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Cement and Concrete Research 37 (2007) 988–995

Cement and Concrete Research

Effect of pumice and fly ash incorporation on high temperature resistance of cement based mortars

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Received 22 September 2005; accepted 12 February 2007

Abstract

The effects of high temperature on the mechanical properties of cement based mortars containing pumice and fly ash were investigated in this research. Four different mortar mixtures with varying amounts of fly ash were exposed to high temperatures of 300, 600, and 900 °C for 3 h. The residual strength of these specimens was determined after cooling by water soaking or by air cooling. Also, microstructure formations were investigated by X-ray and SEM analyses.

Test results showed that the pumice mortar incorporating 60% fly ash revealed the best performance particularly at 900 °C. This mixture did not show any loss in compressive strength at all test temperatures when cooled in air. The superior performance of 60% FA mortar may be attributed to the strong aggregate–cement paste interfacial transition zone (ITZ) and ceramic bond formation at 900 °C. However, all mortar specimens showed severe losses in terms of flexural strength. Furthermore, specimens cooled in water showed greater strength loss than the air cooled specimens. Nevertheless, the developed pumice, fly ash and cement based mortars seemed to be a promising material in preventing high temperature hazards.

Keywords: High temperature; Mortar; Pumice; Fly ash; Mechanical properties; Microstructure

1. Introduction

High temperature effect on concrete is one of the most important physical deterioration processes that affect the durability of structures. 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.

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 [1–4].

In the case of elevated heating conditions, when the temperature reaches about 300 °C, the interlayer Calcium Silicate Hydrate (C-S-H) water, and some of the combined water from the C-S-H 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 to 600 °C may activate a 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]. The decomposition of calcium hydroxide does not generally occur below 350 °C. The conversion of calcium hydroxide into lime and water vapor during heating is not critical in terms of strength loss. Nevertheless, it may lead to serious damage due to lime expansion during the cooling period [8]. The detrimental effects of $Ca(OH)_2$ can be eliminated by using mineral admixtures such as, fly ash and ground

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0008-8846/$ - see front matter © 2007 Elsevier Ltd. All rights reserved.
doi:10.1016/j.cemconres.2007.02.005

granulated blast furnace slag. Due to the pozzolanic reaction between $Ca(OH)_2$ from cement and reactive SiO_2 from these mineral admixtures, the amount of $Ca(OH)_2$ decreases in the system. The useful contribution of fly ash on high temperature resistance was proved by Dias [9] and Sarshar [2].

The porosity and mineralogy of the aggregate seem to exercise an important influence on the behavior of concrete exposed to fire. Siliceous aggregates containing quartz, such as granite and sandstone, may cause distress in concrete at about 573 °C since the transformation of quartz from α to β form is associated with a sudden expansion of the order of 0.85%. In the case of carbonate rocks; a similar distress may begin above 700 °C as a result of the decarbonation reaction. In addition to possible phase transformations and thermal decomposition of the aggregate, the response of concrete to fire is influenced in other ways by aggregate mineralogy. For instance, aggregate mineralogy determines the differential thermal expansions between the aggregate and the cement paste and ultimate strength of the transition zone [5]. Therefore, the first criteria to be considered when selecting a high temperature-resistant aggregate is its thermal stability, both physical and chemically. Also, the aggregate should produce a strong temperature-resistant bond with the cement paste, and be thermally compatible with it whenever possible [1]. The loss of strength is considerably lower when the aggregate does not contain silica such as; limestone, basic igneous rocks, and particularly with crushed brick and blast furnace slag. Concrete with a low thermal conductivity has a better fire resistance, for instance, lightweight concrete stands up better to fire than ordinary concrete [10]. Lightweight aggregates such as pumice, foamed slag, and expanded clay products have high resistance to fire, and concrete made from them has a low heat conductivity [11]. In addition, these aggregates have high resistance to volume expansion and decomposition at elevated temperatures [12].

