Thermal resistance of hardened cement pastes containing vermiculite and expanded vermiculite

S. M. A. El-Gamal · F. S. Hashem · M. S. Amin

Abstract The durability and thermal stability of hardened Portland cement pastes containing vermiculite (V) and expanded vermiculite (EV) exposed to high temperatures were studied. Different mixtures were prepared using 2.5, 5, and 10 wt% of both types of V. Each mixture, after 28 days of hydration, was heated at 300, 600, and 800 °C for 3 h. Two modes of cooling were used; gradual cooling in air and rapid cooling in cold water. The percentage of residual strength, chemically combined water content, change in phase composition, and the thermal stability of the heated specimens were studied. The specimens cooled in water showed greater loss in strength than the air-cooled specimens. The presence of V improved the heat resistance of ordinary type I Portland cement (OPC) pastes. 5 wt% replacement revealed the best performance at all heating temperatures. The EV showed better thermal resistance than the nonexpanded one. Addition of silica fume (SF) with V in OPC pastes lead to superior performance. This can be explained as result of the combined effects of insulation properties of V and pozzolanic reactivity of SF which accounts for the notable increase in the residual strength for these mixes.

Keywords Vermiculite · Expanded vermiculite · Thermal resistance · Portland cement

Introduction

Thermal dehydration, which occurs by exposure of hardened cement pastes or concrete to high temperature, was become of important interest due to economic and safety sides. This effect may decrease the expected service life of structure due to permanent damage. It is possible to minimize the effect of high temperature by taking preventive measures such as choosing the right materials and proper insulation methods [1].

The porosity and mineralogy of the aggregate seem to exercise an important influence on the behavior of concrete exposed to fire. The factors that influence the strength of cement-based mortars and concrete under high temperatures can be divided into two groups: material properties and environmental factors. Properties of aggregate, cement paste and aggregate–cement paste bond, and their thermal compatibility between each other greatly influence the resistance of concrete. On the other hand, environmental factors such as heating rate, duration of exposure to maximum temperature, cooling rate, loading conditions, and moisture regime affect the heat resistance of cementitious materials [2–4]. In the case of elevated heating conditions, when the temperature reaches about 300 °C, the interlayer calcium silicate hydrate (CSH) water, and some of the combined water from the CSH and sulfoaluminate hydrates will evaporate [5]. Microcracks appear first (at about 300 °C) in the areas of $Ca(OH)_2$ concentration and next in the areas of unhydrated grains (at about 400 °C) [6]. High temperatures in the range of 400–600 °C may activate series of reactions in the hardened cement paste. These reactions commence with the complete desiccation of the pore system, followed by decomposition of hydration products and the destruction of C–S–H gels [7].

Silica fume (SF) is one of the materials which can be incorporated as active addition or substitution for ordinary type I Portland cement (OPC) concrete structures. The amorphous silica present in SF reacts with lime, principally originated during the hydration of Portland cement [8–10].

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ucts which enhance the properties and durability of the concrete against fire [8]. In addition, the thermal conductivity of crystalline silica is about 15 times that of amorphous. Therefore, it is natural for the concretes with amorphous silica to have lower thermal conductivity [11, 12].

Vermiculite (V), is a naturally occurring mineral, of chemical composition consists of a complex hydrated aluminum and magnesium silicate. Upon heating, V can expand 8–20 times from its original thickness. Such property called exfoliates. The expanded or exfoliated V has low bulk density, comparative high refractoriness, low thermal conductivity, and chemical inertness make V satisfactory for many types of thermal and acoustic insulations. For this, V can be used as a good additive in lightweight aggregate. Concrete with a low thermal conductivity has a better fire resistance, for instance, lightweight concrete stands up better to fire than ordinary concrete [13]. Lightweight aggregates such as pumice, foamed slag, and expanded clay products have a high resistance to fire, and concrete made from them has low heat conductivity [14]. In addition, these aggregates have high resistance to volume expansion and decomposition at elevated temperatures [15]. However, the use of V with OPC to increase its durability at elevated temperature was not studied earlier, although materials of similar properties studied like perlite [16, 17] and pumice [18, 19].

Our study aims to investigate the effect of high temperature treatment on the mechanical and physical properties as well as the thermal shock resistance of cement pastes containing different mass percentages of V and expanded vermiculite (EV) in presence and absence of SF.

Experimental

Materials

OPC supplied from Suez Cement Factory (Egypt) with a Blaine surface area 3,250 cm^2 g^{-1} was used in this study. V mineral supplied from The Red Sea for Phosphate Company, Egypt. EV obtained by heating V at 500 °C for 3 h. V and EV were ground before their use to particle size <0.125 mm. Table 1 shows the chemical oxide composition for OPC and

V. Condensed SF is a byproduct of silicon or ferrosilicon alloys industries. It is obtained from ferro-silicon Company; Kom-Ombo, Egypt. SF particles are spherical and have an average diameter of about 0.1×10^{-6} m. It is a $\sim$99% amorphous silica with specific surface area 20,000 m^2 kg^{-1}. These characteristics account for the substantial pozzolanic activity of SF in terms of both its capacity of binding lime and rate of reaction. Table 2 shows the percentage composition of the different mixes and their designations. Each dry mix was mechanically mixed in a porcelain ball mill for 12 h to assure complete homogeneity.

