

Strength Properties of Slag/Fly Ash Blends Activated with Sodium Metasilicate and Sodium Hydroxide+Silica Fume

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Abstract

This study presents the strength properties of alkali activated Ground Granulated Blast Furnace Slag (GGBFS)/Fly Ash (FA) blend mortars prepared with sodium meta-silicate and, sodium hydroxide-silica fume combination mixture. The GGBFS/FA ratios were arranged at (100/0, 80/20, 60/40, 40/60, 20/80, 0/100). Water to “GGBFS+FA” ratio was kept constant at 0.5. Na concentration ratio to “GGBFS+FA” was kept constant at 6%. These ratios were in mass basis. Three different mixture parameters were used. In the first series of mixture, only sodium meta-silicate was used as activator. In second and third series, sodium hydroxide and silica fume combination was used as activator. Sodium hydroxide and silica fume was mixed with water and used directly in preparation of second series of mortar mixtures. For preparation of third series of mortar mixture, sodium hydroxide and silica fume were mixed continuously with water for three days to allow dissolution of silica fume in sodium hydroxide solution. At first, 3-days compressive strengths of all alkali activated mortar mixture were measured. After that due to the very low compressive strength of mortars made with high volume fly ash content, only 100/0 and 80/20 slag/fly ash ratios were investigated for 7, 14 and 28 days compressive and 28 days flexural strength. The results of the 3 days tests show that decreasing the slag/fly ash ratio decreases distinguishably the compressive strength of the mortars. The mortars produced with the mixture of sodium hydroxide and silica fume combination as activator showed satisfactory results when compared with those activated with sodium meta-silicate.

Keywords

Fly Ash · Blast Furnace Slag · Silica Fume · Sodium Metasilicate · Sodium Hydroxide

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1 Introduction

The cement production industry is responsible for more than 7% of CO₂ released in the atmosphere [1], the consumption of large amounts of natural resources and nearly 2-3% of global primary energy [2].

On the other hand cementitious compounds are not enough durable against many external influences. Also, in some cases, Portland cement is not economical in terms of cost to use. The technical, economical and environmental problems mentioned, make the production of alternative binders an attractive option. The most interesting work towards the production of a binder without the use of Portland cement is the alkali-activation of industrial wastes such as GGBFS and FA [3].

GGBFS is a by-product obtained during the process of manufacturing of pig iron in the blast furnace. It is produced by the combination of earthy constituents during the melting process of iron ore in the blast furnaces, in the presence of limestone as flux [4].

GGBFS is defined as the glassy granular material formed when molten blast-furnace slag is rapidly chilled as by immersion in water. The fast cooling of the slag minimizes the formation of crystal structures and transforms the molten slag into fine aggregate sized particles composed of mainly amorphous material. Because of its high silica and alumina content in an amorphous state, GGBFS shows pozzolanic behaviour similar to that of natural pozzolans [4, 5].

Whereas fly ash is released by factories and thermal power plants and recently its amount has been increasing to a large extent. Nowadays the disposal of fly ash has become a serious environmental and economical problem. Previously, fly ash was generally released into the atmosphere, but nowadays pollution control measures require that it should be captured prior to release [6].

On the other hand silica fume is also an industrial waste obtained as a by-product from the silicon metal and the ferrosilicon alloy industries, producing alloys with 75 percent or higher silicon content. It contains 85 to 95% non-crystalline silica [7].

Blast furnace slags and fly ashes are well-known materials for their use in blended cements and concretes. Nevertheless,

Tab. 1. Chemical composition of GGBFS, FA and SF (%)

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	LOI
GGBFS	36.7	5.20	0.98	32.61	10.12	0.99	0.76	0.42	2.88
FA	61.81	19.54	7.01	1.77	2.56	0.31	0.99	2.43	2.20
SF	92.63	0.23	0.13	0.95	0.43	0.46	1.2	0.27	3.40

only 20-30% of slags [8], and about 53% of fly ashes [9] are being used and the remaining part is stored in large extensions. Making use of these excesses in the manufacturing of alternative binding materials would contribute to the solution of an environmental problem and to the production of new high-performance materials. By activating these wastes with alkali solutions new binders with high mechanical strengths and also low energy costs in their manufacturing process would be obtained [8].

