

An investigation of resistance of sodium meta silicate activated slag mortar to acidic and basic mediums

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Abstract

This study presents the resistance of sodium meta-silicate activated ground granulated blast furnace slag (GGBFS) to acidic and basic mediums. Geopolymer mortars were prepared with GGBFS activated by sodium meta-silicate activator having 4, 8, and 12% sodium concentrations by mass of slag. These mortars were exposed to two types of acid solutions, H₂SO₄ and HCl with a 5% concentration of acid-water solution. Similarly, some specimens were exposed to a basic medium composed of 25% NaOH solution with water. Exposure time for the acidic and basic mediums was set to one year. Comparison of results obtained from slag based geopolymer mortar was made with normal Portland cement control mortar. Normal control samples lost compressive strength significantly after exposure to acidic and basic medium in comparison to alkali activated slag mortar. Particularly, after exposure to H₂SO₄, the compressive strength of the normal control samples decreased by about 40%. Contrary to this, the slag based geopolymer mortar gained compressive strength after exposure to the acidic or basic mediums. An up to 21% strength increase was observed from the alkali activated slag mixtures made with 4% and 8% Na content.

Keywords: Blast furnace slag, geopolymer, alkali activation, mortar, durability.

Introduction

Concrete is the second most used material used by people after water (Mehta & Paulo, 2006; Luga, 2015). It is an economical construction material, which is widely used all over the world in construction and building industry and in vast amounts due to the ample and easy procurement of its ingredients (Meyer, 2009). Thus, utilization of concrete in

many areas puts Portland cement in an important position as a comprehensive material (Luga, 2015; Criado et al., 2005).

It is known that the production of cement needs intensive energy consumption, thus, production of cement is responsible for 7% of the total carbon dioxide emissions in the atmosphere due to its intensive energy needs and disintegration of calcareous matter during the sintering process. In total, one ton of cement productions releases nearly one ton of carbon dioxide into the atmosphere (Luga, 2015; Sarker et al., 2013). In addition, huge amounts of natural sources are consumed in the production of Portland cement. Researchers reported that nearly 2-3% of the total energy used in the world is consumed for cement production (Luga, 2015; Juenger et al., 2011).

Blast furnace slag and fly ash, as an industrial waste, pollutes the earth and water by leaching heavy metal and radioactive matter, thus causing a great amount of trouble for the environment. Use of these materials as cement admixtures or pozzolanic mineral admixture construction materials is about 20-30% of their total amounts (Luga, 2015; Puertas et al., 2000). Recently, the utilization of these waste or by-product materials by alkali activation in the construction industry has attracted the attention of researchers. With some alkalis, slag and fly ash are activated and alternative binding properties have been obtained as an alternative to Portland cement binder (Coppola, et al., 2018).

Concrete can be conducted to acidic solution. It is known that cement is not resistant to acidic attack. Particularly, cement composites are not durable to acidic solutions. Acid attack to concrete can give tremendous damage when its pH value is lower than 6.5. The lower pH value causes higher acidic attack damage to cement composites (Neville, 1995; Demir, 2017).

The calcium ingredient of chemical compounds of hardened cement mortar begins to be dissolved easily in acidic and basic mediums. With the dissolution process of calcium compounds, the porosity of hardened cement composite materials increase and the process of disintegration accelerates (Hossain, 2015; Chindaprasirt, 2016).

If acid attacks occur on reinforced concrete, the pH levels of the medium drops, then steel reinforcement is corroded and section loss occurs. Concrete needs to be protected from acidic attack because it harms the concrete as it causes disintegration and steel reinforced corrosion in a short time, particularly in coastal areas or in medium with the presence of chemical waste. If the pH value of a medium is higher than 7, it indicates that the medium is basic. Mehta & Paulo (2006) reported that low permeability concrete is harmed when the pH value of the exposure medium of concrete is less than 6 (Mehta & Paulo, 2006; Demir, 2017).