Preparation of the hardened cement pastes

Different cement pastes were prepared using W/S ratio = 0.30. Each paste was prepared by mixing the dry mix with the required amount of water for about 3 min. After complete mixing, the resultant paste was molded into cubic specimens by using 1 inch cube moulds. The moulds, containing the pastes, were cured in 100% relative humidity for 24 h, and then the cubic specimens were removed from the moulds and cured under water for 28 days.

Heat treatment and cooling process

After the curing period (28 days), six specimens from each mixture were exposed to 300, 600, and 800 °C for 3 h in the oven. The heating rate was set at 10 °C min^{-1}. Afterward, the hot specimens were cooled by two different ways. One group of specimens were left in a closed desiccator for slow or gradual cooling while the others were soaked in water ($\sim$20 °C) for rapid cooling. Hot specimens were left to cool until their temperatures drops to 20 °C. The cooling periods varied between 20 min and 2 h depending on heating temperatures and cooling method.

Techniques

The percentage of residual strength

Compressive strength test was performed on the cooled specimens. After the cooling period, three cubes representing each specimen were subjected to compressive

Table 1 Chemical oxide composition of OPC and V/wt%

Materials	Oxide content/%										
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Mn_2O_3	H_2O	LOI
OPC	20.46	5.14	3.53	61.28	3.20	2.90	0.11	2.82	0.11	–	0.40
V	12.30	42.81	12.10	–	13.8	–	6.34	–	–	12.58	–

Table 2 The percentage composition of the different mixes and their designations

Mixes	W/S	Mix proportion/wt%			
		OPC	V	EV	SF
A0	0.3	100	–	–	–
A1	0.3	97.5	2.5	–	–
A2	0.3	95	5	–	–
A3	0.3	90	10	–	–
B1	0.3	97.5	–	2.5	–
B2	0.3	95	–	5	–
B3	0.3	90	–	10	–
C1	0.3	85	5	–	10
C2	0.3	85	–	5	10

strength test and the mean value was recorded. This was accomplished using a Ton-industric machine (West Germany) for maximum load of 60 tons. The percentage of residual strength of cooled specimens was calculated as follows:

$$\text{Residual strength } \% = \frac{(\text{C.S.})_t}{(\text{C.S.})_0} \times 100$$

where $(\text{C.S.})_t$, value of compressive strength at temperature t (°C); $(\text{C.S.})_0$, value of compressive strength at 28 days of hydration.

Chemically combined water contents

The content of chemically combined water, Wn (%), was determined as follows: an exact mass of the dried cement paste was charged to a silica crucible and ignited for 2 h at 1,000 °C (20 °C min^{-1}) in a muffle furnace. The crucible was cooled in a desiccator then weighed at room temperature. Duplicate measurements were carried out for each sample and the mean value was recorded:

$$\text{Wn } (\%) = [(W_o - W_i)/W_i] \times 100$$

where W_o, mass of the dried sample before ignition; W_i, mass of sample after ignition.

Phase composition

The phase composition of the formed hydrates was investigated by X-ray diffraction using cobalt target ($\lambda = 0.17889$ nm), and nickel filter under working conditions of 40 kV and 40 mA.

Differential scanning calorimetry

Differential scanning calorimetry runs were conducted using a Shimadzu DSC-50 thermal analyzer at a heating

rate of 20 °C min^{-1}. The sample chamber was purged with nitrogen at a flow of 30 mL min^{-1}.

Results and discussion

The percentage of residual strength

Figure 1 shows the percentage of residual strength of control and cement mixes containing V and SF gradually cooled in air. As shown in Fig. 1, the compressive strength of the control mix (A0) increased by about 3.5% (relative to the value recorded at 28 days of hydration) by heating at 300 °C. However, the compressive strength decreased by about 48.4% upon heating at 600 °C and reached 100% loss in strength by heating at 800 °C. The cement paste containing 2.5 wt% V (mix A1) showed loss in strength by 12.4, 17.5, and 76% upon heating at 300, 600, and 800 °C, respectively. The cement pastes containing 5 and 10 wt% V (mixes A2 and A3) and that containing 5 wt% V + 10
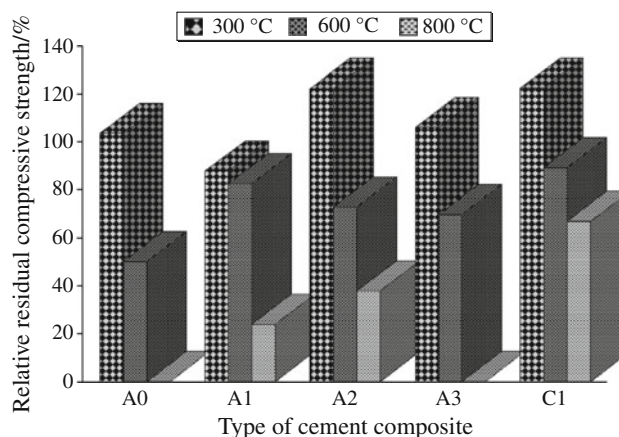

