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STRENGTH OF SILLE STONE AGAINST ACID RAIN

Gizem BOLUK[1], Yasin Ramazan EKER[2],
Şerife Yurdagül KUMCU[3], Yusuf UZUN[4]*

[1]Necmettin Erbakan University, Engineering Faculty, Konya, Türkiye, boluk823@gmail.com
[2]Necmettin Erbakan University, Engineering Faculty, Konya, Türkiye, yeker@erbakan.edu.tr
[3]Necmettin Erbakan University, Seydişehir Ahmet Cengiz Engineering Faculty, Konya, Türkiye, yurdagulkumcu@gmail.com
[4]Necmettin Erbakan University, Seydişehir Ahmet Cengiz Engineering Faculty, Konya, Türkiye, yuzun76tr@gmail.com

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Abstract: *Sille stone, which has mine area in the Sille District of Konya, is a mined structural mineral stone which has been used since ancient times. Structures normally react with acid rain in the nature and acids formed for various reasons. Building materials generally lose their physical and chemical properties because of their reaction with acids. The aim of this study is to investigate the resistance of historical structures, which have been built with Sille stone by various cultures and civilizations, by acid rain. The pH value of rainwater, between 5.6 and 6, shows acidic characteristics. It has been also shown that the pH value of acid rain can be dropped below 5- 3 in some cases. In this study, first, the chemical and physical structure of Sille stone was determined. The strength of Sille stone against atmospheric conditions, in the aquatic environment, citric acid, sulfuric acid, nitric acid and their mixtures is tested to see the effect of acid rain. For this purpose, samples were taken from the stone quarries in Sille District, and they were cut as 7x7x7 cm3 dimensions depending on the TS 1926 Turkish Standards description for strength tests. All samples were put into the acidic mixtures. 1 molar concentration has been prepared for each acidic mixture. Test duration was kept as 3 and 6 months. ResuIts were evaluated in each 3 months. It was seen that, the sample taken from the mine gained strength at first, however, there were decreases in strength with time.*
Keywords: *Sille stone, acid rains, nitric acid, nitric acid, Sille stone mining, mining and process*

1. Introduction

The structures where human activities are carried out and that we are in always react with increasing acid rains and acids originating from different reasons. Building materials lose their chemical and physical properties because of the reactions they enter with acids. Tecer examined the effects of SO_2 and NO_x formed due to environmental pollution on carbonate rocks. [1] investigated the gloss loss rates caused by natural stones such as marble, travertine and granite at the end of a five-month period. There are many external effects on building materials in the atmospheric environment, and because of these effects, corrosion, brittleness, fragility, darkening, contamination, whitening, bleaching, fragmentation, dispersion, breakage, shedding and loss of protective effect occur. This study was carried out in two ways. To directly affect the first, the samples were placed outdoors with their shiny surfaces facing up, while the second samples were kept in a covered environment. Regular monthly brightness measurements were made at the corner points of the 5*5*5 cm^3 samples. The loss of brightness was greater in the marble and travertine that remained in the rainy area compared to the closed environment [1]. In 2005, [2] and his colleagues examined the effects of acid rain on the 1056-hectare El Tajinin archaeological site in Veracruz, Mexico. As a result of the studies, it was determined that 85% of the rainfall in this region was acidic and the limestone building material in this region was dissolved by acid rain and as a result, it eroded over time and lost its shape and resistance [2]. [3] conducted studies in three different regions in the northeastern United States in 1984. In his study, it was observed that the more rain fell on the stone, the more dissolution on the stone surface increased proportionally [3]. [4] investigated the effects of air pollution on historical buildings in the historical city center of Gaziantep in 2018. It was determined that the cause of all air pollution in the province was

* Corresponding author: Şerife Yurdagül Kumcu, Prof. Ph.D, Necmettin Erbakan University, Seydişehir Ahmet Cengiz Engineering Faculty, Konya, Türkiye, yurdagulkumcu@gmail.com

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heating. The study consists of two stages. In the first stage, deterioration in the ceiling due to air pollution was found. In the second stage, the factors affecting the samples taken were examined. In this study, the effects of SO_x and NO_x and particles caused by weather conditions were evaluated and their damages were determined. As a result of the study, air pollution was seen in the form of color change, salt accumulation, crust formation and separation on building facades [4]. Dal examined the causes of deterioration in carbonate stones used in traditional historical structures of the Thrace region in detail in 2010. The study showed that the amount of HCO_3 in rainwater causes calcite separation in the materials [5].

