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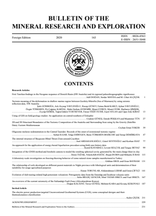

A laboratory-scale investigation on freezing-thawing behavior of some natural stone samples manifactured in Turkey

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Research Article

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ABSTRACT

In the study, the changes in the mechanical properties of the natural stone samples, which had been obtained from different regions of Turkey, before and after the freeze-thaw test were examined. Within the scope of the study, 17 different natural stone samples were used and, after chemical and mineralogical-petrographical properties were determined. Water absorption at atmospheric pressure, real density, porosity, and uniaxial compressive strength tests were performed before and after freezing-thawing. The freezing and thawing test has been performed using TS EN 12371 standard from -12 °C to +20 °C in 56 cycles (2 cycles = 24 hours). After the test, the samples were subjected to uniaxial compressive strength test in order to identify the changes of the compressive strength values. The results of the physical and mechanical tests on these samples were evaluated by grouping according to chemical and mineralogical-petrographical properties of natural stones. It was aimed to determine which natural stone samples are resistant to freeze-thaw, and can be used in external cladding applications according to the findings obtained from the study.

1. Introduction

In this study, uniaxial compressive strength test was performed before and after the freeze-thaw test in order to determine the freeze-thaw effect on the natural stone samples manufactured, and it was aimed to examine the changes in the strength values according to the test results. Natural stones are the oldest construction materials that are mined from the surface by using suitable production methods and then cut-dimensioned and polished if necessary, and can also be mined economically. Since the early ages, natural stones have been used extensively by human beings in their buildings and monuments thanks to their visual beauty as well as their resistance to degradation. Due to the increase in usage of them, they are used especially in areas such as cladding, flooring, sculpture, construction, porcelain and glass industry, ornaments, optical industry, tombstone and stone chips production (T.C. Ekonomi Bakanlığı, 2016). Today, the usage rate of natural stones is increasing, this choice of use increases the reputation of some natural stones and enables new ones to be released. Natural stones create an ever-growing economic value for the region where they are found, and the country itself. However, natural resources and building blocks are not endless; reserves can be depleted, even affected by environmental conditions and degradable (Gökaltun, 2004; 2011).

As a result of this rising demand, mining activities have increased similarly and natural stone reserves with different origins, colors, textures and physical properties have started to be operated in Turkey where the richest natural stone deposits of the world are located. By commercial definition, natural stones

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include all natural rock materials produced for decorative usage (Onargan et al., 2006).

The properties sought in natural stones can be listed as color property and pattern homogeneity, block making feature and cutting and polishing, geomechanical and physical properties, resistance to atmospheric and chemical effects (Şentürk et al., 1995). These properties directly affect the suitability of these natural stones for building cladding depending on the usage place. All natural stones on the earth are constantly affected by physical and chemical processes such as freezing and thawing. However, the harmful effects of these processes are not the same everywhere and in every natural stone sample. The geological type and mineral composition of the natural stones, their resistance to abrasion, the climate conditions on the region where the stones are used, have a direct impact on the degradation; these harmful effects can be clearly seen especially on the surfaces of the materials used on cladding on exterior walls (Akbay et al., 2012). Along with the increase in the rate of natural stone use in building exterior cladding, the examination of the physical changes of natural stones exposed to effects such as rain and cold is an important parameter in terms of the choice of the usage area. In this process, especially in regions where the continental climate is dominant and the temperature difference is high during the day, the physical and mechanical properties of natural stones are weakened by the freeze-thaw effect. In natural stones used in long-term structures such as historical artifacts and monuments and exposed to freezing-thawing processes intensely, it is seen that the degradation that occurs as a result of these processes is very important in the change of physical and mechanical properties (Mutlutürk et al., 2004). Waters intruding by water absorption or capillary water absorption to the natural stones used in the regions where the day and night temperatures are quite different and the cold continental climate is dominant, freeze by increasing its volume by approximately 9% in sub-zero temperatures. As a result of this freeze-thaw cycle which is repeated over and over again and causing the most damage in natural stones, the cracks begin to form due to the increase in pore pressure when the absorbed water turns into ice, and over time, these cracks expand and grow. Thus, this situation causes degradation and fragmentation in the rock (Chen et al., 2004).