Fig. 1 Relative residual compressive strength of heated cement pastes containing V and SF and gradually cooled in air

wt% SF (mix C1) did not show a strength loss up to 300 °C. The strength increased by 22.2, 5.9, and 22.3% for mixes A2, A3, and C1, respectively, at 300 °C relative to their original values recorded at 28 days of hydration. The strength gained in control specimens by heating at 300 °C can be explained due to internal autoclaving process and filling up of pores with additional hydration products. In pastes containing SF an additional reason for increasing the compressive strength was due to its pozzolanic reaction with free $Ca(OH)_2$ leading to the formation of more CSH which form a denser and closer structure [20]. In addition, the consumption of free $Ca(OH)_2$ decreases the conversion of $Ca(OH)_2$ to lime and water vapor during heating which may lead to serious damage due to lime expansion upon the cooling period [21]. Also, the insulation properties of V mineral were responsible for increasing strength for mixes A2 and A3.

At 600 °C, all cement mixes containing V and SF and gradually cooled in air gained residual strength higher than the control mix (A0). Mix C1 showed the highest residual strength. At 800 °C, mixes A1, A2, and C1 lost about 76.03, 62.1, and 33.04% from their original strength, respectively, while mixes A0 and A3 lost all their strength at this temperature. Such results can be attributed to V expansion or exfoliate which occurs at 500 °C and hence leads to a decrease in the bulk density and thermal conductivity of cement pastes due to air voids formation between the V layers upon expansion. This leads to an improvement in the durability of cement pastes containing V at higher temperatures. Also, the presence of SF with V in mix C1 causes a marked increase in its residual strength compared to all the mixes studied.

The percentage of residual strength of cement specimens heated at 300, 600, and 800 °C and suddenly cooled in water are given in Fig. 2. Obviously, the percentage residual 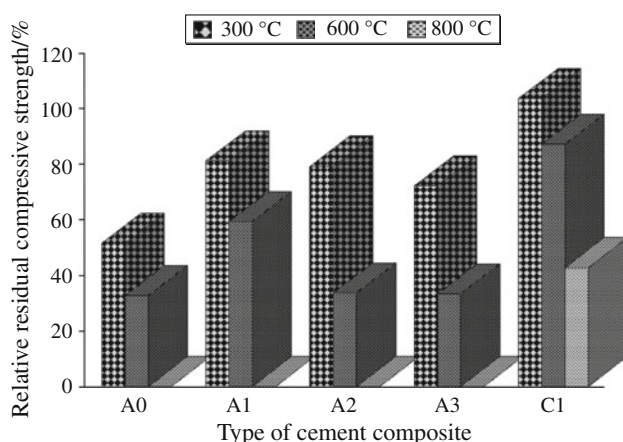strength for suddenly cooled specimens were lower than those of gradually cooled specimens. In addition, A0 showed continuous decrease in the percentage of residual strength with increasing the heating temperature from 300 to 600 °C till reached to zero at 800 °C. Also, mixes A1, A2, and A3 showed a similar behavior as A0 but the degrees of loss in the strength were less than in case of A0. The loss in residual strength increased with increasing the V content (i.e., from 2.5 to 10 wt%). However, mix C1 showed an increase in the percentage of residual strength upon heating at 300 °C by about 3.7%. At 600 and 800 °C the residual strength of mix C1 decreased by 12.9 and 57.21, respectively. The great loss in strength noted for sudden cooled specimens can be explained, besides the effects of high temperatures, in terms of the thermal shock that occurred by sudden cooling (using cooled water) of the heated specimens. This leads to an enlargement in the microcracks and formation of new cracks inside the heated specimens. Increasing the heating temperature increases the effect of thermal shock and this explain the great loss in strength in all specimens at 600 and 800 °C.

The cement mixes containing EV showed percentage of residual strength by heating at 300, 600, and 800 °C and gradually cooled in air or suddenly cooled in water are represented in Figs. 3 and 4. For gradually cooled specimens, the presence of EV improved the heat resistance of all mixes at all temperatures compared to the mixes containing non EV with the same wt% ratios. This behavior was also noticed for sudden cooled specimens heated at 300 and 600 °C. At 800 °C, all cement specimens containing EV lost their strength (mixes B1, B2, and B3). Mix C2 which contains 5 wt% EV + 10 wt% SF showed an increase in residual strength by 74.8 and 49% by heating at 300 and 600 °C, respectively, for gradual cooling specimens. At 800 °C, C2 lost about 28.2% in strength. Upon sudden cooling C2 gained strength by 6% at 300 °C and lost strength by 8.3 and 42.16% upon heating at 600 and
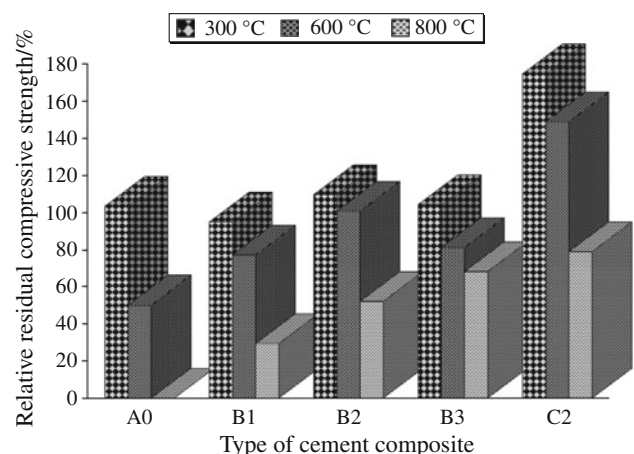

