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EFFECTS OF ACID AND HIGH-TEMPERATURE TREATMENTS ON DURABILITY OF BACTERIAL CONCRETE

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Abstract: Bacterial concrete specimens were produced in this study to investigate the effects of microbially induced calcium carbonate precipitation (MICP) mechanism on concrete durability. Bacterial concrete (BC) samples were produced through supplementation of *Bacillus megaterium* bacteria into concrete mixture and curing water. However, control concrete (CC) samples were produced without bacteria. BC and CC were exposed to acid (HCl) and high temperature (400°C) treatments. In the first phase of the study, 100×100×100 mm cube specimens were immersed into HCl solution for 10 days and compressive strengths and weight losses were determined. Compressive strength of acid-treated samples was measured as 25.08 MPa for BC samples and as 17.90 MPa for CC samples. Such values revealed that BC samples yielded 40.11% greater compressive strength. When CC samples lost 10.99% weight due to acid attack, BC samples lost 8.74% weight. In the second phase of the study, concrete specimens were exposed to 400°C temperature and compressive strength of heat-treated samples was determined. As the result of high temperature, bacterial samples yielded 13.76% greater compressive strength against high temperature. Present findings revealed that $CaCO_3$ formation on concrete improved concrete durability against attacks and high temperatures.

Keywords: Bacterial Concrete, Bacillus Megaterium, MICP, Durability, Acid Attack, High Temperature.

Bakterili Betonun Asit ve Yüksek Sıcaklık Etkisine Karşı Dayanımı

Öz: Bu çalışmada yenilikçi bir yöntem olan bakteriler kullanılarak beton içerisinde kalsit oluşumu sağlanmıştır. Bakterilerin beton içerisinde ürettiği ürünün betonun asit ve yüksek sıcaklık dayanımına etkisi incelenmiştir. Bacillus megaterium türü bakteri betonun karışım suyuna ve kür suyuna katılarak bakterili beton numuneleri (BC) ile bakteri kullanılmadan bakterisiz kontrol numuneleri (CC) üretilmiştir. 100×100×100 mm boyutunda küp beton numunelerinin 28 günlük basınç dayanımı belirlendikten sonra HCl asidine 10 gün boyunca maruz bırakılmıştır. Asit etkisi sonucunda bakterili ve bakterisiz kontrol numunelerinin ağırlık kayıpları ile basınç dayanımı kayıpları ölçülmüştür. Aside maruz kalan bakterili numunelerin basınç dayanımı 25,08 MPa, kontrol numunelerinin ise 17,90 MPa olarak belirlenmiştir. Bu fark bakterili numunelerin %40,11 daha yüksek basınç dayanımı sağladığını göstermiştir. Asit etkisi sonucu kontrol numuneleri ağırlıklarının %10,99'unu kaybederken bakterili numuneler sadece %8,74'ünü kaybetmiştir. Çalışmanın ikinci kısmında beton numuneleri 400°C sıcaklığa maruz bırakılarak yüksek sıcaklık sonrası basınç dayanımları belirlenmiştir. Yüksek sıcaklık etkisi sonucunda bakterili betonun basınç dayanımı %13,76 oranında daha yüksek elde edilmiştir.Tüm bu çalışmanın sonuçları incelendiğinde bakterinin ürettiği $CaCO_3$ ürününün betonun boşluk ve yüzeyinde oluşması betonun geçirimsizliğini arttırdığı sonucuna varılmıştır. Bu geçirimsizlik hali betonun sıcaklık ve asit etkisine karşı olan durabilitesini arttırmıştır.

Anahtar Kelimeler: Bakterili Beton, Bacillus Megaterium, MICP, Durabilite, Asit Etkisi, Yüksek Sıcaklık.

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1. INTRODUCTION

Concrete is the most preferred building material worldwide thanks to its high compressive strength, plasticity and availability. (Gartner, 2004). When the earthquake-damaged buildings were assessed, it was observed that besides compressive strength, durability was also prominent characteristics of concrete. Concrete is exposed to several physical and chemical impacts throughout the service life (Baradan and Yazıcı, 2003). Besides technical and economic problems, various environmental problems are also encountered when the reinforced concrete structures were not able to sustain existing state. Serious quantities of CO_2 are released into atmosphere during the cement production process.

Durability problems in concrete-based structures are generally related to mixture water and transition of hazardous substances and gases into microstructure of the concrete. Such a transition occurs through the surface voids and cracks of the concrete (Baradan and Aydın, 2013).

