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Evaluation of Durability Behavior of Geopolymer Concrete Containing Nano-Silica and Nano-Clay Additives in Acidic Media

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ABSTRACT

The present study mainly aims at the identification and laboratory production of geopolymer concrete with the increased resistance to acid attack. For this purpose, after evaluating the reactivity of the raw materials and their applicability in the production of geopolymer cement among the available aluminosilicate materials, the metakaolin pozzolan was selected as the appropriate raw material. After the selection of the binder and activator of sodium hydroxide, the alkali activation process was performed for the mixtures of raw materials. Then, for the initial estimate of the durability performance of geopolymer cement in acidic media, the durability of concrete samples was investigated in 1M sulfuric acid for 7, 28 and 90 days. The results showed that the nano-silica and nano-clay are effective in improving the performance of geopolymer concrete, as the addition of 3% nano-silica resulted in the 0.44% increase in the strength of the geopolymer concrete. Also, the addition of 3% nano-clay led to the increase in the strength of geopolymer samples up to 0.54. In addition, the samples are more durable in acidic media, so the weight loss of nano-clay-containing geopolymer samples in 90 days is 1.2 times that of geopolymer concrete samples without this additive.

Keywords: Geopolymer, acidic medium, nano-clay, nano-silica, compressive strength, tensile strength, acid

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1. INTRODUCTION

eopolymer cement is a combination of natural -geological materials of silicate and alumina and hence referred to as geopolymer environmentally friendly. It is a green cement and the finished surface is aesthetically pleasing. In order to produce this type of cement, sodium hydroxide and potassium hydroxide (chemical and petrochemical waste) alkali solvent are separately prepared and then added to sodium silicate liquid. This solvent is mixed with natural pozzolan powders such as fly ash, metakaolin, etc. [1-3]. On the other hand, metakaolin has been commonly used as the base material in the concrete samples [4-6], and sodium hydroxide and sodium silicate have been used in the studies for the curing of the base material [7-13]. Various researchers have investigated the geopolymer concrete and its strength in acidic media. Zhuang et al. (2017) investigated the behavior of geopolymer concrete mortars in the acidic and chlorine media. In this study, the researchers conducted a laboratory study on the chemical erosion resistance of geopolymer mortar (GM) to sulfuric acid and sodium chloride. The variations of the mechanical properties and mass of GM after soaking in water, sodium chloride and sulfuric acid solutions were studied for different time periods (30, 60, 90, 180, 270 and 360 days). The results of the experiment showed that GM has good resistance to erosion solutions of sodium chloride and sulfuric acid [14]. Li et al. (2019) investigated the potential deterioration of geopolymer concrete containing metakaolin in the corrosive environments. The geopolymers were exposed to the air cycle, seawater, dry and wet thermal cycle and seawater heat for 30, 60 and 90 days, and finally, the geopolymer concrete structure was analyzed by SEM and X-ray methods [15]. Merabtene et al. (2019) studied the application of calcined kaolin in the production of geopolymer concrete [16]. Toniolo and Boccaccini (2017) used several types of materials such as biomass ash, red mud, recycled glass and heavy metals waste for the evaluation of geopolymer concrete behavior. Because of insufficient SiO2/Al2O3 ratio, it is not sufficient to individually use these materials for the production of geopolymer concrete. Therefore, mixing these materials with fly ash or metakaolin can be a suitable option for the production of geopolymer concrete, which has been

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investigated in this research [17]. Colangelo et al. (2013) discussed the use of metakaolin as a geopolymer base material with epoxy materials in concrete production, and the results showed that the use of new materials can increase the compressive and tensile strength of geopolymer concrete [18]. Luukkonen et al. (2018) reviewed the use of activators in the production of geopolymer concrete. The research suggested that this material is used as an activator and is not easy to work with because of its binding properties. The method of using this alkali material and its processing and the way it reacts with the base material are described in this study [19].

The durability of concrete samples in acidic media is among the issues that have always received the interest of researchers [20-23]. For example, Vafaei et al. (2019) explored the durability of geopolymer concrete made of fly ash in acidic medium. In this study, the 5% acid was used to evaluate the durability of concrete samples, and the results showed that the geopolymer concrete had a better resistance behavior in acidic media [24]. Alzeebaree et al. (2019) evaluated the behavior of geopolymer concrete containing the fibers in acidic

2. MATERIALS AND METHODS

In this part of the research, the used materials and also the method of tests conducted on the concrete samples are reviewed.

