Cement & Concrete Composites 26 (2004) 803–809

Cement & Concrete Composites

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Flexural performance of polyester and epoxy polymer mortars under severe thermal conditions

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Received 4 November 2002; accepted 8 July 2003

Abstract

A comparative study of the influence of thermal effects on flexural strength of two different binder formulations of polymer mortars was performed. For this purpose, specimens of unsaturated polyester and epoxy polymer mortars were exposed to a large range of temperatures, between −20 and +100 °C, and tested afterwards in bending. For each temperature level, flexural strengths at test temperature, and residual flexural strengths after temper, were determined. The strength degradation process occurring in the material, as a consequence of positive thermal fatigue cycles (+20 °C/+100 °C) and freeze–thaw cycles (−10 °C/+10 °C), was also quantified and analysed. Obtained results show the relevance of material properties assessment with temperature.

Keywords: Polymer concrete; Flexural behaviour; Epoxy concrete; Polyester concrete; Thermal degradation

1. Introduction

Durability is one of the most important properties for a material to be used in the building industry. The relevant literature frequently reports the chemical durability in various aggressive environments. However, thermal durability, i.e., the ability of a component to retain its physical–mechanical properties during and after exposure to severe thermal conditions, is also very important [1]. Building components, during their lifetime, are often subject to changing temperatures. These temperatures can vary from well below zero up to about 80 °C in direct sunlight, depending on the geographical location and on the colour of the surface [2]. Therefore, thermal sensitivity is an important subject that must be taken into account in the evaluation of durability and service life of construction materials.

Polymeric composite materials are one of the youngest building materials and the ones that are appearing, time after time, with new and changed properties, as new combinations and formulations are developed. Therefore, long-term behaviour of these materials cannot be deduced from experience with the material over the expected lifetime or from long-time tests [3]. Regardless of its significant advantages compared with conventional construction materials, mechanical properties of polymer composites are highly susceptible to the type of resin and reinforcement (or aggregate) employed, as well as to the dosage of both components [4].

However, the main problem, related with polymeric materials, arises from the viscoelastic properties of the polymer, which result in creep and high sensitivity to temperature [5,6]. Mechanical properties of polymers, undergoing temperature variations, change considerably, especially within the glass transition temperature range. The glass transition takes place over a wide temperature range, which lies for many resins used in civil engineering between 20 and 80 °C. This means that during the service lifetime of the material the glass transition can occur [3]. Therefore, thermal durability becomes one of the most important factors in the assessment criteria of a polymeric composite material, as a potential construction material.

In previous studies, carried out by the authors [7,8], the chemical durability of two specific binder formulations of polyester and epoxy polymer mortars was analysed. In order to continue and complete the characterization

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0958-9465/$ - see front matter © 2003 Elsevier Ltd. All rights reserved.
doi:10.1016/S0958-9465(03)00162-8

process of these two mortars formulations, the present work aims to analyse and quantify the strength degradation process occurring in the material as a consequence of thermal effects.

Previous works on the mechanical behaviour of hydraulic concretes and mortars when subjected to severe thermal conditions, can be found in Sun et al. [9], Balendran et al. [10], Komonen and Pentalla [11], and Shao and Jiang [12]. However, in spite the previous explanation, there appears to be only a very limited amount of work done on the performance of polymer concrete and mortars under the same conditions. Furthermore, most of the related literature is restricted to a limited temperature range and a certain type of polymer. Traditional approaches are usually focused on temperatures above room temperature, which is understandable taking into account that common resins used in polymer concretes present glass transition temperature ranges above this temperature. However, resistance to very low temperatures, depending upon polymer concrete application, could also be a very important and even crucial subject.

Letsch conducted a research work on the mechanical behaviour of a polyester polymer concrete at temperatures of 23, 40 and 60 °C. He concluded that the presence of higher temperatures resulted only in a small decrease in compressive strength, but a relatively large decrease in flexural strength and a very large increase in creep deformation [13].

A comparative study of the influence of temperature on the mechanical strength of three different polymer concretes (epoxy, polyester and acrylic), has also been carried out by Pardo and co-workers [4], with the aim of establishing a criterion to determine the safety factor for precast structures. Concrete specimens were tested in bending and compression, after conditioning at different temperatures (from 20 to 200 °C). Test results showed that, when the specimens were tested at room temperature, after tempering, no important reduction in the mechanical strength occurred. However, when the specimens were tested at aging temperature, a significant decrease of both flexural and compressive strength characteristics took place.