Fig. 2 Relative residual compressive strength of heated cement pastes containing V and SF and suddenly cooled in water

Fig. 3 Relative residual compressive strength of heated cement pastes containing EV and SF and gradually cooled in air
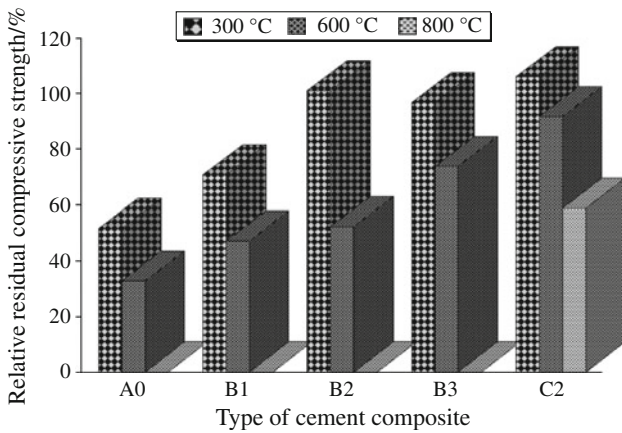
Fig. 4 Relative residual compressive strength of heated cement pastes containing EV and SF and suddenly cooled in water
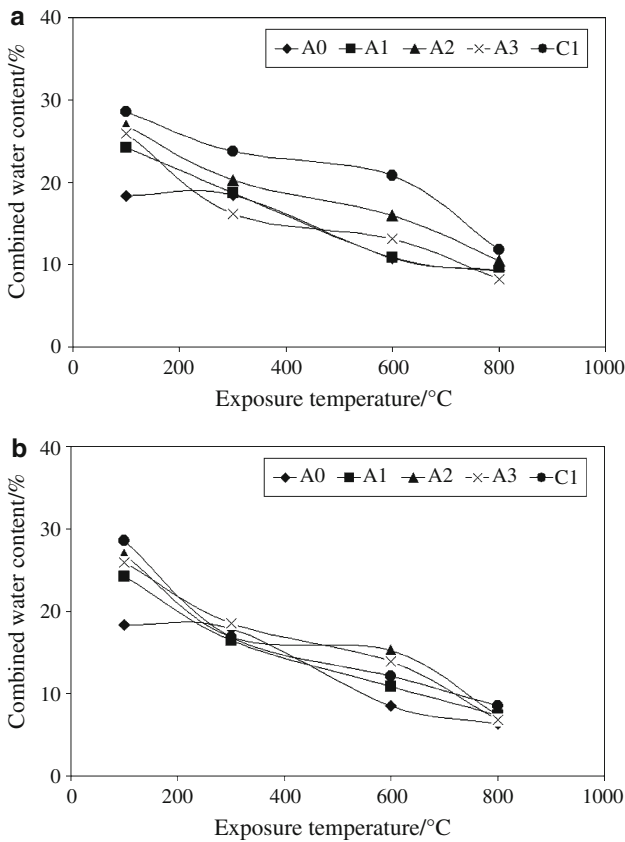

Fig. 5 Combined water content versus exposure temperature for hardened cement pastes containing V and SF. **a** Air cooled, **b** water cooled

800 °C, respectively. EV being a granular aggregate with numerous air voids. These air voids act as a good insulator for heat transfer which decrease the harmful effects occurred either by heating the cement specimens at higher temperatures (>500 °C) or the thermal shock due to sudden cooling of heated specimens in water.

Combined water content (Wn %)

The results of chemically combined water content of the heated specimens with two modes of cooling are represented graphically in Fig. 5a, b. All the blended specimens showed higher values of combined water at 28 days of hydration than the control specimen. Upon heating and cooling gradually in air, the control specimens showed combined water contents at 300 °C comparable to 28 day values while heating at 600 and 800 °C results in a continuous decrease in combined water contents. The specimens containing V (mixes A1, A2, and A3) showed a gradual loss in combined water contents by heating at 300, 600, and 800 °C. However, the values of combined water for these specimens were still higher than those of the control specimen at all treated temperatures. The presence of SF with V (mix C1) results in an increase in the values of Wn % and this mix showed the highest values of combined water at all treated temperatures (Fig. 5a).

