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A RESEARCH FOR BACTERIAL SELF-HEALING IN METAKAOLIN BASEDGEOPOLYMER MORTARS

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ABSTRACT

Cement production is a polluting process for nature. For this reason, new types of concrete which can be produced with recycled materials and without cement continue to be investigated. On the other hand; cracks in structural elements reduce the strength and durability of a building. Extending service life of buildings has eliminated the cost of rebuilding and thus, contributed to both the economy and the ecosystem. Due to this, research on crack healing in Portland cement concretes with various bacteria is continuing for some time. However, there are not enough studies in literature regarding the improvement of metakaolin-based geopolymer mortars produced without using cement by urolytic bacteria. The parameters of temperature, pH and void ratio of bacterial geopolymer mortar affect the viability of bacteria. For example, pH value of the medium required for the survival of bacteria is, generally around nine. During the production of geopolymer concrete, a sudden increase in high alkali environment occurs due to use of activators. This reduces the survival rate of bacteria added to the mixture during the production of geopolymer mortar. In this study, the most suitable environment for geopolymer mortar, and the conditions for the bacteria to survive until the end of the curing process for the mortar to be strengthened were investigated. Analyses on the effects of urolytic bacteria and geopolymer mortar healing process on mechanical strength of the mortar were conducted. *Sporosarcina Pasteurii* were used for the self healing process. Various mixtures of geopolymer mortars were cured under different environmental conditions to observe changes in their mechanical strength and water absorption capacity. As the result of the study, the most suitable mixture ratio and curing medium were identified. It was observed that the nutrient, ensuring the life cycle of the urolytic bacteria, had no negative effect on the mechanical strength of mortar and reduced

capillary water absorption of the mortar.
This study is a specific text in the literature that analyzes bacterial curing conditions and the effect of improving geopolymer mortar.
Keywords: Geopolymer concrete, urea, self healing, bacterial healing, mechanical properties.

1. INTRODUCTION

One of the most important greenhouse gases which cause air pollution is CO_2 gas with a rate of 7%. Today's cement production techniques are among the main factors causing CO_2 emissions. Less harmful waste emissions and reuse of wastes are important for preventing environmental pollution. Therefore, scientific researches on environment friendly materials are continuing

intensively. Currently, geopolymer concretes (cementless concretes) that can replace cement in the construction sector are produced. Cracks in concrete are also not desired in geopolymer concrete because they reduce strength and durability. Therefore, it is necessary to repair and seal the cracks in the concrete in order to increase the life of a building. Bio-cement and self healing crack repairing techniques in Portland cement mortars with various bacteria are currently being investigated extensively. In addition to this, there is a need for researches on the use of these bioagents in geopolymer mortars and their results. However, many studies are required for the use of these biologic agents in geopolymer mortars.

Geopolymer mortars are new generation inorganic binders with a high potential to replace Portland cement mortars [1]. The first geopolymer concrete research was initiated in 1994 by Davidovits, J. Geopolymer cement is a high alkali (K-Ca) -Poly (sialate-siloxo) cement. It is created by an inorganic polycondensation reaction called geopolymerization. Geopolymer cement quickly sets at room temperature and reaches a compressive strength of approximately 20 MPa after 4 hours. 28 days compressive strength is in the range of 70-100 MPa when tested at 20 ° C and in accordance with standards applied to hydraulic binder mortars. It can be used for radioactive waste storage due to its high early strength, low shrinkage, freeze-thaw resistance, sulfate resistance and corrosion resistance. These high alkali cements do not produce an alkali- aggregate reaction [2].

Metakaolin/fly ash/slag, aggregate and alkali catalytic liquids are used in the production of geopolymer mortars. There are different combinations in the alkaline activation system such as alkali silica, hydroxide, distilled water, etc. This process is a complex chemical process involving geopolymerization of alumino-silicate materials, dissolution of raw materials, transport or orientation, and polycondensation of reaction products [3], [4], [5], [6]. Any natural mineral or industrial waste may be used as the binder, provided that it contains amorphous Si and Al. Hydration products of fly ash / metakaolin are sodium aluminosilicate hydrate gels [7].

