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NON-DESTRUCTIVE CHARACTERISATION OF MORTARS REINFORCED WITH VARIOUS FIBRES EXPOSED TO HIGH TEMPERATURE

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Abstract: The objective of this study is to investigate the effect of temperature on the physical and mechanical properties of standard mortar reinforced with steel fibers, polypropylene fibers and hybrid fibers. Non-destructive tests (capillary water absorption, interconnected porosity, gas permeability, ultrasound celerity) were carried out on samples that had been heated, at a temperature ramp of 5 °C/min, to maximum temperature of: 105 °C, 400 °C, 500 °C et 800 °C. The results show a good correlation between the evolution of properties and the damage resulting from the imposed heat exposure treatment. The study shows that there is a significant deterioration of physico-mechanical properties of the fiber mortars above 500 °C. The hybrid fiber mortars show a good compromise: the polypropylene fibers guarantee a thermal stability whereas the steel fibers act to conserve good mechanical behavior.

Keywords: *mortars, steel fiber, polypropylene fiber, temperature, non-destructive test*

1. INTRODUCTION

Cementitious materials are damageable materials under mechanical stresses. The mechanical behavior is nonlinear, brittle or semi-brittle, with poor tensile properties. According to different authors, the fiber reinforcement acts in different ways:

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- by their capacity to control cracking (Saradar at al. 2018; Cuenca et al. 2016) as energy absorbers (Yu at al. 2016; Ding et al. 2012; Sukontasukkul et al. 2010);
- by their capacity to transfer loads under tensile stresses in order to delay, to limit or to prevent quasi-brittle fracture (Köksal et al. 2013; Bencardino et al. 2010);
- by their capacity to improve nonlinear behavior by retarding the localization of the damage, the fibers act to improve deformation capacity of the material and the peak stress (Nili et al. 2010; Sideris et al. 2009; Mohammadi et al. 2008; Sivakumar et al. 2007; Zongcai et al. 2006; Poon et al. 2004);
- after cracking, fibers provide mechanical capacity to the concrete in order to avoid cracking growth and propagation (Sivakumar et al. 2007).

Recently, the fire resistance of fiber reinforced cementitious materials has become a major interest to researchers. Mortars can generally be characterized by a dense microstructure and low permeability. The dense microstructure appears to be a weak point in mortar's thermal stability when such materials are exposed to high temperature (Pliya et al., 2010; Phan et al. 2001). The fires in the tunnels at St. Gotthard (Switzerland 2001), Tauern (Austria 1999), Mont Blanc (France 1999) or the Windsor Tower (Spain 2005) and nuclear reactors have shown that concrete heated to high temperatures can present a risk of instability. Parameters such as fire condition (temperature, duration, calorific energy), the nature of concrete, free water in the concrete, the type of aggregates, the heating rate, and the presence of loading, all have an influence on thermal instability of the material (Kanema et al. 2007).

An effective method for reducing the risk of thermal instability is the addition of polypropylene fibers into a cementitious material mix (Rodrigues et al. 2010; Aydın et al. 2008; Pliya et al. 2008; Xiao et al. 2006; Zeiml et al. 2006; Bilodeau et al. 2004; Kalifa et al. 2001). The polypropylene fibers melt at relatively low temperatures (~170 °C) thus creating a supplementary porosity that allows the transport of humidity in the concrete towards the exterior, and limits the development of high interstitial water pressures within the cement matrix (Bangi et al. 2011; Pliya et al. 2011; Sukontasukkul et al. 2010; Sideris et al. 2009).

For safety reasons, it is necessary that the material conserves it mechanical properties for as long as possible during a fire to maintain safe conditions, limit the propagation of the fire and to leave time for evacuation. Furthermore, it may be important that the moderate degradation of the material enable a good probability of renovation after the fire (Denoël 2007). To this end, the residual mechanical performance of the concrete must be kept at a level as close as possible to the initial state. The addition of steel fibers in a cementitious mortar, subjected to high temperatures, provides a possible way in which the residual mechanical properties could be improved. Steel fires would not limit high interstitial water pressures in the mortar but strengthen the mortar against cracking and spalling. Steel fibers provide a mechanical rather than thermal role.

