



# Accelerated aging laboratory tests for the evaluation of the durability of hydrophobic treated and untreated andesite with respect to salt crystallization, freezing–thawing, and thermal shock

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## Abstract

The processes of deterioration related to the interaction of the petrographic, physical, and mechanical characteristics of the rocks with physical and chemical actions of water are important. The aim of the article was to study the deterioration of andesite by accelerated weathering tests. For this purpose, laboratory studies were carried out in two stages. First, the main characterization analysis of the andesite was performed. Main technical values and parameters such as mineralogical–petrographic (polarize microscope, XRD, and SEM), chemical (XRF), and mechanical–physical properties were studied. Moreover, calculations on some physical and mechanical tests as well as the resistance to dry weight change caused by weathering were assessed. The change of rock strength and ultrasonic wave velocity measurements were experimented. These experiments were carried out on untreated and hydrophobic treated andesite before and after accelerated alteration tests such as cyclic freezing–thawing, salt crystallization, and thermal shock. The ultrasonic wave velocity and uniaxial compressive strength test results on the samples subjected to salt crystallization and freezing–thawing tests showed decreased values for all the andesite samples. However, the physical integrity of the andesite samples did not deteriorate. Laboratory test results showed that all andesite samples were suitable for use as building stones in a humid and salty environment. It was found that treated samples were less affected by the destructive effect of accelerated weathering tests compared with the untreated samples. Therefore, for the outdoor use of these stones, it is recommended that the block surfaces are to be treated with chemicals against weathering.

**Keywords** Accelerated weathering tests · Andesite · Salt crystallization · Cyclic freezing–thawing · Thermal shock · Water repellent

## Introduction

İscehisar andesite is a volcanic rock from Ağın Mountain (NE Afyonkarahisar, Turkey), which has been widely used in cultural heritage including mosques, fountains, public buildings, and residences, especially from Ottoman times onward, in Afyonkarahisar. Andesite is widely used in ex-

terior and interior of buildings. In addition to walkways and landscaping in parks and gardens, it is also used as covering and paving stones. Andesite is generally used without polishing because of the cavity structures.

It is important to realize that any building stone used will change in appearance and undergo at least some decay as an inevitable consequence of deterioration due to long-term atmospheric effects. Most building stones, exposed to long-term atmospheric (climatic conditions, various gases and compounds) or environmental effects (badly construction or maintenance), inclined to deterioration. These processes, named as stone decay, weathering and degradation, or simply deterioration, refer to an altered state or damage of the building stone (Frasca and Yamamoto 2006). Building stone deterioration is influenced by total effective porosity, pore size, pore size distribution, tensile strength, ionic type and concentration of pore

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solutions, frequency of wetting and drying, thermal shock, and freezing–thawing action.

Weathering processes are associated with water in the pore network. Water absorption and desorption and water transport and migration of natural stones are hygric processes. The hygric properties of natural stones are related by their porosity characteristics. Hygric properties are considered for the characterization of stone materials and their state of weathering as well as for stone treatment and the design of protective agents (Fitzner 2016). Freeze–thaw cycle is a typical weathering process and has strong impacts on the physical and mechanical behaviors of rock in the cold regions (Chen et al. 2004). As water freezes, it expands by about 9.05% of its volume. Thus, expansion of water inside pores, cracks, and joints will act to stress the surrounding rocks on freezing, and this process causes rock deterioration in cold regions (Park et al. 2015). Chemical processes need water, occurring more rapidly at higher temperature, so they are more common in warm and wet climates. Weathering of building stones is caused by the chemical action of water containing atmospheric oxygen, carbon dioxide, and some organic acids in solution on the rock-forming minerals leading to an adjustment of the mineralogical composition with the formation of new minerals, like hydrous phyllosilicates, iron oxides/hydroxides, soluble salts, and other alteration products, consisting of rock decay by chemical decomposition (Duarte et al. 2018).

Estimating the response of a building stone to environmental decay conditions is very important in detecting the life of historical buildings. A variety of engineering tests are employed to predict building stones' weathering behavior with time and under given climatic conditions accurately. Most of the tests are designed to accelerate the normal physical weathering response of the building stone under the controlled laboratory conditions. Some of these tests are freeze–thaw (FT), salt crystallization (SC), and thermal shock (TS) (Ca'rdenes et al. 2014). With the help of these tests, some occurred deteriorations in the building stones can be examined. The salt crystallization test will not reproduce the environment in which the stone is expected to do but rather accelerate the effects of isolated aspects of the natural environment. Deteriorations will occur caused by freezing–thawing action of water in porous building stones by the freeze–thaw test. Cracking and bending will be occurred by thermal changes of specific minerals in certain building stones by the thermal shock test.