Microbially Induced Calcium Carbonate Precipitation (MICP) methods have been recently studies to improve concrete durability through reducing gas and water permeability of concrete. With this method, more environment-friendly, economic and self-healing concrete production is possible. Microorganisms have been started to be used within microstructure of concrete since they are environment-friendly and economic organisms. With the bacteria supplementations, resultant microbiological classification within the microstructure of concrete may improve mechanical, physical and chemical characteristics of the concrete. Besides, such a process is also largely used to fill up the cracks and voids.

In calcite production mechanism of microorganisms, bacteria reduce urea into ammonia and carbonate through urease enzyme. In this way, existing calcium ions combine with carbonates and turn into calcium carbonate ($CaCO_3$), in other words, to calcite. Resultant $CaCO_3$ quantity depends on bacteria activity and optimum concentration. For instance, the biochemical reaction realized by Bacillus megaterium bacteria are presented in Equations 1 and 2 (Wang et al., 2016).

$$CO(NH_2)_2 + 2H_20 \xrightarrow{\textit{Bacteria urease enzyme}} 2\,NH_4{}^{+} + CO_3{}^{2-} \tag{1}$$

$$Ca^{2+} + CO_3{}^{-2} \rightarrow\ CaCO_3 \tag{2}$$

It was reported that bacteria could fill up the voids of concrete and improved compressive strength and mechanical characteristics of the concrete (Andalib et al., 2016; Krishnapriya et al., 2015). Acid-containing hazardous chemicals and high temperatures are among the most significant physical and chemical impacts to which concrete is exposed throughout the service life. Therefore, besides improved compressive strength, bacteria also have significant effects on durability. Previous studies revealed that bacterial concrete was more resistant against some strong acid types than the normal concrete (Reddy et al, 2012; Andalib et al., 2014). Bacteria fill up the concrete voids and cracks and thus reduce porosity (Achal et al., 2013; Meera and Subha, 2016). This study was conducted to investigate possible contributions of Bacillus megaterium bacteria to concrete resistance against acid and high temperature through filling concrete void spaces with $CaCO_3$.

2. MATERIAL AND METHOD

2.1. Materials

2.1.1. Bacteria

Bacillus megaterium were used as a biological additive in concrete mixtures. The bacteria were supplied from the Culture Collection of Refik Saydam Hygiene Institute (Ankara, Turkey).

2.1.2. Bacteria Activation and Nutrient Media

Different nutrient media were used for activation and storage of Bacillus megaterium strains. Nutrient Agar, TSB Agar media were used for bacteria activation. Nutrient agar, Nutrient broth (CM0001, OXOID), Trytic Soy agar (105458, MERCK), Urea (U5378, SIGMA), $CaCI_2$ (102378, Merck) chemicals were used for curing media of bacterial concrete samples. Curing water composition of bacterial concrete samples is provided in Table 1.

Table 1. Curing water composition of concrete samples.

Additives	Quantity
Nutrient broth	3 g/L
$CaCl_2$	2.8 g/L
Urea	20 g/L
Bacillus Megaterium	30×10^5 cell/ml

2.1.3. Cement

CEM I 42.5R type Portland cement was used in present experiments. Chemical composition of cement is provided in Table 2.

Table 2. Chemical composition of cement (% by weight).

SiO_2	CaO	Al_2O_3	MgO	Fe_2O_3	K_2O	Na_2O	SO_3	Loss of Ignition
20.12	62.38	5.88	1.87	2.40	0.93	0.38	3.28	1.82

2.1.4. Aggregate

Aggregate gradation with 16 mm maximum grain size designed based on TS 706 EN 12620 is presented in Figure 1. Specific gravity of coarse aggregates was 2.75 and specific gravity of fine aggregates was 2.65.