In the context of this research, resistance of alkali activated slag to acidic and basic mediums were taken into account. In the following sections, brief information and discussion are provided from some research done on the resistance of alkali activated slag mixtures to acidic and basic mediums.

Luga (2015), studied the resistance of NaOH alkali activated slag and fly ash geopolymer mortar mixtures to acidic and basic mediums. Prismatic alkali activated slag mortar specimens with size of 40x40x160 mm were prepared and cured at 100°C for 72 hours, and control cement mortar was prepared and cured in water at 23°C for 28 days. The specimens were exposed to acidic and basic mediums for a year. Resistance to hydrochloric acid (HCl), sulphuric acid (H₂SO₄), and base (NaOH) were evaluated by measuring strength changes after immersion of samples for one year in the acidic and basic solutions. The concentrations of the acid and base were 5 and 25% in water solution. After HCl exposure, compressive strength losses were 28, 30, 32, and 46% for the control cement mortar, slag and fly ash blended mortar, slag mortar, and fly ash mortar, respectively. Similarly, after H₂SO₄ exposure, compressive strength losses were 62, 33, 31, and 59%, respectively. Slag containing mortar lost 31% compressive strength. After NaOH exposure, all alkali activated mortars were disintegrated and compressive strength values could not be obtained. However, the cement mortar gained compressive strength (Luga, 2015).

Topçu & Canbaz (2008) worked on acid effect on mortar made with slag as a replacement for Portland cement. They replaced slag with cement from 0% to 100% with a 25% interval. After exposing mortar to an acidic solution, the mechanical and physical loss of the mortar was measured. They prepared cubic samples with a side measuring 7 cm, cured them in water for 28 days, and exposed them to sulphuric acid for 56, 90, and 250 days. They concluded that as the slag amount was increased, the physical loss was reduced. Cement mortars showed higher loss in unit weight, ultrasonic pulse velocity, and compressive strength in comparison to slag containing mortars. In addition, mortar containing 100% slag as a binder activated by NaOH and Na₂SiO₃ blends showed the best resistance to an acid effect (Topçu & Canbaz, 2008).

Zhuang et al. (2017) evaluated the strength and mass loss of geopolymer mortars under acidic effect. They prepared 40x40x160 mm sized geopolymer prism mortar samples with metakaolin, polypropylene fiber, sand mixture, and potassium silicate solutions. The samples were cured at an ambient condition for 35 days and, then they were immersed in H₂SO₄ acid solutions and sodium chloride solutions. Immersion in acidic medium took up to 30, 60, 90, 180, 270, and 360 days. The decreases in compressive strength of samples subjected to sodium chloride and sulfuric acid solutions were 13.7, and 10.9%, respectively. No comparison was reported with the control samples (Zhuang, 2017).

There are not many studies published on the resistance of alkali activated slag mixtures to acidic and particularly basic mediums. In this work, powdered blast furnace slag was activated by sodium meta-silicate and alkali activated slag mortar was obtained. Sodium meta silicate activated slag mortars were prepared with 4, 8, and 12% sodium content of slag, then 40x40x160 mm sized prismatic samples were cast. Samples were cured at different conditions for various curing durations. After the curing duration, hardened samples were exposed to acidic and basic environments in solution to evaluate the resistance of alkali activated slag mortars to acidic and basic mediums. Samples were kept in acidic and basic environments for a year, then, their flexural and compressive strength losses were determined. For comparison purposes, the same testing was also done with the control samples.

Methodology

Properties of materials used and experimental program

Cement

The Portland cement used was CEM I 42.5 R type cement. It complied with the relevant standard TS EN 197-1 (2012). It was procured from the Tokat Adoçim Cement Concrete Factory.

Ground slag

The slag used in this study was obtained as waste from the Iskenderun Iron and Steel factory. The waste slag was cooled down purposely by water in the factory to obtain an amorphous matter, then, it was milled and powdered at the Adana Cement factory. The specific gravity of the slag used was 2.88 gr/cm³, the Blaine specific surface area or fineness was 5990 cm²/gr. The chemical oxide composition of the slag is presented in Table 1.

