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Mechanical Examination of Fly Ash and Zeolite-Based Geopolymer Mortars

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Abstract

This study intends to study compressive and flexural strength properties, Ultrasonic Pulse Velocity (UPV), weight changes, and splitting tensile of geopolymer mortar made with fly-ash and zeolite as binder materials, silica sand and basalt stone powder as filler materials with different ratios (25%, 50%, and 75%). The prepared geopolymer mortar was activated by sodium silicate and sodium hydroxide solutions (12mol) and the Ground granulated blast-furnace slag was added with 13% of the completely prepared mix. The suggested specimens were exposed to compressive and flexural strength test at 7, 28, 56 days, weight changes were investigated after the 28th day, the splitting tensile test was tested at 28 and 56 days while the Ultrasonic Pulse Velocity tests was examined during 3, 7, 28, and 56 days.

The acquired experimentally results reveal that all the manufactured geopolymer mix's compressive and flexural strength properties increase with time and the highest value was attended on the 56th day. Moreover, the weight changes value were between 7.70% and 9.19%, splitting tensile test increase at the 56th day and were between 3.94 and 5.13 same as ultrasonic Pulse Velocity through time and the highest value was obtained on the 56th day for B2 mix with 4134 m/s.

Keywords: *Geopolymer, binder, fly-ash, zeolite, strength properties, UPV.*

1. Introduction

In general, an alkali-activated cement is mainly composed of an alkaline liquid solution (for example soda or sodium silicate) and a chemically reactive pulverulent solid, made for example of blast furnace slag or clays calcined, which are sources of reactive aluminum and/or calcium silicates (Fu et al., 2012). As for Portland cement, additives (plasticizers, etc.) can be added to them, in particular, to implement them (Dupuy et al., 2019). Geopolymers are special cases of alkali-activated cement. Although the term geopolymer was proposed by (Davidovits et al., 1991).

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This term defines a group of predominantly amorphous inorganic materials, having an aluminum-silicate skeleton, based on SiO2 and Al2O3. An alternative term is "inorganic polymer cement" or inorganic polymer cement (IPC) (Kamseu et al., 2012). Geopolymers are among the alkali-activated materials poor in calcium Ca (Gharzouni et al., 2018). They are often qualified as "green cement", on the one hand, due to the low emission of CO2 into the atmosphere, linked to their manufacture (Kenne Diffo et al., 2015), and on the other hand, their compressive strength, which may be similar or superior to that of certain Portland cement mortars or concretes (Cyr & Pouhet, 2015). Geopolymers are the product of a reaction between an alumino-silicate source and an alkaline or acidic activator solution. The materials used as the alumino-silicate source can be activated clays (by calcination at more than 500 ° C) (Kenne Diffo et al., 2015), such as metakaolin, obtained from kaolinite), fly ash (Criado et al., 2010), silica fume or a mixture of these different sources (Krivenko et al., 2007). The alumino-silicate source is usually in the form of a solid powder, and it is the only component that provides aluminates to the geopolymer (Duxson et al., 2007). The alumino-silicate source also provides silicates, but the reaction between aluminates and silicates, giving the geopolymer, must be activated chemically.

The activation solutions are:

- Acids, such as phosphoric acid (H3PO4) (Célerier et al., 2018) or mixtures of acids.
- Alkaline solutions such as sodium hydroxide (NaOH), potassium hydroxide (KOH), or an alkaline silicate of the type (n SiO2 -m M2O) where M is an alkaline cation (generally Na, K, or even Cs or Li).

In what follows, the alkali-activated binders with alkali silicates (Hajjaji et al., 2013), for which the literature is abundant and which have the best performance. Their behavior and performance are the best known and could be applied industrially for the design of grouts for the treatment of solids entering reservoir rocks.