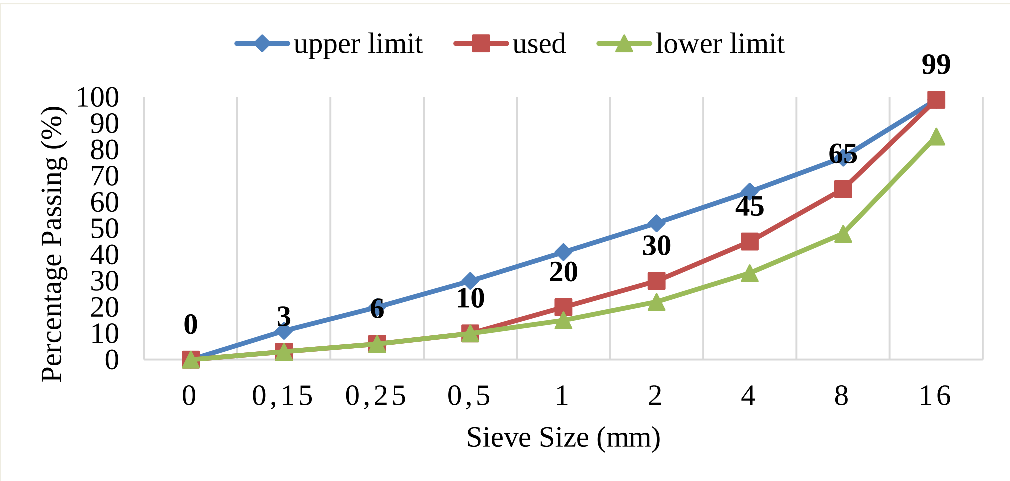

Figure 1:
Aggregate gradation curve.

2.1.5. Mixing Water

Municipal water supply was used in control concretes (CC). On the other hand, in bacterial concrete (BC) samples, *Bacillus megaturium* bacteria were added (30×10^5 cell/ml) into mixture water.

2.1.6. Acid

Hydrochloric acid (HCl) was used for acid treatments of the concrete samples.

2.2. Experiments

2.2.1. Preparation of Concrete Mixtures

Concrete production was performed in accordance with TS 802 standards. Concrete cube specimens (100x100x100 mm) were produced for acid and high temperature experiments. Based on these dimensions, maximum aggregate diameter (D_{max}) was selected as 16 mm. Water/cement ratio was selected as 0.45. Concrete mixture water was supplemented with *Bacillus megaterium* bacteria at a ratio of 30×10^5 cell/ml. Cement, coarse and fine aggregate were dry-mixed, then bacteria supplemented mixture water was used to produce concrete mixture. Materials used in concrete mixture are provided in Table 3**.**

Table 3. Concrete mixture materials for 1 m^3

Materials	Quantity	Volume (m^3)
Water (30×10^5 cell/ml bacteria-supplemented)	195 kg	0.195
Cement	433.34 kg	0.138
Air	%2.5	0.025
8-16 mm aggregate	618.3 kg	0.225
4-8 mm aggregate	353.33 kg	0.128
0-4 mm aggregate	766.02kg	0.289
Total	2367 kg	1 m^3

Bacterial concrete (BC) specimens were cured in nutrient broth-supplemented water (Table 1). Mixture water of control concrete (CC) specimens did not contain bacteria and also its curing water did not contain bacteria.

2.2.2. Acid Attack Tests

Concrete is exposed to various chemicals throughout the service life. Hydrochloric acid (HCl) is among the most significant ones. Since HCl is among the mostly encountered chemicals in industrial waters, concrete should have high durability against HCl which is also among the strongest acids.

Bacteria supplementations reduce concrete porosity and thus prevent acid transition into microstructure of the concrete and increase durability accordingly. Bacterial concrete (BC) and control concrete (CC) samples were produced to observe such behaviors.

For each experiment, 3 samples of 28-day cured BC and CC were used. Acid effect test was conducted in two phases (weight loss and compressive strength loss). Initially, pre-weighed oven-dried BC and CC specimens were immersed into 6% HCl solution for 10 days. Then, samples were kept in tap water for 1 day and dried in an oven at 105 °C for 24 hours. These dry samples were weighed and its lost weights were determined. In the second phase, compressive strength of acid-exposed specimens (for 10 days) were measured to investigate the effects of bacteria supplementations on acid resistance of concrete. The compressive strength test was achieved according to TS EN 12390-3.

2.2.3. High-Temperature Tests

Throughout the experiments, “Eurocode 4- Part 1-2: Structural fire design” standards were taken into consideration.

Again, for each experiment, 3 samples of 28-day cured BC and CC were used. Concrete specimens were exposed to high temperature (400 °C) in a muffle furnace for 150 minutes. The samples were kept in the furnace to cool down to room temperature. Cooled specimens were then broken in compressive strength press to get compressive strength. Thus, the effect of high temperature on the compressive strength of bacterial and control concrete was observed. The compressive strength test part of experiment was made according to TS EN 12390-3. A high-heat treated concrete specimen in compressive strength test is presented in Figure 2.