In one study that analyzed the impact of curing temperature on concrete strength in geopolymer mortars, the mortars were cured at 10, 20, 30, 40, 60, and 80 ° C. It was determined that curing time decreased from 40 days to 1 hour as temperature increased. However, the cavity rates of high temperature cured mortars also increased. The researchers stated that high- temperature curing provides early strength, but the effect of curing heat on late strength is the opposite [8]. There are different ways in the literature to cure geopolymer concrete. Although heat curing is known to give the highest resistance in the shortest time, successful results were also obtained by trying self-curing methods with biocomponents [9].

Research in the field of geopolymer has shown that these mortars have the potential to be used in concrete repair [10]. Some authors [11] have shown that geopolymeric mortar-repaired concrete specimens with one-day curing have higher binding strength than samples repaired with commercial repair products available after 28-day curing. It has also been reported that metakaolin-slag-based geopolymers are the best composites in terms of acceptable mechanical and durability performance as well as being environment friendly [12].

Apart from the type of the binder, another important issue in concrete is the cracks that occur due to various reasons and affect durability. As a result of this cracking, strength loss occurs in concrete or reinforced concrete element and security problem arises. Then, either the reconstruction of the structure or the repair of the concrete will be necessary. Since reconstruction is a costly, time consuming, and environmentally harmful method, concrete cracks are repaired. However, repairing and reinforcing concrete with existing techniques is as laborious as reconstruction of concrete. Various researches are conducted to solve this problem more economically and environment friendly. At this point, self-healing concrete researches stand out. Self-healing concretes, very popular in concrete in recent years, are biologically limestone producing systems for covering cracks on concrete surfaces.

Bacterial species, capable of precipitation of carbonate, are mixed with a calcium-based feeder and added to the mortar. These bacteria can form endospores and remain in the concrete structure for up to 200 years. When the

water starts to get into the cracks of the structure, these spores are activated if the necessary nutrients are present for the bacteria. The bacteria then feed on the calcium source and consume the oxygen in the environment. Limestone solidifies in the crack area and prevents water from entering the crack. Thus, the dissolved calcium source becomes limestone and the reinforcement is protected against corrosion since there is no oxygen left inside.

In the literature, one of the most common and first studied ulcerative soil bacteria in self- healing concrete is *Sporosarcina pasteurii* [13], [14] [15]. This bacterial species breaks down the urea with the unease enzyme and precipitates $CaCO_3$ using free Ca + ions. The reason for using *Sporosarcina pasteurii* pasteurii bacteria in this study is that this species can survive by forming endospores even in insufficient environmental conditions such as high pH and low nutrients. The studies have shown that 2% of *Sporosarcina pasteurii* cells can remain viable for 330 days when they are added into the cement mortar together with the Urea-Yeast Extract medium. [16], [17]. The nutrients necessary for sporosarcina pasteurii are urea, peptone and some salt in liquid medium.

There are many studies on self-healing of cement-bound concrete in the literature. It has been observed that *Sporosarcina pasteurii* in fly ash/silica fume concretes improve cracks, strength, and durability [18], [19]. In many other studies, it has been reported that the improvement increases the compressive strength of conventional concrete by approximately 20%. [20]. However, it does not affect flexural strength [21]. In another study on bacterial conventional concrete, it was observed that the compressive strength values of the mortar containing bacteria decreased slightly after 90 days. The reason for this situation is due to the gaps resulting from the dissolution of the protein structure when the bacteria lose their viability [22]. This has also been attributed to degradation of biomass, which may create additional porosity [23]. There are parameters that affect healing, the first of which is the cell density of the bacteria [18], [24].

Almost all of the similar studies in the literature are about cement-bound concretes and no study has been found on investigation of the appropriate method for healing in geopolymer mortar and on the effect of healing cures on the mortar. The aim of this study is to investigate the

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ideal cure method and nutrient effect on the mechanical properties of geopolymer mortar in early and mid term.

2. EXPERIMENTAL PROGRAM

Materials and Mix Design

Bacterial growth: In this study, *Sporosarcina pasteurii* (DSMZ 33) was used as the bacterial agent. The contents of the designed liquid medium are as follows: 20 grams urea (MERCK), 20 grams peptone (NEOGEN), 5 grams NaCl for 1 liter. Bacterial culture of the samples was obtained by growing the bacteria in the shaker incubator at 150 rpm for 24 hours. At this stage, the bacterial density was checked by spectrophotometric analysis (OD600-Thermo brand microplate reade).