In general, the chemical composition of a geopolymer can be written as:

$$aAl_2O_3 - bSiO_2 - cM_2O - nH_2O$$

with a, b, c, and n being the mole amounts of aluminates, silicates, alkaline activator, and water, respectively, and M the type of alkaline activator (usually Na, K, Li, or Cs). Sodium and potassium are the most commonly used activators. In the literature, the different formulations are expressed using molar ratios, which are either (1) those of the oxides, for example, SiO2/ Al2O3 (Chen et al., 2005), or (2) the chemical elements, for example, Si / Al. In the case of aluminum and silicon, there is a factor of two between the molar ratios of the element and those of the oxide. For example, a molar Si / Al equal to 1.8 corresponds to a molar $SiO_2 / Al_2O_3 = 3.6$.

In (Zhang et al., 2014) notably, the molar ratio (SiO2 / M2O) of an alkali silicate, where M is an alkali metal, is expressed as an oxide ratio and called the modulus of the silicate.

To evaluate the properties of geopolymers concerning the stresses of oil wells, for which Portland cements are usually used, (Elder et al., 2013) proposed composition limits for geopolymers based on metakaolin and solution. sodium-based activator (M = Na).

The fly ash from the incineration of household waste is made up of a large number of chemical compounds, some of which - based on heavy metals: lead, cadmium, etc. - are a threat to the environment. The properties of fly ash differ depending on the characteristics of the coal and the method of burning. It is generally useful as an additive in cement and concrete by showing pozzolanic properties due to its siliceous and aluminous composition. It increases the workability of fresh concrete due to its fine and spherical grains; it also reduces the heat of hydration. By reacting with the lime formed as a result of cement hydration, it forms an additional binder gel, fills the gaps in the cement paste, and adds strength to the concrete. Lime rate in fly ash obtained by burning lignite coal is generally high and such ashes also show hydraulic, ie binding properties. The deliberate use of fly ash in various fields provides an economic advantage for both the user and the ash producer, and the environment is protected as waste material is removed.

In addition, the user gains various technical advantages in new products or applications they produce. Despite all these positive aspects, the amount of fly ash evaluated using it cannot exceed a small percentage of the amounts obtained in the power plants, and the world average is around 15%. Zeolite is an aqueous alumina silicate crystal of porous, alkali (Na and K) and alkaline earth (Ca) elements with a three-structure network. The smallest structural unit of any zeolite crystal is SiO_4 or AlO_4 tetrahedra (He et al., 2012). Single and double ring secondary structure units and high symmetry parameters are formed by the combination of primary structure units formed by Si and Al tetrahedra. By arranging these polieder and secondary structure units in different shapes in three dimensions(Kong et al., 2007), a zeolite skeleton with micropores emerges.

Main physical and chemical properties of zeolites (Ranjbar et al., 2015); There is an ion change, adsorption and related molecular sieve structure, silica content, and also light color and lightness in sedimentary zeolites.

The structures of zeolites contain voids and have a honeycomb or lattice appearance. Cations and water, which are generally alkali and alkaline earth metals, can be found in cavities (Oh et al., 2010). The honeycomb or lattice structure of zeolites has a channel or gap size between 2-12 Ao. Since cations are weakly bound to the zeolite, they can easily exchange ions, so zeolites are used as ion exchangers (He et al., 2013). The water molecules in the pores can also be easily heated, leaving the zeolitic structure, or can be re-adsorbed.

Natural zeolites are a group name consisting of more than 40 minerals. The most known of these are; It is analcime, chabasite, clinoptilolite, erionite, ferrierite, heulandite, mordenite, stilbite, and phylipsite, laumonite, natrolite, faujasite, synthetic zeolite, synthetic zeolite X.

There are also synthetic zeolites. Pure and properly structured synthetic zeolites were first synthesized in 1938, and their production was carried out in 1948. These are not equivalent to natural zeolites and there are 200 types. These zeolites, especially detergent and consumed in the chemical industry in Turkey are not output at this time.

This work presents a geopolymer mortar manufactured by fly-ash and zeolite as binder materials activated with chemical solutions sodium silicate and sodium hydroxide, while as filler materials basalt stone powder were used replaced silica sand with different ratio (25%, 50%, and 75%). The mechanical performance of the manufactured geopolymer specimens was conducted at 7, 28, and 56 days. Visual appearance, the strength properties, UPV, weight changes, and splitting tensile of the manufactured samples were obtained experimentally.