Figure 2 :
Breaking a high-heat treated concrete specimen in concrete press.

3. RESULTS AND DISCUSSION

3.1. Results of Acid Effect Tests

3.1.1. Weight Loss

Physical destructions and spilths were observed in 10-day acid-treated concrete specimens. The acid-induced physical destructions on concrete specimens are presented in Figure 3. Weight loss was measured through weighing acid-treated and untreated samples. Average weights of the specimens before and after acid treatments are provided in Table 4. About 10.99% weight loss was observed in control samples. On the other hand, 8.74% weight loss was observed in bacterial concrete samples. Such values indicated that BC samples were 20.47% more resistant against acid effects than the CC samples.

Table 4. Change in weight of concrete samples before and after acid treatments

Samples	Average weight (g)		Weight loss (g)	Weight loss (%)
	Before acid treatment	After acid treatment		
CC (without bacteria)	2299.37	2046.54	252.83	10.99
BC (bacterial)	2335.87	2131.84	204.03	8.74

Although the only variation between BC and CC is bacteria, the difference in weight loss is remarkable. This difference showed that bacteria are active in concrete and shows the effect of bacterial product. The formation of bacterial product calcium carbonate ($CaCO_3$) on the BC sample surface was visible to the naked eye before acid treatment and showed its effect at the end of the experiment. $CaCO_3$ formation resulted in more solid and impervious layer over the concrete surface and prevented acid reaction with cement mortar and acid-solving of mortar. It was indicated in previous studies conducted on acid-treated bacterial concrete that calcite formation with bacteria supplementations increased concrete durability against type of different acids and reduced weight losses (Andalib et al., 2014).

Figure 3:
A concrete specimen exposed to acid effect.

3.1.2. Compressive Strength Loss

Compressive strength of acid-treated and untreated BC and CC samples were measured to observe the effects of acid on compressive strength of the concrete.

Effects of acid treatments on compressive strength of CC and BC samples are clearly seen in Figure 4. Compressive strength of CC samples decreased from 47.36 MPa to 17.90 MPa with about 62% loss. On the other hand, compressive strength of BC samples decreased from 51.87 MPa to 25.08 MPa with around 52% loss.

The bacterial product ($CaCO_3$) reduces the porosity of concrete and therefore concrete was more resistant to external influences (Achal et al., 2013). Bacteria supplements prevented the acid from penetrating into the microstructure of the concrete and thus preserved the compressive strength of the concrete. Final compressive strength of BC samples was 40.11% greater than the compressive strength of CC samples. The main reason of the difference is that the concrete pore was filled with calcium carbonate by bacteria. In this way, more void-free and durable concrete was obtained. Therefore, BC samples strength were determined to be higher than CC samples. In previous studies, greater compressive strength values were also reported for bacterial concrete samples (Andalib et al., 2014; AL-Ridha et al., 2018; Krishnapriya et al., 2015).

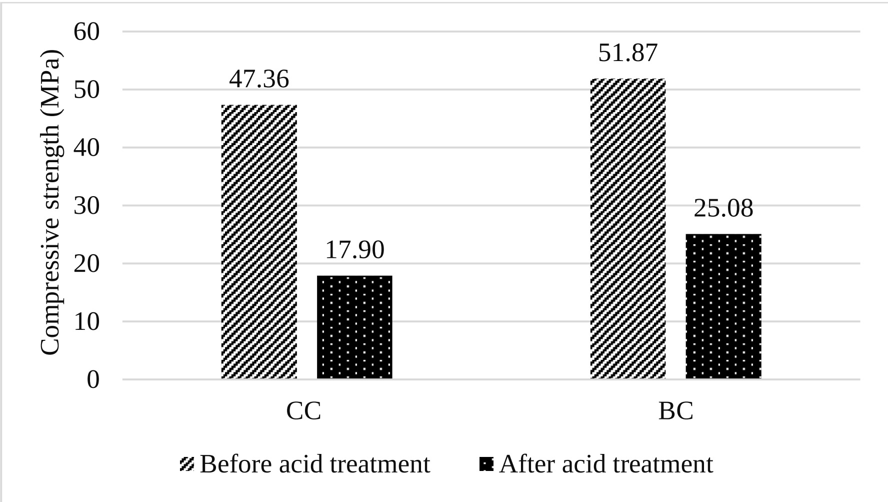

Figure 4:
Compressive strength before and after acid treatments (MPa).

