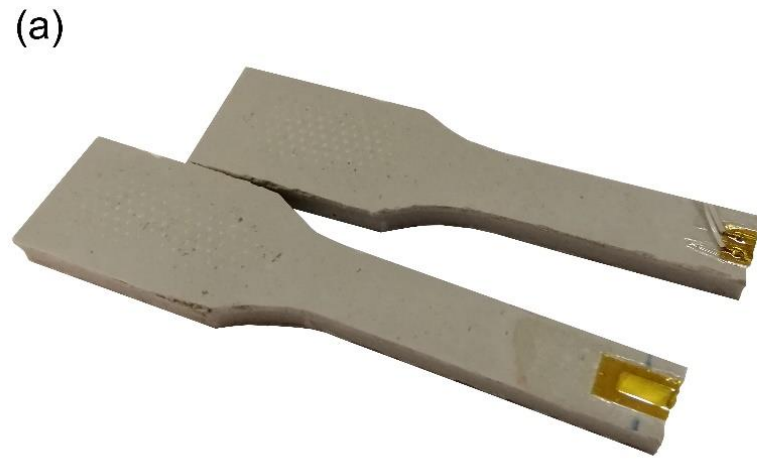
# Effect of post-curing temperature on mechanical properties

- Increase in the post-curing temperature up to 60 °C → Reactivation of cross-linking → Tensile strength increased
- For post-curing temperatures of 70 °C, a reduction in tensile strength and modulus was observed.
- The  $Tg_{\infty}$  of the adhesive might be in the range of 60 °C to 70 °C.
- Post-curing temperature at 40 °C → largest benefit in the ultimate compressive load
- Beyond 40 °C, a decrease in the compressive mechanical properties took place.





# Typical failure modes



Tensile failure mode



Compressive failure mode



# Conclusions

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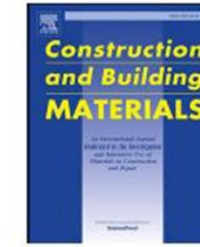
- ❑ Curing and post-curing temperature affected  $T_g$  differently. In other words, with increase in the curing (or post-curing) temperature up to the  $T_g^\infty$ , the  $T_g$  of the epoxy increased. On the other hand, for the curing (or post-curing) temperature beyond the  $T_g^\infty$ , the  $T_g$  of the epoxy decreased.
- ❑ With increase in the testing temperature, mechanical properties of epoxy adhesive were decreased. In this sense, a sharp reduction was observed at the temperature near or beyond the  $T_g$  of the epoxy adhesive and due to complete rubbery state of the epoxy, the elastic modulus was almost negligible.
- ❑ The inclusion of an extra cycle of post-curing barely affected the mechanical properties of epoxy specimens.
- ❑ Curing temperatures below  $T_g^\infty$  improved the mechanical properties because of the cross-linking promotion. On the other hand, curing temperatures beyond  $T_g^\infty$  resulted in a reduction in the mechanical properties due to a possible thermal degradation on the adhesive.
- ❑ No considerable effect of an extra post-curing was observed, which was due to stabilization of epoxy because of post-curing at the same curing temperature.
- ❑ Similar to curing temperature, post-curing the epoxy at a temperature below the  $T_g^\infty$ , 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 tensile strength took place for post-curing temperature equal to 60 °C, whilst for compressive strength it happened at 40 °C.



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

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Post-curing

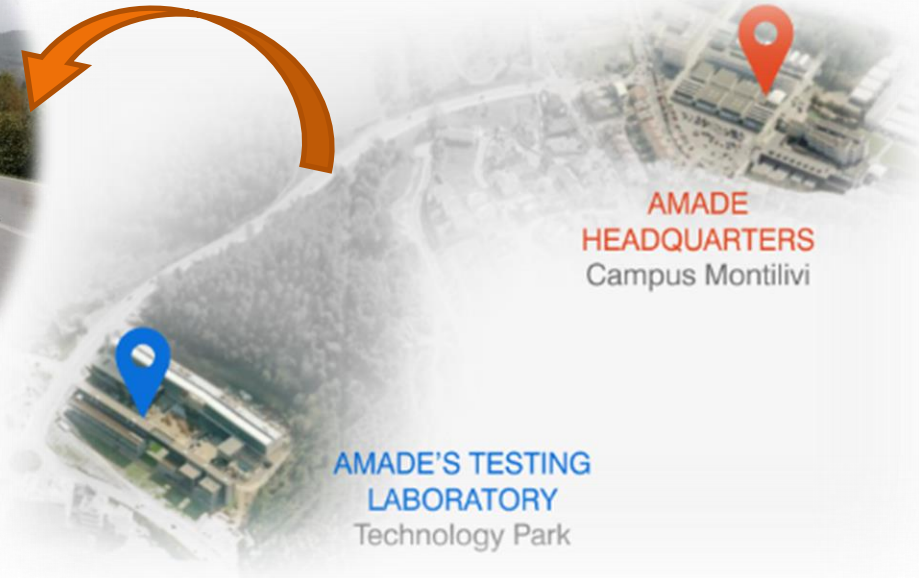
$T_g$

Mechanical properties

#### 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.

# Thanks for your attention



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