The importance of the alkali-activation is not restricted in converting waste materials to useful products, but also in its ability to produce a high-performance binder from materials such as fly ash or blast furnace slag. [10]

Known also as geopolymer, these binders are produced as a result of the reaction of materials which contain both alumina and silica with alkaline solutions, forming an aluminosilicate structure [11]. Davidovits [12, 13] defines geopolymer as amorphous three-dimensional aluminosilicate materials with ceramic like properties which are formed through mixing solid silicate-aluminate raw materials with alkali or alkali silicate solutions.

The primary input is usually sources of amorphous alumina silicates with SiO₂+ Al₂O₃>80 wt %. The geopolymer structure consists of chain, sheet-like and three dimensional networks made of various monomeric or polymeric structures formed after the geopolymerization referred as Q unit types of connected SiO₄ (S) and AlO₄ (A) tetrahedral. In contact with a high pH alkaline solution, the input materials (amorphous or semi crystalline aluminosilicates) dissolve progressively to form oligomers; geopolymers are then precipitated [14].

The mechanical properties and outer appearance of geopolymer mortars or concrete are very similar to ordinary Portland cement mortars or concrete. At the same time geopolymer is known to have a very good performance when exposed to high temperatures or acidic environment [15, 16].

Researchers report the use of different alkali-activators such as liquid sodium silicate, sodium meta-silicate, sodium hydroxide and sodium carbonate in the production of geopolymeric mortar [17–19].

Provis et al. [20] reported that blended slag/fly ash binders are attracting attention of the researchers and, their coexistence and use in the most convenient combinations have been investigated.

In the present study the strength properties of GGBFS/FA blends activated with sodium meta-silicate or solution of sodium hydroxide and silica fume combination cured at room temperature have been investigated.

2 Properties of the materials used

2.1 Ground granulated blast-furnace slag (GGBFS)

GGBFS was provided from Iskenderun Iron–Steel Factory located in southern Turkey. Its chemical composition is given in Table 1. The specific gravity of GBFS was 2.81 g/cm³. The blast furnace slag was ground granulated in Iskenderun Cement Factory to have a Blaine specific surface area about 4250 cm²/g. According to ASTM C989 [21] hydraulic activity index, the GGBFS used was classified as a grade of 80 slag.

2.2 Fly ash (FA)

In the study, Class F fly ash provided from Sugozy thermal power plant established in Yumurtalik-Adana country of southern Turkey. The chemical properties of fly ash are given in Table 1. According to the standard limits in EN 450-1[22] and ASTM C618-94a [23] the value of SiO₂+ Al₂O₃+ Fe₂O₃ is more than 70% and, its CaO amount is less than 10%, therefore, current FA is classified as class F (low lime) fly ash. The specific gravity of the FA used is 2.39 g/cm³, and the Blaine specific surface area is 2900 cm²/gr. Pozzolanic strength activity index determined according to ASTM C 618 [23] was 78 at 28-days.

2.3 Silica fume

Silica fume was supplied from Antalya-Etibank ferro-chrome factory located in west-southern Turkey. The chemical oxide composition of silica fume is given in Table 1. The specific gravity and unit weight were 2.32 and 245 kg/m³, respectively. The pozzolanic strength activity index determined according to ASTM C 618 [23] was 122% at 28-days. The amount of silica fume remaining on a 45 µm sieve was 4.8%.

2.4 Sodium Metasilicate

Sodium meta-silicate was used in this study as an alkali activator for the activation of the GGBFS and FA. It was provided from Silmaco Silicates. SiO₂/Na₂O modulus of sodium metasilicate by mass is 1 (Ms=1). The chemical properties are given in Table 2.