Sporosarcina Pasteurii has a suitable living temperature of approximately 30 ° C. It is known that when it is dried at 40 ° C, it convert to the sport form but loses its vitality in rapid temperature and pH increases. The reason for this is that these rapid changes have serious impact on the bacteria. For this reason, it is necessary to control sudden temperature and pH increases in geopolymer mortars where no encapsulation is performed. In this study, firstly the resistance of non-spore bacteria to alkali activator fluids was determined by bacteria-NaOH titration test. The purpose of the titration is to determine whether there will be a chemical reaction between the bacteria culture and the NaOH substance, as well as is to detect the behavior of bacteria against activators. For this, 12 M NaOH solution was prepared. The pH of the bacterial culture was measured to be pH = 9. It was determined that 1 ml of NaOH solution taken by pipette corresponds to 30 drops and instead of the liquit medium placed on the magnetic stirrer, NaOH was added at rates ranging from 2-20 drops. Samples were taken from the NaOH-bacterial culture titration and solid agar medium was seeded and reproduction was shown in Figure 1.

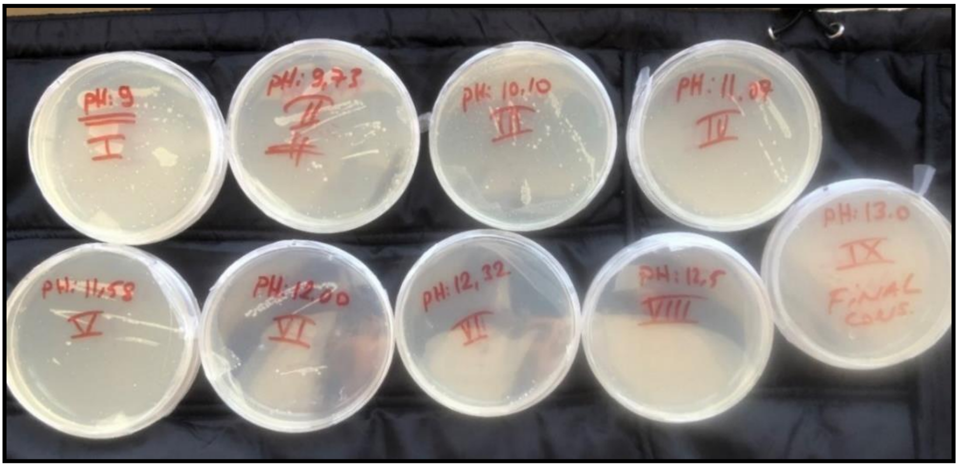

Figure 1. The bacteria-NaOH interaction in agar medium

According to the results of the titration test, directly mixing of bacterial culture and NaOH liquids was not considered appropriate. Alternatively, The bacterial spores were collected from the solid agar surface to pure water. Bacterial density was adjusted to $1x10^9$ by McFarland method, it is known as effective density in the literature. And spores added to the mortar with pure water.

NaOH solution prepared with pure water according to its molarity was allowed to cool completely, then NaOH and Na_4O_4Si were mixed at 1: 2 ratio at room temperature.

Geopolymer Mortar: The specific gravity of metakaolin used in the geopolymer mortar is 2.52 g / cm^3 and has high pozzolanic activity. The chemical compositions of the metakaolin and slagused are shown in Table 1.

Table 1. Chemical analysis of metakaolin and slag (%)

	Metakaolin	Slag		Metakaolin	Slag
SiO_2	56,10	40.55	MgO	0,16	5,87

Al_2O_3	40,23	12,83	K_2O	0,51	0,68
Fe_2O_3	0,85	1,10	Na_2O	0,24	0,79
TiO_2	0,55	0,75	B_2O_3	-	-
CaO	0,19	35,58	Glow effect	1,10	0,03

Other materials used in mortar are CEN-Standard Sand (EN 196-1) as aggregate, NaOH (%99 purity, prepared in various molarities by dissolving with water) and Na_2SiO_3 (ρ:1,35 g/cm^3 at the 20°C) as activator and 6 mm microsynthetic fiber (specific gravity: 0,90) to prevent cracks.

Mix Design: As the first step of this study, the 1st series of geopolymer mortars were cured in 3 different medium conditions in order to gain their early strength. These are room temperature curing, oven curing, and hot water curing. At the same time, 3 different molarity NaOH activators were used in these mixtures (Table 2).