2. Materials and Methods

In this work, fly ash and zeolite were used as binder materials silica sand and basalt stone powder was used as filler materials. Liquid sodium silicate (SiO_2/(Na)_2O = 3.29 M) ratio and sodium hydroxide (12mol) was once used for alkaline activation taken from AS Kimya (Istanbul/Turkey). The sodium hydroxide was prepared by adding 1liter of distilled water to 480g of sodium hydroxide pellets to obtain 12mol. The obtained sodium hydroxide was stored at room temperature for 24 hours before being used with sodium silicate/sodium hydroxide in a 2:1 ratio. To enhance the tenacity of the mix blast furnace slag was used. Basalt samples were homogenized and dried at 105^{o}C for 24 hours. From INCI Group Company (Sakarya/Turkey) the basalt powder stone was extracted. In this work as aggregate, the silica sand with less than 0,25 mm particle diameter was used correspondent to TS 706 EN 12620. The mixing procedure has been finished, the mortar was used to the molds 50x50x50 mm, 40x40x160 prisms, and 300*150 mm cylinders and vibrated, and then the geopolymer samples were kept for 24h in the ambient temperature. All the specimens were held for 24 hours in the drying oven at 100^{o}C. After the curing, the samples were preserved in room temperature conditions. Moreover, the mechanical tests, compressive strength test according to ASTM C 109 was executed after 7, 28, and 56 days utilizing the 50x50x50 mm cubes, and the Flexural strength test quoted by ASTM C 348 utilizing the 40x40x160 prisms samples was carried also after 7, 28 and 56 days (Nikolić et al., 2015).

Table 1: The Fly ash, zeolit, and slag chemical properties.

Chemical Analysis(%)	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3
Fly-Ash	59,94	22,87	4,64	0,94	3,08	1,55	2,19	0,62	0,35
Zeolite	71	-	1,70	-	3,40	1,42	2.70	-	-
Slag	40,55	12,83	1,1	-	32,58	5,87	-	0,79	0,18

Table 2. Sodium silicate chemical properties.

Chemical Analysis (%)	NaOH	Na_2CO_3	CL	SO_4	Al	Fe
SH	99,1	0,3	≤0,01	≤0,01	≤0,002	≤0,002

Table 3. Sodium hydroxide chemical propertimes

Chemical Analysis (%)	SiO_2	Na_2O	Fe (%)	Density (g/ml)	Heavy metals (%)
SS	27,0	8,2	≤0,005	1360	≤0,005

Table 4. Silica sand and basalt stone powder chemical properties

ChemicalAnalysis (%)	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	CaO_2	K_2O	Na_2O	SO_3
Silica sand	99,70	0,17	0,016	-	0,01	92,9	-	-	-
Basalt stone powder	56,9	17,6	8,1	0,9	7	-	1,9	3,8	-

Table 5. Mix of control sample geopolymer composites (g)

Fly ash	zeolite	Slag	SS (Si)	SH (NaOH (12 mol))	Silica Sand
700	300	133	667	333	2000

Table 6. The mix of basalt stone powder replacing silica sand with different ratios (g).

Fly ash	Zeolit	Slag	Si	NaOH (12mol)	Silica sand	Basalt powder
					1500	500
700	300	133	667	333	1000	1000
					500	1500

3. Results and Discussion

The mixes were named as following control (100% silica sand), B1 (75% silica sand and 25% basalt powder), B2 (50% silica sand and 50% basalt powder) and B3 (25% silica sand and 75% basalt powder).

Fig. 1. The manufactured geopolymer samples

3.1. Strength properties

Table 7. Compressive strength results (Mpa) at 7, 28 and 56 days.