For the heated specimens and suddenly cooled in water, the decrease in combined water contents in A0 specimens began by heating at 300 °C and continued to 600 and 800 °C. All the blended specimens showed higher values
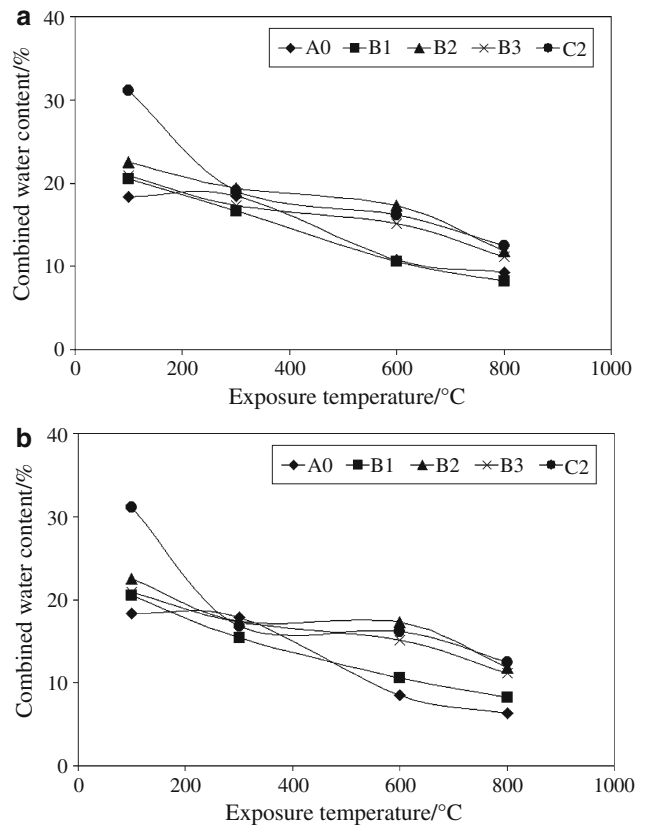

Fig. 6 Combined water content versus exposure temperature for hardened cement pastes containing EV and SF. **a** Air cooled, **b** water cooled

of combined water especially at 600 and 800 °C than those of the control specimens (Fig. 5b). A similar trend was observed for specimens containing EV with noticeable higher values of combined water at 600 and 800 °C than those of the control specimens (Fig. 6a, b).

Heating at 300 °C lead to evaporation of loosely bounded water in cement hydrates [5]. In addition, a self-autocleaving reaction occurred between the unhydrated cement grains leading to formation of more CSH which results in an increase in the gained Wn %. The net of these two opposite effects determines the obtained values of Wn % either comparable or less than 28-day values. Heating at higher temperatures, 600 and 800 °C leads to decomposition $Ca(OH)_2$ and CSH. Here, chemically combined water contents could be considered as a relative quantitative measure of the degree of degradation of these hydration products at the heating temperature. Therefore, the higher values of combined water recorded for cement specimens containing V and EV reflected the insulation properties of this material. Presence of SF, which acts as a pozzolanic material, with V was responsible for increased values of Wn %.

X-ray diffraction

XRD patterns of blank mix after 28 days of hydration and heated for 3 h at 300, 600, and 800 °C and gradually cooled in air are shown in Fig. 7. The XRD pattern of control mix, hydrated for 28 days, showed that the main phases formed in the hydration were portlandite (CH), CSH and $CaCO_3$. Unhydrated cement phases like β-C_2S as well as quartz also appeared in this pattern. Heating the control specimens at 300 °C caused an increase in the intensities of the peaks characterized to the hydration products. This is

attributed to the internal autoclaving reaction occurred to the unhydrated cement grains by heating at 300 °C [8, 20, 22]. Also, such increase in the intensity of the peaks of the hydration products confirms the increase in the compressive strength noticed by heating at such temperature. At 600 °C, the peaks characterized to CH are nearly disappeared due to its decomposition. In addition, the intensities of the peaks characterized to anhydrous phases (β-C_2S and C_3S) were increased which attributed to their recrystalization [20]. XRD patterns at 800 °C showed unexpected peaks for CH which should be completely decomposed at this high temperature. This is due to a recrystallization of the amorphous part of portlandite [22] after the heat treatment during the cooling in the furnace. This shows the importance to make the tests very quickly after a fire [23].

Presence of 5 wt% V mineral with OPC did not affect the hydration of OPC as shown from the XRD patterns of specimens A2 in Fig. 8. The same hydration products were formed with similar peaks intensities as A0 after 28 days of hydration. Also, after heating at 300, 600, and 800 °C, the same behavior of change of hydration products with temperature was noticed in A2 specimens as in control specimens. Such results lead to an important conclusion that, the thermal resistance found in specimens containing V was due to its insulation character rather than its pozzolanic reactivity. However, at 600 and 800 °C, in XRD of A2, new peaks were appeared which characterized to oyelite $[(CaO)_x \cdot SiO_2 \cdot ZH_2O]$ phase.