Table 1
Physical, chemical and mechanical properties of cement and fly ash

Chemical composition (%)	Cement	Fly ash
SiO_2	19.30	42.14
Al_2O_3	5.57	19.38
Fe_2O_3	3.46	4.64
CaO	63.56	26.96
MgO	0.86	1.78
Na_2O	0.13	–
K_2O	0.80	1.13
SO_3	2.91	2.43
Loss on ignition	2.78	1.34
Insoluble residue	0.42	–
Free CaO (%)	1.22	4.34

Physical and mechanical properties of cement	
Specific gravity	3.15
Blaine specific surface area (m^2/kg)	352
Initial setting time (min)	145
Final setting time (min)	275
Volume expansion (mm)	1.00
Compressive strength (MPa) of cement	
2 days	27.2
7 days	42.4
28 days	52.7
Pozzolanic activity index of (%) FA	
7 days	79
28 days	88
Specific surface of fly ash (m^2/kg)	290

Table 2
Grading, physical and chemical properties of pumice

Chemical composition (wt.%)	
SiO_2	75.51
Fe_2O_3	1.10
Al_2O_3	9.94
CaO	0.25
MgO	0.04
Na_2O	2.04
K_2O	5.12
TiO_2	<0.01
P_2O_5	<0.001
LOI	4.27
Physical properties	
Bulk specific gravity	2.03
Water absorption (%)	6.38
Unit weight (kg/m^3)	
Loose	1220
Compacted	1380
Sieve size (mm)	**% passing**
4	100.0
2	91.5
1	75.4
0.5	52.5
0.25	27.4

Pumice is essentially an aluminum silicate of igneous origin with a cellular structure formed by a process of explosive volcanism, because the cellular structure, lightweight and insulating properties of pumice has been extensively used as a building construction material. Türker et al. [12] have investigated the role of aggregate type on high temperature resistance of mortars and they have found that the pumice aggregate mortar, which does not show compressive strength loss up to 500 °C, is more resistant to high temperature than quartzite, and limestone. Yazıcı et al. [13] also investigated high temperature resistance of pumice mortar and they have found that the pumice aggregate mortar gain a compressive strength value of 41% at 600 °C, while conventional natural river sand mortar lost 39% of their strength.

The purpose of this study is to develop a high temperature-resistant mortar by using pumice and fly ash that are known as high temperature-resistant materials.

2. Materials and experimentation

The cement used in mortar mixtures was ordinary Portland cement (CEM I 42,5) a product of Cimentas Cement Plant, Izmir, TURKEY. The chemical and physical properties of cement and C type of fly ash (FA) procured from Soma B power

Table 3
Mix design of all mortar mixtures

Mixture	Fly ash (%)	Water/binder	Flow (mm)	Unit weight in fresh state (kg/m^3)
FA0	0	0.72	111	1968
FA20	20	0.74	110	1920
FA40	40	0.76	110	1900
FA60	60	0.78	111	1868

plant are presented in Table 1. Pumice was procured from Menderes region of Izmir. Its gradation, physical and chemical properties are shown in Table 2.

Four types of mortar mixtures were prepared by replacing cement with fly ash at different ratios of 0%, 20%, 40%, and 60%. The material compositions of all mixtures are presented in Table 3. The pumice to binder (cement and fly ash) ratio of 3 was maintained for all mixtures. All batches were prepared by using a mechanical mixer confirming to the requirements of ASTM C305. Cement, fly ash and pumice were dry mixed for 3 min initially and then water was gradually added while mixing continued for about 5 min. Water was added at different ratios to provide a constant fluidity of about 110±1 mm. The flow test was performed according to ASTM C230. Fresh mixtures were cast into prismatic (40 × 40 × 160 mm) steel molds. The specimens were left in the molds for 24 h at room temperature of 20 °C. After demolding, the specimens were kept in a curing room at a temperature of 20 °C and relative humidity of 90±5% for 27 days. After the curing period, twelve specimens from each mixture were exposed to 300, 600 and 900 °C temperatures for three hours in the oven. The heating rate was set at 10 °C/min. Afterwards, the hot mortar specimens were cooled by two different ways. One group of specimens were left in laboratory conditions for slow cooling while the others were soaked in water (~ 20 °C) for rapid cooling. Hot specimens were left to cooling until their temperatures drops to 20 °C. The cooling periods varied between 20 min–2 h depending on heating temperatures and cooling method. After the cooling period, the prismatic specimens were subjected to flexural strength test according to ASTM C348. Six specimens were tested for each stage and average values were recorded. The specimens were loaded from their mid span and the clear distance between simple supports was 120 mm. The compressive strength tests were performed following the flexural tests. The two broken pieces left from flexural test were subjected to compressive strength test. The flexural and compressive strength test results were compared with the test results of unheated mortar specimens.