Mix ID	7 days	28 days	56 days
Control	60,21	65,34	66,11
B1	62,84	67,27	68,08
B2	65,33	69,77	72,15
B3	63,12	65,44	66,79

Fig. 2. The diagram of compressive strength for the manufactured samples

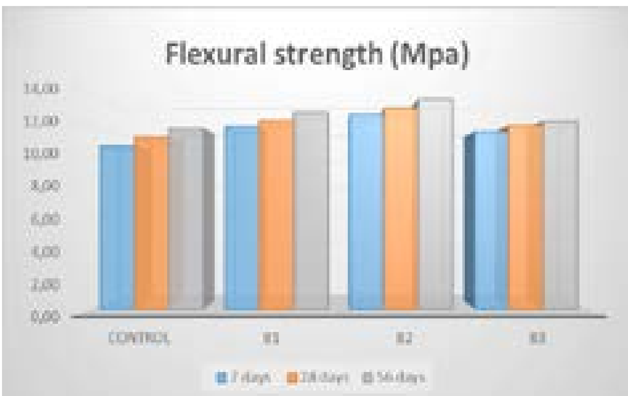

Fig. 3. The diagram of Flexural strength for the manufactured samples

Table 8. Flexural strength results (Mpa) at 7, 28 and 56 days.

Mix ID	7 days	28 days	56 days
Control	10,66	11,23	11,81
B1	11,94	12,34	12,87
B2	12,72	13,13	13,72
B3	11,59	11,98	12,25

The different combinations used in this work affect the results of compressive and flexural strength, Four samples were mixed with basalt powder and partial replacement of silica sand was observed as a new class in sequence to consider the acquired results. Tables 7 and 8 show the results below. According to compressive strength, the mixes with partial replacement of basalt powder with a ratio of 50% (B2) confirmed higher consequences when in contrast to 100% silica sand as a control mix. The combination of 25% basalt powder (B1) satisfied demonstrated a changeable achievement from 7 to 56 days, the same as 75% basalt (B3). Also, the results obtained for the mixe of 50% basalt powder (B2) 7 to 56 days the performance increased with time and the final result was 72.15 Mpa. Furthermore, the samples show an increase in flexural strength and the best results obtained was 50% basalt powder (B2) with a value of 13.72 Mpa at 56 days. The whole results are showin in Fig.2, Fig.3, Table 7, and Table 8.

3.2.Water absorption and porosity

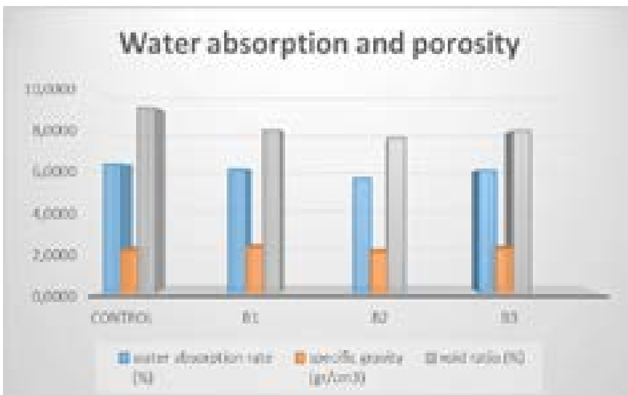

Fig. 3. The diagram of Flexural strength for the manufactured samples

Observing the physical properties of manufactured geopolymer sample about the water absorption and porosity of control sample 6.93% in terms of porosity 9.19% of water absorption. The basalt stone powder samples (B1, B2, B3) confirmed good overall performance in contrast with the control samples, the values were 6.11%, 5.67%, and 6.06% respectively for porosity and 8.08%, 7.70%, and 8.03% of water absorption respectively, The fine particles of basalt stone powder could explain the behavior bellow and provide a good transportation property of the matrix.