The aim of this study was to characterize the damage of different mortars subjected to temperatures of 105 °C, 400 °C, 500 °C and 800 °C. The temperature limit is 800 °C. Previous studies have shown that at upper temperatures, the material is too much damaged and measurements become difficult (Ezziane et al. 2011; Komonen et al. 2003).

The mortars tested were as follows: standard mortar without fibers; steel fiber reinforced mortars; Polypropylene fiber reinforced mortars and mixed fiber reinforced mortars (50% steel fibers plus 50% polypropylene fibers).

The fiber content of the three last mortars was 0.58% by volume.

The characterization and quantification of the damage induced by heat treatment were carried out by non-destructive methods. The NDT consisted of capillarity water absorption, interconnected porosity, gas permeability (helium (He)) and ultrasound celerity.

2. EXPERIMENTAL METHODS

2.1. SAMPLE PRODUCTION AND CONSERVATION

Four types of mortar mix were produced: standard mortar (SM), mortar with steel fibers (SFM), mortar with polypropylene fibers (PFM) and hybrid fiber mortar (SPFM: 50% polypropylene fibers plus 50% steel fibers). The cement used for the fours mortar mixes was CEMI 52.5N. The water to cement ratio (W/C) was 0.5 and the sand to cement ratio (S/C) was 3. For the three fiber mortars, the fiber content was 0.58% by volume (i.e., 45 kg/m^3 of steel fibers or 5.2 kg/m^3 of polypropylene fibers). This choice allowed the conservation of workability without the need for a superplasticizer (Beaudoin 1982).

All the mortars were mixed following the same mixing protocol (CEN196-1 standard): water was added to the cement and mixed to obtain a homogeneous paste; sand was then gradually added to the paste and mixed to homogeneity. In the case of the fiber mortars, the fibers were added and manually dispersed as the final step. The mixes were mixed for two minutes after the introduction of the last constituent. No adjustment to the water content was made to account for the presence of the fibers: the workability of the mortars was therefore different. Workability was determined using a mini slump cone (EN 12350-2 standard with a modified one-half scale cone); the air content was measure using an air content meter (EN 1015-7).

The test samples were cylindrical, 40 mm diameter and 60 mm long. The samples were conserved in an environmental chamber (20 °C, 95% RH) for 24 h, and then conserved in a water bath for 28 days. The samples were then stored in a dry chamber (20 °C, 50% RH) until sample weight stabilized. Under these conditions, a significant portion of the free water in the mortar matrix had evaporated (Zhang et al. 2008).

2.2. HEAT TREATMENT

The physical and mechanical characteristics of a sample are notably influenced by the sample size and the temperature ramp rate. A temperature gradient within the sample may induce micro cracking due to thermal expansion. On the other hand, the chemical and mineralogical transformations are mainly due to the maximum temperature and the exposure period (Georgali et al. 2005; Castellote et al. 2004). The heat treatment was carried out in an electric muffle furnace with a temperature ramp rate of 5 °C/min. The duration at the peak temperature was one hour. The cooling to room temperature was carried out with the furnace turned off and the door left closed. The cooling rate could not be controlled but was of the order of –0.3 °C/min.

2.3. NON-DESTRUCTIVE TESTS

Characterization and quantification of damage induced in fiber and non-fiber mortars, subjected to high temperatures, was carried out by non-destructive techniques, these tests being good indicators of the evolution of the microstructure and cracking within the mortar. Determinations of capillary water absorption, interconnected porosity, gas permeability reflect the importance of the interconnectivity of pores that corresponds to the pathways intrusion of aggressive agent; whilst ultrasound celerity reflects the global level of damage within the sample.

To ensure reproducibility of the results, a reference state was established for all samples: samples were dried at a temperature of 105 °C until weight stabilization. This process slightly changed certain hydrates (ettringite) and partially changed the microstructure. However, the process did avoid a large dispersion of results due to variations in sample drying.

2.3.1. CAPILLARY WATER ABSORPTION

The capillary water absorption tests were carried out in accordance with AFPC-AFREM recommendations (AFPC-AFREM 1997). The lower face of the dry cylindrical samples was immersed in water to the depth of 3 mm. The lateral faces of the samples were covered with aluminium foil to promote an axial flow of water and thus avoiding evaporation at the side. The weight of the sample was monitored. The kinetic capillary absorption coefficient, expressed as $kg \cdot m^{-2} \cdot s^{-1/2}$, is defined as the tangent through the origin of curve with respect to the square root of time.