In terms of thermal and fire insulation, coefficient of thermal conductivity of materials is of great importance. The coefficients of thermal conductivity of mortars were measured on unheated 40 × 40 × 160 mm specimens. A quick thermal conductivity meter based on ASTM C 1113–90 Hot Wire Method has been used to measure the thermal conductivity [14]. The specimens were dried at the age of 28 days in an oven at 105±5 °C for 24 h prior to testing.

3. Results and discussion

There are three test methods available for determining the residual compressive or flexural strength of concrete at elevated temperatures: stressed test, unstressed test and unstressed residual strength test. In stressed test method, there is a constant load on the specimen during heating period. When the test temperature is reached, the specimen is loaded up to failure. In unstressed test, specimen is heated to the test temperature in unloaded condition and then the load is increased until failure occurred. In unstressed residual test, the specimen is heated in unloaded condition to test temperature. After the cooling period, the load is applied on the specimen. The first two types of test are suitable for assessing the strength of concrete at high temperatures, while the latter is proper for determining the residual properties after the high temperature exposure [15,16]. It is well known that the last method gives the lowest strength values and is therefore more suitable for finding the limiting values and due to this fact third method has been selected for this research.

Compressive and flexural strength of air cooled and water cooled mortar specimens at 20 °C (control specimen), 300 °C, 600 °C and 900 °C are given in Table 4. As seen in Table 4, FA replacement resulted in a decrease of compressive and flexural strength of mortar specimens at room temperature.

3.1. Residual compressive strength

Relative residual compressive strength of air cooled and water cooled mortar specimens are shown in Figs. 1 and 2, respectively. As shown in Fig. 1, the pumice mortar with fly ash did not show a strength loss up to 600 °C. The residual compressive strength of all air cooled specimens increased

Table 4
Compressive and flexural strength of all mixtures

Mixture	Compressive strength (MPa)							Flexural strength (MPa)						
	20 °C	300 °C		600 °C		900 °C		20 °C	300 °C		600 °C		900 °C	
		Air	Water	Air	Water	Air	Water		Air	Water	Air	Water	Air	Water
FA0	45.6	55.9	37.0	43.7	31.5	14.4	12.8	9.0	8.6	5.6	6.1	3.6	1.8	1.0
FA20	39.4	49.3	31.5	42.2	27.7	21.3	18.8	8.6	7.4	4.2	6.0	3.4	2.8	1.2
FA40	35.8	44.5	27.3	40.8	24.1	28.3	25.9	8.4	7.3	3.9	4.8	2.4	3.2	1.4
FA60	22.2	27.5	15.0	22.6	13.8	26.4	24.0	5.8	5.1	2.1	3.1	1.4	3.5	1.1

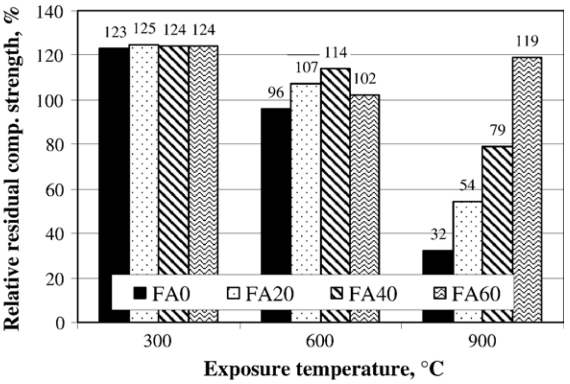

Fig. 1. Relative residual compressive strength of air cooled mortars.