The same research team also analysed the mechanical behaviour of polymer concrete after exposure to 100 thermal fatigue cycles. Six different thermal cycles have been developed between 20 °C up to 40, 60, 80, 120, 140 and 160 °C, respectively for each cycle. After 100 thermal cycles, one specimen series was tested at 20 °C and other series was tested at the maximum cycle temperature. They concluded that thermal fatigue does not affect strength if polymer concrete is taken back to its initial conditions. However the combination of mechanical and thermal actions up to 60 °C, implies the obligation of applying the thermal partial safety coefficients depending on the type of resin and load [14].

Galán [15] also used thermal fatigue cycles to evaluate thermal durability of binder formulations of unsaturated polyester polymer mortars. The aim of his research work was to analyse the viability of application of 'Polialbero' [1] as local construction material. In order to simulate local weathering conditions (temperature and humidity fluctuations), specimens of both formulations were exposed to distinct thermal fatigue cycles. Temperature ranges, simulating Spring/Summer and Autumn/Winter season conditions, were defined according to the maximum and minimum temperature values, recorded locally in the last years. After 360 cycles, corresponding to one year of outdoor exposure, no significant influence was found on mechanical strength of 'polialbero' specimens.

A different approach was used by Oshima et al. [16], to analyse thermal sensitivity of polymer concretes. Tests were made on temperature-dependent mechanical properties such as dynamic Young's modulus, logarithmic decrement, creep coefficient, compressive and flexural strengths. The study was conducted in order to determine the inflexion points in temperature in the respective properties where temperature dependency becomes dominant. Tests results showed that the inflection points were related to the heat distortion temperature (HDT) of the resins used, rather than glass transition temperature (T_g), and were not affected by the resin content.

Following these studies, the present work aims to analyse the influence of different thermal conditions on flexural behaviour of two specific binder formulations of both unsaturated polyester and epoxy mortars. For this purpose, specimens of both formulations were exposed to the following thermal conditions: conditioning at constant temperatures, for temperature levels between −20 and +100 °C, positive thermal fatigue cycles (+20 °C/+100 °C) and freeze–thaw cycles (−10 °C/+10 °C). Residual flexural strengths, during and after exposure to these severe thermal conditions, were obtained. Analysis of flexural strength degradation process occurred allowing for the assessment of material thermal durability.

2. Experimental program

2.1. Binder formulations

Mortar formulations were prepared by mixing foundry sand with unsaturated polyester and an epoxy resin.

[1] 'Polialbero' was named by the researcher, to polymer mortars prepared with a specific aggregate called Albero. This aggregate can only be found in the province of Sevilla (Spain), and its use is very widespread in Andalusian region, Spain.

Table 1
Thermal and mechanical properties of polyester and epoxy resins

Resin properties (after one week at 25 °C)		Polyester resin	Epoxy resin
Glass transition temperature–T_g	ISO 6721-5	87 °C	45 °C
Heat distortion temperature–HDT	ISO 75	50 °C	34 °C
Tear strength	ISO 527	58 MPa	40 MPa
Flexural strength	ISO 178	119 MPa	70 MPa

Resin content was 20% by weight and no filler was added in both formulations.

Previous studies by the authors, [17,18], considering an extensive experimental program supported by the Taguchi method, allowed an optimisation of mortar formulations that are now being used in the present work.

The polyester resin used in this investigation was s226e (NESTE®), an unsaturated orthophtalic polyester diluted in 44% styrene. The resin system is pre-accelerated by the manufacturer and the initiator used was methyl ethyl ketone peroxide (2 phr).

The epoxy resin system was eposil 551 (SILICEM®), based on a diglycidyl ether of bisphenol A and an aliphatic amine hardener. This system has low viscosity, and is processed with a maximum mix ratio to the hardener of 2:1.

Thermal and mechanical properties of both resins are presented at Table 1.

Foundry sand was a siliceous one, with very uniform grain and a mean diameter, d_{50}, of 342 μm. The sand was previously dried before being added to the polymeric resins in an automatic mixer.

Polymer mortars, with these binder formulations and mix proportions, were mixed and moulded to prismatic specimens 40×40×160 mm, according to the RILEM norm TC-113/PC2 [19]. For each formulation five batches with 18 specimens were casted. All the specimens were allowed to cure, for seven days at room temperature and then post-cured at 80 °C for 3 h, before being exposed to the defined environmental conditions.