Table 2. 1st series samples and cure medium for early strength

Sample no	1.Cure type	Cure temp.($^\circ$c)	NaOH molarity
G8-D	Dry	60	8 M
G12-D	Dry	60	12 M
G16-D	Dry	60	16 M
G8-W	Watery	60	8 M
G12-W	Watery	60	12 M
G16-W	Watery	60	16 M
G8-M	Liquit medium	30	8 M
G12-M	Liquit medium	30	12 M
G16-M	Liquit medium	30	16 M

The aim of this part of the study is to determine the appropriate cure type, in which the geopolymer mortar completes its hydration as quickly and with as high strength as possible. Because hydration time is important for the survival of the bacteria. In addition, pH is a factor that directly affects the viability of bacteria. Thus, the compressive strengths of the geopolymer mortars on the 3rd, 7th, 28th and 60th days were determined. Accordingly, 2nd series of samples were prepared by oven curing and 12 moles/L NaOH solution which provided the highest compressive strength. The purpose of these series is to see the effect of bacteria on the amount of gap of geopolymer mortar samples, which are in different medium conditions. The samples of the second series were cured in pure water, Liquit medium, bacteria containing liquid culture medium and dry medium (no intervention), at 30 degrees up to 60th day for healing (Table 3). And optimal cure medium for self healing were evaluated according to the water absorption amount of the mortars.

Table 3. 2nd series samples and cure medium for healing

Sample no	Conte nt	Naoh molarty	Cure medium
K1	Control	12 M	Culture
K2	Control	12 M	Liquit medium
K3	Control	12 M	Anythink
K4	Control	12 M	Pure water
1_1	Only wıth liquit medium	12 M	Culture
1_2	Only wıth liquit medium	12 M	Liquit medium
1_3	Only wıth liquit medium	12 M	Pure water
1_4	Only wıth liquit medium	12 M	Anythink
2_1	Bacteria culture added	12 M	Culture
2_2	Bacteria culture added	12 M	Liquit medium
2_3	Bacteria culture added	12 M	Pure water
2_4	Bacteria culture added	12 M	Anythink

Curing

The 1st series of samples were placed in 40x40x160 mm molds for flexural test and 50x50x50 mm molds for compressive test. They were kept covered in the mold for 24 hours at room temperature. Afterward, the samples were kept for 48 hours at 60 ° C in an oven, 60 ° C in hot water and 30 ° C at ambient temperature.

The second series of samples were poured into the molds. The molds were kept covered inside the room for 24 hours at room temperature. Samples were then removed from the molds and incubated in an oven at 60 ° C for 48 hours. All the samples were stored at room temperature for 7 days without losing moisture and were exposed to the medium shown in Table 5 from 7^{th} day until the 60^{th} day.

Test Procedure

In this study, EN 13057, EN 1015-3, EN 12504-4, EN 12390-5 and EN 12390-3/AC standards were used for capillary water absorption determination, spreading detect, UPV, flexure- and compressive-strength, respectively.

3. RESULTS AND DISCUSSION

The temperature and the pH of the geopolymer mortar are important for the maintenance of the bacterial life cycle. During the production of geopolymer mortars, the temperature can rise to very high levels in a very short time. It has also been reported that the workability of geopolymer concretes largely depends on the activator molarity, water content and the use of superplasticizer additives [25]. As a result of the tests, it was determined that the most important parameter affecting this was NaOH solution. The results are shown in Table 4 and Fig 1.

Table 4. Reaction temperatures, pH, spread and setting time.

	8 M	12 M	16 M
NaOH t=0 temp. ($^\circ$C)	73	90,7	98
NaOH+Na_4O_4Si mixed t=0 temp. ($^\circ$C)	35	45	5

				5
Geopolimer mortar temp. (○C)		29	31,6	34,2
Geopolimer mortar pH		12,2	12,5	13
Spread (cm)		20,6	18,5	16,5
Setting Time		8 M	12 M	16 M
Control Sample	Start (min)	65-130	55-100	40-60
Control Sample	Finish (min)	500-620	450-550	380-470
Mortar sample with bacteria	Start (min)	75-140	60-110	55-80
Mortar sample with bacteria	Finish (min)	600-720	550-650	450-580