about 24% at 300 °C in comparison with control specimens. The strength gain up to 300 °C can be partially due to the strengthened hydrated cement paste during the evaporation of free water, which leads to greater Van der Waal's forces as a result of the cement gel layers moving closer to each other [1,9,17]. Because the transportation of moisture in mortar is rather gradual, residual moisture in mortar allowed accelerated hydration at the early stage of heating mortars to high temperatures. Further hydration of cementitious materials is another important cause of the hardening of hydrated cement paste. For FA0 mortar, the temperature leads to additional hydration products from the unhydrated cement grains. For FA incorporated mortars, besides unhydrated cement, unhydrated FA particles can react with calcium hydroxide and produce C-S-H like gels [1,17,18]. The residual compressive strength of specimens at 600 °C is also higher than room temperature except FA0. This composition showed a negligible strength loss at this temperature level. The superior performance of pumice mortar is closely related to similar behavior of cement paste and aggregate at high temperatures. Pumice is lightweight aggregate with shrinking properties under high temperatures in contrast with normal aggregates that have expanding characteristics. Harmathy and Allen found that pumice concretes exhibit considerable shrinkage at temperatures above 315 °C [19]. In this study, length measurement on 40*40*160 mm mortar specimens also showed that pumice mortar has shrinkage potential under high temperature in accordance with Harmathy and Allen [19]. For instance, FA60 mortar showed a shrinkage of about 0.2, 1.2 and 2.5% after exposure to 300, 600 and 900 °C, respectively. Cement paste also shrinks at temperatures above 149 °C [20]. The similar shrinking behavior of aggregate and binder prevents the development of microcracks at ITZ. However, when the temperature elevated to 900 °C, FA0 specimens exhibited severe strength loss about 68%, while FA60 mixture gained 19% strength compared to the control specimens. The beneficial effect of FA replacement was observed clearly at 900 °C. At this temperature level, the relative residual compressive strength of the specimens increased depending on their FA content. The relative residual compressive strength values for FA0, FA20, FA40, and FA60 were found as; 32%, 54%, 79% and 119%, respectively. Furthermore, FA0 specimens revealed the highest residual compressive strength values up to 600 °C, but when the temperature elevated to 900 °C, FA0 specimens exhibited lower residual compressive strength values than the mixtures with fly ash. The severe strength loss of FA0 specimens between 600 to 900 °C is due to the decomposition of C-S-H gel [5,8,15,21]. The maximum residual compressive strength was found for FA40 specimens with a value of 28.3 MPa (Table 4).

Relative residual compressive strength of water cooled mortar specimens is shown in Fig. 2. The residual compressive strength values of water cooled mortar specimens are smaller than the air cooled specimens at all temperatures. This result can be attributed to the formation of microcracks as a result of large thermal gradients to be set up within the concrete (thermal shock), and the increment of water saturation degree of specimen [2]. In addition, above 400–500 °C, free calcium hydroxide turns to CaO (quick lime) by losing water. If CaO gets in contact with water, it rehydrates to $Ca(OH)_2$ accompanied by an expansion in volume. Both of these phenomena are potentially detrimental to strength. However, the differences between air cooled and water cooled strength values were more pronounced at 300 °C than 600 °C and 900 °C. This is due to most of the microcracks having taken place at relatively high temperatures of 600 °C and beyond [3]. As shown in Fig. 2, the compressive strength of mortar specimens decreased about 19–32% at 300 °C and 30–38% at 600 °C. With further increase in temperature up to 900 °C, the specimens which contain FA up to 40% exhibited strength loss of 28–72%. However, FA60 specimens gained strength about 8% compared to the control specimens. Nevertheless, at 900 °C, FA40 mixture still had the highest strength value of 25.9 MPa. This was followed by FA60, FA20, and FA0 mixtures. For 300 °C and 600 °C, with the increasing FA content, the relative residual compressive strength tend to decrease while at the 900 °C the relative residual compressive strength increased.