2.1. MATERIALS AND PROPERTIES

The materials used in this study include nano-clay and nano-silica, which are utilized with the metakaolin base material for the production of geopolymer concrete. In this part of the study, the materials used in the mix design of geopolymer samples are introduced. Sand and gravel:

medium. In this study, the BFRP and CFRP wrapping were used in acidic medium, and the results showed that the use of wrapping increased the durability of samples in acidic medium [25]. Özcan and Karakoç (2019) also investigated the geopolymer concrete made of slag in acidic media, and examined the sample weight change in acidic medium, amount of compressive strength and also ultrasonic test on acidic samples [26]. The review of literature on the geopolymers and their durability in acidic media indicates the good durability of geopolymer samples in corrosive environments [27-28]. This type of concrete can create corrosion protection, which makes it as a practical option for retrofitting the structures in deleterious environmental conditions.[29] Valikhani has investigated the use of this type of concrete for retrofitting the bridges in corrosive environments.[30] In the present study, two types of nano-silica and nano-clay additives were added to the geopolymer concrete. Therefore, in this research, after describing the process of producing the geopolymer samples, the compressive and tensile strength tests are performed and the results of the work are finally plotted on the graphs.

According to the ASTM classification, the aggregates smaller than 4.75 mm and larger than 0.075 mm (75 μm) are referred to as sand. According to the unified soil classification system (USCS), sand is grouped into three categories: fine, medium and coarse. Nano-silica: The silicon dioxide nanoparticles, also known as silica nanoparticles or nano-silica, provide the basis for a large volume of biomedical research due to the high stability, low toxicity, and applicability along with a wide range of molecules and polymers [31-33]. Figure 1 shows the nano-silica particles used in the research.



Figure 1. Nano-silica particles used in geopolymer concrete

Nano-clay: Nano-clays are very fine-grained materials with a particle size of less than 5 μm . The clay particles are very active in such dimensions. Nano-clay is actually a compound (or composite) of inorganic silicates which

has the electric charge and is classified into different categories depending on the composition and morphology of the nanoparticles. Figure 2 shows the nano-clay particles used in the research.



Figure 2. Nano-clay powder used for production of geopolymer concrete

Sodium hydroxide: Sodium hydroxide or caustic soda (NaOH) is a white and solid mineral composition with the melting point of 318 °C and density of 2.13. It can easily absorb the moisture and carbon dioxide existing in the air. Sodium silicate: Commonly known as sodium metasilicate and also water glass, it is recognized as one of the widely used and important materials due to the

widespread industrial and commercial uses. The main constituents of sodium silicate include silica, sodium oxide and water, where water plays a part in the hydrous and anhydrous compounds derived from this product, and silica is recognized as the major component in this compound.

2.2. MIX DESIGN OF STUDY SAMPLES

In the present study, six mix designs were used where the percentage of nano-silica and nano-clay varied in this

study. <u>Table 1</u> shows the mix design of the produced samples.

Table 1. Mix design of geopolymer concrete produced in laboratory

Material	Sand	Gravel	Metakaolin	Sodium silicate	Sodium hydroxide	Nano- clay	Nano-silica
Group 1	800	1200	300	95	85	0	0
Group 2	800	1200	300	95	85	1	0
Group 3	800	1200	300	95	85	2	0
Group 4	800	1200	300	95	85	3	0
Group 5	800	1200	300	95	85	0	1
Group 6	800	1200	300	95	85	0	2
Group 7	800	1200	300	95	85	0	3

In <u>Table 1</u>, group 1 is the control sample and groups 2-7 are the geopolymer concrete samples made of nano-clay and nano-silica.