2.2. Testing procedures

2.2.1. Conditioning at constant temperature

To determine the influence of temperature on flexural strength of polymer mortars, two types of study were carried out:

- In the first one, specimens were heated/cooled to the different test temperatures in a climatic chamber (three specimens for each temperature level), and after that, they were immediately tested in bending at aging temperature (conditioning temperature). Heating/cooling rate was, respectively, +5 °C/min and −2 °C/min. Temperature test was maintained for 3 h in order to establish a steady state condition of heat transfer.
- The second one was carried out as the first one, but specimens were quickly tempered, by immersion in water, before being tested in bending at room temperature (23 °C).

Bending tests were performed in a mechanical testing machine (displacement controlled type), at a crosshead movement rate of 1 mm/min, according to the RILEM norm TC-113/PCM-8 [20].

2.2.2. Thermal fatigue cycles

To evaluate the flexural strength deterioration of polymer mortars induced by repeated thermal actions, specimens of both formulations were also exposed to the following thermal fatigue cycles:

- Positive thermal fatigue cycles between +20 and +100 °C: 2 h at 100 °C followed by 6 h at 20 °C; heating/cooling rate of 0.67 °C/min; moisture content of 50%.
- Freeze–thaw cycles between −10 and +10 °C: 2 h at 10 °C followed by 2 h at −10 °C; heating/cooling rate of 0.17 °C/min. This experimental program was divided into two series: in the first series, test specimens were heated and cooled by air (dry cycles), in the second series, test specimens were frozen and thawed by water in order to accelerate degradation process (wet cycles).

Bending tests were performed, at room temperature, after 50 and 100 cycles (three specimens for each cycle period and for each cycle type). The weight of the specimens, that were subjected to freeze–thaw cycles, was recorded before and after exposure to thermal cycles.

3. Experimental results and discussion

3.1. Influence of temperature on flexural strength

Flexural test results (average flexural strength and correspondent standard deviation) are presented at Table 2. Relationships between conditioning temperature and flexural strength of both formulations of polymer mortars, for each test condition, are shown in Figs. 1 and 4. In order to assess test temperature effect on ductility and stiffness of polymer mortars, typical shapes of stress deflection curves obtained from flexural tests were determined and plotted (Figs. 2 and 3).

3.1.1. Flexural tests carried out at aging temperature

As shown in Fig. 1, flexural strength of epoxy mortars presents a strong and marked temperature dependency. Characteristic value of flexural resistance remains stable

Table 2
Flexural test results after exposure to different test temperatures

Test temperature (°C)	EPOXY MORTARS flexural strength|S.D. (MPa)[a]		POLYESTER MORTARS flexural strength|S.D. (MPa)[a]	
	Aging temperature	Room temperature	Aging temperature	Room temperature
−20	51.30|2.32	41.35|1.78	20.22|0.97	22.50|0.44
−10	48.10|1.45	–	19.67|2.61	–
0	45.66|0.82	40.97|2.20	20.02|1.26	22.90|1.14
10	41.92|2.21	–	20.29|2.19	–
20	41.26|0.73	–	20.89|2.08	–
23 (Reference)	**41.09**|1.24	**42.64**|0.79	**20.33**|0.26	**23.80**|2.05
30	33.84|0.51	–	18.98|0.85	–
40	18.37|0.78	42.25|0.68	18.87|0.46	22.98|1.95
50	6.86|0.43	–	15.21|0.25	–
60	3.09|0.13	41.72|0.81	12.89|0.20	23.32|2.32
80	1.26|0.52		6.92|0.18	
100	0.80|0.61	45.48|0.44	2.44|0.19	24.26|1.21

[a] Average result of three specimens.

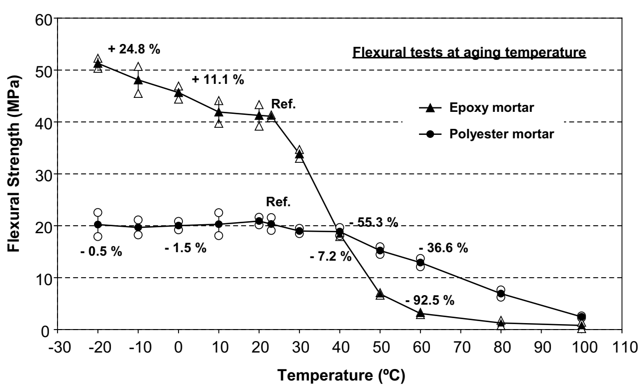

Fig. 1. Flexural strength of epoxy and polyester polymer mortars as function of test temperature.