Three specimens were tested for each condition (mortar type and heating temperature).

2.3.2. INTERCONNECTED POROSITY

The interconnected porosity was determined from three weights of the same sample: the weight of the sample dried at 105 °C, the weight of when saturated after 24 h in a vacuum chamber, and the hydrostatic weight of the saturated sample. The weights were used to determine the interconnected porosity and the apparent and real densities.

Three specimens were tested for each condition (mortar type and heating temperature).

2.3.3. GAS PERMEABILITY

The intrinsic permeability was measure at the reference state (105 °C) for all the mortars. The gas permeability was determined using the Klinkenberg method, and taken from 5 measurements of apparent permeability at various helium percolation pressures and at a constant confining absolute pressure of 0.8 MPa. For the degraded samples (400 °C, 500 °C and 800 °C), one apparent permeability was measured that being for a percolation absolute pressure of 0.2 MPa with the same confining pressure.

Three specimens were tested for each condition (mortar type and heating temperature).

2.3.4. P-WAVE VELOCITY TEST

P-wave velocity is calculated using the transmission travel time of an acoustic pulse along the axial direction of the samples. The experimental setup includes a waveform generator (SOFRANEL 5800PR), two piezoelectric transducers (with a resonant frequency of 500 kHz) mounted on the sample holder, and a numerical oscilloscope board connected to a computer. The sample holder and the transducer were immersed in water to ensure a reproducible contact the specimen and the transducers: the coupling agent is the water film which keeps the same thickness (5 mm). Fast Fourier Transform (FFT) was used for spectral analysis of the received wave thus enabling an evaluation of absorbed sound energy.

Three specimens were tested for each condition (mortar type and heating temperature).

3. RESULTS

3.1. CHARACTERIZATION OF FRESH MORTAR

The characteristics of the fresh mortar mixes are set out in Table 1. The addition of fibers tended to reduce the workability, as determined by a mini cone and increased the entrained air content. The steel fibers had a relatively small effect whereas the polypropylene fibers, which have a large specific surface, required a large quantity of mix water for surface wetting and therefore greatly reduced the workability. There was a high entrained air content in the polypropylene fiber mortar. The slump and entrained air content were reduced in the mortar SPFM due to the smaller polypropylene fiber content than in PFM.

Table 1. Characteristic of fresh mortar

Mortar	SM	SFM	PFM	SPFM
Slump [mm]	39	34	14	24
Entrained air content [%]	3.2	3.5	5.2	4.6

3.2. NON-DESTRUCTIVE CHARACTERISATION OF HARDENED MORTARS

Characteristics of the mortars in the reference state differed due to the different mix formulation. The evolution of these characteristics also differed due to the very different modification induced by the heating treatment. In order to compare the different mixtures at different temperatures and illustrate the relevant changes, the NDT values after the heat treatment have been normalized with respect to the initial value measured at 105 °C.

3.2.1. CAPILLARY WATER ABSORPTION

The absorption kinetics is expressed by the variation of the quantity of water absorbed per unit surface with respect to the square root of time (Fig. 1). For each type of mortar, the curves stabilize at different levels, the physical limit being when the capillary absorption front reaches the top of the sample.