Table 1. Chemical oxide composition for slag used. Source: Bingöl (2018).

| Oxide | Weight (%) |
|--------------------------------|------------|
| SiO ₂ | 38.89 |
| CaO | 28.94 |
| Al ₂ O ₃ | 13.48 |
| MgO | 5.53 |
| SO ₃ | 1.51 |
| Fe ₂ O ₃ | 1.36 |
| MnO | 1.16 |
| K ₂ O | 0.78 |
| TiO ₂ | 0.53 |
| BaO | 0.45 |
| P ₂ O ₅ | 0.41 |
| Na ₂ O | 0.36 |
| SrO | 0.10 |
| Loss on Ignition | 6.50 |

Sand

In the study, crushed river sand obtained from the Tokat region in Turkey was used. The maximum grain size of the sand was approximately 3 mm, and its density was 2.2 g/cm³.

Water

The clean drinkable water used to prepare the mortar mixture was taken from the city tap water supply. It complied with the relevant standard (TS EN 1008, 2003).

Alkali-activator

The alkali activator was a sodium meta silicate, which was provided from a private company, Tekkim Chemical Industry. The properties and chemical information of the sodium meta silicate are given in Table 2 (Tekkim Kimya, 2016).

Table 2. Chemical oxide composition and properties of sodium meta-silicate. Source: Bingöl (2018).

| Parameter | Rate (%) |
|-------------------|-----------|
| Purity | ≥95.0 |
| Na ₂ O | 48-51 |
| SiO ₂ | 44.4-47.4 |
| Fe | ≤0.1 |
| Non-water soluble | ≤0.5 |
| pH | >12.0 |
| Melting point | 1089 °C |

Mixture proportions, preparation, curing, and testing

The mortar mixture proportions were 3 for the slag-sand ratio and 0.5 for the water-slag ratio. Sodium amounts used in the mortar for alkali activation were 4, 8, and 12% sodium GGBFS in mass basis. While the geopolymer mortars were being prepared, sodium silicate was added to the slag in powder form. Sodium silicate with slag was mixed homogeneously and the mortar was prepared in a Hobart mixer. No cement was used in preparing the alkali activated slag mortar. Cement was used only in the preparation of the control samples for the reference cement mixture.

After mixing the fresh alkali activated slag mortar and Control samples, a three-cell casting mold was used to cast 40x40x160 mm sized prism samples. The Portland cement mixture samples were prepared according to the relevant standard (TS EN 196-1, 2016) and were cured in water at 21±1 °C for up to 28 days (DW), then, they were immersed in the acidic and basic solutions. Alkali activated slag mortars were subjected to three different curing conditions. After casting the three-cell prisms specimens, the first curing was , immediate heat curing at 75°C for 24 hours with the mold in an oven (DO), then, they were demolded and immersed in the acidic and basic solutions. The second curing was the same curing, same testing, and exposure conditions with the Control samples (DW). For the third curing conditions, after the preparation and casting of the alkali activated mortar mixture, it was left in laboratory conditions for 24 hours before demoulding and kept in laboratory air condition for 27 more days at 21±1 °C with 50% relative humidity (DA).

After completing 28 days, the samples were immersed in HCl and H₂SO₄ acidic and NaOH basic solutions for one year. The concentrations of water-acid solutions contained 5% hydrochloric (HCl) acid or sulphuric (H₂SO₄) acid. The concentration of the water-base solutions contained 25% sodium hydroxide (NaOH). After a year, flexural and compressive strength loss and mass loss were determined. The results obtained were compared with the Control samples and between themselves. Average of three specimens were used for compressive strength and mass loss testing, and average of six specimens were used for flexural strength results.