3.3 Ultrasonic pulse velocity

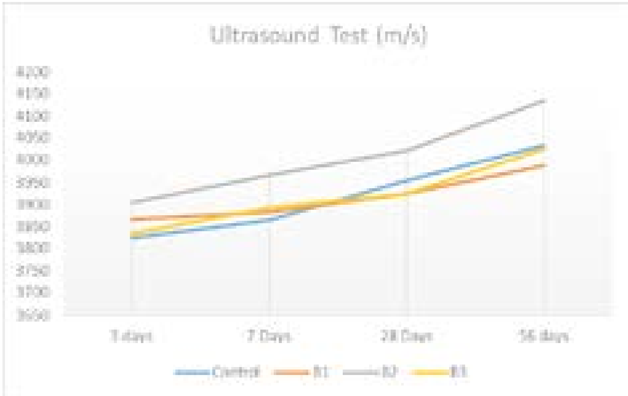

Fig.4. Ultrasonic pulse velocity values (m/s).

To investigate the homogeneity of the matrix, the ultrasonic pulse velocity test was investigated. Fig 4 shows the result obtained (Chakkor & Altan), as can be remarked B2 (50% basalt stone powder + 50% silica sand) sample confirmed a certain boom in velocity and, besides they exceedingly yielded higher effects in contrast with the other mixes the value yielded were 3905 m/s, 3965 m/s, 4022 m/s, and 4134 m/s respectively for 3, 7, 28 and 56 days. B1 and B3 mixes showed an increase from 3 to 56 days and the final value was 3988 m/s and 4025 m/s respectively. Furthermore, the control sample yielded 3825 m/s, 3864 m/s, 3955 m/s and 4034m/s respectively for 3, 7, 28 and 56 days.

3.4. Split tensile strength

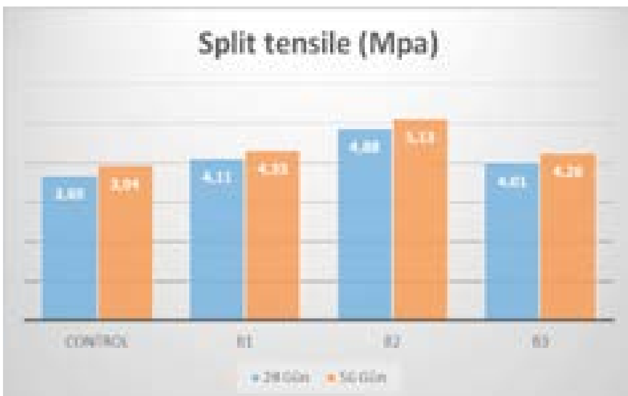

Fig.5. Ultrasonic pulse velocity values (m/s).

The split tensile strength of the manufactured geopolymer mortar samples with different filler ratios of silica sand and basalt stone powder related to 28 and 56 days is shown in the bar diagram in Fig.5. From the bar diagram the geopolymer mortar sample B2 blended with 50% basalt powder and 50% silica sand shown maximum split tensile strength values from the other mixes with 4.88 Mpa and 5.13 Mpa. Moreover, it was observed that the split tensile strength for the other mixes B1 and B3 increases gradually with age from 28 to 56 days.

4. Conclusion

The current work propose was to study the mechanical composition impact of adding basalt stone powder as a filler material with silica sand with different ratios (25%, 50%, and 75%), and the main binder materials of this study, fly ash and zeolite based on geopolymer composites.

Moreover, the experimental study approved that by observing compressive and flexural strength a significant increase estimate was obtained in terms of time 7, 28, and 56 days for all the manufactured samples. Furthermore, the higher value when in contrast to the control were basalt stone powder B2 (50% basalt stone powder + 50% silica sand) geopolymer samples.

Regarding water absorption and porosity effects in contrast to the control sample, the basalt stone mixes improved amelioration in weight loss due to abrasion.

The basalt stone powder is used as a filler material with silica sand in different ratios to produce composites with proper inside traits and applicable interfacial for the geopolymeric matrix.

As an established conclusion, growing waste materials, as a filler replacing silica sand with different ratios, basalt powder concerning the control samples contributed to the mechanical behaviors development of the composite. On the other hand, the use of fly ash and zeolite as a binder material with a ratio of 70% - 30% was given the best result according to other ratios used in the trail mixes.

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