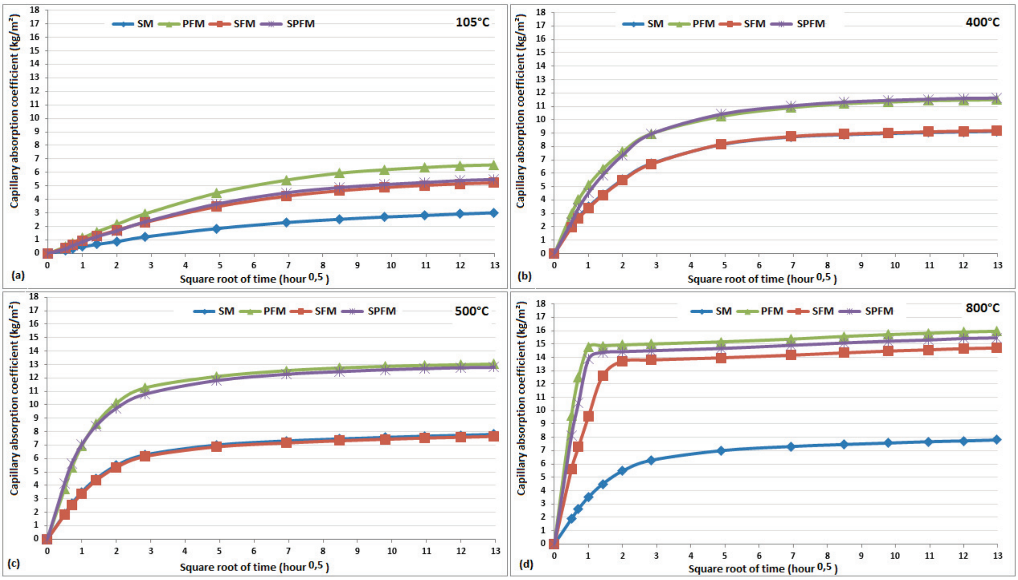

Fig. 1. Capillary water absorption of sound and heat-treated samples

Comparing the results of the fiber and non-fiber mortars (Fig. 1a), it appears that the introduction of fibers, regardless of type, results in an increase, to a greater or lesser extent, of the capillary absorption coefficient (SM: 0.63 ± 0.12; SFM: 1.18 ± 0.44; PFM: 1.53 ± 0.30; SPFM: 1.26 ± 0.30·10^{-2} kg·m^{-2}·$s^{-1/2}$). This type of behavior has already been reported by other researchers (Abdou et al. 2007).

After heating to 400°C the variation in capillary absorption between SM mortar and SFM mortar is very small (Fig. 1b). In terms of absorption kinetics and peak value PFM and SPFM mortars show similar behaviors and exhibit higher capillary absorption coefficients than the SM and SFM mortars. This increased absorption is directly related to the melting of the polypropylene fibers.

At 500 °C (Fig. 1c), the difference in absorption between mortars with (SM and SFM) or without polypropylene fibers (PFM and SPFM) increases.

After heating to 800 °C (Fig. 1d), the absorption of the three types of fiber mortar greatly increases while for non-fiber mortars there is quite no changes between 500 °C and 800 °C. It can be seen in the 800 °C curves, that mortar saturation is rapidly attained in comparison with the 400 °C and 500 °C curves. The SM mortar shows the lowest capillary absorption; and the polypropylene fiber mortars systematically show the greatest absorption.

3.2.2. INTERCONNECTED POROSITY

In the reference state (105 °C), mortars, with and without fibers, have the similar porosities of the order of 17% (a minimum of 17.1% ± 0.5 for SFM and a maximum of 17.8% ± 0.2 for PFM). Between the reference state and 400 °C there is a relative increase in porosity of on average 24% for non-fiber mortar (Fig. 2). A slightly lower

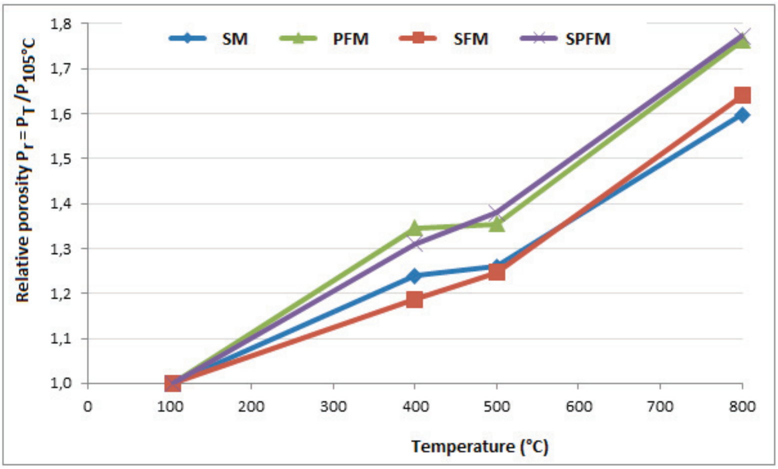

Fig. 2. The evolution of mortar porosity with respect to the maximum temperature in the heating cooling cycle

increase is seen in the case of the steel fiber mortar. On the other hand, in the case of polypropylene fiber mortar and the hybrid fiber mortar, the increase is far greater: 35% for PFM and 31% for SPFM. For all mortar types it is noted that there is a small increase in porosity between 400 °C and 500 °C. Above this temperature all mortars exhibit a rapid increase in porosity. At 800 °C one can distinguish two levels of porosity: 31% for PFM and SPFM, 28% for SM and SFM.