2.3. GEOPOLYMER PRODUCTION AND RESEARCH OBJECTIVE

The design of geopolymer concrete, as with the mix design of ordinary concrete, is selected based on future performance. In the geopolymer concrete made of metakaolin, for the mass ratio of alkali solution to base material, the values ranged from 0.3 to 0.45, the mixing time was 5 min, and the curing was done in the pond of

water for 24 h after the molding [30-32]. Various studies have shown that the use of sodium hydroxide in different quantities leads to the dispersion of the compressive strength of concrete samples. Also, the change in the ratio of sodium hydroxide to sodium silicate results in the change in compressive strength of concrete samples and the liquid limit of samples [33-39]. This study investigates the effect of adding nano-clay and nano-silica on the compressive and tensile strength properties of geopolymer samples and also the durability of samples in acidic medium. Figure 3 shows the produced samples and the pond of water for the 7- and 28-day curing of the samples.



Figure 3. Concrete sample curing pond

2.4. EXPERIMENTS

2.4.1. COMPRESSIVE STRENGTH TEST

The test was performed on standard 15*15*15 cubic samples and the amount of compressive strength was obtained by applying to the sample at 7 and 28 days per square centimeter during the sample failure. Also, the

effect of adding nano-silica and nano-clay was investigated. Figure 4 shows the compressive strength of concrete samples.



Figure 4. Compressive strength test of geopolymer samples

2.4.2. TENSILE STRENGTH TEST OR BRAZILIAN TEST

In the Brazilian tensile test, the sample is positioned between the plates of the device so that its axis is horizontal and then the load is increased to generate the failure as the splitting in the plane containing the vertical diameter of the sample. This test method is recommended in section 117 of ASTM C496-71 and BS 1881-8371 (revised in 1979 and 1990). The dimensions of the cylinders are standard and equal to 150*300 mm, as shown in Figure 5.

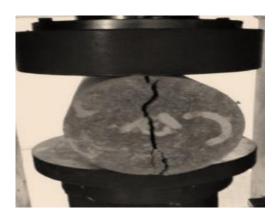


Figure 5. Tensile test of geopolymer samples

2.4.3. SAMPLE STRENGTH TEST IN ACID SOLUTION

The weight changes of concrete samples in acidic medium were reviewed on a weekly basis. In the test method, the samples were exposed to acidic medium for 7 and 28 days after the production and the weight changes were reported, and the percentage of concrete corrosion in the acidic medium was given on the diagrams of Figures 6-7. The test results of exposing the concrete samples to acidic medium showed that the acid attack onto the concrete surface led to the corrosion of the concrete, so that the

surface of the geopolymer concrete samples was corroded to a relatively large extent after the 28-day exposure to the acidic medium. The sample weight after the exposure to acidic medium for 28 days reached 80% of initial value in the control sample, indicating that the durability of samples in acidic medium is increased with increasing the age of concrete. In this study, the 7- and 28-day samples were subjected to the acidic medium for 7 and 28 days.

3. RESULTS AND DISCUSSION

This section presents the results of compressive strength, tensile strength and weight changes of concrete samples in acidic medium, and <u>Figures 6-8</u> show these results, respectively. As can be seen in all the figures, the addition of nano-clay and nano-silica for 3 wt.% of cement had the highest compressive and tensile strength, while the corrosion rate of the geopolymer samples in acidic media was greatly reduced. In the following, it is attempted to

clarify the cause of this issue by discussing the results of the present study and previous research. The results presented in the diagrams of <u>Figures 6-8</u> correspond to the average values of all samples produced in the laboratory, which were separately obtained for the ages of 7 and 28 days. In each group, 3-5 samples were produced to control and ensure the accuracy of the results

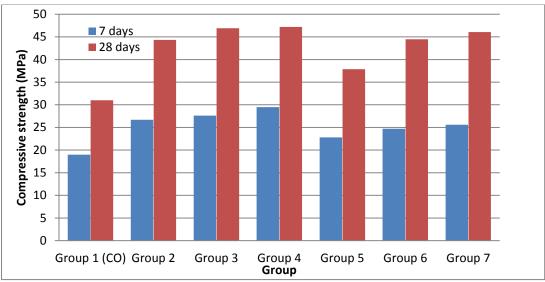


Figure 6. Compressive strength results of geopolymer samples

Figure 6 shows the compressive strength of the samples produced for this study. As shown in the figure, using nano-silica and nano-clay increases the compressive strength of the geopolymer concrete, so that for 2% increase in the amount of nano-clay, the compressive strength of the geopolymer samples is 1.38 times that of the 7-day control concrete samples and 1.41 times that of the 28-day control concrete samples. The use of 3% nano-clay also caused that the compressive strength of 7-day samples to be 1.39 times that of the control sample for 7-day concrete and 1.44 times that of the control sample of 28-day concrete for nano-clay. This value for nano-silica is 1.30 times for the 7-day concrete and 1.29 times for the 28-day concrete strength. Figure 7 shows the tensile

