Beneficial Use of Coal Bottom Ash in Production of Geopolymer Mortars

İ. B. Topçu, M. U. Toprak

***Abstract*—** The alkali activation of waste materials is a chemical process that allows the user to transform glassy structures into inexpensive cement-like composites called geopolymers. Coal bottom ash (CBA) can be used as a raw material in geopolymer without any thermal activation. The present study supply results of an extensive experimental program that aims identifying the optimal mix composition and curing parameters for CBA geopolymers (CBAG) in terms of early and later age strengths. Compressive strengths of alkali (NaOH, Na_2SiO_3) activated CBAG were 18 and 27 MPa after 7 and 28 days of storage at room temperature. CBAG cured at 80 ° C oven for 24 h produced a material with compressive strength of 25 MPa. CBAG possessed superior thermal stability up to 400 °C. The strength loss of CBAG was only 5.71 % after 50 freeze-thaw cycles according to TS EN 12091.

***Keywords*—** Coal bottom ash, geopolymer, curing parameters, compressive strength, high temperature, freeze-thaw effect.

I. Introduction

Coal-fired power generation is a principal energy source throughout the world. Approximately, 70-75% of coal combustion residues are fly ash (FA) and the remainder is CBA. CBA can not be used because of the limitations in cement and concrete standards. Thus it is stored giving serious space problems, potential risks of air pollution, contamination of water due to leaching. Furthermore, the transportation and storage of this type of ash causes significant costs. The process of activation of CBA allows obtaining a material with similar cementing features with ordinary Portland cement (OPC). But this process is also the origin of important economical and environmental benefits when compared with the manufacture of OPC, the emissions of CO_2 to the atmosphere and the energy consumption would decrease, the destruction of natural quarries would be attenuated [1]. Thus in the last few decades particular attention has been paid to activate silicate binders with low or zero OPC content. The extended usage of these binders is motivated by a number of considerations related to energy savings, economy and ecology [2-4]. Scientific studies indicated a certain potential for geopolymers to become cement for the future [5-6].

İ. B. Topçu and M. U. Toprak are with Eskişehir Osmangazi University Civil Engineering Department, BatıMeşelik Campus, Eskişehir, Turkey (corresponding author to provide phone: +90-222-2393750; fax: +90-222-2393613; e-mail: utoprak@ogu.edu.tr).

Geopolymers can be formed from silica-alumina oxides mixed with alkali hydroxides and alkali silicates. The main product of reaction is three-dimensionally networked amorphous to semi-crystalline alumina-silicate material [7]. Geopolymers possess rapid strength gaining, durability of environmental effects, low thermal conductivity and high volume stability, fire and chemical resistance. Geopolymer technology can also be employed for waste disposal and immobilize toxic metals. Therefore, they have been gradually attracting attention as potentially revolutionary materials [8-10]. Properties of geopolymers will mainly depend on the experimental conditions (nature and concentration of the activator, curing temperature and pressure, "solid/liquid" ratio, etc.) [11-12].

Because the geopolymers harden slowly at room temperature, these systems are usually subjected to mild curing temperatures. Most research on geopolymers have been conducted with curing conditions of about 95% relative humidity and temperatures ranging from 30 to 85 °C. Accordingly, curing times may vary from several hours to several days [13-14]. The results show that both FA and CBA can be utilized as an aluminasilicate source for production of geopolymer. The objective of this study is to investigate the effects of mix composition and curing parameters on compressive strength of CBAG. Besides durability performance of CBAG will be evaluated considering freeze-thaw and high temperature resistance of CBAG.

III. Materials and Method

CBA from Seyitömer Power Plant in Turkey used in this study were ground by a ball mill for 45 min. to increase the surface area and reactivity of CBA. Blaine fineness of CBA has been increased to 4100 cm^2/g from it's original 930 cm^2/g. The chemical composition and physical properties of CBA are given in Table 1. The alkaline solutions were sodium hydroxide (98% purity) and sodium silicate (8.9% Na_2O, 28.7% SiO_2 and 64.8% H_2O, d=1.38 g/cm^3). The experimental study was conducted in three sections; (i) determination of the optimum alkaline solution, (ii) determination of optimal curing parameters for CBAG and (iii) durability of CBAG to high temperature and freeze-thaw effects.