Tab. 2. Chemical composition of sodium metasilicate

Properties	Molar ratio (SiO ₂ /Na ₂ O)	Mass ratio (SiO ₂ /Na ₂ O)
Values	1	0.91

2.5 Sodium Hydroxide

NaOH sodium hydroxide (SH) was used as an alkali activator for the activation of the GGBFS and FA blend. Chemical com-

Tab. 3. Chemical composition of sodium hydroxide (%)

Oksit (%)	NaOH	Na ₂ CO ₃	Cl	SO ₄	Pb	Al	Fe
	≥ 97	≤ 1	≤ 0.01	≤ 0.01	≤ 0.002	≤ 0.002	≤ 0.002

position of sodium hydroxide was presented in Table 3. It was supplied from Akca Chemical Company.

2.6 Sand

Rilem-Cembureau Standard dry sand produced in Trakya Set Çimento Sanayi T.A.S. Cement Factory was used for the preparation of the mortar samples. The grading of the sand is given in Table 4.

Tab. 4. The Grading of Standard CEN Sand

Sieve Size (mm)	2	1.6	1	0.5	0.16	0.08
Cumulative Percentage	0	7	33	67	87	99

3 Experimental work

3.1 Preparation of the Specimen Mixtures

In this study, mortar made with GGBFS/FA blend was alkali activated. In the production of these mortars RILEM sand, GGBFS and FA, water, sodium meta-silicate or a combination of sodium hydroxide and silica fume were used. 1350 g Rilem Sand in conformity to TS EN 196-1 [24] was used as fine aggregate. The amount of the binder (GGBFS+FA) was kept constant at 450 g in conformity to the same standard. Water to “GGBFS+FA” blend ratio was kept constant at 0.5. Sodium to “GGBFS+FA” blend ratio was kept constant at 6% in mass basis. The slag/fly ash ratios were arranged at (100/0, 80/20, 60/40, 40/60, 20/80, 0/100).

Three mixture parameters were used. For first series of mortar mixtures, sodium meta-silicate was used, and accepted as first mixture parameter. For second and third series of mortar mixtures sodium hydroxide and silica fume solution was used as activator. In second series of mortar mixtures, sodium hydroxide and silica fume mixed in water and directly used in mortar mixture. Direct using was assumed a second mixing parameter. For third series of mortar, sodium hydroxide and silica fume mixed with water continuously three days to allow dissolution of silica fume in sodium hydroxide solution. This is also assumed another mixing parameter and called third mixing parameter.

All mortar specimens were cured in normal curing conditions (at 100% relative humidity) according to TS EN 196-1 [24]. The materials used for the preparation of the specimens were weighed separately according to their rates on a precision scale. The ingredients of the mortar mixtures made standard for 40 mm x 40 mm 160 mm sized three-cell mould are given in Table 5.

3.2 Casting of the Specimens

Mortar mixtures were prepared according to the mix proportions given in Table 5 and mixed in the Hobart mixer in a certain order. The Hobart mixer was used in automatic adjustment according to TS EN 196-1 [24] standard.

In the first series (named as SM1 and SM2), the water and sodium meta-silicate were mixed in a glass jar according to the mix proportions given in Table 5, until it was completely dissolved. After that the solution and 450 g of binder (GGBFS and FA blend) were put in the mixer at the low gear for 30 seconds. In the second step while the mixer is still working the sand is added.

In the second series (named as SHD1 and SHD2), the water, sodium hydroxide and silica fume were mixed in a glass jar according to the mix proportions given in Table 5, until sodium hydroxide were completely dissolved. Then, the solution and 450 g of binder (GGBFS and FA blend) were put in the mixer at the low gear for 30 seconds, in the second step while the mixer is still working the sand is added.

In the third series (named as SHP1 and SHP2), the water, sodium hydroxide and silica fume were mixed in a glass jar according to the mix proportions given in Table 5 until they were completely dissolved, the solution was mixed continuously for 72 hours so that silica fume can dissolve in sodium hydroxide solution. Then, the solution and 450 g of binder (GGBFS and FA blend) were put in the mixer at the low gear for 30 seconds, in the second step while the mixer is still working the sand is added.