This study aims to examine the behavior of Sille Stone, which is used as a carrier, exterior cladding and restoration material, against acid rain. Sille Stone is mined in Sille district of Konya province in Turkey. In this study, nitric acid, sulfuric acid, carboxylic acid and the mixture obtained from these acids will be used to represent acid rain. Sille Stone samples were cut into 7x7x7 cm^3 sizes to measure the compressive strength according to the TS1926 standard. Samples kept in water and open air were used as reference samples. A total of 36 samples were tested, including reference samples, samples kept in acids and samples kept in the open environment. The samples exposed to acids were analyzed twice with the compressive strength test at the end of the 3rd and 6th months. As a result, acid rain simulation was performed on Sille Stone samples and their mechanical resistance to acids and their changes over time were investigated. It was observed that although the strength increased initially, the compressive strength decreased over time.

2. Sille Stone

Sille stone is produced in volcanic mountains settled in the form of ash-blocks. Although the sille stones extracted from the quarries in Sille are andesite tuff, andesites are rarely found. Andesite stones are less preferred in the market because they are more difficult to process than andesite tuff. Sille stone has not lost its feature of being used as a building stone and covering stone from ancient times to the present day and is still preferred today [6]. This type of stone has been known and used since the time of the Greeks.

Fig.1. Sille Stone mine

3. Acid Rains

After the industrial revolution, there has been an increase in the use of fossil fuels to meet the increasing energy demand due to industrialization, urbanization and increasing technology. Increasing fossil fuel use and many other natural and anthropogenic (human-sourced) sources cause air pollution. Clean air normally contains 78.09% nitrogen, 20.95% oxygen, 0.093% argo, 0.03% carbon dioxide, almost no smoke, dust particles, sulfur and nitrogenous gases [7]. The cleaning of natural or artificial pollutants from the air is called auto purification. The cleaning process can be carried or by physical, chemical, biological decomposition. If the amount and speed of pollutants mixed into clean air exceeds the speed and capacity of the air to clean itself (auto purification), clean air begins to become polluted. As a result of this situation, the presence of more pollutants than necessary in clean air creates air pollution [7]. The effects of air pollutant gases can be divided into 3 groups as global, regional and local. Global effects affect the entire earth, regional effects affect certain regions, and local effects affect residential and industrial areas [8].

The mixing of toxic gases into the natural water cycle causes acid rain. Sulfur dioxide and nitrogen dioxide, which are defined as primary pollutants, are acid gases that cause acid rain. When primary pollutants join the water cycle and combine with water vapor, they turn into secondary pollutants such as sulfuric and nitric acids because of chemical reactions.

$SO_2 + H_2O\ H_2SO_4$ (Sulfuric Acid) (1)

$CO_2 + H_2O\ H_2CO_3$ (Carbonic Acid) (2)

$NO_2 + H_2O\ HNO_3$ (Nitric Acid) (3)