TABLE I
CHEMICAL COMPOSITION AND PHYSICAL PROPERTIES OF CBA

Chemical composition (%)	CBA
Al_2O_3	18.76
Fe_2O_3	9.57
SiO_2	51.51
S+A+F	79.84
CaO	5.08
MgO	0.93
P_2O_5	0.07
K_2O	2.56
Na_2O	0.52
SO_3	0.007
Cl	0.005
LOI	10.85
Physical properties	
Specific gravity	2.21
Blaine fineness (cm^2/g)	930

The Na_2O and SiO_2 mass ratios of 12 different alkaline solutions were given in Table 2. In preparation of the CBAG, alkaline solutions and water were first mixed in a beaker and cooled down to room temperature. Then CBA and sand were added into the alkaline solution and mixed for 2 min. After that, the fresh geopolymeric matrix is rapidly poured into metallic prismatic moulds (4x4x16 cm), which were cured in an oven at 80 ºC and 40% RH for 20 h (OC). Sand/CBA (S/CBA) and water/CBA (W/CBA) ratios of 2:1 and 1:2 are used constant in these specimens. After cooling to ambient temperature, demoulding of the specimens were carried out and the prisms were submitted to unit weight, ultrasound velocity, flexural and compressive strength tests at 1 d according to TS EN 196-1.

TABLE II
MIX PROPORTIONS OF CBAG

Sample	W/CBA	S/CBA	Na_2O %[a]	SiO_2 %[a]	SiO_2/Na_2O[a]
SH8SS0	0.5	2	8	0	-
SH8SS4	0.5	2	8	4	0.5
SH8SS8	0.5	2	8	8	1
SH8SS12	0.5	2	8	12	1.5
SH12SS0	0.5	2	12	0	-
SH12SS4	0.5	2	12	4	0.33
SH12SS8	0.5	2	12	8	0.66
SH12SS12	0.5	2	12	12	1
SH16SS0	0.5	2	16	0	-
SH16SS4	0.5	2	16	4	0.25
SH16SS8	0.5	2	16	8	0.5
SH16SS12	0.5	2	16	12	0.75

[a]: Percentage with respect to CBA content (in mass).

The optimum alkaline solution was determined in terms of 1 d compressive strengths of OC specimens. Using the optimum alkaline solution determined in previous section, the optimal mix composition and curing parameters (such as S/CBA and W/CBA ratio, the optimum curing time at 80 ºC oven, precuring time at room temperature) for CBAG were investigated in terms of compressive strengths of the CBAG mortars; (i) OC for 1 d (σ_{OC1D}), (ii) OC for 1 d and air cured till 7 d (σ_{OC7D}) and (iii) 7 d air cured (AC) at room temperature (σ_{AC7D}). Lastly effect of mix composition (S/CBA and W/CBA ratios) and curing conditions (OC or AC) on mass, volume and compressive strength properties of CBAG exposed to freeze-thaw effects (TS EN 12091) and high temperature effects were investigated. Freeze-thaw and high temperature tests are conducted on 28 d CBAG mortars. CBAG mortars were subjected to temperatures of up to 400 and 600 °C at a gradual incremental rate of approximately 6 °C/min from room temperature. As soon as the target temperature (400 or 600 °C) was attained, it was maintained for an additional 180 min before the furnace was shut down to allow the specimens in the furnace to cool down to room temperature.

III. RESULTS AND DISCUSSION

The specific objective of this section was to study the effect of the activator type and molar ratios on the properties CBAG. Finally the optimum alkaline mix was determined as SH12SS8. All the test results obtained for determining optimum alkaline mix are given in Table 3. The SH8SS0 and SH8SS4 specimens were very stiff with no flow value and difficultly compacted to the molds. The workability of the mixes increased with the increase in the Na_2O/CBA ratio.