Beier and Sego (2009) and Proskin et al. (2010) showed that some engineering properties of rocks, such as stress and strength, are severely affected by the freeze-thaw cycles. Binal et al. (1997; 1998) investigated the changes in the physical and mechanical properties, after the freeze-thaw cycles, of volcano-sedimentary rocks in the Eskişehir region. Simonsen and Isacsson (1999), on the other hand, conducted a study on the dissolution resistance of paving stones laid in cold regions where the continental climate is dominant. In the study conducted by Binal and Kasapoğlu (2002), 30 freeze-thaw cycles have been applied on ignimbrite samples of the Aksaray region. At the end of these cycles, increases in some physical properties such as porosity and water absorption by volume and weight have been observed. On the other hand, it has been stated that some other mechanical properties like shear strength and uniaxial compressive strength decrease. Alyıldız (2003) and Altındağ and Alyıldız (2004) investigated the effects of freeze-thaw cycles on the physical and mechanical properties of tuff samples from Isparta region in a laboratory environment. As a result of the experiments carried out by applying 55 cycles in total, while the change of physical properties started to show differences after the 10th cycle, some other mechanical properties after 25th cycle; it has been stated that these differences are most effective in the point load strength index in mechanical test results and in water absorption values by weight through physical test results. Tuğrul (2004) examined the effect of degradation on the properties of rocks and stated that there is an increase in porosity and a decrease in strength values as a result of the experiments conducted on rock samples exposed to degradation at different levels. Nicholson and Nicholson (2000) examined the effect of defects (discontinuity etc.) on degradation by repeated freeze-thaw cycle tests on 10 sedimentary rock samples already having those structures. As a result, they have suggested that the defects existed before the test should also be evaluated in addition to rock strength and textural properties and that the loss of mass takes place due to the degradation in the structure of the rock. Binal et al. (2004) have used the Ankara ignimbrite rock in their study and compared the changes of physical parameters between the samples exposed to the freeze-thaw process due to the effect of atmospheric conditions in the nature and other samples, which the freeze-thaw test has been applied on, in laboratory environment. They stated that the changes in the physical results of the samples that underwent freeze-thaw for one year under natural conditions and the samples that had 10 freeze-thaw cycles in the laboratory environment are closely compatible with each other. In addition, they stated that the uniaxial compressive strength results of the samples with 5 freeze-thaw cycles applied in the laboratory were similar to the results of the samples exposed to freeze-thaw process under the effect of atmospheric conditions in nature.

Martínez-Martínez et al. (2013) conducted freeze-thaw tests on 6 different carbonated rock samples with 12-24-48-96 cycles, respectively. P wave propagation velocity, macro and micro-textural properties, elasticity modulus, volume loss, porosity and uniaxial compressive strength results have been examined with the tests performed after the relevant cycle numbers on the samples. They have stated that there is a non-linear relationship between those properties and freeze-thaw cycle time. However, after the given cycles, they have showed that there is a slight increase in porosity values for all rock samples determined before the cycle. As a result, P wave velocity and uniaxial compressive strength have decreased.

2. Material and Method

2.1. Identification Process of the Material

Mineralogical and petrographic analyzes of the natural stone samples subjected to the relevant tests have been carried out in Mineralogy-Petrography laboratories operating under Department of Mineral Analysis and Technology, General Directorate of Mineral Research and Exploration. Mineralogical and petrographic analyzes of the samples (Table 1) were made on thin section based on TS EN 12407 standard. Chemical analyzes of natural stone samples were made in the geochemistry laboratories operating under

Table 1- Mineralogical and petrographic analyzes of the samples.

