Effect of thermal aging on shear strength of concrete-epoxy interface in extreme moisture conditions

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Abstract
A series of shear strength measurements are carried out, to study the effect of curing temperature and the effect of thermal ageing on the bond strength of concrete-epoxy interface. The results show that the curing temperature and water immersion play an important role. At early age, the shear strength of the samples cured at 40°C in air for 24h is more than twice the one at 20°C. The samples treated at 80°C in air could maintain a strong interface strength until 32 days, however, a drop due to interface debonding is observed for the samples treated at 80°C in water for 16 days.

Mots-clefs Époxy, béton, interface, vieillissement thermique, résistance au cisaillement

Key-words Epoxy, concrete, interface, thermal aging, shear strength

I. INTRODUCTION

Epoxy resin are widely used in concrete repair and structural reinforcement, thanks to its fast curing, wide application temperature range, good adhesion with concrete and excellent interface bond strength. When composite materials interact with the environment, the matrix oxidation involves a weight loss and a density increase and as a consequence, a shrinkage of the skin layer of the material [Decelle et al., 2003]. A higher temperature of curing reduces the time required to achieve a higher glass transition temperature (T_g) value while guaranteeing a high degree of crosslinking, who plays an important role in the mechanical performance of the material [Michel et Ferrier, 2020]. On the contrary, the tensile strength and stiffness of FRP (fiber-reinforced polymer) sheets or plates drop significantly once the temperature exceeds their T_g due to the accelerated softening of the polymeric matrix [Wang et al, 2021]. Moreover, the effectiveness of entire strengthening or retrofitting scheme highly depends on the bond performance of concrete-epoxy interface, where moisture plays an important role on durability. Zhou et al. [2017] conducted a comprehensive experiment focusing on the coupled effect of sustained load and moisture on the bond property of concrete-epoxy interface, and observed a drastic deterioration of interfacial fracture toughness up to 77%; Lau and Büyüköztürk [2010] obtained a significant decrease of interface fracture toughness affected by moisture, up to about 50%; with the increase of aging duration to 120 days under wet-dry cycles in NaCl solution, the tensile strength and the elastic modulus of epoxy primer had substantially decreased, which would lead to a reduction of the interface fracture energy at the CFRP-concrete bonding interface [Cui et al., 2021]. The research of Daneshvar et al. [2021] shows that the casting and curing temperature dramatically impacts the mechanical properties of the epoxy bonded concretes by wedge splitting and bi-surface shear tests. The interface debonding and the degradation of bonding adhesives play an important role in dominating the bond failure modes and performance degradation of FRP-strengthened concrete members [Wang et al, 2021].

The objective of this work is to study the effects of cure temperature, thermal aging and water aging on interfacial bond strength by measuring its shear strength, it provides a simple and intuitive method for evaluating concrete-epoxy interface durability, and may benefit the engineering application of concrete reparation and reinforcement.

II. MATERIAL AND METHODES

A. *Preparation of samples*

The commercial epoxy supplied by society SPPM for the research project ANR DUREVE (ANR-18-CE06-0028), and a steel fiber reinforced concrete UHPRFC (R_c=150MPa) were used in this study. The list of samples is shown in Table 1, including the number of samples, curing/treatment condition and time, for the different tests.

TABLE 1. Samples and curing/treatment condition

Test	Sample	Number	Curing/treatment	Time (d)
Mass loss/ Shrinkage	Epoxy disk	3	80°C Air	1/2/4/8/12/16/32/48/64
		3	80°C Water	1/2/4/8/12/16/32/48/64
Shear strength-effect of curing temperature	Concrete-epoxy	4	20°C Air	1/4/8/16
		4	40°C Air	1/4/8/16
Shear strength-effect of thermal ageing	Concrete-epoxy	4	80°C Air	4/8/16/32
		4	80°C Water	4/8/16/32

6 epoxy thick disk samples of 65mm in diameter and 0.7mm in thickness were made for the measurement of mass loss and shrinkage, stored in an oven at 80°C and in a water bath at 80°C for 1, 2, 4, 8, 12, 16, 24, 32, 48 and 64 days. We define ΔD/D as shrinkage rate and ΔM/M as mass loss rate.