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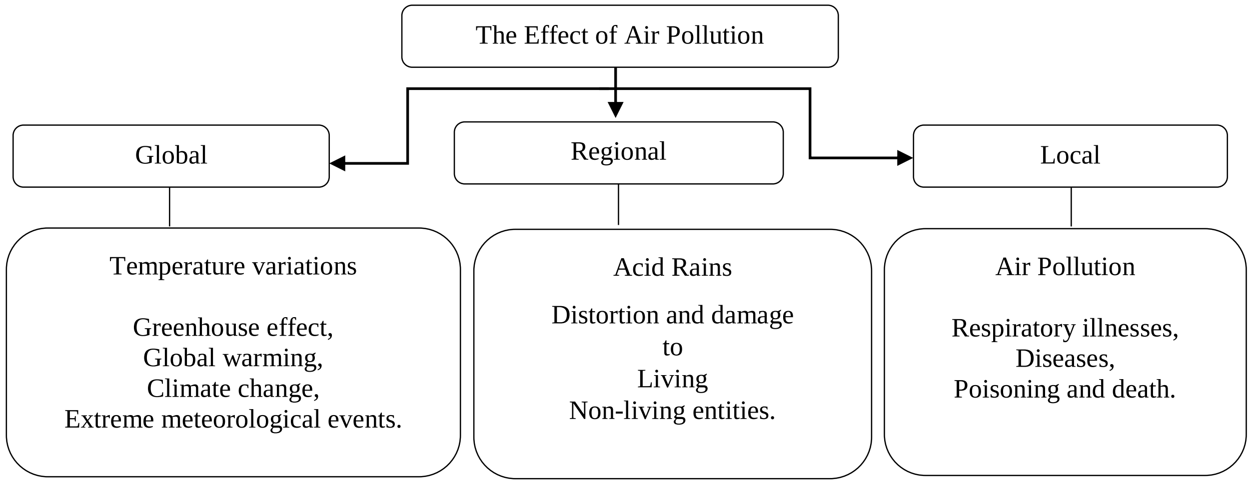

Fig.2. Global, regional and local effects and consequences of air pollution

As a result of the reactions, sulfuric acid, nitric acid and carbonic acid are formed. These acids are called secondary pollutants. Secondary pollutants rain down on regions hundreds and thousands of kilometers away from where they are released into the atmosphere as dry particles or rain, snow, frost, fog and dew. Secondary pollutants rain down on regions hundreds and thousands of kilometers away from where they are released into the atmosphere as dry particles or rain, snow, frost, fog and dew. Secondary gases are harmful not only to the region where they are formed by reacting, but to the entire world. Acids make dry storage and wet storage on the earth [9]. Wind is an important factor in dry storage and carries acidic gases and particles. Gaseous emissions can accumulate as dry storage 30 km away from the source. Therefore, it is defined as short-distance storage. After dry deposition, precipitation can increase acidity. It is assumed that approximately 50% of the acidity accumulated in the atmosphere is dry deposition [10]. Wet storage falls to the earth with rain, snow and avalanches. In wet storage, it occurs because of the reaction of dilute acids with water vapor. Half of the storage in the atmosphere is known as dry storage [10, 11, 12]. In this study, the effects of acid rain, which is a regional effect of air pollutants, on Sille stone, which is used in many historical monuments and various construction stages of buildings in Konya and has a reserve in Sille district, were examined. In this study, the behavior of Sille stone in secondary pollutants such as nitric acid, sulfuric acid, citric acid and their mixtures was observed.

3. Material and Method

Rapid increase in industrialization, have brought by advancing technology, air pollution, which has become one of the important environmental problems of our day, causes acid rain. In this study, Sille stone, which has a reserve in Sille District of Konya Province and is a geological heritage, was examined the physically and chemically. Acid rain environments were tried to simulate by various acidic environments. Nitric acid (HNO_3), sulfuric acid (H_2SO_4), which are common in acid rains, and citric acid ($C_6H_8O_7$) were used to simulate acid environments because their pH value is close to acid rains and are also a carboxylic acid. Acid solutions were prepared to be 1 mole. During the experimental study, in order to see the differences and to compare the effect of the acidic environments, the samples were kept in 6 different environments: atmospheric environment, water, nitric acid (HNO_3), sulfuric acid (H_2SO_4), citric acid ($C_6H_8O_7$) and acid mixture for 6 months. The testing period, which started at the beginning of the winter season, ended at the end of spring. Thus, the sample, especially the one kept in the atmospheric environment, was exposed to different weather conditions during seasonal transitions. After 6 months, the mechanical and chemical changes of the Sille stones were analyzed.