3.2. Residual flexural strength

The variation of flexural strength of air cooled and water cooled mortar specimens are shown in Figs. 3 and 4, respectively. The deteriorating effect of elevated temperatures

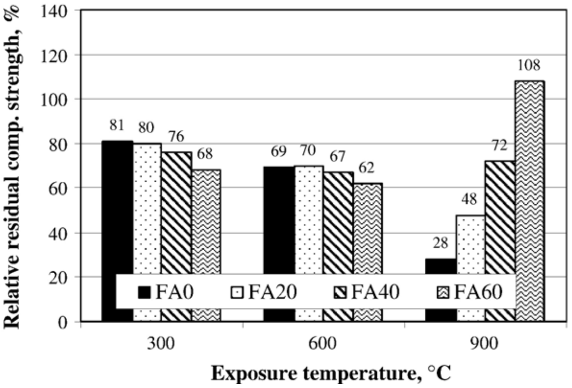

Fig. 2. Relative residual compressive strength of water cooled mortars.

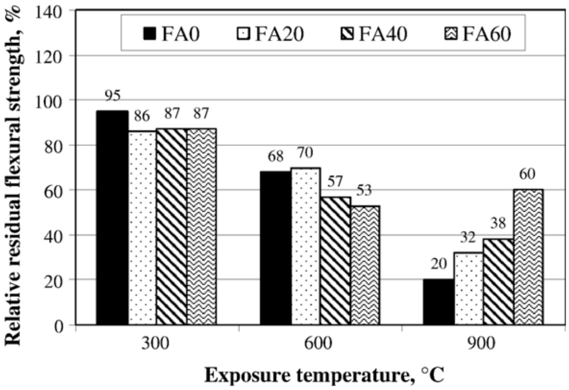

Fig. 3. Relative residual flexural strength of air cooled mortars.

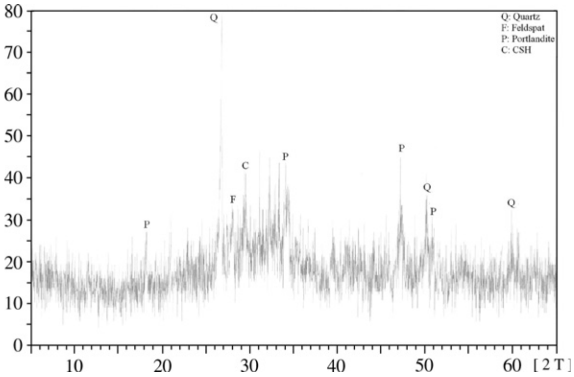

Fig. 5. X-ray analysis of cement paste (60% FA) at 20 °C.

on flexural strength of mortar specimens was more severe than on compressive strength. All mixtures have lost some flexural strength at all temperature levels, distinct from the case of compression. The existence of cracks reduces a valid area of cross sections and the existence of tensile stress causes expansion of cracks. Due to this fact, microcracks that form at elevated temperatures are more destructive on flexural strength than compressive strength [22–24].

Fig. 3 shows the variation of relative residual flexural strength of air cooled mortar specimens. The maximum relative residual flexural strength was obtained for FA0, FA20 and FA60 mixtures for 300 °C, 600 °C and 900 °C, respectively. The increment of FA content results in an increase of relative residual flexural strength at 900 °C. Similar to compressive strength, the maximum strength loss at 900 °C was observed for FA0 mixture, as 80%. As shown in Table 4, the maximum residual flexural strength was obtained for FA60 specimens with a value of 3.5 MPa. This was followed by FA 40, FA20, and FA0, respectively.