For shear strength, the dimension of the cylindrical concrete BFUP samples was 65mm×25mm (diameter height), three holes evenly distributed of 10mm in diameter were drilled on each sample, and finally 1g of epoxy was injected in each hole to form with the help of a silicone cylinder (10mm×5mm) placed at the bottom of the hole, a thin epoxy cylinder of diameter 10mm, and thickness about 3mm (see Fig.1b). To evaluate the effect of curing temperature, measurements were performed on 4 samples cured in the oven at 20°C and 4 samples cured at 40°C, after 1, 4, 8, 16 days. Furthermore, 4 samples treated in the oven at 80°C and 4 samples treated in the water bath at 80°C, were tested after 4, 8, 16, 32 days, to evaluate the effect of thermal ageing and water ageing. The samples treated in water were wiped and dried in the oven at 80°C for 1 hour, and naturally cooled before measuring.

B. *Shear strength test of interface*

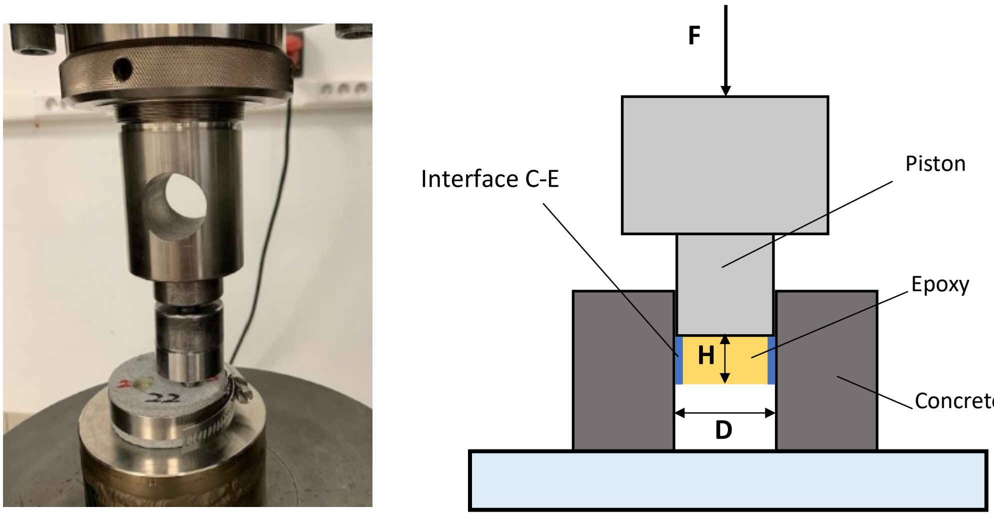
The shear strength measurements of interface were performed with a Zwick/Roell Z250 press. A cylindrical stainless-steel piston with a bottom diameter D of 9.9 mm was employed to push the epoxy cylinder under the axial loading (Fig.1a). The loading rate was 0.12mm/min. According to the schematic diagram shown in Fig.1b, a cylindrical shear plane of interface is obtained and then the shear strength (τ) can be calculated as:

$$\tau = \frac{F}{\pi D H} \quad (1)$$

F - peak force

D - diameter of epoxy sample

H - height of epoxy sample



(a) (b)

FIGURE 1. Shear strength test device and schematic diagram

C. *Permeability measurement*

In order to evaluate the permeability properties of epoxy coatings, the apparatus detailed in [Quelennec et al, 2023] was used (Fig.2). The difference lies in the addition of a diffuser on either side of the test sample, which provides a better lateral seal. The principle is to measure the evolution of the pressure in the downstream reservoir (R2) to compute the flow going throw the specimen. The permeability evolution is seen as an indicator of the microstructure evolution and consequently of the eventual ageing of the material.