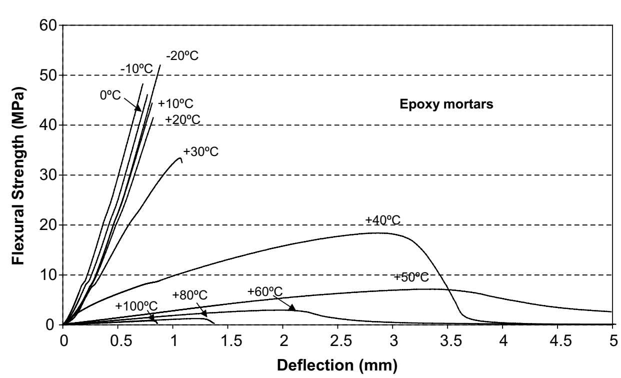

Fig. 2. Stress–deflection curves of epoxy mortar specimens obtained from flexural tests carried out at aging temperatures.

only within a limited range of temperatures, between +10 and +23 °C. For temperatures above room temperature, flexural strength of this particular mortar decreases drastically. Between +30 and +50 °C, a decrease of 85% of initial resistance occurs, and at +100 °C, the load carrying capacity becomes almost insignificant. On the other hand, for temperatures below +10 °C, flexural

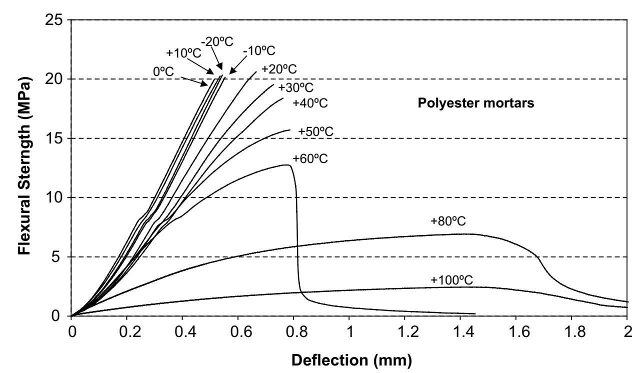

Fig. 3. Stress–deflection curves of polyester mortar specimens obtained from flexural tests carried out at aging temperatures.

strength tends to increase as the temperature test decreases. This tendency is very clear, and it appears to indicate, that for lower temperatures than −20 °C, a higher increase of flexural strength should be expected.

Relatively to polyester mortar formulation, it seems that its flexural strength is not significantly affected by environment temperature within temperature range between −20 and +40 °C. For higher temperatures, a gradual decrease of flexural strength occurs, but the decrease rate is lower than that one occurred for epoxy binder formulation.

As shown in Figs. 2 and 3, for both mortar formulations, the loss of load bearing capacity with increasing temperature, is associated with a progressive loss of material stiffness. As temperature test increases, flexural elasticity modulus decreases and failure becomes more ductile, and less brittle.

The temperatures of the inflection points, when temperature dependency becomes dominant, are related to HDT values of resins used in each mortar formulation. These points, named by other researchers [16] as 'heat distortional temperatures of polymer mortars', are both located around 10 °C lower than HDT of resins used.

3.1.2. Flexural tests carried out at room temperature after specimens tempering

According to test results presented in Figs. 4, temperature during the exposure time had no significant influence on flexural strength of both formulations, as long as specimens were taken back to its initial environmental conditions and tested at room temperature. A slight increase of flexural strength was observed for epoxy mortar specimens that have been exposed to the highest temperature, possibly due to post-curing.

No significant differences, induced by temperature conditioning, occurred also on the flexural elasticity modulus or failure mode of mortar specimens.

The recovering of initial flexural properties, observed on both formulations, can be explained by molecular mobility of polymeric chains. This mobility increases with temperature, and is associated to local motions of the less hindered side groups around their equilibrium position, and as temperature reaches the glass transition region, molecular mobility increases profoundly leading to configurational rearrangements of the polymer chain backbones [21]. Loss of mechanical strength is one consequence of this process. However, when polymers are tempered, there is not energy enough to maintain this mobility and therefore, initial cohesion between polymeric chains is recovered.

3.2. Influence of thermal fatigue cycles on flexural strength

Table 3 presents average flexural test results of mortars specimens that have been subjected to thermal fatigue cycles. Weight change of mortars specimens immersed in water during freeze–thaw cycles is also presented. Residual flexural strengths of both formulations after 50 and 100 exposure cycles, for each different thermal cycle, are shown in Figs. 5 and 6.