3.2.3. GAS PERMEABILITY

In the reference state, all the mortars have a relatively low intrinsic permeability of the order of 10^{-17} m^2. It can be seen that the permeability of mortars without fibers ($3.25 \pm 0.01 \cdot 10^{-17}$ m^2) and with steel fibers ($3.12 \pm 0.55 \cdot 10^{-17}$ m^2) are lower than the mortars with polypropylene fibers ($6.55 \pm 0.69 \cdot 10^{-17}$ m^2) and with hybrid fibers ($4.98 \pm 0.73 \cdot 10^{-17}$ m^2).

In the degraded state, the residual permeability increases with temperature (Fig. 3). The relative permeability of mortars progressively increases with increased temperature up to 500 °C. Above this temperature the permeability increases rapidly. As seen in the measurement of porosity, the polypropylene fiber mortars and the hybrid fiber mortars have the greatest increases in permeability. Between 105 °C and 500 °C there is a small variation in relative change in permeability of the non-fiber mortars and those with steel fibers (less than 2%). Above 500 °C the increase in permeability of the steel fiber mortar is greater than that of non-fiber mortar. At 800 °C, the relative difference between the two materials reaches 30%.

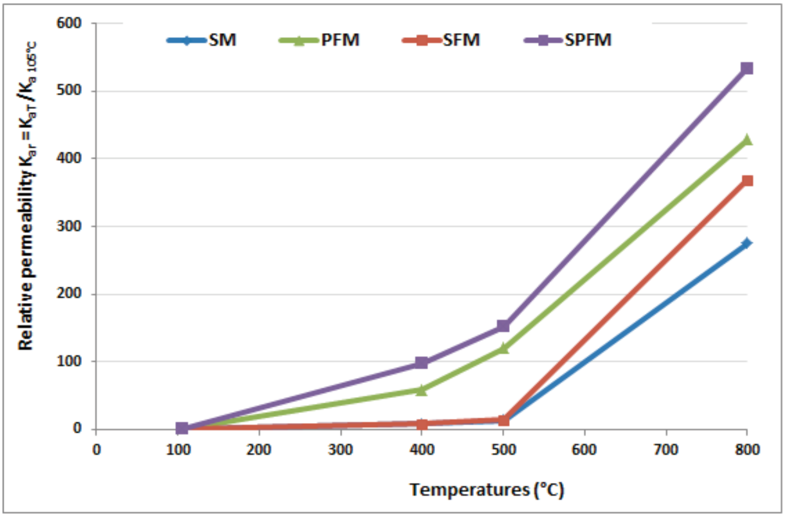

Fig. 3. Evolution of relative apparent permeability of mortars
with respect to the maximum temperature in the heating cooling cycle

3.2.4. ULTRASOUND CELERITY AND ATTENUATION

The measurement of celerity was carried out on samples, in the immersion mode, before and after heat treatment. Samples in the reference state had celerity between 4535 ± 20 m/s (PFM) and 4710 ± 65 m/s (SFM). The steel fiber mortars had a slightly higher celerity than the non-fiber mortars SM (4705 ± 20 m/s); the polypropylene fiber mortars (PFM) and the hybrid fiber mortars (SPFM) had a lower celerity (4600 ± 85 m/s). The differences in celerity between the mortars were linked to the nature of the fibers, the modification in their microstructure and their mechanical characteristics (elastic modulus and compressive strength).

The evolution of celerity with respect to temperature is shown in Fig. 4. For all the mortars, to a greater or lesser extent, a progressive decrease in celerity was observed for increased temperature up to 500 °C. Above this temperature the decrease in celerity is significantly greater. This observation indicated that the heat treatment induces an irreversible transformation that modifies the ultrasound pathway.