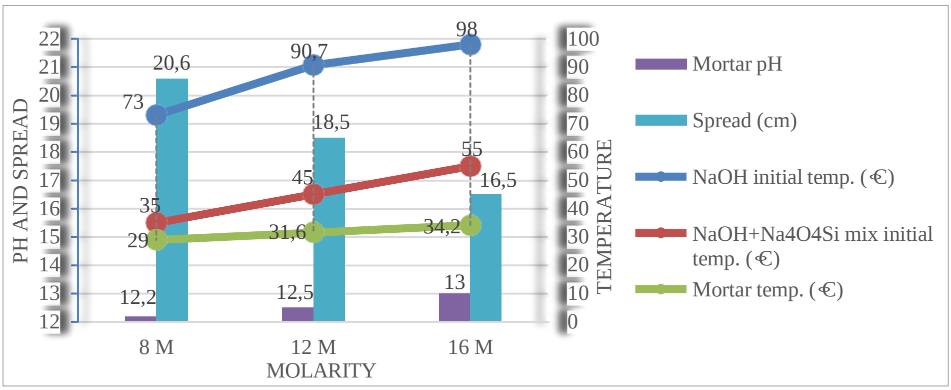

Figure 2. Temperature, spread and pH changes associated with molarity

As shown in Fig 2, the temperature of the solution increased in direct proportion to the concentration of NaOH in the solution. Similarly, when NaOH and Na_4O_4Si are mixed, the reaction temperature increased linearly with molarity. When the mixture which is cooled to room temperature is added to the mortar, temperature of the mortar increased due to the reaction, but this increase was limited to 5 $^{\circ}C$. The impact of NaOH concentration on the workability of the mortar was in the opposite way. As the amount of NaOH in the solution increased, the concentration of the solution increased. Accordingly, the spread of the mortar was reduced by approximately 20% per 4 moles.

The compressive strength of the 1st series of geopolymer mortars is given in Table 5 and Fig 3.

Table 5. Compressive strength according to curing type of 1st series geopolymer mortars

				COMPRESSIVE STRENGHT (MPa)				
Sample no	Cure type	Cure temp.	NaOH molarity	3 days	7 days	28 days	45 days	60 days
G8-D	Dry	60	8 M	48.4	57.8	59.14	46.21	32.36
G12-D	Dry	60	12 M	68.75	82.52	84.25	54.87	44.24
G16-D	Dry	60	16 M	65.25	79.16	80.67	51.25	42.78
G8-W	Watery	60	8 M	43.7	51.26	53.01	41.55	29.71
G12-W	Watery	60	12 M	63.1	75.31	79.48	48.47	41.3
G16-W	Watery	60	16 M	58.62	69.47	71.26	46.68	38.92
G8-M	Liquit medium	30	8 M	22.44	25.11	29.94	30.06	32.88
G12-M	Liquit	3	12 M	26.4	30.0	39.73	41.14	43.06

	medium	0			4			
G16-M	Liquit medium	30	16 M	25.92	29.96	36.5	39.9	41.08

Fig 3 shows that the highest compressive strength occurred in mortars containing 12 moles of NaOH. When the NaOH concentration was reduced to 8 moles the late compressive strength decreased by almost 20%. When it was increased to 16 moles it decreased by almost 5%. In addition, the oven curing provided about 10% higher compressive strength than hot water curing. When looking at the late strengths, it was seen that the samples cured in the laboratory and those with low early strength reached the hot cured samples. This was thought to be caused by sudden and rapid hydration of hot cured mortars.