When the mixer stopped, the mortar mixtures were cast into prismatic moulds with 40 x 40 x 160 mm sized three-cell mould. By using an appropriate spoon, the fresh mortar is put into the moulds in two layers and shaken in the jolting apparatus, where it is impacted 60 times in one minute and then the mortars surface is finished. All the specimens were de-moulded after 24 hours curing normal curing conditions according to TS EN196-1[24].

Firstly, the three day compressive strength of the whole range of GGBFS/FA ratio was measured. After that, due to low compressive strength development of the mortars made with high fly ash content, only 100/0 and 80/20 GGBFS/FA ratios were chosen for further investigation of longer term compressive strength at 7, 14 and 28 days. Flexural strength was also measured at 28-days.

3.3 Determination of the Flexural Strength

The flexural test of the specimens was performed according to TS EN 1015-11 (2000) [25] standard. In order to determine the flexural strength of the mortars 40x40x160 mm specimens were

Tab. 5. The Ingredients of the Mortar Mixtures

Specimen type	Water to GGBFS+FA	Water (g)	Binder (g)	Slag /FA	Sodium Metasilicate (g)	Sodium Hydroxide (g)	Silica Fume (g)	Sand (g)
SM1	0.5	225	450	100/0	72	-	-	1350
SM2	0.5	225	450	80/20	72	-	-	1350
SM3	0.5	225	450	60/40	72	-	-	1350
SM4	0.5	225	450	40/60	72	-	-	1350
SM5	0.5	225	450	20/80	72	-	-	1350
SM6	0.5	225	450	0/100	72	-	-	1350
SHD1	0.5	215	450	100/0	-	47	35	1350
SHD2	0.5	215	450	80/20	-	47	35	1350
SHD3	0.5	215	450	60/40	-	47	35	1350
SHD4	0.5	215	450	40/60	-	47	35	1350
SHD5	0.5	215	450	20/80	-	47	35	1350
SHD6	0.5	215	450	0/100	-	47	35	1350
SHP1	0.5	215	450	100/0	-	47	35	1350
SHP2	0.5	215	450	80/20	-	47	35	1350
SHP3	0.5	215	450	60/40	-	47	35	1350
SHP4	0.5	215	450	40/60	-	47	35	1350
SHP5	0.5	215	450	20/80	-	47	35	1350
SHP6	0.5	215	450	0/100	-	47	35	1350

used. The specimens were tested at 28-days for flexural strength under three-point loading with the span between supports being 100 mm. The average of results obtained from three prismatic specimens was reported as flexural tensile strength.

3.4 The Determination of the Compressive Strength

The compressive strength test was carried out in accordance with relevant specification TS EN196-1[24]. The two broken parts of the 40 x 40 x 160 mm retained after the flexural strength test were used for compressive strength. The loading rate is 500 N/s. The loading area is 40 x 40 mm. The average of results obtained from six broken pieces was reported as compressive strength.

4 Results and discussion

The test results of compressive strength carried out at 3-days were presented in Table 6 and Fig. 1 show the evolution of the compressive strength of the mortars cured at room temperature.

The graph of the 3 days compressive strength values versus GGBFS/FA ratio (see Fig. 1) for three series of mortars indicate that the compressive strength decreases as the GGBFS/FA ratio decreases [8, 20]. It is concluded that, increasing the amount of fly ash in mortar mixture result with a decrease in compressive strength. The mortars produced with 100% fly ash shown almost no compressive strength. This is attributed to very slow activation rate of fly ash at room temperature.

At the end of 3-days curing, at room temperature, the highest compressive strength value was obtained from SM1, 24 MPa produced with a 100/0 slag to fly ash ratio and activated with sodium meta-silicate. This is attributed to high activation rate of GGBFS at room temperature.