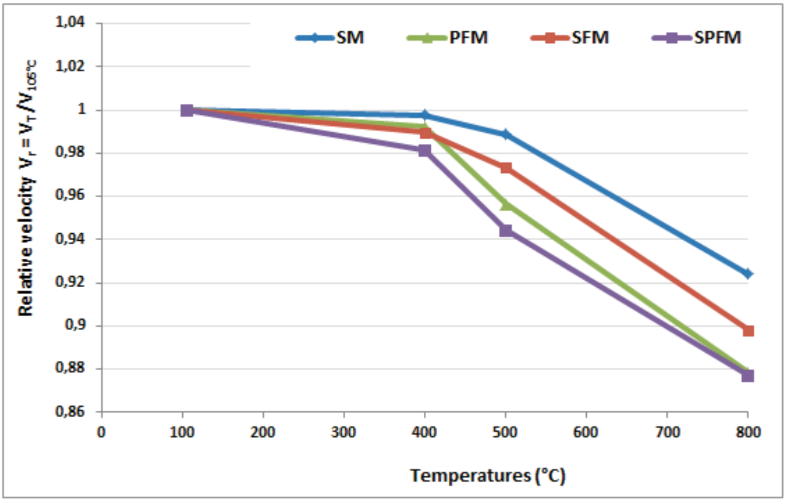

Fig. 4. Evolution of relative celerity of the mortars
with respect to the maximum temperature in the heating cooling cycle

The spectral analysis of frequencies and amplitudes helps to describe the relative importance of the wave damping in the material due to porosity, heterogeneity and to diffusive elements (presence of fibers) and low acoustic impedance (pores and fissures). The effect of damage to the material can be observed in the evolution curves of frequency spectra with respect to temperature (Fig. 5).

Two clear consequences of the sample deterioration can be identified: the attenuation of signal amplitude and the shift towards lower frequencies. These curves clearly testify to the level of damage in the samples. The evolution of attenuation of signal amplitude as a function of temperature is shown in Fig. 6.