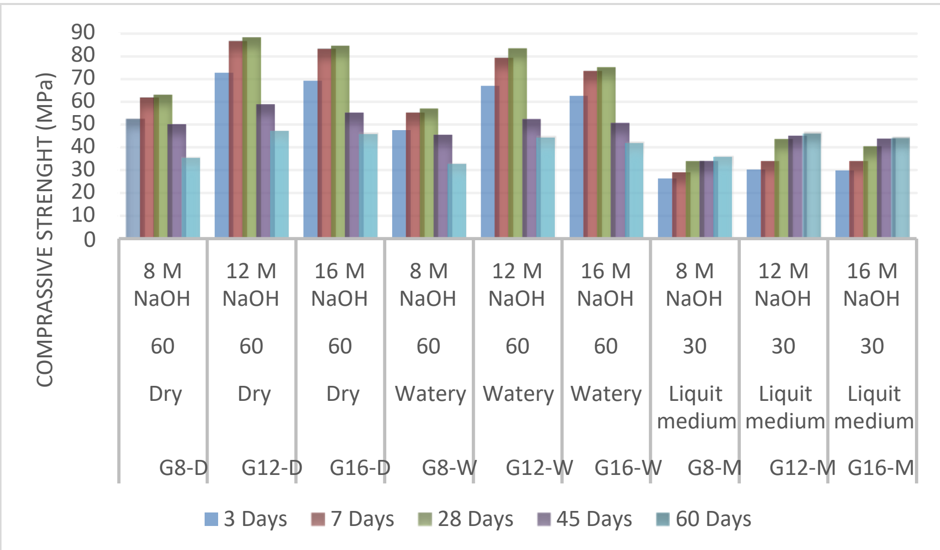

Figure 3. Compressive strenght according to curing type of 1st series geopolymer mortars

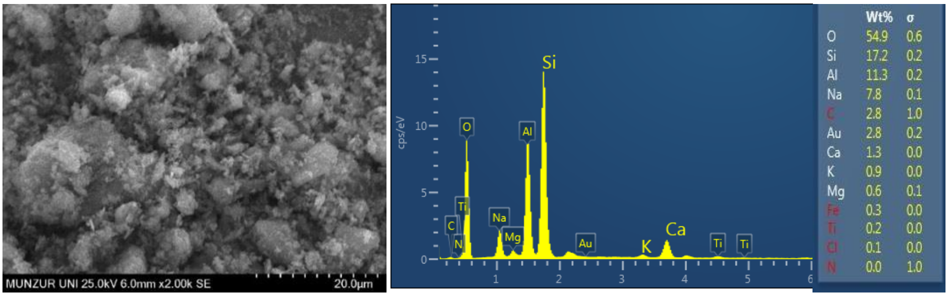

Figure 4. 7th day SEM image and EDS analysis of G8-D series

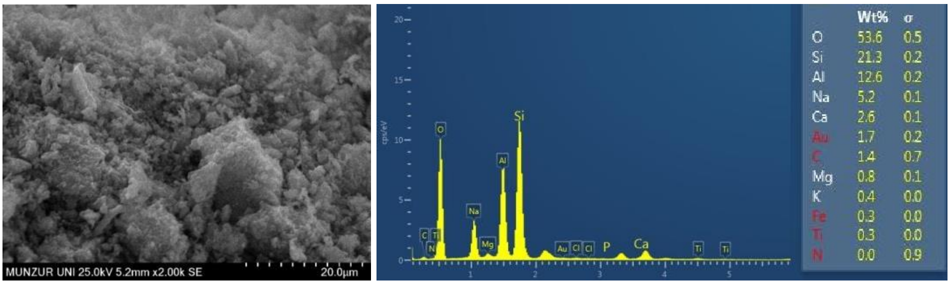

Figure 5. 7th day SEM image and EDS analysis of G12-D series

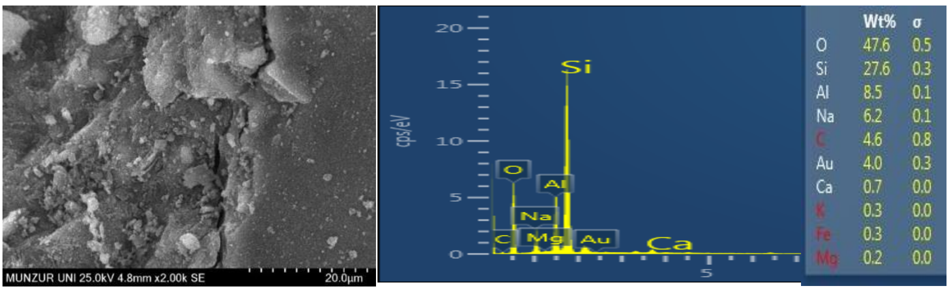

Figure 6. 7th day SEM image and EDS analysis of G16-D series

According to the results of SEM and EDS analysis, as the pH increased, the bond strength of the mortar increased and more stable $CaCO_3$ structures were formed (Fig 4, 5 and 6). Zhang et al. [26] stated that depending on Si / Na ratio in geopolymer mortars, it can form nanosized crystals of crystalline zeolite or another zeolite. This showed that the presence of bacteria does not change the pH-compressive strength relationship in the geopolymer mortar.

The compressive strength results of this series are given in Fig 7 and

Table 6.