The main material of this study is Sille stone was investigated for two different conditions which are varnished and unvarnished which are protected from outside effects. [13] states that the required sample size for the pressure test in natural stones should be 7x7x7 cm^3. Therefore, in this study tests were lasted 6 months, the size of the Sille stone sample was cut into a cube of 7x7x7 cm^3. The pressure strength test was performed on at least 5 test samples for each environment [13]. In addition, 1 test sample is required for chemical experiments. In this case, 5+1 test samples were prepared for each environment. Table 1, Figure 3 and Figure 4 show the tested samples; a view from the preparation stage of stone samples and acidic materials used during the test, respectively.

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Table 1. Testing samples

	No. of sample (varnished)	No. of sample (unvarnished)
Reference	5	5
Nitric Acid	5+1	5+1
Sulfiric Acid	5+1	5+1
Sitric Asic	5+1	5+1
Mixture	5+1	5+1
Outside environment	5+1	5+1
3. Month	30	30
6. Month	30	30
Total	130	

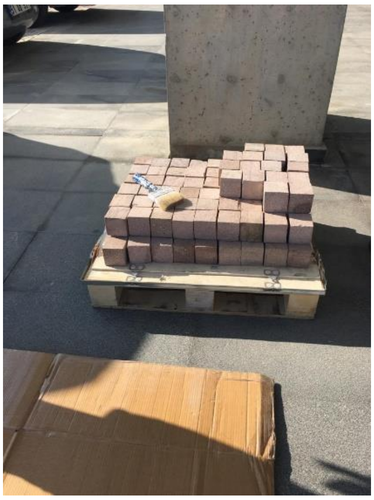

Fig.3. Preparation of testing samples

a) b) c)

Fig.4. Acidic materials used during the tests

The chemical information of the acids to be used in the study and the amounts of acids required to prepare the experimental environment are given in Table 1. Testing samples are given in Table 2, where Ma=the molecular mass, d= the density, m= the mass and n is the number of moles.

Table 2. Testing samples

Acid	Ma (g/mol)	d (g/L)	Concentration (%)	m (g)	n (mol)	Volume (ml)
HNO_3	63.02	1.40	65	2275	36.1	27.6
H_2SO_4	98.079	1.84	96	4416	45.03	22.2
$C_6H_8O_7$	192.12	1.66	-	192	-	-

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Using Table 2, 110.4 ml HNO_3, 88.8 ml H_2SO_4, 768 g $C_6H_8O_7$ were used to obtain 4 L of per solution. 36.67 g HNO_3, 29.6 g H_2SO_4, 256 g $C_6H_8O_7$ were used to prepare the mixture mediums. The preparation stages carried out in the laboratory are shown in Figure 5. Acid-resistant storage containers of 3x700 ml were used to keep the cubic stone samples in the acidic mediums. In order to prevent various damages such as explosion during freezing and thawing against air changes that may occur to samples that will be kept in the open air, a hole was made in the lids of acid-resistant containers. Prepared containers were kept labeled so that acidic mediums and samples do not mix.

The cubic stone samples were placed in to the containers, after that each mixture was added up to mixture liquid level rises above the sample and containers were placed in to external environment. A view from the laboratory study and a view from the external environmental study are shown in Figure 5 and 6.

Fig.5. A view from the external environmental study

Fig.6. A view from the laboratory study

4. Results and Discussion

4.1. Pressure Tests

The pressure test was made with the pressure device in the civil engineering laboratory of Necmettin Erbakan University and prepared according to the TS1926. Sille stones were prepared as cube samples of $7x7x7cm^3$ dimensions. 5 test samples were made for each prepared environment. As stated in TS1926, the loading speed was set to be less than 1.50 MPa/s, and the speed in the compressive strength test was set to 1.36 MPa/s. Before the sample was placed in the pressure device with the loading speed set, it was checked that the surface on which the sample would be placed was clean and smooth. The sample was carefully placed in the center of the loading bed so that the load would affect the test device exactly in the middle, i.e. axially. After the placement process, the loading was started and the maximum force that the sample could withstand was measured from the device.