strength of geopolymer concrete samples. The tensile strength of concrete samples for the addition of 2% nanoclay was 1.35 times that of control samples at 28 days and 1.30 times for nano-silica control samples at the age of 28 days. At the age of 7 days, the geopolymer samples showed an increase in the strength of concrete samples for 2% nano-clay up to 1.31 times the control sample and 1.24 times the control sample for nano-silica. The use of nano-clay for 3 wt.% of cement resulted in the tensile strength of geopolymer concrete made of metakaolin up to 1.38 times the control sample, and the addition of nanosilica resulted in the compressive strength of geopolymer concrete up to 1.29 times that of the control sample.

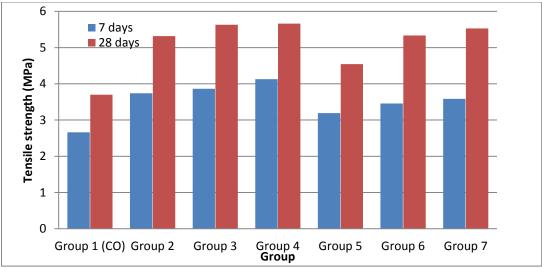


Figure 7. Tensile strength results of geopolymer samples

Figure 8 shows the durability of the geopolymer samples for 7, 28, and 90-day exposure to acidic medium where the weight loss was not evident at 7-day age of concrete. However, the exposure of concrete to acidic medium for 90 days resulted in the weight loss of concrete samples and reached 0.67 and 0.70% of initial value in the geopolymer samples containing 2 and 3% nano-silica, respectively. The change in the nanomaterial did not have a significant effect on preventing the weight loss of the samples, but the samples containing nano-clay had less cracking and corrosion than the control samples.

In recent years, nanotechnology has made a great breakthrough in human knowledge. As a product of nanotechnology, nanoparticles have been recognized as a highly active synthetic pozzolan in concrete technology and influenced and improved the structure of cement-based materials with the application in their production. In this study, due to the covering of concrete voids, the use of nano-clay leads to the increase in the compressive and tensile strength of the materials, and increasing the percentage of nano-clay and nano-silica to 3% increases the strength and stability of concrete in acidic media. Also, the better curing of cement in concrete due to the addition of nano-clay and nano-silica is one of the main reasons that the compressive and tensile strengths of concrete were increased with increasing the nanomaterial.

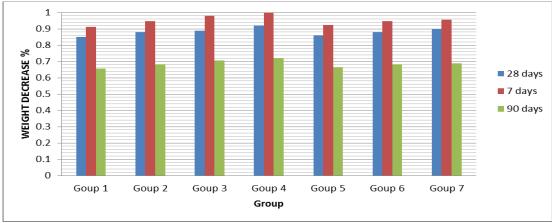


Figure 8. Weight changes of concrete samples in acidic medium after 28 and 7 days (Group 1 is control sample)

Adak et al. (2017) used nano-silica in concrete, and for the use of 6% nano-silica, the compressive and bending strength of concrete reached 1.3 and 1.20 times the control sample, respectively [38]. However, in the present study, the increase in strength was greater in the samples made of metakaolin. On the other hand, in the both studies, the compressive strength of the samples was increased with the age of concrete and reached to 1.35 times in the present study and 1.40 times in the 7-day sample of Adak et al. Assaedi et al. (2016) [39] investigated the effect of nano-clay on geopolymer samples. According to this study, adding 2% nano-clay to concrete samples increases the compressive and tensile strength of the sample, and the compressive strength of the sample is 1.33 times the original sample for the optimal amount of nano-clay. The relationship between tensile strength and compressive strength in the present study reaches 0.7 time the compressive strength, which is 0.74-0.84% in previous studies in the same concrete grade [41].