Table 6. Compressive strenght of 2nd series geopolymer mortars

Series	7. Days compressive strenght (MPa)	60. Days compressive strenght (MPa)	Spread (cm)	Unıt volume mass ın fresh mortar (g/cm^3)
K1-K	79.1	46.60	15	1780
K2-BY	80.2	48.45	15	1780
K3-SS	79.6	45.86	16	1780
K4-B	78	48.20	16	1780
1_1-K	68.18	43.13	16	1720
1_2-BY	71.70	45.25	16	1720
1_3-SS	69.88	44.37	16	1720
1_4-B	69.57	43.92	16	1720
2_1-K	80.92	49.58	16	1720
2_2-BY	80.44	51.04	16	1720
2_3-SS	77.92	44.48	16	1720
2_4-B	78.35	46.66	16	1720

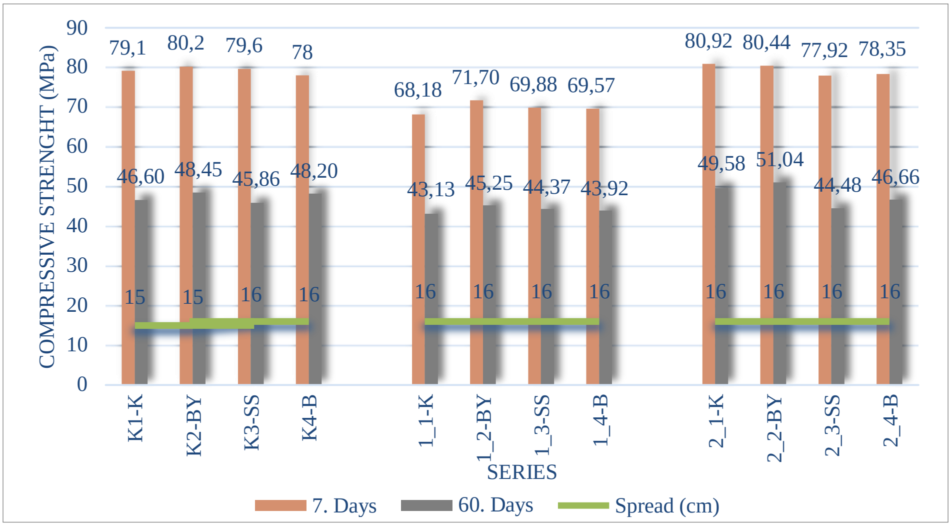

Figure 7. Compressive strength and spread values of 2nd series geopolymer mortars

The compressive strength of geopolymer mortars reached very high values 7 days after curing in a hot furnace environment and started to decrease after 28th day, which is the opposite of the expected strength gain trend. This is due to the rapid hydration, increase in void volume and amount, and sudden hardening of the structure. It is also thought that bacteria, which lost their vitality after the completion of the cure, fell as in conventional concrete. It eventually reached its final value on the 90th day.

In many studies in the literature, it has been stated that the improvement of conventional concrete with bacteria increases the compressive strength [19], [27], [20]. In the geopolymer mortars in this study, there is a slight increase in the compressive strength of the samples 2 produced by adding bacteria.

In addition, Andalib et al. showed that Geopolymer bacterial concrete had the least weight and strength losses than ordinary bacterial concrete at different ages [28].

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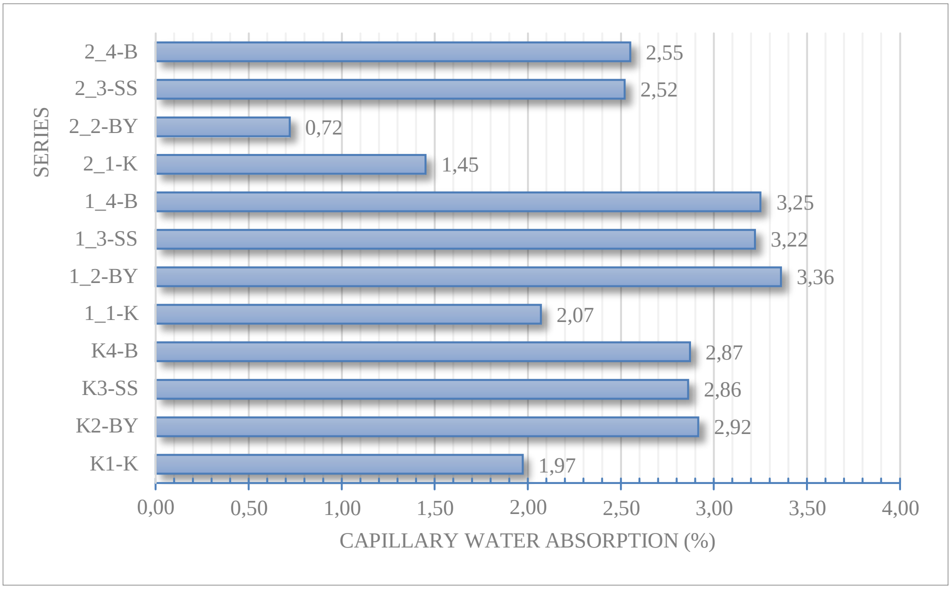