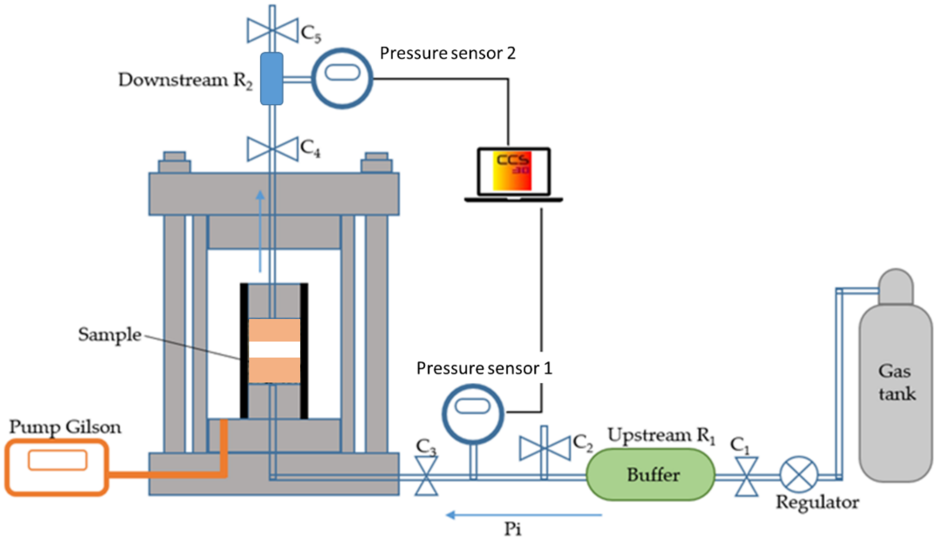


FIGURE 2. **Experimental device for permeability measurement**

III. RESULTS

A. Mass loss and shrinkage

The mass loss rate and shrinkage rate from 0 to 64 days are respectively shown in Fig. 3 and Fig.4. We observed that both of the samples treated in air at 80°C (group 1) and in water at 80°C (group 2) had rapid mass loss in the early days (Fig. 3), with almost the same average mass loss rate about 4.2% after 8 days. Afterwards, mass loss kinetic of group 2 significantly decreased, and after 16 days their mass was stabilized. In the same time, samples of group 1 presented a mass loss evolution reaching about 6.3% after 64 days. The shrinkage of group 2 is faster than case 1, but from 16 days the shrinkage rate remains stable at around 1.6% (see Fig. 4), whereas group 1 continues to shrink and reaches 2.0% at 64 days. Clearly, the presence of water inhibits further mass loss and shrinkage. We can more clearly observe a linear relationship between shrinkage rate and mass loss rate in average value for these two aging conditions from Fig. 5, and the shrinkage rate and mass loss rate of aging in air are greater than those of aging in water after 64 days, about 25%.