Fig. 4 shows the relative residual flexural strength of water cooled mortar specimens. Similar to compressive strength, the relative residual strength of water cooled specimens is less than air cooled specimens. At 300 °C and 600 °C, strength loss increases with the increasing FA content, but at 900 °C, the relative strength increases with the increasing FA ratios.

According to the test results, the beneficial effect of fly ash in terms of high temperature resistance is more pronounced at 900 °C. This result is in contrast with the findings of some other researchers [9,15].

3.3. Microstructure investigations

Microstructure investigations consist of X-ray analyses and SEM investigations. SEM investigations realized on FA0 and FA60 mortar specimens while the X-ray analyses realized on pumice aggregate, and 60% FA incorporated cement paste with 0.35 water/binder ratio. Cement paste specimens were cured and exposed to temperature under same conditions. These analyses were performed on air cooled specimens.

3.3.1. X-ray analyses

X-ray analyses of pumice showed that pumice consists of quartz and feldspat at 20 °C and also at 900 °C. In other words, no visible change has been observed in the pumice at 900 °C.

X-ray analyses of cement paste which incorporates 60% of FA at 20 and 900 °C are given in Figs. 5 and 6, respectively. For the 20 °C case, the paste has been composed of quartz, feldspat, portlandite, and C-S-H phases. After exposure to 900 °C, portlandite and C-S-H phases have disappeared. And, the gehlenite formation in glassy phase has appeared in a molten media in place of C-S-H and portlandite. The increase of compressive strength of mortars at 900 °C heat level can be attributed to the compact structure formation due to filling of pores by this molten phase. However, in hot state mechanical

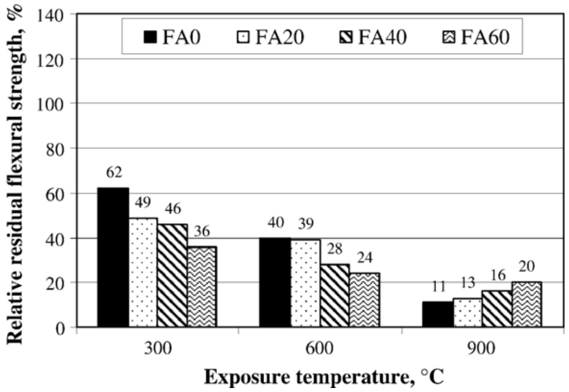

Fig. 4. Relative residual flexural strength of water cooled mortars.

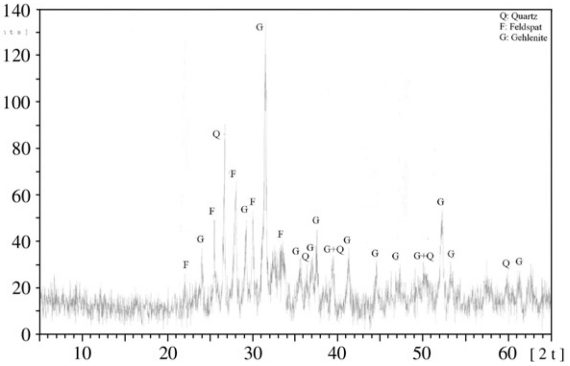

Fig. 6. X-ray analysis of cement paste (60% FA) at 900 °C.

properties of the similar gehlenite formed mortars have been decreased according to the Altun studies [25].

3.3.2. SEM investigations

SEM observations of FA0 mortars at 900 °C are shown in Fig. 7. After exposure to 900 °C, all the hydrated phases including C-S-H and CH appeared as amorphous structures by losing their characteristic crystal structure. Small, rounded formations were observed in place of C-S-H crystals (Fig. 7a). Crystals with rounded shapes may be β-C_2S, which is one of the decomposition products of C-S-H at elevated temperatures, as confirmed by literature [12]. Furthermore, cement paste tend to separate from the aggregate at ITZ thereby creating gaps that leads to cracks in cement paste at 900 °C (Fig. 7b).