Sample Code	Petrographic Identification	Texture Type	Grain Size	Constituent Distribution	Rock Classification
AND-1	Andesite	Porphyritic	Fine grained	Homogeneous	Magmatic
AND-2	Andesite	Massive, Hipocrystalline- porphyritic	Medium-Fine grained	Homogeneous	Magmatic
AND-3	Andesite	Massive, Hipocrystalline- porphyritic	Fine-Medium grained	Homogeneous	Magmatic
BAZ-1	Basalt	Massive, porphyritic	Fine grained	Homogeneous	Magmatic
GRN-1	Granite	Massive, Holocrystalline grain texture	Fine grained	Homogeneous	Magmatic
İGN-1	Ignimbrite	Massive, Detritic	Mostly fine-medium grained, Occasionally very coarse grained	Homogeneous	Magmatic
RKRÇ-1	Recrystallized Limestone	Massive, Detritic	Fine grained	Heterogeneous	Metamorphic
RKRÇ-2	Recrystallized Limestone	Massive, Detritic	Fine grained	Heterogeneous	Metamorphic
RKRÇ-3	Recrystallized Limestone	Massive, fine grained Granoblastic texture	Medium-Coarse-Very coarse grained	Homogeneous	Metamorphic
RKRÇ-3	Recrystallized Limestone	Massive, Detritic	Fine grained	Homogeneous	Metamorphic
RKRÇ-5	Recrystallized Limestone	Massive, Cryptocrystalline detritic	Fine grained	Homogeneous	Metamorphic
MER-1	Marble	Massive, granoblastic occasionally heteroblastic	Fine grained	Homogeneous	Metamorphic
TRV-1	Travertine	Vesicular, Microcrystalline	Fine grained	Homogeneous	Sedimentary
TRV-2	Travertine	Massive porous, Fine grained	Fine grained	Homogeneous	Sedimentary
TRV-3	Travertine	Massive porous, Fine grained	Fine grained	Homogeneous	Sedimentary
TÜF-1	Glassy Tuff	Massive, Detritic	Fine grained	Homogeneous	Magmatic
TÜF-2	Glassy Tuff	Massive, Detritic	Fine grained	Homogeneous	Magmatic

the Mineral Analysis and Technology Department. Chemical analyzes of the samples (Tables 2 and 3) were made by using the XRF device based on the TS EN 15309 standard.

2.2. Method

2.2.1. *Sample Preparation Processes*

The rock samples to be analyzed were prepared in the accredited natural stone laboratory of Mineral Analysis and Technology Department affiliated to General Directorate of Mineral Research and Exploration. Samples were prepared to a cube block of 50x50x50 mm in the relevant standards with large and small cutting-sizing machines, respectively. The cube samples were prepared for the related tests by removing the roughness with the related devices (Figure 1).

Table 2- Chemical analyzes of the samples with magmatic origin.

Chemical Composition	**Sample Code**							
	AND-1	AND-2	AND-3	BAZ-1	GRN-1	İGN-1	TÜF-1	TÜF-2
Al_2O_3	15.61	15.51	15.65	21.39	17.17	16.88	15.10	15.83
CaO	1.42	1.87	1.79	9.14	4.13	3.32	3.16	1.11
Fe_2O_3	1.81	1.92	1.87	10.23	3.57	4.03	1.79	1.69
MgO	0.61	0.52	0.45	4.63	2.08	1.37	0.64	0.37
SiO_2	68.46	68.56	69.17	45.72	62.00	62.78	68.73	70.28

Table 3- Chemical analyzes of the samples with Sedimentary-metamorphic origin.

Chemical Composition	**Sample Code**								
	RKRÇ-1	RKRÇ-2	RKRÇ-3	RKRÇ-4	RKRÇ-5	MER-1	TRV-1	TRV-2	TRV-3
Al_2O_3	0.26	0.26	0.44	0.50	0.53	0.10	0.17	0.20	0.22
CaO	54.56	54.65	54.12	40.30	53.02	36.2	54.77	53.86	54.51
Fe_2O_3	0.11	0.09	0.21	0.30	0.24	0.10	0.07	0.60	0.11
MgO	0.51	0.50	0.45	12.1	0.89	17.10	0.27	0.68	0.41
SiO_2	0.62	0.66	1.69	1.80	1.20	0.30	0.44	0.80	0.87

Figure 1- The visuals of the natural stones used.

2.2.2. *Experiments on the Samples*

In order to determine the real density, total and open porosity of the rock samples, tests based on TS EN 1936 “Determination of Natural Stones-Experiment Methods-Real Density, Total and Open Porosity” standard were performed by using cube samples (Tables 4 and 5). The samples of natural stones were powdered (grounded, -63 µm) and the pycnometer method was used for the real density test. Tests based on TS EN 13755 “Natural Stones-Experiment Methods-Determination of Water Absorption at Atmospheric Pressure” were performed by using cube samples (Tables 6 and 7) to determine the water absorption coefficients of the samples.