TABLE III
PROPERTIES OF CBAG

Sample	Unit Weight g/cm^3	Ultra. Vel. Km/s	Flext. Str. MPa	Comp. Str. MPa
SH8SS0	1.538	0.96	0	2.38
SH8SS4	1.589	1.12	1.56	4.62
SH8SS8	1.678	1.45	1.74	5.14
SH8SS12	1.690	1.42	1.41	4.43
SH12SS0	1.709	1.13	1.33	3.54
SH12SS4	1.694	1.35	1.45	4.96
SH12SS8	1.827	2.47	3.87	12.24
SH12SS12	1.713	1.64	2.52	8.52
SH16SS0	1.615	1.44	3.11	5.76
SH16SS4	1.686	1.75	2.33	7.58
SH16SS8	1.808	1.92	2.64	9.26
SH16SS12	1.764	1.56	2.12	6.17

The values of unit weights measured in this work range between 1538 and 1827 kg/m^3 are significantly lower than PC mortars (2000-2400 kg/m^3). Thus, the results prove that lightweight construction materials can be manufactured starting from these CBAG. The lower compressive strength of SH12SS4 and SH12SS12 specimens having lower and higher SiO_2/Na_2O ratios from SH12SS8 highlighted the importance of determining the optimal molar ratios for CBAG. Compressive strength variation of the specimens due to Na_2O/CBA and SiO_2/Na_2O mass ratios was investigated in Fig. 1. Optimum values of strength were obtained if the SiO_2/Na_2O mass ratios varied from 1.0 to 1.4 [1, 15, 16] while it was 0.85 in present experimental study. Note that optimal molar ratios significantly changes with the geopolymer raw materials and curing methods as different successful molar ratios are reported in literature.

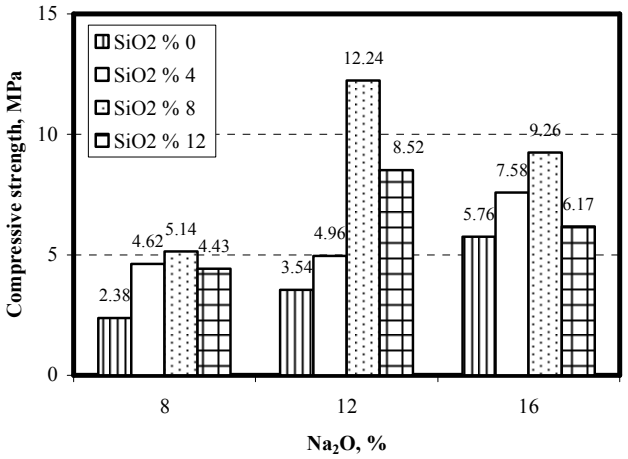


Fig. 1 Effect of Na_2O/CBA and SiO_2/Na_2O on comp. str. of CBAG

The mechanical strength of the material is enhanced by a higher initial SiO_2/CBA ratio, which increases the number of strong Si-O-Si bonds in the final product. The amount of Na_2O/CBA is assumed to play an important role to build up the alkaline medium responsible for the geopolymerization reactions. The dissolution and hydrolysis of silica in alkaline water solution were significantly improved with increase in Na_2O/CBA ratio. But excessive alkalinity (16% Na_2O) adversely affected the strength of the final product due to the excess concentration of OH as demonstrated in literature by Lee and Van Deventer [17].

S/CBA ratio of 2:1 was used in relevant articles [1, 15]. Taking the W/CBA ratio and curing time at 80 ºC oven as 0.5 and 24 hours respectively effect of different S/CBA ratious (such as 0:1, 1:1, 2:1 and 3:1) were tested in terms of σ_{OC1D}, σ_{OC7D} and σ_{AC7D} of CBAG mortars. The optimal sand/CBA ratio was determined as 1:0 in Fig. 2. The decrease in sand content is found to be effective in improving the compressive strength of CBAG. The improvement is mainly due to the better compaction of CBAG mortars prepared with lower sand content.

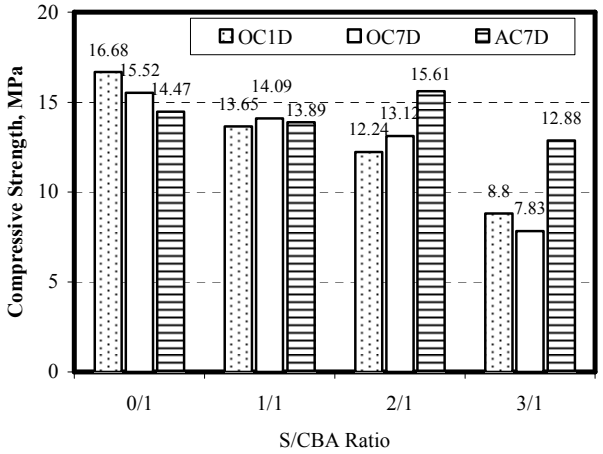


Fig. 2 Effect of S:CBA ratio on comp. str. of CBAG

After 0:1 determined as optimal sand/CBA ratio, it is kept constant and water/CBA ratios of 0.40, 0.45 and 0.50 were tested. The strength of mortars was improved with the decrease in water/CBA ratios. For each case, compressive strength of OC1D and OC7D were measured and compared with the AC7D specimens as shown in Fig. 3.