Results and discussion

Resistance of alkali activated slag exposed to acidic and basic mediums

The Control samples, after curing in water, were immersed in acidic solutions made with hydrochloric (HCl) acid, or sulphuric acid (H₂SO₄) to evaluate the acidic resistance of the mixtures. Alkali activated slag mortar, after one day of heat curing, was immersed in acidic solutions. Slag samples cured in water for 28 days were immersed in acidic solutions. Air cured slag samples were immersed in acidic solutions after 28 days of curing. All samples exposed to the acidic test were left in the acidic solutions for one year. The compressive and flexural strength of the samples were

measured before and after exposure to the acidic medium. Strength loss and mass loss of the samples were determined and the results are presented in tabular form. The results of the samples exposed to HCl acid are presented in Table 3. The results of samples exposed to H₂SO₄ acid are presented in Table 4. Mixture coded with DO means cured in oven, with DA means cured in air, and with DW means cured in water.

Table 3. Results of samples exposed to hydrochloric acid. Source: Bingöl (2018).

| Sodium Amount | Curing | Flex. Str. | Flex. Str. | Comp. Str. | Comp. Str. | Mass Exchange (%) |
|---------------|--------|-----------------------|----------------------|-----------------------|----------------------|-------------------|
| | | Before Exposure (MPa) | After Exposure (MPa) | Before Exposure (MPa) | After Exposure (MPa) | |
| 4% | 1d DO | 7.43 | 8.14 | 56.50 | 53.04 | -7.9 |
| | 28d DA | 7.16 | 4.56 | 37.02 | 23.16 | -1.9 |
| | 28d DW | 7.43 | 7.42 | 45.10 | 57.35 | -1.5 |
| 8% | 1d DO | 6.09 | 8.06 | 59.00 | 62.98 | -8.2 |
| | 28d DA | 8.71 | 8.22 | 58.79 | 69.75 | -5.0 |
| | 28d DW | 9.86 | 9.67 | 76.78 | 78.56 | -2.4 |
| 12% | 1d DO | 5.12 | 7.31 | 63.73 | 58.08 | -9.0 |
| | 28d DA | 9.02 | 8.77 | 62.71 | 64.85 | -6.0 |
| CEM I 42.5 R | 28d DW | 7.70 | 10.91 | 58.48 | 78.33 | -2.0 |
| | 28d GW | 9.26 | 9.06 | 54.78 | 43.12 | -1.5 |

Table 4. Results of samples exposed to sulphuric acid. Source: Bingöl (2018).

| Sodium Amount | Curing | Flex. Str. | Flex. Str. | Comp. Str. | Comp. Str. | Mass Exchange (%) |
|---------------|--------|-----------------------|----------------------|-----------------------|----------------------|-------------------|
| | | Before exposure (MPa) | After exposure (MPa) | Before exposure (MPa) | After exposure (MPa) | |
| 4% | 1d DO | 7.43 | 7.05 | 56.50 | 41.2 | -3.0 |
| | 28d DA | 7.16 | 7.86 | 37.02 | 44.92 | -2.0 |
| | 28d DW | 7.43 | 9.08 | 45.10 | 53.21 | 3.0 |
| 8% | 1d DO | 6.09 | 8.30 | 59.00 | 53.37 | -5.0 |
| | 28d DA | 8.71 | 9.06 | 58.79 | 70.94 | -3.5 |
| | 28d DW | 9.86 | 8.53 | 76.78 | 62.5 | 3.0 |
| 12% | 1d DO | 5.12 | 12.87 | 63.73 | 58.56 | -5.0 |
| | 28d DA | 9.02 | 11.4 | 62.71 | 67.56 | -6.0 |
| CEM I 42.5 R | 28d DW | 7.70 | 11.64 | 58.48 | 64.23 | 0.0 |
| | 28d GW | 9.26 | 7.96 | 54.78 | 33.00 | -1.4 |

For the hydrochloric acidic exposure case, mass loss data shows that the slag mixtures cured in the oven for one day lost some mass. Other slag mixtures and the control cement mortar mixtures lost a very small amount from its mass when compared to the oven curing case.

When strength variation was closely observed, the 28-day air cured slag samples containing 4% sodium, lost a significant amount of its flexural and compressive strength. However, flexural strength did not change and compressive strength increased a significant amount (about 20%) for slag mixture cured in water for 28 days after exposure to acidic medium.