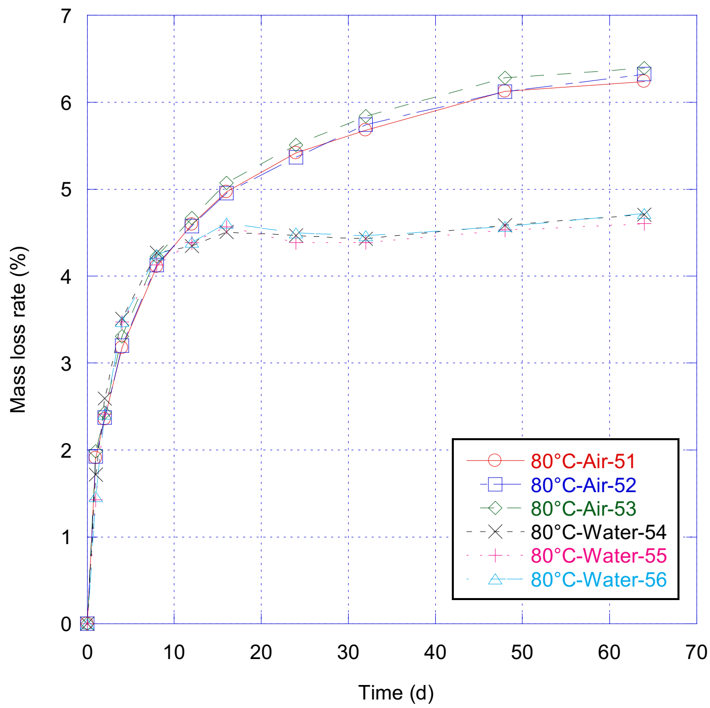


FIGURE 3. Mass loss in water and in air at 80°C

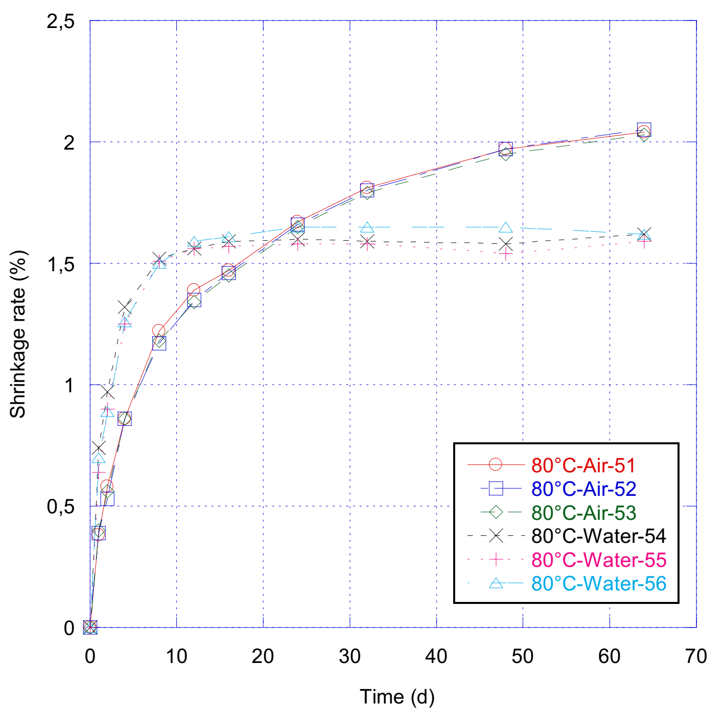


FIGURE 4. Shrinkage in water and in air at 80°C

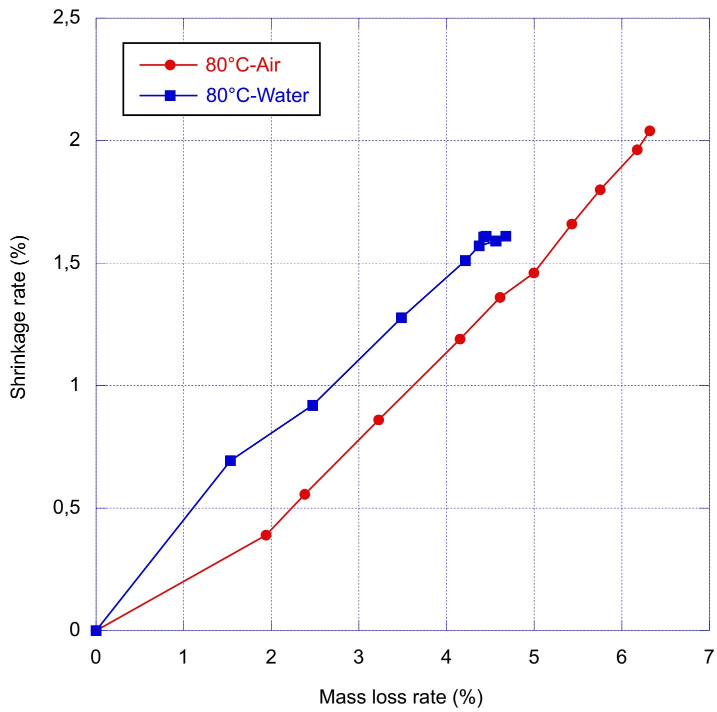


FIGURE 5. **Correlation between shrinkage rate and mass loss rate**

B. Shear strength of interface

For 3 samples cured at 20°C, an average shear strength of 11.38MPa is obtained at 1 day, and it increases progressively to 23.79MPa (+109%) at 16 days, see Fig. 6. In comparison, it reaches a value of 25.95MPa from 1 day, i.e. more than twice as high as that at 20°C, which is maintained until 16 days, in the case of curing at 40°C. For reference, the Fig. 6 also shows the results obtained by Yu et al. [2020] in similar studies, the average of shear strengths 8.16MPa for the specimens no treatment and unconstrained, cured at room temperature for 7 days. As we know, crosslinking plays an important role in the mechanical performance, the stronger the crosslinking effect, the stronger the corresponding mechanical strength. Further, T_g consistently increase with the curing temperature, owing to the crosslinking, it can be confirmed by the study of Michel and Ferrier [2020], a higher T_g about 70°C under a curing temperature of 40°C for 14 days, against a T_g about 50°C for 20°C. Therefore, we get naturally a stronger mechanical property of interface at early age, with a higher T_g obtained. The results of shear strength obtained show clearly the importance of curing temperature on the interfacial strength.