In order to determine the uniaxial compressive strength of natural stone samples, tests based on TS EN 1926 “Natural Stones-Experiment Methods-Determination of Uniaxial Compressive Strength” standard were performed by using cube samples (Table 8 - 9). During the test, a uniformly distributed load was applied on 10 samples of each natural stone type in accordance with the relevant standards, the load was continuously increased until fracture occurred through the sample and the results were given in MPa units. Standard deviation was calculated by 10 results obtained for each natural stone sample after the compressive strength test. Tests based on TS EN 12371 “Natural Stones-Test Methods-Determination of Frost Resistance” standard were performed by using cube samples to determine the effect of freeze/thaw cycles in natural stones. The samples were tested in the freezing tank at temperatures between -12°C and +20°C in 56 cycles (2 cycles = 24 hours). After the test, uniaxial compressive strength tests were carried out after freezing to determine the compressive strength changes of the samples.

Table 4- Open-total porosity and real density values of the samples with magmatic origin.

Test Name	**Sample Code**							
	AND-1	AND-2	AND-3	BAZ-1	GRN-1	İGN-1	TÜF-1	TÜF-2
Open Porosity (%)	18.0	10.7	14.5	5.0	0.9	38.4	10.7	26.6
Total Porosity (%)	25.7	15.2	21.0	11.9	1.7	49.9	13.6	38.9
Real Density (gr/cm³)	2.6	2.6	2.6	3.0	2.7	2.5	2.5	2.5

Table 5- Open-total porosity and real density values of the samples with sedimentary and metamorphic origin.

Test Name	**Sample Code**								
	RKRÇ-1	RKRÇ-2	RKRÇ-3	RKRÇ-4	RKRÇ-5	MER-1	TRV-1	TRV-2	TRV-3
Open Porosity (%)	0.3	0.2	5.2	1.1	0.8	0.5	9.0	4.7	9.1
Total Porosity (%)	1.3	1.4	6.6	2.5	1.3	1.3	17.3	11.5	16.8
Real Density (gr/cm³)	2.7	2.7	2.7	2.8	2.7	2.9	2.7	2.7	2.7

Table 6- Water absorption rates of the samples with magmatic origin under atmospheric pressure by mass.

Test Name	**Sample Code**							
	AND-1	AND-2	AND-3	BAZ-1	GRN-1	İGN-1	TÜF-1	TÜF-2
Water absorption at atmospheric pressure (%)	10.6	5.1	7.8	1.5	0.4	32.9	5.0	10.4

Table 7- Water absorption rates of the samples with sedimentary and metamorphic origin under atmospheric pressure by mass.

Test Name	**Sample Code**								
	RKRÇ-1	RKRÇ-2	RKRÇ-3	RKRÇ-4	RKRÇ-5	MER-1	TRV-1	TRV-2	TRV-3
Water absorption at atmospheric pressure (%)	0.1	0.2	1.9	0.4	0.5	0.2	2.5	2.0	3.9

Table 8- Compressive strength values of the samples with magmatic origin before and after freezing.

Test Name	Sample Code							
	AND-1	AND-2	AND-3	BAZ-1	GRN-1	İGN-1	TÜF-1	TÜF-2
Before Freezing (MPa)	75	135	112	146	167	19	166	80
Standard Deviation	6	10	14.6	6	34	1	9	4
After Freezing (MPa)	65	131	114	142	185	16	160	80
Standard Deviation	12	18	12.4	3	8	1.1	8	6

Table 9- Compressive strength values of the samples with sedimentary and metamorphic origin before and after freezing.

Test Name	Sample Code								
	RKRÇ-1	RKRÇ-2	RKRÇ-3	RKRÇ-4	RKRÇ-5	MER-1	TRV-1	TRV-2	TRV-3
Before Freezing (MPa)	167	151	79	154	183	237	39	56	29
Standard Deviation	12	26	8	23	27	7	19	6	7.5
After Freezing (MPa)	165	156	80	130	171	224	30	55	29
Standard Deviation	8	18	8	28	14	5	13	6	8

3. Research and Results

3.1. Relationship Between Pressure Values and Water Absorption Rate Before and After Frost Resistance Test

According to the results of compressive strength tests before water absorption and frost resistance test, it has been determined that the pressure values before freezing decreased due to the increase of water absorption rates in natural stone samples of magmatic origin.