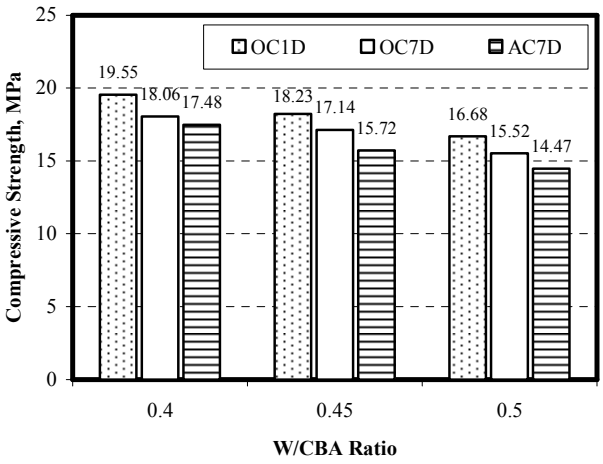


Fig. 3 Effect of W/CBA ratio on comp. str. of CBAG

It can be noticed that when the W/CBA ratio changes 0.5 to 0.4, compressive strength of OC1D, OC7D and AC7D increases 17.2%, 16.3% and 20.8 respectively. Reducing the water content caused higher PH values and thus enhanced rapid dissolution and reaction of the geopolymer. Finally optimal W/CBA ratio was found as 0.4. Using the determined optimal W/CBA and S/CBA ratio, six different time of oven curing at 80 º C in oven were investigated in Fig 4. For all curing times, specimens were cooled down to room temperature before comp. str. Test. Considering compressive strengths of OC1D, OC7D and AC7D specimens, it was obvious that the optimal total cure time was 20 h, while 16 h was not enough to accelerate the strength development and 24 h caused formation of microcracks.

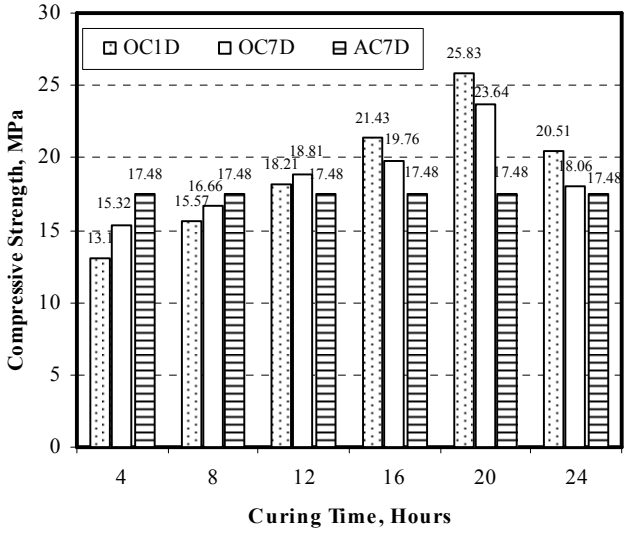


Fig. 4 Effect of OC time on comp. str. of CBAG

Based on the obtained comp. str. results in Fig. 4, increase in curing time improves the strength of concrete at 1 d (OC1D); however, it affects the 7 d results (OC7D) negatively, and these effects are clearer on the specimens cured 12 h and longer at 80 ºC in oven. These compressive strength development behaviours of CBAG are similar with the cement based construction materials. Increase in curing time and temperatures improves the strength of concrete at early ages; however, it affects the 28-day results negatively [18, 19]. Finally effect of precuring time at room temperature before application of heat was examined with three different precuring time (such as 0, 6, 12 and 24 h) in Fig. 5. With an increase in precuring time, comp. str. of the specimens decreased as a result of the decreasing effect of heat on hardened CBAG mortars. It can be concluded that precuring has no beneficial effect on comp. str. of CBAG mortars.