SEM observations of FA60 mortar at 900 °C can be seen from Fig. 8. At this temperature level, the space ratio in cement paste has increased significantly (Fig. 8a). The formation of ceramic bond has been observed in cement paste at this temperature. XRD analyses of FA60 pastes after 900 °C confirm this finding. At this temperature one of the main ceramic mineral of gehlenite were detected (Fig. 6). Besides, its known that hydraulic binding break down at high temperatures of about 800 °C, and ceramic binding takes place at higher temperatures with an increase in residual, though not hot, strength [1]. The bond between aggregate and cement paste seems to be quite strong (Fig. 8b). This indicates that thermal compatibility of pumice with FA incorporated cement paste is greater than the paste which consist of only cement. Length measurements showed that FA incorporation affected the shrinkage behavior of pumice mortar significantly. After exposure to 900 °C, FA0 mortar showed 1.9% shrinkage, while it was 3.5% for FA60. The compressive strength gain at 900 °C may be attributed to conversion of microstructure of the paste from hydraulic to ceramic type. Also the bond at ITZ is much stronger than the normal room-temperature state.

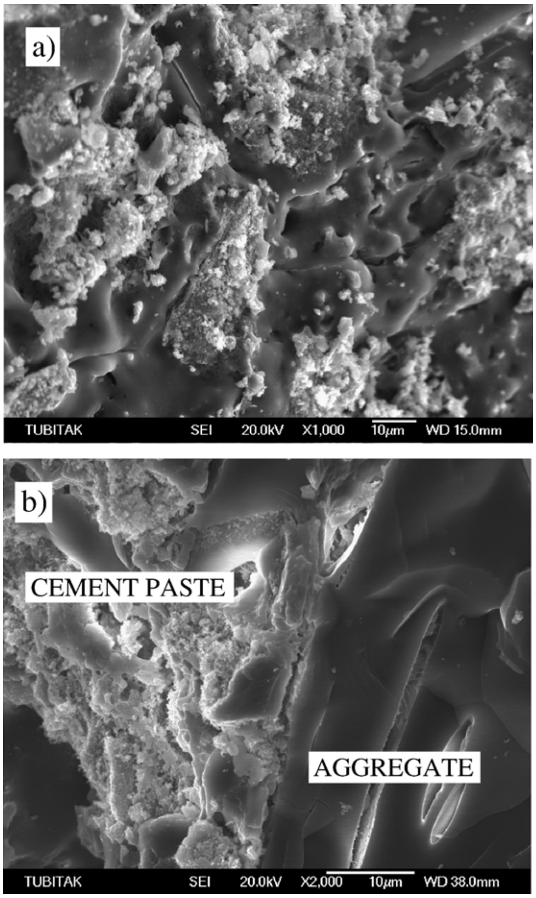

Fig. 7. SEM analysis of FA0 mixture at 900 °C, a) cement paste, b) ITZ.

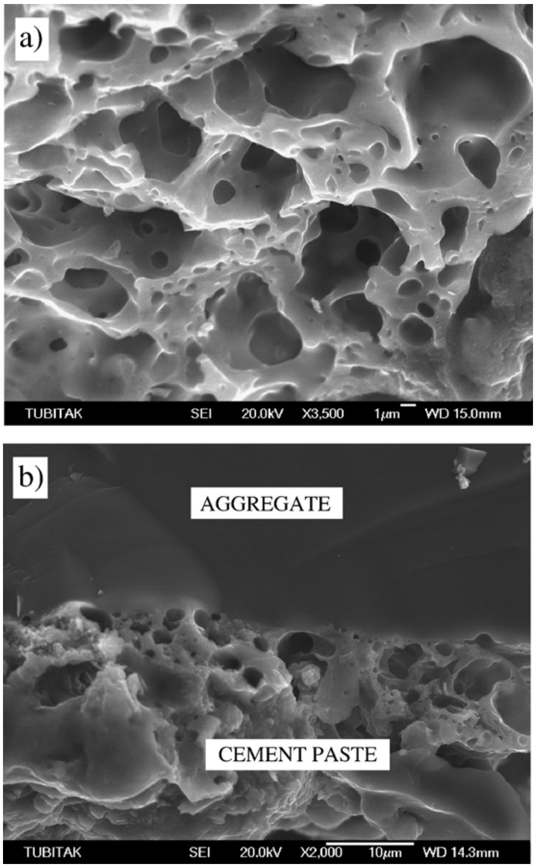

Fig. 8. SEM analysis of FA60 mixture at 900 °C, a) cement paste, b) ITZ.