The slag mixtures containing 8% sodium showed very small differences in flexural strength while compressive strength increased after exposure. A similar observation was made for slag mixtures containing 12% sodium. The Control cement mortar mixture lost a small percentage from its flexural strength; however, its compressive strength was reduced in the order of 20% after exposure. In comparison to the Control samples, all slag mixtures performed better, except for the slag mixtures containing 4% sodium and cured in air for 28 days. It could be considered that alkali activated slag mortars showed very good resistance to the hydrochloric acid solution.

For the sulphuric hydrochloric acidic exposure case, mass loss data showed that the control Portland cement mixture and alkali activated slag mixtures for all curing conditions showed a small mass loss percentage. All alkali activated slag mixtures showed an increase in flexural strength and compressive strength, except for the slag mixture containing 4%

sodium and cured in an oven for 24 hours. Other slag mixtures showed a significant amount of strength development after exposure to the sulphuric acid solution. Portland cement mixture reduced in flexural and compressive strength in a considerable amount after exposing them to the sulphuric acidic solution. All slag mixtures performed better than the Control sample mixtures. It was concluded that alkali activated slag mortars showed very good resistance to sulphuric acid solution.

Mass loss data and strength data obtained before and after immersing in a NaOH solution of alkali activated slag mortar and Control samples are exhibited in Table 5.

Table 5. Results of samples exposed to NaOH. Source: Bingöl (2018).

| Sodium Amount | Curing | Flex. Str. | Flex. Str. After exposure (MPa) | Comp. Str. | Comp. Str. | Mass Exchange (%) |
|---------------|--------|-----------------------|---------------------------------|-----------------------|----------------------|-------------------|
| | | Before exposure (MPa) | | Before exposure (MPa) | After exposure (MPa) | |
| 4% | 1d DO | 7.43 | 10.33 | 56.50 | 59.22 | -4.0 |
| | 28d DA | 7.16 | 9.80 | 37.02 | 57.21 | 1.5 |
| | 28d DW | 7.43 | 12.8 | 45.10 | 68.26 | 2.3 |
| 8% | 1d DO | 6.09 | 7.42 | 59.00 | 61.77 | -4.0 |
| | 28d DA | 8.71 | 7.21 | 58.79 | 56.93 | -7.0 |
| | 28d DW | 9.86 | 13.4 | 76.78 | 78.82 | 8.0 |
| 12% | 1d DO | 5.12 | 8.35 | 63.73 | 63.89 | -5.0 |
| | 28d DA | 9.02 | 9.47 | 62.71 | 66.68 | -5.0 |
| | 28d DW | 7.07 | 13.00 | 58.48 | 74.56 | 1.3 |
| CEM I 42.5 R | 28d GW | 9.26 | 8.00 | 54.78 | 41.60 | 2.0 |

Mass loss data showed that the Control samples did not lose weight while alkali activated slag lost a very small amount of their weight. The flexural and compressive strength of all slag mortars significantly increased in the NaOH solution and showed excellent resistance to basic medium. However, the Control samples reduced their strength properties in the order of 14 and 24%, respectively.

Conclusions

In the study, the resistance of sodium meta-silicate activated slag mortar to acidic and basic mediums was evaluated experimentally. From the study results, the following conclusion were made: Sodium meta silicate activated slag mortar showed excellent resistance to acidic and basic mediums by showing an increase in strength properties while the Portland cement mixtures showed a reduction in strength. Only the slag mortar mixtures, cured in air for 28 days and containing 4% sodium binder mass, showed lower performance than the Portland cement mixture's performance.