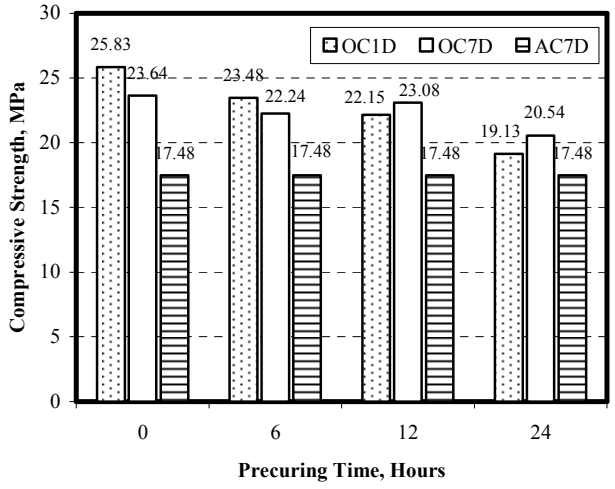


Fig. 5 Effect of precuring time on comp. str. of CBAG

Effect of time on compressive strength development of OC and AC specimens were given in Fig. 6. CBAG mortars cured with the optimal curing conditions determined above (S/CBA and W/CBA ratios of 0:1 and 0.40, 20 h at 80 ºC in oven) showed compressive strength of 25.83 MPa at 1 d but afterwards specimens showed deterioration of strength up to 12.23% at 90 d. AC showed an attractive strength development up to 17.48 and 29.58 MPa at 7 and 90 d respectively. The strength development rate was higher especially up to 7 d and afterwards gradually decreased.

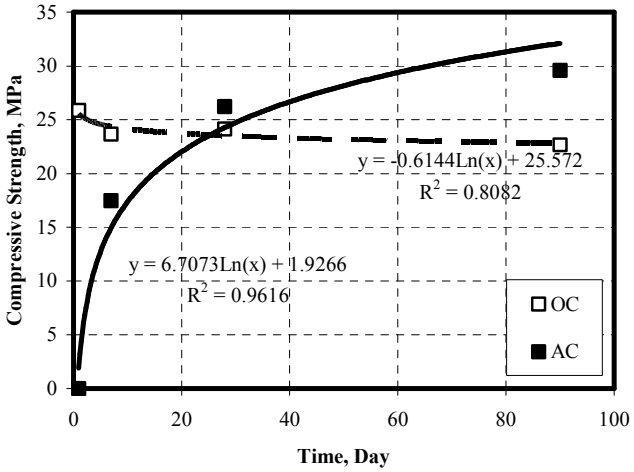


Fig. 6 Effect of time on comp. str. development of CBAG

The compressive strength loss of 28 d CBAG mortars having three different S/CBA and W/CBA ratios and two different curing conditions (OC or AC), were investigated before and after 50 freeze-thaw cycles (FT) according to TS EN 12091. Slight damage or deformation especially at the corners were detected as seen in Fig. 7.



Fig. 7 CBAG mortars before and after 50 FT cycles

Specimens are coded starting with the cure type, W/CBA and S/CBA ratios and finally before and after exposure to FT or HT effects. As an example AC-0.40-1:0-AFT represents the specimen cured at room temperature having W/CBA and S/CBA ratios of 0.40 and 0:1 after 50 FT cycles. The compressive strengths of the specimens decreased after FT. Fig. 8. showed that the specimens with higher S/CBA ratio showed higher resistance to FT cycles. AC mortars generally achieved better durability performances. The best specimen with only 5.71% compressive strength loss was the AC specimen having 3:1 S/CBA ratio. It can be seen that the correlation coefficients of specimens subjected OC are lower than that of the AC specimens.