After the freeze-thaw determination test, it has been determined that there was no change in the compressive strength values of some samples and there was an increase or decrease in the compressive strength values of some other samples (Figure 2).

Similarly, it was found that pre-freezing pressure values decreased due to the increase of water absorption rates in natural stone samples of metamorphic and sedimentary origin. After freezing-thawing, just like the samples of magmatic origin, there was no change in the compressive strength values of some samples and there was an increase or decrease in the compressive strength values of some other samples (Figure 3).

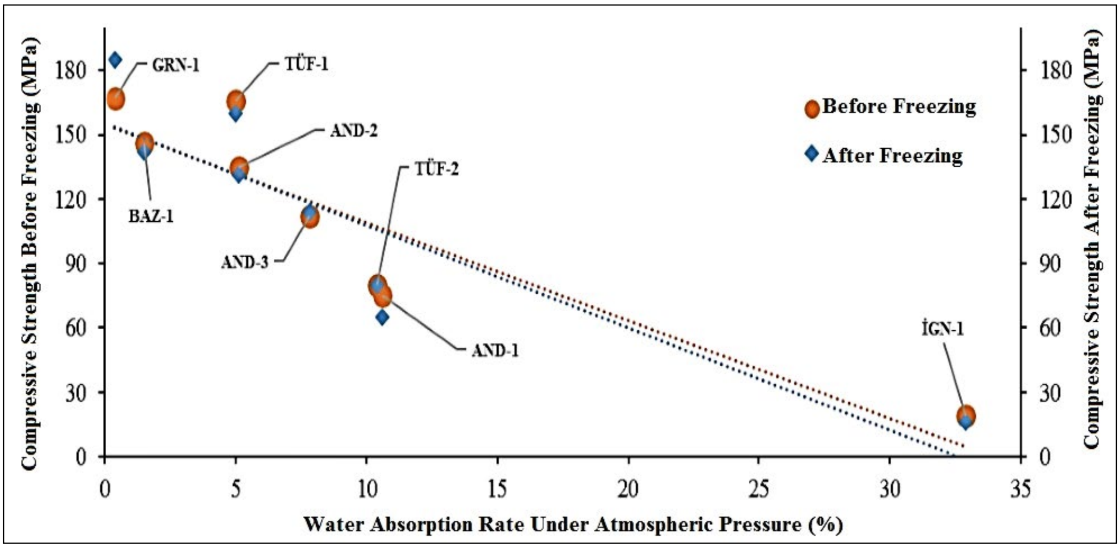

Figure 2- Water absorption rates and changes in compressive strength of the samples with magmatic origin, before-after freezing.