XRD patterns of C1 specimens (85% OPC + 10% SF + 5% V) at various heating temperatures with gradual cooling in air are shown in Fig. 9. It is noticeable that the decrease in the intensities of CH peaks after 28 days of hydration compared to those of A0. It is attributed to consumption of CH by the pozzolanic reaction of SF with

Fig. 7 XRD for A0 mix after heating at various temperatures and gradually cooled in air
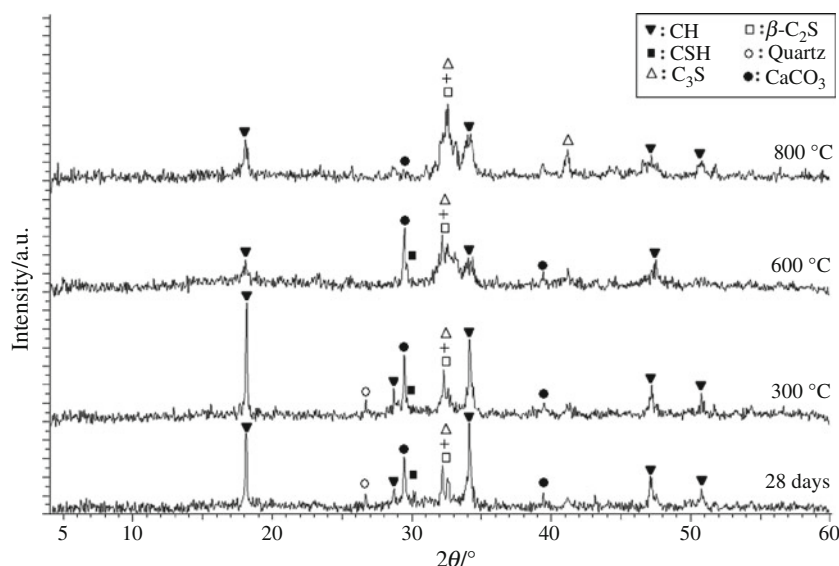
Fig. 8 XRD for A1 mix after heating at various temperatures and gradually cooled in air
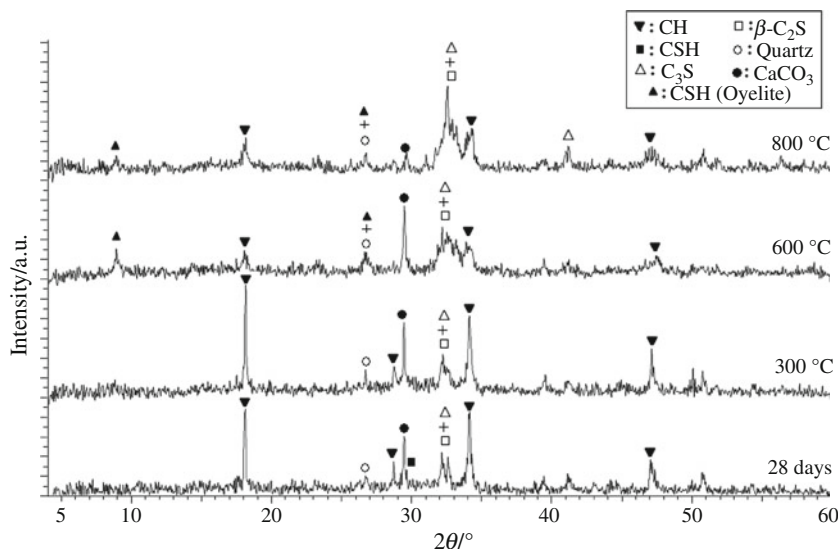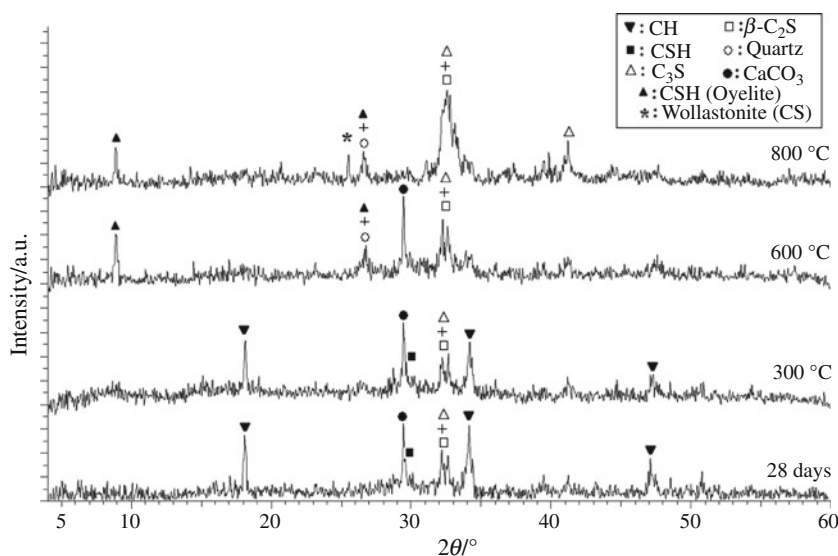

Fig. 9 XRD for C1 mix after heating at various temperatures and gradually cooled in air

it. Likewise, this explains the decrease in CH peaks at 300 °C. At 600 and 800 °C, no peaks observed to CH while the peaks characterized to unhydrated phases and oyelite phases appeared.