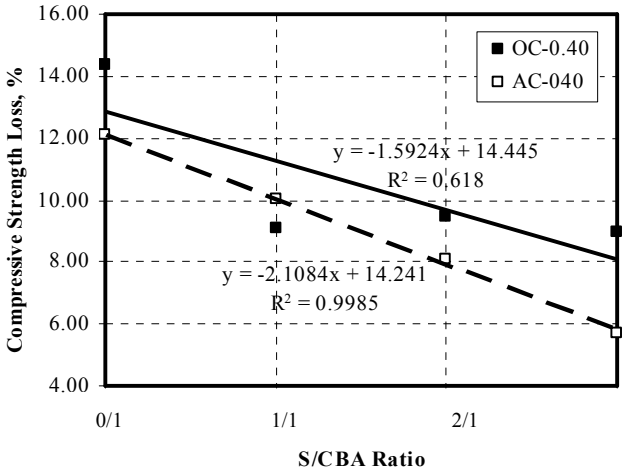


Fig. 8. Comp. str. loss-S/CBA ratio relationship after 50 FT cycles

It appears that the greater the amount of water, the lower the resistance to FT cycles as seen in Fig. 9. It is crucial to note that these mixes were cured for only 28 days before FT cycles. The results may have been quite different if the specimens were cured for a longer period of time. It is evident from the results obtained that the investigated CBAG possessed an excellent FT resistance.

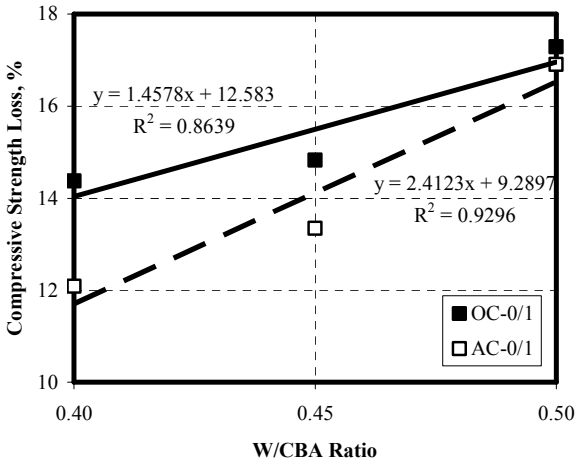


Fig. 9. Comp. str. loss-W/CBA ratio relationship after FT cycles

Results showed correlation with other studies conducted on FA geopolymers. [16, 17, 20]. Finally after 50 FT cycles, the comp. str. of CBAG mortars has not decreased more than 20%. The correlation coefficients were high enough to show that there is a strong relationship between W/CBA ratio-comp. str. loss of the CBAG mortars exposed to FT cycles. The mass loss, thermal expansion and comp. str. loss of the oven or air cured CBAG mortars with and without sand were determined after exposure to HT effects (400 and 600 ^{o}C for three h). It can be noticed from Fig. 10 that, the mass loss is about 15% at CBAG mortars produced without sand while it significantly decreases up to 5% for the specimens having S/CBA ratio of 1:3 when exposed to 600 ^{o}C. This is due to the lower water content of the CBAG mortars produced with sand. When the temperature changes from 400 to 600 ^{o}C only 2-3% increase in mass loss was observed.

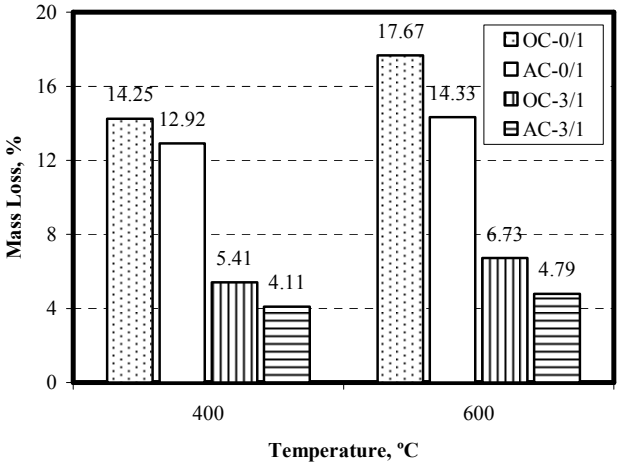


Fig. 10 Mass loss-S/CBA ratio relationship after exposure to HT

With destruction of the chemical structure due to HT effect, the color of the CBAG changed from pink to grey when the temperature increased from 400 to 600 ^{o}C as seen in Fig. 11.