Pressure tests were made for 5 samples belonging to samples kept in each environment. A pressure test was performed on Sille stone extracted from the quarry, too. Visuals of the pressure test of the samples extracted from the quarry are given in Figure 7. Compressive strength is the maximum stress formed in concrete under the effect of axial pressure load, the resistance it shows to not break. The reasons why the most used strength type in this study and in the field of engineering is compressive strength. The test applied to determine compressive strength is simpler than the tests applied to determine other types of strength. The compressive strength value of the carrier has an important role in structural designs. If the compressive strength is known, it is possible to have an idea about the other strength sizes of the samples and to make comments.

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Fig.7. Strength tests of the sample extracted from the mine

When the samples were compared at the end of three months, it was observed that the strength increased in all environments compared to the strength when they first came out of the quarry. This is an indication that the strength of the samples coming out of the quarry increased when exposed to oxygen. When acidic environments were compared with natural environments, it was observed that there was an increase in the strength of all of them. All acidic environments contain oxygen. This oxygen increases the strength of the stone. In addition, different chemical reactions occur in acidic environments. These oxidation and chemical reactions play a role in increasing the strength of the sample. An increase in strength was observed at the end of the first three months after the experiments were carried out. However, a decrease occurred at the end of the sixth month (Figure 8).

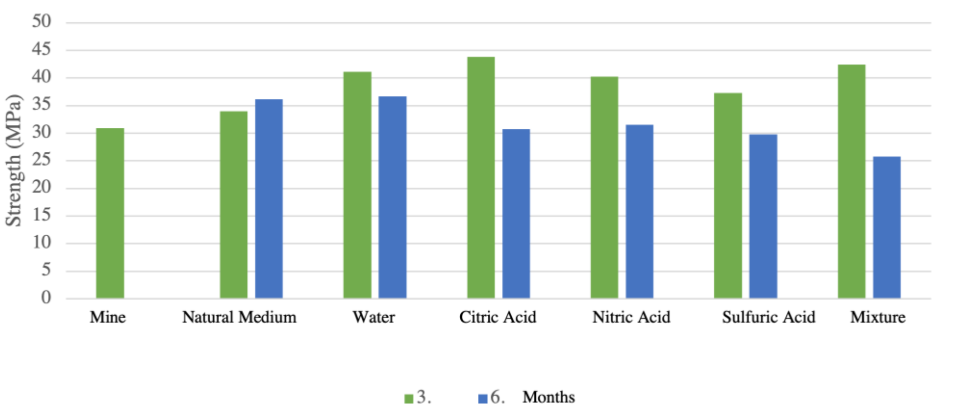

Fig.8. Strength tests after 3. and 6. months

There was an increase in strength in the environment in the first three months. However, it is seen that the strength started to decrease at the end of the sixth month. The system that reacted and increased the strength of the sample created a side or additional reaction that would reduce the strength of the material over time and the values started to decrease. However, there is still an increase in the natural environment.

4.2. SEM-EDS Analysis

Chemical analyses are useful in solving the components of the chemical structure of substances. Chemical analysis can be defined briefly as the act of separating the components of matter. The components determined in chemical analyses can be used to determine the contents in elemental or molecular form, that is, in compound form, depending on the research topic. SEM-EDS analysis is an X-ray crystal science analysis. This analysis is used to examine very small details. Scanning Electron Microscope (Scanning Electron Microscope) takes images using electrons, not high-wavelength rays like normal microscopes. Since electrons provide focusing at nanometer size, more detailed and high-resolution images are obtained. SEM analysis is used in many branches of science [14, 15].

The parts taken from the samples subjected to compression test were subjected to the following processes in order; first, polyester resin was poured onto the sample in a cylindrical mold and frozen. Figure 9. shows the first stage in the preparation of the samples for SEM analysis. Figure 9. shows the pouring of the samples into the molds with epoxy resin. This process makes it easier to disperse the sample prepared for analysis during the sanding stage and to hold it by hand.

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Fig.9. Pouring of the samples into the molds with epoxy resin

Secondly, the parts taken from the samples were sanded. figure 10 shows the sanding of the samples molded with epoxy resin. After the rough sanding done on the sanding device, the samples were sanded again with the more delicate sandpaper located next to the polishing device.