Nearly, the XRD patterns obtained for C2 specimens at various heating temperatures (85% OPC + 10% SF + 5% EV) were similar to that of the XRD patterns of C1 specimen. This indicates that the same hydration products were formed in C2 specimens as in C1 (Fig. 10). Also, the mechanism of EV in increasing the thermal resistance of OPC specimens is as that of normal V.

Differential scanning calorimetry

The results of DSC test of A0 specimens heated at various temperatures and cooled gradually in air are shown in Fig. 11. It can be noticed that the DSC curve for A0

specimen hydrated for 28 days shows three main endothermic peaks. The first located between 108 and 187 °C which results due to dehydration of CSH and calcium sulpho-aluminate hydrates. The second peak, at 458.7–480 °C, which represents a major mass loss, is corresponding to the dehydroxylation of portlandite [23–25]. At 718–761 °C, a third endothermic peak appears corresponding to the decarbonation of calcium carbonate [25, 26].

Let us consider the behavior of A0 specimens after heat treatment. The first endothermic peak correlated to dehydration of CSH nearly disappeared for specimens previously heated at 600 and 800 °C. While, the second peak, corresponding to dehydroxylation of Ca(OH)$_2$ remains for samples fired beyond 600 °C although, the samples were previously heated at 600 and 800 °C for 3 h, and the dehydroxylation of Ca(OH)$_2$ should be completed. Moreover, it is noticeable that the beginning of the endothermic

Fig. 10 XRD for C2 mix after heating at various temperatures and gradually cooled in air
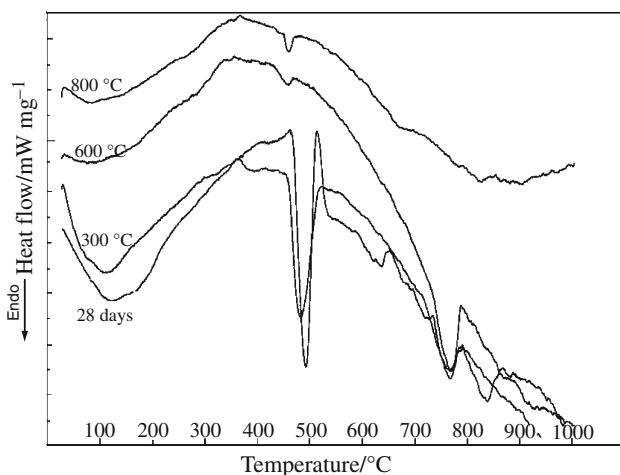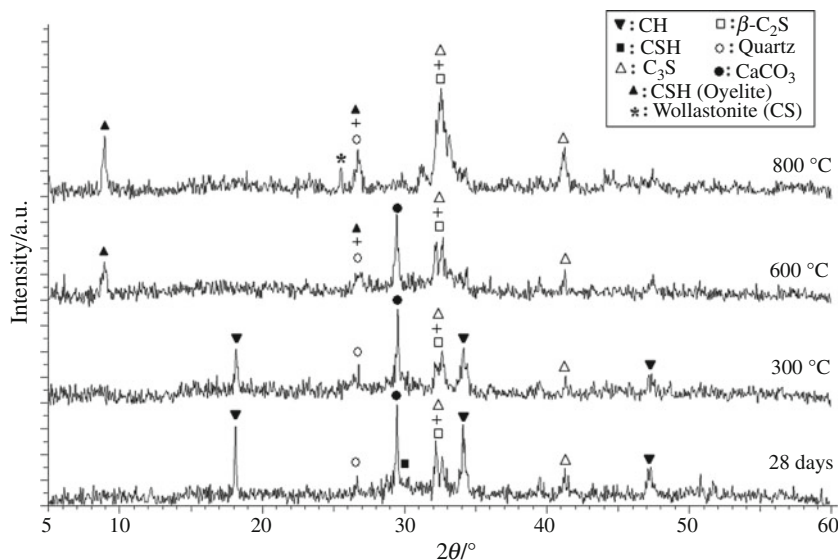

Fig. 11 DSC curves for A0 mix after heating at various temperatures and gradually cooled in air
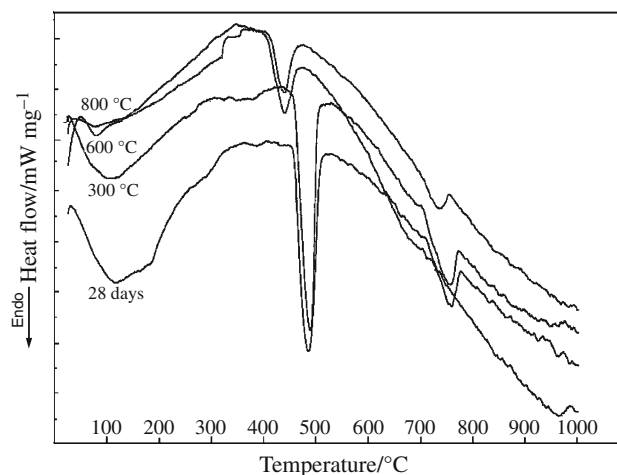