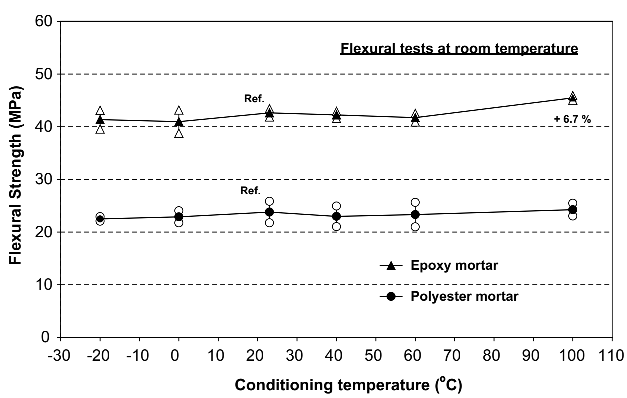

Fig. 4. Flexural strength of epoxy and polyester polymer mortar, after specimens tempering, as function of conditioning temperature.

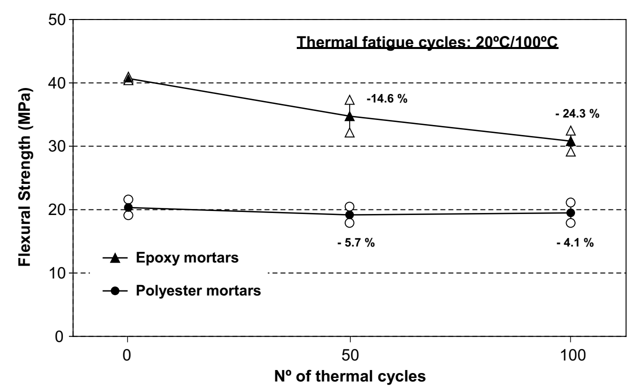

Fig. 5. Residual flexural strength of epoxy and polyester polymer mortars after exposure to 50 and 100 positive thermal fatigue cycles (20 °C/100 °C).

Table 3
Flexural test results after exposure to thermal fatigue cycles

Thermal fatigue cycles		Polyester mortar		Epoxy mortar	
		Flexural strength|S.D. (MPa)[a]	Mass change (%)[ab]	Flexural strength|S.D. (MPa)[a]	Mass change (%)[ab]
+20 °C/+100 °C	0 cycles	**20.33**|1.24	–	**40.70**|0.27	–
	50 cycles	19.17|1.27	–	34.75|2.58	–
	100 cycles	19.49|1.62	–	30.82|1.66	–
−10 °C/+10 °C (Air)	0 cycles	**22.11**|0.96	**0**	**40.85**|1.11	**0**
	50 cycles	23.40|0.20	0.031	40.56|2.69	0.010
	100 cycles	22.11|0.63	0.052	39.18|1.86	0.017
−10 °C/+10 °C (Water)	0 cycles	**22.11**|0.96	**0**	**40.85**|1.11	**0**
	50 cycles	23.20|0.83	0.240	38.27|0.61	0.094
	100 cycles	23.19|0.30	0.355	38.66|1.66	0.137

[a] Average result of three specimens.
[b] Mass change was determinate in accordance with RILEM standard TC113/PC-11[22].

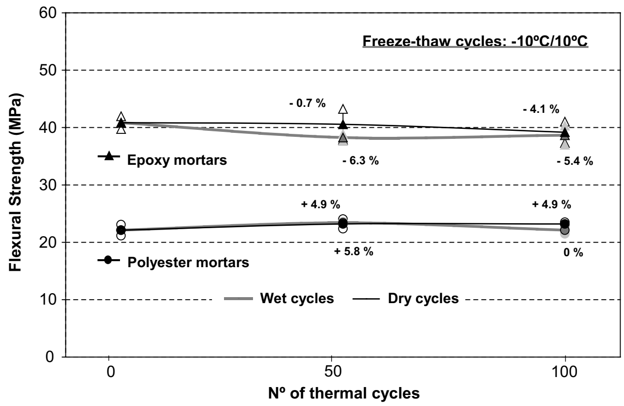

Fig. 6. Residual flexural strength of epoxy and polyester polymer mortars after exposure to 50 and 100 freeze–thaw cycles (dry cycles and wet cycles).