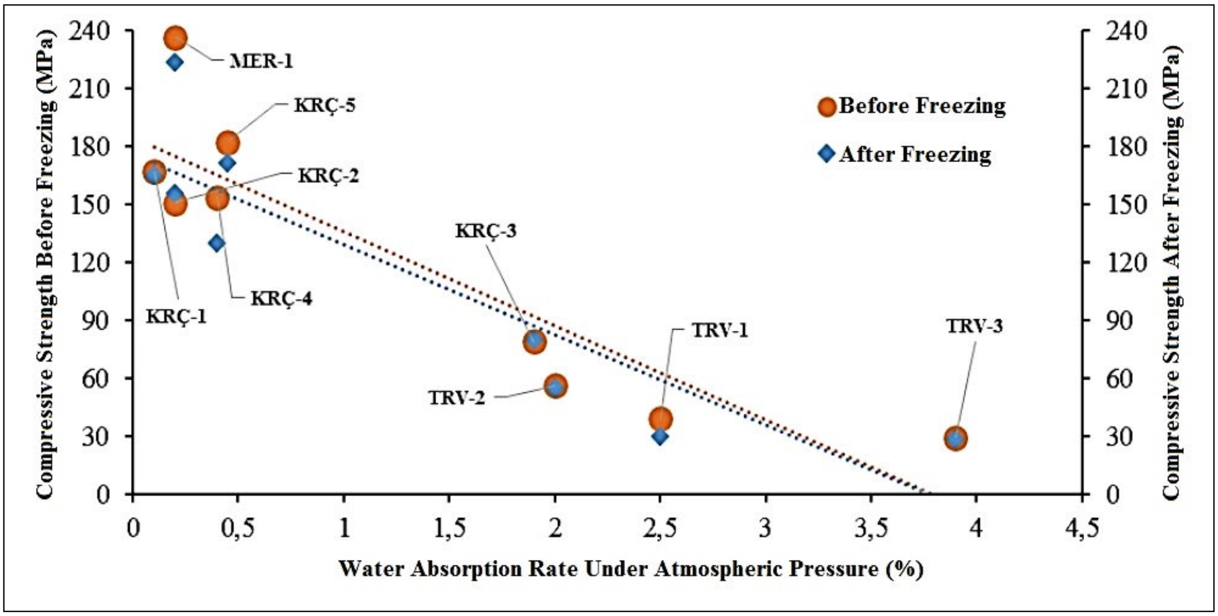

Figure 3- Water absorption rates and changes in compressive strength of the samples with sedimentary-metamorphic origin, before-after freezing.

4. Conclusion and Discussion

According to the results of water absorption and open-total porosity tests in magmatic, metamorphic and sedimentary origin stone samples, it has been determined that the open and total porosity rates increased due to increase in water absorption rates. According to the results of compressive strength tests performed before frost resistance tests, it has been concluded that the compressive strength values decreased due to the increase of water absorption rates in natural stone samples, therefore the compressive strength values would be lower in porous rocks. After the freeze-thaw test, there was a decrease in the compressive strength values of AND-1, BAZ-1, İGN-1, TÜF-1 and AND-2 samples with magmatic origin, however, increase in the values of AND-3 and GRN-1 samples. There was no change in compressive strength value of TÜF-2 sample. There was a decrease in the compressive strength values of RKRÇ-1, RKRÇ-4, RKRÇ-5, MER-1, TRV-1, TRV-2 samples with metamorphic-sedimentary origin, however, increase in the values of RKRÇ-2 and RKRÇ-3 samples and it has been observed that the value of the TRV-3 sample did not change.

The increase in water absorption and porosity ratio in atmospheric pressure is directly related. The reason for the decrease in pressure resistance values of some samples in both groups after frost resistance test is that the water is filled into the rock pores and subsequently expands the existing or newly formed cracks as a result of the water freezing and expanding at -12 °C and thus causes degradation through the rock structure. Compressive strength values of some samples in both groups at the end of freezing, no change was observed contrary to expectations or an increase was observed. From 20 samples prepared homogeneously for the test, 10 were selected for the pre-freezing compressive strength test and the rest for the post-freezing compressive strength test. During these selections, it is not possible to determine the ones having more or less compressive strength before the compressive strength test. After this sample selection, the sample or samples with high compressive strength were put into frost resistance test and they were not fully affected by the freeze-thaw cycle. As a result, it has been observed that the compressive strength values of these samples increased and the average compressive strength values increased slightly in the compressive strength tests performed after frost resistance tests. Momeni et al. (2015) have conducted uniaxial compressive strength tests on igneous rocks with different numbers of freeze-thaw cycles. In the related study, it has been revealed that compressive strength values increased after 50 cycles, but compressive strength values decreased at 100 cycles and over. In this study, it has been observed that some of the natural stone samples had some increase (GRN-1 sample), some decrease (RKRÇ-4 sample) or remained constant (TRV-3 sample) in uniaxial compressive strength values after freezing-thawing. However, in this observation, it could not be concluded that the number of freeze-thaw cycles has a definite reducing effect on uniaxial compressive strength as expected. As a result, while applying the TS EN 12371 "Natural Stones-Test Methods-Determination of Frost Resistance" test standard, the samples should be subjected to frost resistance test for longer periods instead of 56 cycles to completely see the freeze-thaw effect. Such natural stones should not be used especially on the building exterior since the compressive strength values of natural stones resulted with degradation during the freeze-thaw cycle decrease over time. Natural stones with low porosity and low water absorption rate should be especially used in the regions where continental climate is dominant, with high temperature difference between day and night. Thus, the lifetime of natural stones used on exterior of buildings or over the areas that may be exposed to atmospheric conditions will increase and the most appropriate usage of the these stones will be economically provided.

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