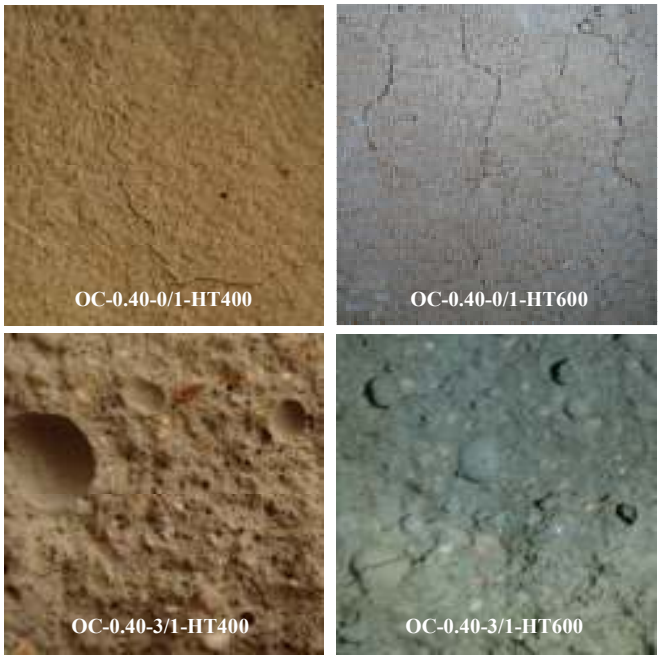


Fig. 11 CBAG mortars with different S:CBA ratio after HT effects

For 600 °C all CBAG with and without sand affected negatively. Note that microcracks are seen in parts exposed to HT and with the increase in temperature length and depth of the cracks increased. Volumetric shrinkage-S/CBA ratio and curing type relationship after exposure to HT effects were investigated in Fig. 12. All the CBAG mortars showed little swelling up to 80 °C. Approximately 2.25% shrinkage by length were observed at specimens without sand while the specimens produced with sand (S/CBA ratio of 3:1) showed much lower shrinkage percentages of maximum 0.8% at 600 °C. This indicates that geopolymer mortars exposed to high temperature, resulted in higher volumetric expansion when produced without sand. This behavior was similar with the mass loss of geopolymers exposed to high temperature.

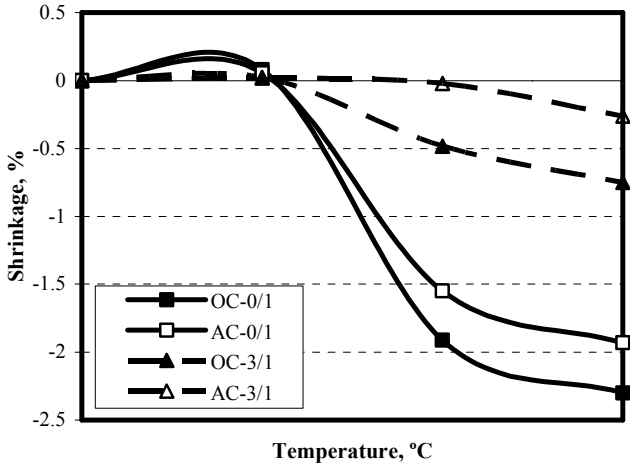


Fig. 12. Volumetric shrinkage-S/CBA ratio after exposure to HT

When exposed to 400 °C for three hours, OC-0:1 and AC-0:1 specimens showed slight compressive strength decrease of about 5% since the specimens produced with sand (OC-3:1 and AC-3:1) showed deterioration of strength up to 30%. It was obviously seen that sand usage negatively affects the mechanical properties of geopolymers exposed to high temperature.