3.2. Results of High Temperature Effect Tests

Initially, 28-day cured CC and BC samples were broken to investigate the effects of bacteria treatments on compressive strength before heat treatments. The 28-day compressive strength of BC samples was obtained 9.52% greater than the compressive strength of CC samples that result was showed in Table 5. This result shows that the compressive strength of concrete increases with the addition of bacteria without any external factors. In addition to this, it is also important to maintain this strength under high temperature effect.

Concrete specimens were exposed to high temperature (400 °C) in a muffle furnace, cooled down to room temperature and broken with the use of compressive strength press. Significant decreases were observed in compressive strength of heat-treatment CC and BC samples. Compressive strength of CC samples decreased from 47.36 MPa to 33.72 MPa with 28.8% reduction and compressive strength of BC samples decreased from 51.87 MPa to 38.36 MPa with 26.04% reduction. BC samples were able to better preserve compressive strengths than the CC samples. The reason of this conservation is the high temperature resistance of $CaCO_3$.

Microbially Induced Calcium Carbonate Precipitation (MICP) yielded about 13.76% greater resistance against high temperatures that result was showed in Table 5. Resultant $CaCO_3$ structure is destructed at quite higher temperatures than the concrete reinforcement and majority of concrete constituents. Addition of bacteria supplementation was caused reduction porosity thanks to calcite precipitation which increase the high temperature resistance of concrete. In previous studies, it was observed that bacteria positively affect the porosity-related properties of concrete such as water absorption and rapid chloride permeability. (Chahal et al., 2012; Siddique et al., 2016).

The main result of the high temperature test, $CaCO_3$ which is the product of the bacterial additive added to the concrete was formed in the pores and surface of the concrete. Thus it made the concrete more filled. In addition, thanks to the temperature resistance of this product and compactness of bacterial concrete, it has become more resistant to high temperatures.

Table 5. Effect of high temperature on compressive strength of 28-day CC and BC samples.

Sample status	Sample name	Compressive strength (MPa)	Compressive strength difference (%)
Not exposed to high temperature	CC	47.36	--
	BC	51.87	+9.52
Exposed to high temperature	CC	33.72	--
	BC	38.36	+13.76

Aggregate color of broken samples could be used to assess if the threshold temperature values, at which concrete destructions are encountered, were reached. For instance, pink or red color indicates temperatures of between 300°C and 600°C, grey color indicates temperatures of between 600°C and 900°C, yellowish beige color indicates temperatures of between 900°C and 1000°C (Kızılkanat and Yüzer, 2008). Figure 5 presents the images of heat-treated and untreated samples broken in compressive strength press. As can be seen in Figure 5, aggregate color of heat-treated samples turned into pink.

Figure 5:
Color difference between heat-treated (left) and untreated (right) samples

4. CONCLUSIONS

Bacterial concrete specimens were produced in this study to investigate the effects of MICP mechanism on concrete durability. Specimens were exposed to HCl acid and 400°C temperature.

Acid attack tests revealed that compressive strength values of CC samples decreased from 47.36 MPa to 17.90 MPa with about 62% reduction and compressive strength of BC samples decreased from 51.87 MPa to 25.08 MPa with about 52% reduction. At the end of the acid attack tests, BC samples had 40.11% greater compressive strength value than the CC samples (25.08 MPa vs 17.90 MPa). $CaCO_3$ formation prevented acid penetration into microstructure of the concrete and thus better preserved compressive strength values. Besides prevention of penetration into microstructure, formation over the concrete surface also reduced acid-induced weight losses. Following 10-day acid exposure, CC samples had a weight loss of 10.99%. On the other hand, such a value was 8.74% in BC samples. Microbial calcite formation improved concrete durability through preventing acid-induced weight losses.

High temperature effect tests revealed that compressive strength of 28-day heat-treated samples was 33.72 MPa for CC samples and 38.36 MPa for BC samples. BC samples had 13.76% greater compressive strength than CC samples. As compared to initial compressive strength values, there was about 29% reduction in CC samples and 26% in BC samples. Bacteria supplementation improved concrete resistance against high temperatures. Bacteria-induced $CaCO_3$ formation exhibited greater resistance against high temperatures and were able to hold the concrete together more.

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