Tab. 6. Three Days Compressive Strength Tests Results

Specimen type	Slag /fly ash	Compressive Strength (MPa)
SM1	100/0	24
SM2	80/20	15.1
SM3	60/40	7.6
SM4	40/60	4.33
SM5	20/80	2.2
SM6	0/100	0.23
SHD1	100/0	17.7
SHD2	80/20	9.1
SHD3	60/40	5.1
SHD4	40/60	2
SHD5	20/80	0.5
SHD6	0/100	0
SHP1	100/0	18.6
SHP 2	80/20	9.8
SHP 3	60/40	1.9
SHP 4	40/60	0.6
SHP 5	20/80	0.4
SHP 6	0/100	0

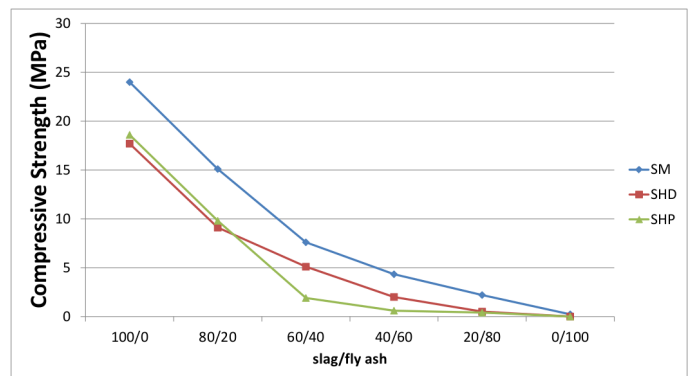


Fig. 1. The evolution of 3 days compressive strength according to slag/fly ash ratio

Tab. 7. Compressive Strength and Flexural Tests Results

Slag/Fly ash		Compressive Str. (MPa)			Flexural Str. (Mpa)
		7 days	14 days	28 days	28 days
100/0	SM1	47.4	72	77.5	6.8
80/20	SM2	35.3	46.8	61.2	7.4
100/0	SHD1	30.2	36.2	66.2	7.4
80/20	SHD2	27.15	30.45	52.1	8.42
100/0	SHP1	47.7	69.1	79.1	6.7
80/20	SHP2	38.9	46.5	59.3	7.25

The mortars produced with a GGBFS/FA ratio lower than 80/20 demonstrated very low values of 3 days compressive strength. Therefore, they were not investigated for 7, 14 and 28 days compressive strength.

Further investigation was carried out with the mixtures that were named SM1, SM2, SHD1, SHD2, SHP1 and SHP2. Compressive strengths of those mixtures were presented in Table 7 and in Fig. 3. The test results presented in Fig. 3 and Fig. 4 show the evolution of the compressive strength of the mortars cured for 7, 14 and 28 days.

It can be seen from Table 7 and Fig. 3 that compressive strength of all mortars increased as curing time increased. Compressive strength of mortar containing 100% GGBFS was higher than that of mortar mixture made with 80% GGBFS and 20% FA regardless of other mixing parameters. This is attributed to low activation rate of fly ash at room temperature.

Direct using of sodium hydroxide and silica fume solution in mortar mixtures (SHD1 and SHD2) meaning second mixture parameter developed lower strength when compared to mortar mixtures made with sodium meta-silicate (first mixture parameter) and mortar mixtures prepared with third mixture parameter (mixing sodium hydroxide and silica fume with water three days continuously).

First mixture parameter (using meta-silicate as activator) and third mixture parameter (mixing sodium hydroxide and silica fume with water three days continuously) developed equivalent compressive strength to each other. It can be seen from Fig. 4 that compressive strength of corresponding mortar mixtures (the same slag to fly ash ratio) almost overlap. This is attributed to that mixing sodium hydroxide and silica fume with water three days continuously can dissolve silica fume in high alkaline medium and forms sodium silicate solution. It could be a good and less expensive alternative to sodium meta-silicate.

Regardless of the mixture parameter, compressive strength of all mortars presented in Table 7 developed high strength at 28-days curing time. Therefore, they can be named as high strength geopolymer mortar.