3.2.1. *Positive thermal fatigue cycles*

The influence of thermal fatigue cycles, between +20 and +100 °C, on flexural strength of epoxy polymer mortars is not irrelevant. Regardless of its recovering capacities showed by last tests, the continued cyclic exposure of epoxy polymer mortars to high temperatures has a negative effect on its flexural strength. After 50 thermal fatigue cycles, a percentage drop of 14% occurs on flexural strength, and after 100 cycles, this property falls to 75% of its initial value.

It seems the continuous cycling to temperatures up to 100 °C, more than twice epoxy T_g, leads to some degradation of the epoxy network. This phenomenon, not noticeable for one cycle, becomes notorious after a certain number of cycles, as degradation progresses cycle after cycle.

The described phenomenon has no significant expression in the case of unsaturated polyester mortars. In this case, the maximum cycling temperature is only 15% higher than the polyester T_g, so it constitutes a much less severe condition to the polyester polymer network.

The flexural elasticity modulus and failure mode of both type of mortars are not affected by exposure to these thermal actions.

3.2.2. *Freeze–thaw cycles*

Exposure to thermal fatigue cycles between −10 and +10 °C had no relevant influence on flexural strength of both formulations of polymer mortars. Even for specimen series immersed in water, subjected to frost attack, flexural strength retained after 100 freeze–thaw cycles was still very high. In addition, no scaling surface was observed and any sediment appeared on water containers. Therefore, specimens weight change was totally due to water absorption.

Frost damage is very clearly linked to uptake of water during freeze/thaw exposure or the degree of water-saturation. The unexpected result may be explained by an insufficient water uptake of polymer mortar specimens, resulting of their low degree of water absorption and high watertightness, already known from previous studies [7].

This explanation is supported by Setzer research work [23]. According to this researcher, the high number of freeze/thaw cycles is primarily needed to saturate the specimens artificially by a micro-ice-lens-pump, and not for the progress of a fatigue fracture process. Micro-ice-lens-pump model, described by Setzer, is based on the phenomenon that the three phases of water coexist between 0 and −60 °C with thermodynamical stability. This is only possible because a high negative pressure is generated in the unfrozen gel–water. In the cooling phase the gel-matrix is compressed, water is squeezed out and trapped at the ice. During subsequent heating, the gel expands and water is sucked in from external sources since the ice particles are still frozen. With every freeze–thaw cycle, the degree of saturation is increased until a critical value is reached and damage sets in. Until this point, only a relative small damage should be observed.

4. Conclusions

This paper briefly discussed the results of an experimental study into the influence of temperature and thermal fatigue cycles, on flexural behaviour of two specific binder formulations of polymer mortars.

Based on test results, the following conclusions can be drawn:

- Flexural properties of unsaturated polyester and epoxy mortars are strongly affected by environment temperature. Except within a limited temperature range, flexural strength of these mortar formulations decreases drastically as temperature increases. The inflection points, where temperature dependency becomes notorious, are related to the HDT of the resins used rather than the glass transition temperatures, in accordance with the conclusions of Oshima and co-workers [16]. For both formulations used in this study, those points are around 10 °C lower than the HDT of respective resins.
- Epoxy mortars are more sensitive to temperature than polyester mortars. The 'HDT of epoxy mortars is lower than corresponding 'HDT of polyester mortars'. After such critical points, decrease of flexural strength for epoxy mortars occurs at a higher rate. For temperatures lower than +10 °C, flexural behaviour of epoxy mortars improves with further decreases in temperature.
- Exposure to constant or changing temperatures (thermal fatigue cycles) has no significant influence on flexural properties of both formulations of polymer mortars, as long as specimens are taken back to its initial environmental conditions. However, recover-

ing capacity of epoxy polymer mortars is gradually reduced by repeated exposure to high temperatures.
- One hundred freeze–thaw cycles between −10 and +10 °C produce very little damage on both epoxy and polyester polymer mortars, possibly due a reduced degree of water absorption. It will be necessary a larger number of cycles to evaluate froze resistance of these materials.

These results show the relevance of material properties assessment with temperature. In particular, with epoxy polymer mortar formulation, it can be concluded that the use of such material is restricted to lower temperature environments in order to benefit from its total potential in strength. In the case of higher temperatures, care should be taken in both formulations, as properties may drop significantly.

Acknowledgement

The financial support of Fundação para a Ciência e a Tecnologia under grant POCTI/EME/35955/1999, "Long-term properties of polymer concrete", is gratefully acknowledged.

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