References

- Demir, İ., Sivrikaya, B., İlhan, O., & Yaprak, H. (2017). Effect of Ph Value of The Curing Water on Cement. *Turkish Science Research Foundation*, 10 (4), 39-46.
- Bingöl, Ş. (2018). *Investigation of mechanical and durability properties of alkali activated blast furnace slag mortars*. Phd Thesis. Institute of Sciences. Erciyes University. <https://tez.yok.gov.tr/UlusalTezMerkezi/> (Thesis no: 506168).
- Chindaprasirt, P., & Rattanasak, U. (2016). Improvement of durability of cement pipe with high calcium fly ash geopolymer covering. *Construction and Building Materials* 112, 956-961. DOI:10.1016/j.conbuildmat.2016.03.023
- Coppola, L., Coffetti, D. & Crotti, E. (2018). Pre-packed alkali activated cement-free mortars for repair of existing masonry building and concrete structures. *Construction and Building Materials*, 173, 111-117. <https://doi.org/10.1016/j.conbuildmat.2018.04.034>
- Criado, M., Palomo, A., & Fernandez-Jimenez, A. (2005). Alkali Activation of fly ashes Part 1: Effect of curing conditions on the carbonation of the reaction products. *Fuel* 84, 2048-2054. DOI: 10.1016/j.fuel.2005.03.030
- Juenger, M. C. G., Winnefeld, F., Provis, J., & Ideker, J. H. (2011). Advances in alternative cementitious binders. *Cement and Concrete Research* 41(12), 1232-1243. DOI: 10.1016/j.cemconres.2010.11.012
- Hossain, M. M., Karim, M. R., Hossain, M. K., Islam, M. N., & Zain, M. F. M. (2015). Durability of mortar and concrete containing alkali-activated binder with pozzolans. *Construction and Building Materials* 93, 95-109. DOI: 10.1016/j.conbuildmat.2015.05.094

- Luga, E. (2015) *Properties of Fly Ash and Blast Furnace Slag Geopolymer Mortars*. Phd Thesis, Institute of Science, Erciyes University. <https://tez.yok.gov.tr/UlusalTezMerkezi/>. (Thesis No: 387461).
- Mehta, P.K., & Monteiro, P.J.M. (2006). *Concrete Microstructure Properties and Materials*. 3rd edition, New York : McGraw-Hill Companies.
- Meyer C. (2009). The greening of the concrete industry. *Cement and Concrete Composites*, 31 (8), 601-605, DOI: 10.1016/j.cemconcomp.2008.12.010
- Neville, A. M. (1995). *Properties of concrete*. 4th edition, Longman.
- Puertas, F., & Martinez-Ramirez, S. (2000). Alkali-activated fly ash/slag cement strength behaviour and hydration products. *Cement and Concrete Research* 30 (10), 1625-1632. DOI: 10.1016/S0008-8846(00)00298-2.
- Sarker, P. K., Haque, R., & Ramgolam, K. V. (2013). Fracture behaviour of heat cured fly ash based geopolymer concrete. *Material and Design* 44, 580-586. DOI: 10.1016/j.matdes.2012.08.005
- Topçu, İ. B., & Canbaz, M. (2008). Acid Effect in Alkali Activated Blast Furnace Slag Mortar, *Dumlupınar University, Science Institute Journal* 16, 69-80. <http://dergipark.org.tr/tr/pub/dpufbed/issue/36104/406992>
- TS EN 1008 (2003). Ankara: Turkish Standardization Institute. TSE Mixing water for concrete - Specifications for sampling, testing and assessing the suitability of water, including water recovered from processes in the concrete industry, as mixing water for concrete.
- TS EN 196-1 (2016) Ankara: Turkish Standardization Institute. TSE Methods of testing cement - Part 1: Determination of strength.
- TS EN 197-1 (2012). Ankara: Turkish Standardization Institute. TSE Cement - Part 1: Composition, specifications and conformity criteria for common cements.
- Tekkim Kimya, Retrieved February 11, 2016 from Tekkim Kimya website. Interact website: www.tekkim.com.tr
- Zhuang, H. J., Zhang, H. Y., & Xu, H. (2017). Resistance of geopolymer mortar to acid and chloride attacks. *Procedia Engineering* 210, 126-131. DOI: 10.1016/j.proeng.2017.11.057