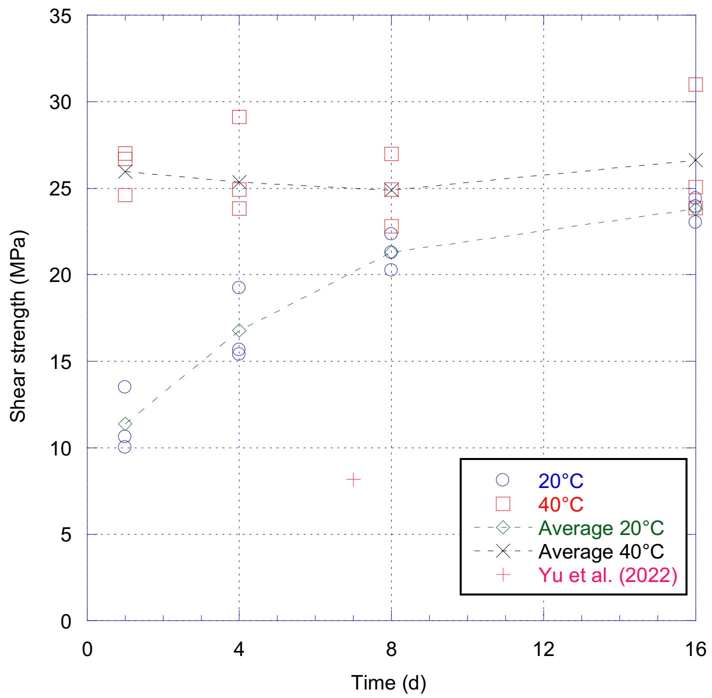


FIGURE 6. **Effects of cure temperature**

The results of shear strength interface evolution due to thermal and moisture ageing effect are displayed on Fig. 7. One can remark that at 4 days the average shear strengths for three different treatment conditions are in the following order: case 1-80°C in air (30.75MPa) > case 2-80°C in water (19.38MPa) > case 3-20°C in air (16.77MPa). Afterwards the samples treated at 80°C in air could maintain a strong strength until 32 days (31.45MPa). However, a partial interface debonding is observed for two of the three samples treated at 80°C in water at16 days, which leads to the very weak shear strengths obtained (1.54 and 3.09MPa). At 32 days, all three samples present an important debonding.