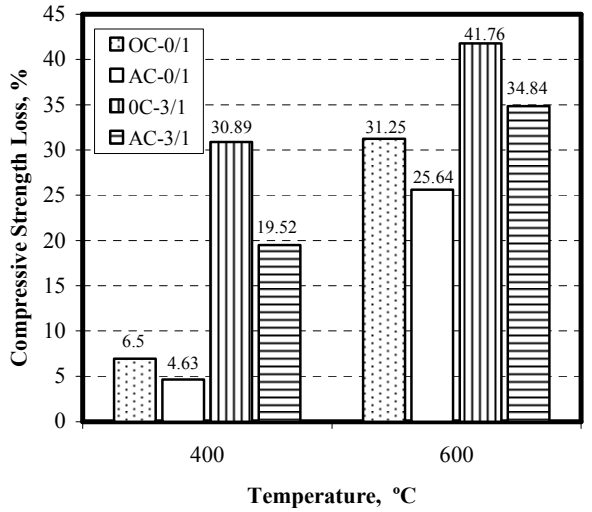


Fig. 13 Comp. str. loss-S/CBA ratio after exposure to HT

This is because the different thermal expansion of geopolymer paste and the sand particles leading to the formation of microcracks inside the specimen as it can be seen in Fig. 11. Another important reason can be the more porous structure of specimens produced with S/CBA ratio of 3:1 that allows the heat transfer easily. OC-3:1 exposed to 600 °C for 3 hours loosed a substantial part of it's strength which dropped to 60 percent of it's original value. With the increasing temperature, area of surfaces exposed to high temperature increased evidently. The color of the specimens reflects the degree of high temperature applied to the CBAG in Fig 11. Finally coal bottom ash geopolymers investigated in this paper can be considered as a construction material with acceptable comp. str, FT and HT resistance. Besides CBAG can be used as a medium for the encapsulation of hazardous or intermediate level radioactive waste as FA or metakaolin geopolymers in literature [21-23]. Although they have been used in several applications their widespread use is restricted due to lack of long term durability studies, detailed scientific understanding and lack of reproducibility of raw materials.

IV. CONCLUSIONS

CBAG has excellent compressive strength, FT and HT resistance. Thus they are suitable for structural applications. They exceed the requirements for construction materials if they are cured at 80 °C for more than 6 h or at room temperature for more than 7 days. The rate of increase in compressive strength was rapid up to 20 hours of curing time; beyond 20 hours, compressive strength was negatively affected. Therefore, heat-curing time need not be more than 20 hours in practical applications. Besides, it is significant, that initial curing at higher temperatures does not increase compressive strength substantially above that achieved by curing at room temperature. AC specimens reached 90 d compressive strength of 29.58 MPa while the OC specimen achieved 22.67 MPa for 90 d. This study showed that the specimens with higher sand:CBA ratio showed higher resistance to freeze-thaw exposure. Specimens which show either a very large or a negligible expansion or shrinkage at HT do not generally show the highest strengths; the best HT resistance was provided by CBAG mortars produced without sand. Air cured specimens generally achieved better FT and HT resistance compared with the specimens cured at 80 °C in oven.

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İlker Bekir TOPÇU is currently professor in the Civil Engineering Department, Eskişehir Osmangazi University, Eskişehir, Turkey. He graduated from Engineering and Architecture of State Academy in 1980, and obtained his Masters degree from the Institute of Science, Anadolu University, Eskişehir, Turkey, in 1984 and joined the teaching profession as a research assistant in 1982 at the Anadolu University. He obtained his Ph.D. from the Institute of Science, İstanbul Technical University, İstanbul, Turkey, in 1988. He has worked as a visiting scientist at the Lehigh University, from 1990 to 1992, and Northwestern University (ACBM), from 1996 to 1997. He has published 94 refereed papers in international and national journals, 134 proceedings papers in international and national congress and symposiums and 43 actual papers. His papers cited more then 250. He supervised 6 Ph.D. and 36 Ms Thesis. His research interests are lightweight, heavyweight, high temperature resistance and high-strength concretes, concretes using industrial wastes such as silica fume, fly ash, blast furnace slag, waste tire, waste glass, waste concrete, concrete as a composite material, Ferrocement, Horasan mortars, corrosion in reinforced concrete and application of fuzzy logic and artificial neural networks on concrete technology. He has refereed articles in many international and national journals. He has examined many structures which destroyed in earthquakes, and he has written many technical laboratory reports on concrete and various materials of structurs. He is married and knows English.

Mehmet Uğur Toprak is research assistant in the Civil Engineering Department, Eskişehir Osmangazi University, Eskişehir, Turkey. He graduated from Engineering and Architecture Faculty of Eskişehir Osmangazi University in 2000, and obtained his Masters degree from the Institute of Science, Eskişehir Osmangazi University, Eskişehir, Turkey, in 2002. He has been working the usage of industrial wastes in cement and concrete technology concerning Ph.D. studies since 2005. He has published 4 refereed papers in international and 1 in national journals, 2 proceedings papers in international and 1 in national congress and symposiums. His research interests are low or zero cement binders, geopolymers, microwave curing , concretes using industrial wastes such as fly ash, coal bottom ash, waste glass. He is married and knows English.