Fig.10. Sanding of the samples molded with epoxy resin

In the third stage, the sample was polished as shown in figure 11 and its dimensions were reduced with the stone cutting device to fit into the SEM device.

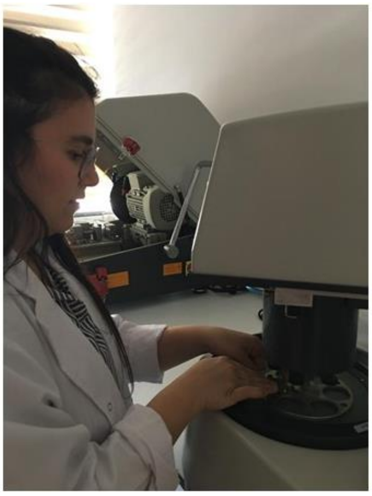

Fig.11. Polishing of samples

The samples were coated with gold in the last step of the preparation stage. The samples prepared by going through these four stages were analyzed on the SEM-EDS device. Figure 12 shows the samples to be gold plated and the gold plating process.

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Fig.12. Samples to be gold plated and the gold plating process

4.3. XRD Analysis

X-ray diffractometry (XRD) method is used to examine the crystal structure of a substance and find its mineralogical composition. It is a method used to define the crystal structure properties of minerals with very small grain sizes. This method is carried out by hitting the atoms of the sample crystals with X-rays consisting of electromagnetic waves with very short wavelengths [16]

Samples from the quarry to be analyzed by XRD were prepared using citric acid, acid mixture, water and unvarnished samples that were kept in an atmospheric environment. The samples to be analyzed were ground into powder with the help of a wire brush. It was sufficient for the powdered sample to be analyzed to be 1-2 grams. Samples to be analyzed by XRD are shown in Figure 13.

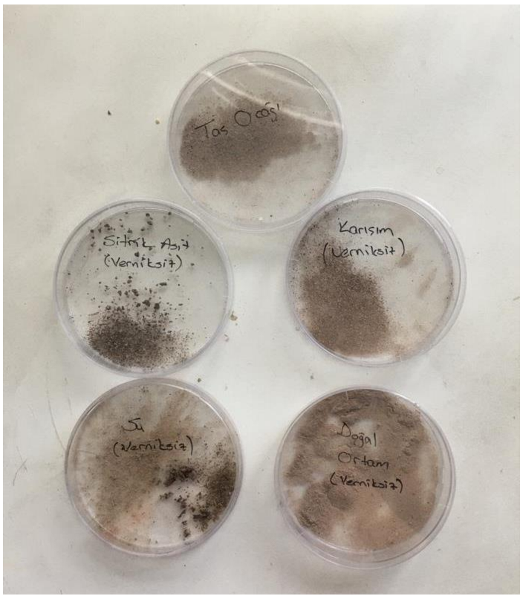

Fig. 13. XRD samples analysis

5. Conclusions

• When the analyses made in all environments were evaluated, it was concluded that the main components of Sille stone are albite and quartz minerals.

• Both physical and chemical transformations occur in Sille stone over time. The physical transformation of the stone occurs when the pressure on the Sille stone extracted from the quarry decreases and it becomes lower in energy. The chemical transformation of the stone is the changes in the compressive strength because of chemical reactions that occur in Sille stone samples kept in different environments.

• The compressive strength of the Sille stone extracted from the quarry without exposing it to any effect increases in all environmental conditions in the first 3 months. The increase in compressive strength causes transformations in the chemical structure of the stone.

• This transformation positively affected the compressive strength of the Sille stone sample kept in the atmospheric environment, water environment and acidic environments for 3 months. However, it started to damage the sample over time and negatively affect it, and at the end of the 6th month, there was a decrease in compressive strength.

• To examine the transformations in the chemical and physical properties of Sille stone in more detail, a single aging environment can be selected, and more detailed findings can be determined and evaluated by performing strength tests at different temperatures and different intervals over a longer period.

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