The brittle behavior and decrease of flexural strength may be attributed to the typical behavior of ceramics.

3.4. Thermal conductivity of mortar

The mortars incorporating pumice has been tested comparatively in order to determine thermal conductivity properties. Also, a standard mortar of natural sand:cement:water mass ratios equal to 3:1:0.5 were tested under the same testing conditions. The coefficient of thermal conductivity of standard mortar was found as 1.516 W/mK whereas, the coefficient of thermal conductivity of FA0, FA20, FA40 and FA60 mortars were found as 0.657, 0.630, 0.596 and 0.522 W/mK, respectively. These results indicate that FA replacement causes considerable reduction in thermal conductivity. The reduction for FA60 was 20% compared to FA0. Reductions were partly due to reduced density with increasing FA content (Table 3) and partly to the amorphous structure of minerals, since the thermal conductivity of crystalline silica is about 15 times as that of amorphous, it is natural for the mortars with amorphous silica to have lower conductivity [26,27]. Furthermore, the coefficient of thermal conductivity of FA60 mortar is about one-third of standard mortar as a result of usage of lightweight aggregate of pumice in place of sand and using of FA in place of cement.

Due to low thermal conductivity parameters the mortars incorporating pumice and fly ash seems to be an ideal insulation composition especially for use in plastering jobs and building blocks and panels.

4. Conclusions

Pumice aggregate mortar with cement as a binder (FA0) has a good high temperature resistance up to 600 °C. This mortar showed only about 4% compressive strength loss and 32% flexural strength loss at 600 °C (cooled in air). However, at the temperature of 900 °C, the mechanical properties of this mixture dropped significantly. The residual compressive strength and flexural strength at this temperature was merely 32% and 20%, respectively.

The positive effect of fly ash incorporation on high temperature resistance has been observed clearly at 900 °C. The replacement of cement with fly ash improved high temperature resistance of pumice mortar significantly. At 900 °C, 68% of the compressive strength has been lost for FA0 specimens whereas FA60 specimens gained 19% strength compared to control specimens (cooled in air).

The best performance has been observed with FA60 specimens. These specimens cooled in air did not exhibit any loss in compressive strength at all test temperatures. The superior performance of this mixture may be attributed to the formation of ceramic type microstructure and stronger aggregate–cement paste ITZ at 900 °C.

X-ray analyses showed that pumice did not display any significant changes at 900 °C. However, 60% FA incorporated cement pastes undergo important changes at 900 °C. At this temperature, gehlenite phase formation was observed instead of C-S-H and portlandite phases.

Specimens exposed to elevated temperatures showed higher decrease in flexural strength than compressive strength due to microcracks and brittle microstructure formation which were more destructive when the specimens were subjected to tensile stress.

The mechanical properties of mortars are noticeably affected by the cooling method. A remarkable reduction in strength was observed for mortars cooled in water than cooled in air, probably due to formation of microcracks created by thermal shock imposed on hot specimens. The differences between air cooled and water cooled strength were more pronounced at 300 °C than 600 °C and 900 °C.

The developed pumice-fly ash-cement composition seems to be a promising insulation material (blocks, mortar) for structures that are exposed to high temperatures. This mortar seems to be a suitable material for thermal insulation purposes due to its low thermal conductivity with a value of 0.522 W/mK which is one-third of standard mortar.

Acknowledgement

The authors would like to thank The Scientific and Technical Research Council of Turkey (TÜBİTAK) for the financial support, Project no: ICTAG I679.

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