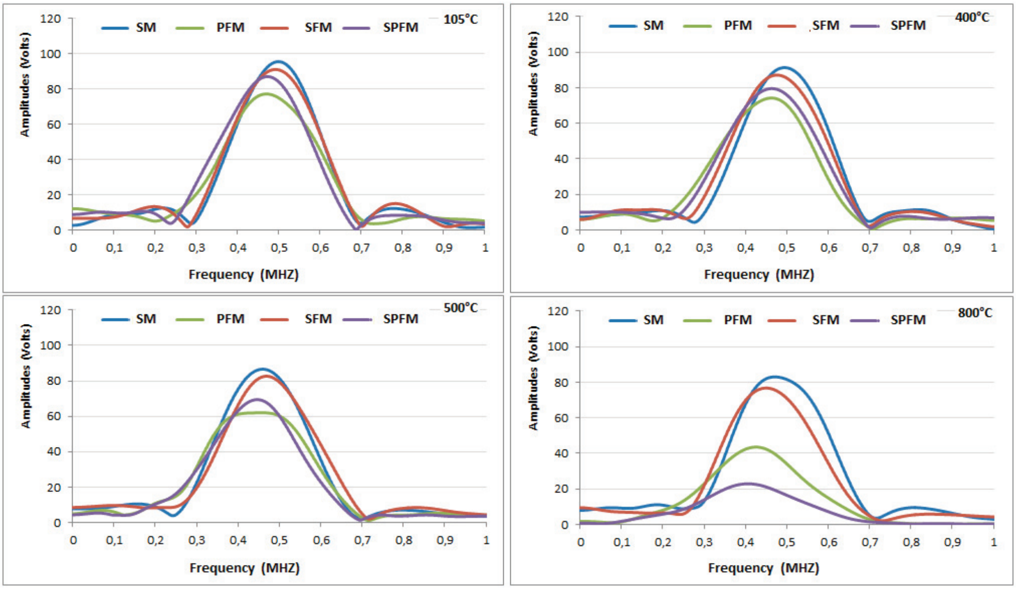

Fig. 5. Evolution of compressive strength

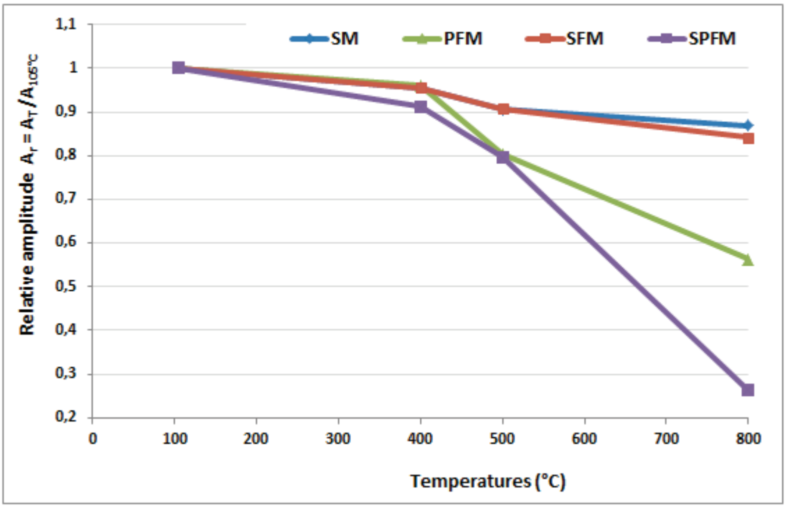

Fig. 6. Evolution of the relative maximum amplitude attenuation of the signal for the mortars with respect to the maximum temperature in the heating cooling cycle

The maximum amplitudes measured for the fiber mortars are less than those measured in non-fiber mortars: 95 ± 3 V for SM and 91 ± 4 V, 87 ± 4 V, 77 ± 4 V for SFM, SPFM and PFM respectively. The mortar containing polypropylene fibers showed attenuation greater than that seen in the case of steel fibers. It can also be seen that in the case of relative signal amplitudes, the behavior is identical for non-fiber and fiber mortars up to 500 °C (Fig. 6). Above this temperature the relative maximum amplitudes of the fiber mortars are less than those measured in non-fiber mortar.

4. DISCUSSION

In the reference state the capillarity results showed that the introduction of fibers, regardless of type, increases the capillarity; the same trend was found in the case of gas permeability. However, the interconnected porosity appeared to be little affected by the presence of fibers. It is therefore proposed that the increase in permeability and capillarity are linked to the increase in connectivity and/or to a decrease in the tortuosity associate with the presence of the fibers. The fibers induce a bridging effect due to the more porous nature of the fiber - cement paste interface (Miloud et al. 2005). This increase in porosity at the fiber interface does not represent a sufficiently large volume to affect the global porosity. The results of the ultrasound tests (celerity and attenuation) showed the same influence of fibers as described above: the fibers tended to reduce celerity and the amplitude to the transmitted wave. This shows that the fibers act as a diffusive constituent that damps the amplitude of the waves. This attenuation was highly correlated with the type of fiber. The attenuation results show that the interface of the polypropylene fibers with the cement paste is not as sound as the steel fibers interface thus confirming the gas permeability results.

After the heat treatment there was degradation in the mortar characteristics. With the increase in temperature it was noted that there was an increase in capillarity, porosity, permeability and a decrease in the celerity of ultrasound and energy transmission.

The evolution of capillarity with respect to temperature showed that above 400 °C deterioration is linked to the presence of fibers, regardless of their type, and not to the cement matrix: the latter being degraded at temperatures much lower than 400 °C. In the case of polypropylene mortars, the phenomenon of absorption is accentuated due to the melting of the fibers at 170 °C. Consequently, the pores thus created improve the absorption kinetics.

The evolution of porosity confirms the results obtained by capillarity and shows that, during heating, the microstructure of the mortar is modified and the material becomes more porous. This opening of pores allows the migration of water vapor thus reducing the moisture clogging (vaporization front) which has been described by several authors as a factor causing thermal instability of concrete (Behnood et al. 2009; Jansson et al. 2009; Zeiml et al. 2006; Hertz et al. 2003). With the increase in temperature, the porosity of the polypropylene fiber mortars increased more rapidly than that of steel fiber and non-fiber mortar. The studies carried out by the authors (Pliya et al. 2011; Noumowé 2005; Gaweska 2004; Komonen et al. 2003) confirms that the presence of polypropylene fibers in a heated mortar gives rise to an increase in the pore volume and modifies the kinetics of pore formation. It is noted that the addition of a steel fiber volumetric content of 0.58% did not have an influence on the porosity of the heated mortar. With the hybrid fiber mortar (steel + polypropylene), the porosity increased less rapidly with temperature than mortar containing only polypropylene fiber; however, the hybrid fiber porosity was greater than the porosity of non-fiber mortar. Up to 500 °C the presence of steel fibers appears to retard the increase of porosity (the porosity of SFM was less than SM and the porosity of SPFM is less than PFM). Beyond 500 °C this retarding effect of the increase in porosity was no longer visible.