Fig. 12 DSC curves for A1 mix after heating at various temperatures and gradually cooled in air

peak seems to be shifted toward lower temperatures when the temperature of the previous heat treatment exceeds 600 °C. Such result is in consistence with the results of Alarcon-Ruiz et al. [27]. He studied the effect of high temperatures on cement pastes using thermal analysis techniques and deduced that, the dehydroxylation reaction of $Ca(OH)_2$ which occurs at ≥ 500 °C is reversible.

A0 specimen heated at 800 °C did not show the third endothermic peaks corresponding to the decarbonation of calcium carbonate. This is attributed to the complete of the decarbonation process for previously heated specimens.

The cement specimens containing 5 wt% V (A2) and thermally treated at various temperatures show the same endothermic peaks in their DSC curves as A0 specimens (Fig. 12). In addition, the change of these endothermic peaks with heating temperature shows the same trend as A0

samples. However, there is a relative increase in the first endothermic peak for A2 specimens heated at 300 °C compared to those of A0. This peak corresponding to dehydration of CSH involved here also the dehydration of V. This led to evaporation of bounded water inside the layers of V and increasing its volume [28, 29].

Figures 13 and 14 show the DSC curves of C1 and C2 specimens, respectively. Here in specimens containing SF with OPC, the endothermic peaks correspond to dehydroxylation of $Ca(OH)_2$, were relatively lower than those of A0 specimens at 28 days of hydration and at 300 and 600 °C and completely disappeared at 800 °C. This endothermic peak could be used as a quantitative measure for the presence of $Ca(OH)_2$. The disappearance of $Ca(OH)_2$ peak correlated to its consumption in these specimens via the reaction with SF to form more CSH.
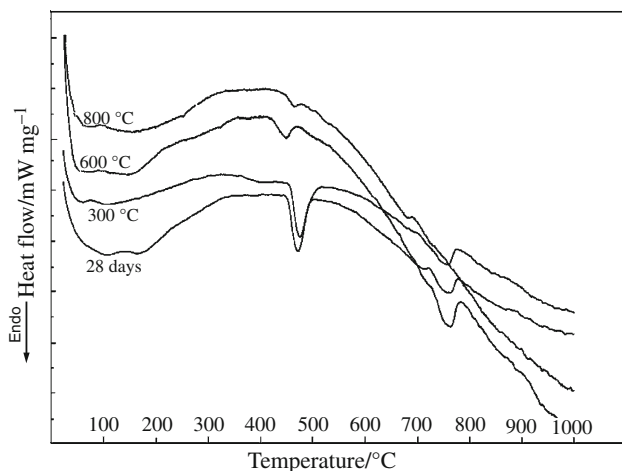
Fig. 13 DSC curves for C1 mix after heating at various temperatures and gradually cooled in air
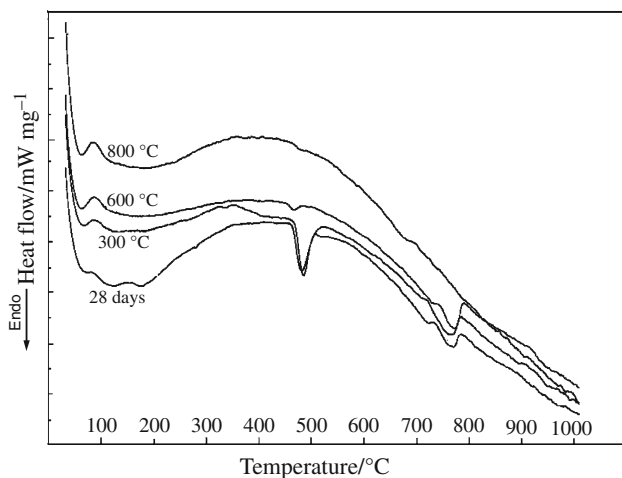

Fig. 14 DSC curves for C2 mix after heating at various temperatures and gradually cooled in air

Conclusions

On the basis of the results of our study, we concluded that:

1. OPC pastes blended with V have good heat resistance properties, especially with 5 wt% addition.
2. EV improves the heat resistance of the OPC hardened pastes more efficiently than the non-EV.
3. The mechanical properties of hardened pastes are noticeably affected by the cooling method. A remarkable reduction in percentage residual strength was observed for pastes cooled in water than those cooled in air, probably due to formation of microcracks created by thermal shock.
4. Addition of 10% SF to the OPC pastes blended with 5% V causes a noticeable increase in the values of percentage residual strength and chemically combined water content.
5. From the XRD and DSC analyses; the presence of V does not affect the hydration reaction of the OPC; but increases its heat resistance through its insulation properties.

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