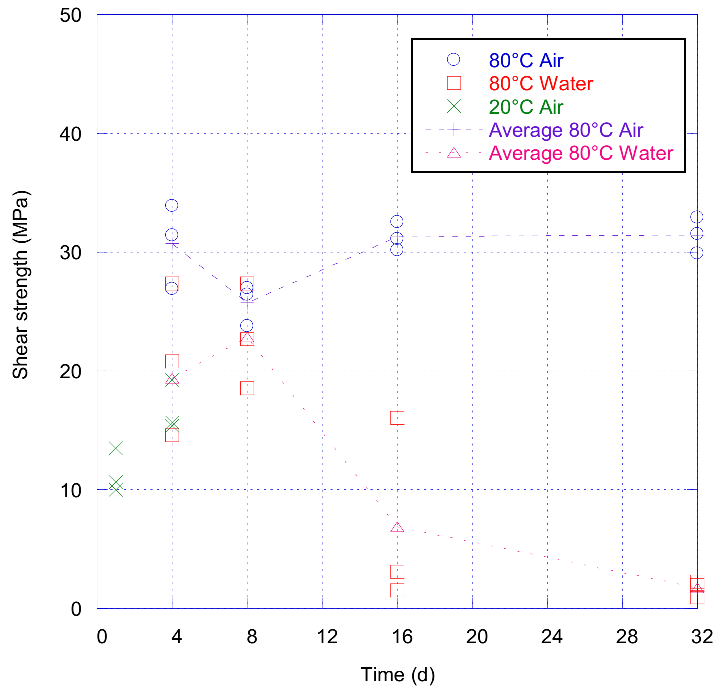


FIGURE 7. Effect of thermal ageing and water immersion

Review the results shown in Fig. 4, although case 1 and case 2 have a similar shrinkage rate about 1.5% at 16 days, case 1 could maintain a good bond with concrete and interface integrity, we note that there is no thermal aging effect for 80°C in air treatment within 32 days, on contrary with the ones treated at 80°C in water. Although the shrinkage is greater in air at 80 °C than in water, a greater decrease in interfacial resistance is observed for samples treated in water. Therefore, there is a cross effect between the amplitude of the shrinkage and the presence of water (Luo et al. [2020]). At a first glance, one can suspect that the chemical reaction of epoxy with water (thermoactivated hydrolyze) will lead to a microstructural evolution of the epoxy (especially at a temperature above the glass transition temperature) but shrinkage measurements in air exhibit higher value. Visible debonding between the epoxy and concrete for specimens cured in water (explaining the very low shear stress) are deemed also to be a consequence of a loose of adhesion due to chemical interaction with concrete (re-saturation of concrete lead to an interstitial solution with a pH closed to 12 which is clearly deleterious for the tensile strength of the interface). Indeed, during curing, shrinkage of the epoxy will produce tensile strength normal to the interface which can lead to progressive failure of the interface.

C. *Consequences of the thermal aging on permeability properties*

On the Fig. 8, one can see the evolution of the permeability for epoxy immersed in water during up to 30 days. Permeabilities measurements remains very low and only very small evolution from the initial value to 3 days (slight decrease) and from 3 days to 30 days (slight increase) cannot be related to an evolution of the microstructure and an eventual degradation of the material. It is worth noting that permeabilities as low as 10^{-23} m^2 must be interpreted carefully and must be taken as an upbound. A period of 30 days in water did not significantly modify the overall permeability and microcracks of important modification of the microstructure of the material can be dismiss.

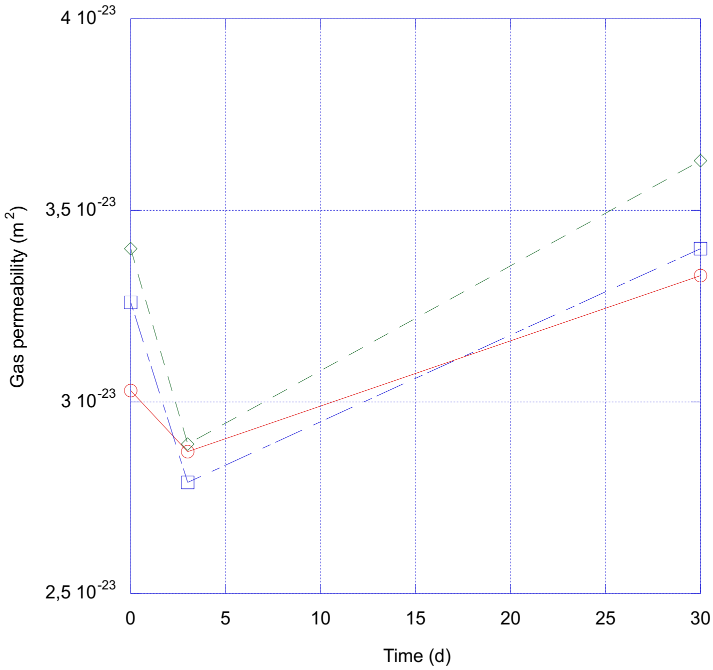


FIGURE 8. Evolution of epoxy's permeability according a thermal aging at 90°C in water

IV. CONCLSIONS

The results of this experimental study show that the curing temperature play an important role in the early strength of the concrete-epoxy interface. Even if, there is no thermal aging effect on the interfacial shear strength for 80°C air treatment until 32 days, an interface failure since 16 days is observed due to a combination of thermal aging and water aging for 80°C water treatment, highlighting the effect of thermo-hydraulic coupled phenomena for concrete epoxy interfaces. Further investigation will be conducted to evaluate the durability of interface by measuring the interfacial shear strength under different temperature and relative humidity treatment conditions in the long term but first results on permeabilities seems to show that the strength decrease in water curing conditions is not related to an evolution of the epoxy microstructure.

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