From the permeability tests two similar observations can be made: Up to 500 °C the reference mortar and the steel fiber mortars show the same deterioration; above this temperature the steel fiber mortar deteriorates more rapidly. This increase in deterioration can be attributed to the difference in the additional heterogeneity associated with the presence of the fibers. The new interfaces created by the fibers are more susceptible to thermal degradation. The connectivity is then increased following the degradation of these interfaces. The effect of temperature on permeability of polypropylene and hybrid fiber mortars differs at all temperatures; the hybrid fiber mortars having a greater permeability than the polypropylene fiber mortars. This difference in behavior can be explained by the nature of the fibers incorporated in the mortar. In the case of polypropylene fiber mortar, the fibers have a coefficient of expansion that is different to that of the cement paste. During heating ($T < 150$ °C) delamination occurs between the fibers and the cement paste that increases the permeability (and pore connectivity). Above 170 °C, the polypropylene fibers melt and improve the interconnection within the porous network, and thus induce an increase in permeability. In the case of the hybrid fiber mortar, there is a cumulative effect of polypropylene and steel fibers on the mortar permeability.

The decrease in celerity of ultrasound in the mortars can be attributed to the dehydration of the cement matrix, to discontinuities in the cement paste that occur during heating, and to the delamination occurring at the fiber cement paste interfaces and aggregate cement paste interfaces resulting from their differing coefficients of expansion (Hager 2013). These defects induce a decrease in mechanical rigidity in the material and therefore a decrease in celerity. A part of the ultrasound energy is dissipated by these defects (cracks, porosity, and delamination) which appear in mortar subjected to heat treatment. The shift towards low frequencies indicates that the higher frequencies (shorter wave lengths) are attenuated more readily than the lower frequencies (longer wave lengths): the high frequencies are less able to “cross” defect such as cracks, that the low frequencies.

The test results show that the fibers act as diffusive component that damps the amplitude of the wave. This attenuation is closely correlated to, the fiber type, the deterioration during heating, the cement paste – fiber bond, and to the degradation of the cement matrix. Once again, it can be noted that the mortars containing polypropylene fibers deteriorated to the greatest extent. As in the case of permeability, the hybrid fiber mortars underwent a greater deterioration (an accumulation of the effects of polypropylene and steel fibers).

The 500 °C threshold can be identified as the temperature above which all the characteristics deteriorate rapidly. This is most certainly a cumulative effect of the alterations to the cement matrix, the total melting of the polypropylene fibers, possible effects on siliceous aggregates and the beginning of steel fiber oxidation that occurs at high temperatures (Ezziane et al. 2011).

A previous study (Ezziane et al. 2015) that investigated the mechanical characteristics of the same mortars revealed a loss of compressive strength associated with the deterioration of the matrix; an increase in porosity induced by the deterioration of the fibers; the loss of rigidity that is more strongly linked to the deterioration of the matrix than the fibers; finally, the loss of flexural strength which is associated with delamination at the fiber matrix interface.

5. CONCLUSION

From this study, one can conclude that the high temperature heat treatment induced an increase in capillarity, porosity, permeability and a decrease in celerity and energy transmission of ultrasound.

Steel fiber mortar exhibits the best mechanical behavior, however non-destructive testing indicates a deterioration of the fiber cement paste interface: capillarity and permeability increased after heat treatment, and there was deterioration in the propagation of ultrasound.

Polypropylene fiber reinforced mortar had a significant decrease in mechanical properties. This decrease can be attributed to the melting of the fibers and the consequential creation of porosity.

A 500 °C threshold is identified above which the physical and mechanical properties degrade rapidly. This threshold is associated with the accumulated alteration in the cement matrix, the melting of the polypropylene fibers and the degradation of the interfaces between fibers and cement matrix.

The hybrid fiber mortar (50% steel fibers, 50% polypropylene fibers) shows a good compromise. The polypropylene fibers guarantee a thermal stability due to the fibers melting and giving supplementary porosity that limits spalling; and the steel fibers limit cracking and thus maintain a good physical behavior.

The results of this research have the potential to contribute significantly to the extension of the use of hybrid fiber reinforced concrete in thermal stabilization and structural reinforcement, especially constructions in enclosed environments such as tunnels, mines, underground construction, etc.

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