In Fig. 2 and in Table 7, the flexural strength of alkali activated mortars cured at room temperature for 28 days has been presented. The flexural strength values of the six mortar types selected, ranged between 6.7 MPa to 8.42 MPa. These flexural

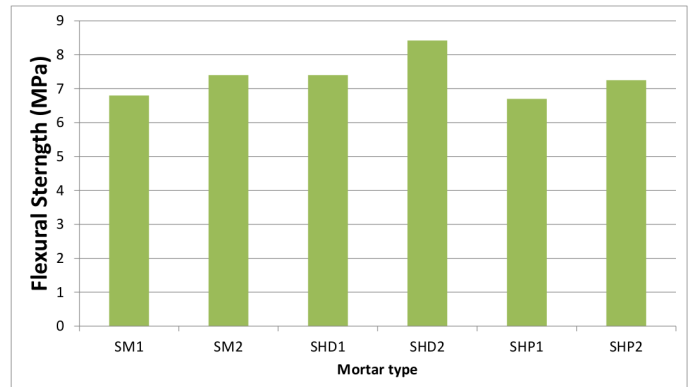


Fig. 2. The 28 days flexural strength of 100/0 and 80/20 slag/fly ash mortars

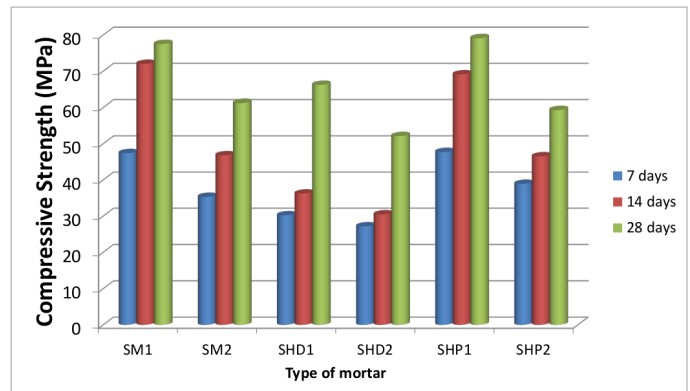


Fig. 3. The compressive strength of 7, 14 and 28 days cured mortars

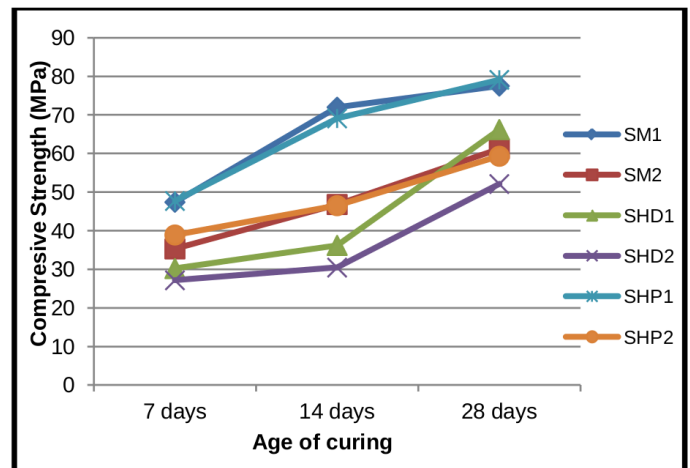


Fig. 4. The evolution of compressive strength according to age

strength values are found to be adequate and comparable with conventional Portland cement concrete.

5 Conclusions

In this study the alkali activation of GGBFS/FA blend mortars activated with sodium meta-silicate and a sodium hydroxide+silica fume mixture was investigated. According to the test results the following conclusion can be drawn:

Increasing the fly ash content decreases the compressive strength of the alkali activated mortars cured at room temperature.

Mixing sodium hydroxide and silica fume with water three days continuously and using it as activator, improves the activation rate and increases the compressive strength of the mortars when compared to direct usage of them.

The premixed solution prepared for the third series behaves very similarly to sodium meta-silicate and that it could be a good and less expensive alternative to sodium meta-silicate.

Activating GGBFS/FA blend developed high compressive strength and flexural tensile strength in the order of 80 and 8 MPa respectively, at 28 days.

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