Figure 8. Capillary water absorption percentage of 2nd series geopolymer mortars

Fig 8 shows that the lowest capillary water absorbing mortar is produced by adding bacteria and heals on the medium. The highest capillary water-absorbing mortar is produced by adding only liquit medium and heals the liquit medium. These results and previous studies coincide that they investigated the effect of NaOH concentration on geopolymer concrete compressive- and flexural-strength as well as water absorption rate [29].

4. CONCLUSIONS

In this study investigated the basic conditions for self-repair of cracks in geopolymer mortars and techniques for preparing geopolymer mortars. For this purpose, the effect of curing conditions on geopolymer mortars, the most suitable methods for adding bacterial agent to the mortar without encapsulation, and its effect on the compressive strength of the mortar were examined. The results are as follows:

- As the NaOH concentration increases, the temperature of mortar rises rapidly. Bacteria are not able to survive at very fast rising temperatures. The activator solutions should therefore be added to the mortar after cooling.

- The NaOH solution and bacterial culture should not be mixed directly.
- Besides, the presence of bacteria prolongs the setting time of the geopolymer mortar.
- The highest compressive strength of the mortars formed with 8, 12 and 16 moles NaOH concentrations was obtained by using 12 moles NaOH solutions.
- As the amount of NaOH in the mortar increases, the density of the solution increases, and the setting time, gap ratio and processability decreases. The spread value in the mortar increases by approximately 20% with every 4 moles / l NaOH increase. The increase in the amount of space is required for the bacteria to meet the living space and oxygen requirements. However, this reduces the mechanical strength of the mortar.

- It is the curing type at 60ºC which provides the highest and early compressive strength values. However, if the bacteria will be added to the mortar in all 3 curing medium, it should be put into sport form and nutritional supplement should be made.
- In this study, it was determined that the most suitable method for geopolymer mortar in bacterial healing was the dry curing technique at 60°C with the highest early resistance.
- The results of the tests showed that all the mortars prepared with the liquit medium were 10% lower than the early and late strength control samples. In addition, the water absorption values of this series are higher than those produced by bacterial culture. Mortars that were put into bacterial culture and cured in liquit medium were found to have a 5% higher late strength. The late strengths of the samples which contained bacterial culture and were cured in pure water or without interference were again 5% lower than the control sample.
- The compressive strength of the bacterial mortars that are healed in the liquit medium is higher than the samples using other curing methods. It was observed that the bacteria in the mortar contributed to the strength by filling the cracks even if they were superficial. This was evidenced by the capillary water absorption values of the samples.
- According to the results of the tests, it was found that the samples in which the bacteria were added to the mortar by suitable methods had

fewer cavities than the samples produced without bacteria. If bacteria were present in the mortar, curing with bacterial culture had a negative effect on capillary water absorption values.

- Compressive strength of geopolymer mortars reached very high 7 days after curing in hot furnace environment and started to decrease after 28th day and fixed on 90th day. This is due to rapid hydration, an increase in void volume and amount, and sudden hardening of the structure. It is also thought that bacteria, which lost their vitality after the completion of the cycle, fell as in conventional concrete.
- According to this study, mixing the bacteria planted on the solid agar surface with pure water into the geopolymer mortar is an easy and effective method for self healing. This method is a first in the literature to heal geopolymer mortars. Using this method, the healing of geopolymer mortar cracks can be investigated.

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NOMENCLATURE

CO_2 : Carbon dioxide
K : Potassium
Ca : Calcium
Si : Silica
Al : Aluminum
$CaCO_3$: Calcium carbonate
NaCl : Sodium chloride
NaOH : Sodium hydroxide
Na_2SiO_3 : Sodium silicate

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