Davidovits et al. (1990) [42] also evaluated the behavior of geopolymer samples in acidic medium, and the results showed that the exposure of geopolymer samples to acidic medium leads to the corrosion of the sample and its weight reaches 0.92 times the initial weight. Also, by increasing the sample age to 30 days according to the

4. CONCLUSION

In the present paper, the effect of adding nano-clay of nanoplate dimension to concrete was investigated in the laboratory to improve the mechanical properties of concrete including the compressive and tensile strength. Nano-clay and nano-silica were added to the concrete at three different percentages proportional to the mass of cement at constant mixing percentages and the concrete samples were cured at two ages and tested according to ASTM C116 standard method. The study results are summarized below for the average values of all samples produced for a particular group as the overall research results.

- The use of nano-clay for 3 wt.% of cement caused the average compressive strength of geopolymer concrete made of metakaolin in all samples to reach 1.44 times the control samples. The addition of nano-silica also caused that the compressive strength of geopolymer concrete to reach 1.35 times that of the control sample.
- The use of nano-clay for 3 wt.% of cement caused that the average tensile strength of geopolymer concrete made of metakaolin in all samples made in 7 and 28 days to reach 1.38 times the control sample. The addition of nanosilica also caused that the compressive strength of

study of Cevik et al. (2018) [40], the sample weight reaches 0.85 times the initial weight. In the present study, the exposure of the samples to acidic medium resulted in the sample weight to reach 0.80 times the initial value, and if the age of the concrete samples is increased, this corrosion is also increased, so that the exposure of the concrete samples in the acidic medium for 90 days leads to the increased corrosion, and the weight of the samples in the control samples reaches 0.650 times the control samples. The use of nano-clay and nano-silica leads to the increased stability of samples in acidic medium, as the use of nano-clay caused the weight of samples for the optimum percentage of nano-clay and nano-silica to reach 0.72 and 0.70 times the initial value. In general, the nanoparticles act as the crystallization centers, and the fineness of nanoparticles makes it possible to fill the pores and voids simply. Also, as a filler, they have a greater effect on the pores and good cohesion between the cement paste and the aggregate and improve its compressive strength. One of the desirable effects of using nano-sized particles is to increase the viscosity of the concrete before the setting time, which improves the aggregate suspension and increases the concrete workability. The effect of increase in the compressive and tensile strength of the concrete samples was observed in the present paper.

geopolymer concrete to reach 1.29 times that of the control sample.

- The replacement of 2 wt.% of cement with nano-clay and nano-silica in the mix design of geopolymer concrete resulted in the increased compressive and tensile strength of concrete samples, so that the average compressive strength of geopolymer samples in all samples (7 and 28 days) is 1.41 times the control sample for the addition of nano-clay and 1.30 times the control sample for the addition of nano-clay. The tensile strength of concrete samples was 1.3 times that of control samples for the addition of 2% nano-clay, and 1.25 times that of control samples for nano-silica.
- The durability of concrete samples in acidic media was increased with the increased optimum percentage of nano-clay and nano-silica, as the weight loss of concrete samples made of nano-clay and nano-silica was lower in acidic media. In the control sample, after 28 days exposure to acidic medium, the weight of geopolymer concrete reached, on average, 0.84-0.90 times the initial weight for all the samples made in this group, whereas if the nano-clay is used for the production of the samples, they lose 0.12 of their weight. The use of nano-silica also

led to 0.14 weight loss for the 28-day concrete. Increasing the exposure of concrete samples to acidic medium to 90 days caused that the average weight of concrete samples to reach 0.62-0.70 times the initial samples, and the use of nano-clay at its optimum percentage can reduce the concrete corrosion, so that for 3 wt.% of nano-silica, the average corrosion value for all the samples made for this group reaches 0.70 times the initial value and 0.72 times for the nano-clay.

In general, the use of nano-silica and nano-clay in the samples produced in this study caused that the addition of

both nanomaterials to affect the mechanical properties of geopolymer concrete and also increase the durability of concrete to the destructive agent of acidic media which reduces the process of gaining compressive strength at higher ages. Nano-silica minimizes the porosity in hydrated cement by filling the voids between the larger particles and greatly reduces the permeability. Nano-clay also increases the durability and compressive and tensile strength of geopolymer samples by filling the voids between the aggregates and with the good compatibility with cement and the better curing.

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AUTHORS CONTRIBUTION

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CONFLICT OF INTEREST

The author (s) declared no potential conflicts of interests with respect to the authorship and/or publication of this paper.

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