Many experimental and theoretical studies have been carried out to explain freeze–thaw action on the deterioration of various building stone samples (Akin and Ozsan, 2011; Tan et al. 2011; Ruedrich et al. 2011; Bayram 2012; Jamshidi et al. 2013; Iñigo et al. 2013; Martínez-Martínez et al. 2013; Ghobadi and Babazadeh 2015; Park et al. 2015; İnce and Fener 2016; Gökçe et al. 2016).

Several studies were performed in accelerated laboratory evaluation of surface treatments for protecting building stones from salt crystallization. Ziyaettin and Tanaçan (2011) studied the influence of various water-repellent chemicals on the salt crystallization in building stones. They declared that treated building stones have a water absorption rate to decrease by 90%, ultrasound speeds increase by approximately 5–10%, and pressure resistance increase by 20–25%. Salazar-Hernández et al. (2015) investigated TEOS/colloidal silica/PDMS-OH effect in the treatment of building stone used to restore several historic buildings in the city of Guanajuato, Mexico. They reported changes of the properties of the tested stone after treatment, such as salt crystallization resistance and variations of porosity. Sena da Fonseca et al. (2017) compared heating and freeze–thaw action with accelerated laboratory tests for consolidants on porous limestone.

Many researchers have used accelerated laboratory tests for the evaluation of the durability of andesite by the freeze–thaw cycles. Yavuz (2011) investigated the effect of freeze–thaw and thermal shock cycles on the deterioration of andesite and its durability. Liu et al. (2012) described the deterioration of the basic laws of frost damage and the changing trend of physical and mechanical properties of granite and andesite under the freeze–thaw cycles through simulating the temperature environment in the Qinghai-Tibet Plateau. Fener and İnce (2015) investigated the changes in physical and mechanical properties of Sille (Konya) andesite during the freeze–thaw cycles. Heidari et al. (2017) evaluated the effects of salt crystallization and freeze–thaw tests on the engineering properties of andesites of Anahita Temple.

Salt damage, which is in literature also known as salt attack, salt crystallization, or salt decay (Doehne 2002), can exhibit itself by honeycomb weathering (alveolarization), powdering or sanding (granular disintegration), efflorescence, or flaking (contour scaling) (Collepari 1990). Many studies have been carried out using experimental laboratory studies to assess building stone deterioration produced by salt weathering (Shahidzadeh-Bonn et al. 2010; Gonçalves and Brito 2014; Çelik and Aygün 2018; Çelik and Tıǧlı 2018). Many researches have been carried out about experimental laboratory studies of the building stones durability using a combination of salt crystallization and other tests (ultrasound waves, SEM, physico-mechanical) (Kramar et al. 2010; Yu and Oguchi 2010; Barbera et al. 2012; Molina et al. 2013; Alves et al. 2013; Matovic et al. 2014; Ergeç et al. 2015).

A few experimental studies have been carried out to assess andesite decay rates occurred by salt crystallization. Zedef et al. (2007) studied the chemical, mineralogical, and engineering properties of andesites and dacites and additionally the effect of salt crystallization on the durability. Yavuz et al. (2017) determined the durability of green andesite by accelerated aging tests such as (Na<sub>2</sub>SO<sub>4</sub>) salt crystallization and freeze–thaw cycles. Heidari et al. (2017) evaluated the

physico-mechanical properties of andesites by salt crystallization and freeze–thaw tests.

Many researchers have investigated the effects of thermal shock cycles on the properties of different building stones. Labus and Bochen (2012) studied accelerated weathering tests and the sandstone petrography influence on the weathering process. Demirdag (2013) experimentally investigated the practice of cement filling procedure in travertine and the effects of thermal shock and freezing–thawing cycles on the travertine. Ghobadi and Babazadeh (2015) studied the weathering behaviors of nine sandstones from the Qazvin area (Western Iran). For this purpose, they conducted accelerated weathering processes, namely, freezing–thawing (F–T), salt crystallization (SC), heating–cooling (H–C), and heating–cooling–wetting (H–C–W). Heidari et al. (2017b) developed a model for reliably estimating the degree of deterioration of limestones exposed to a series of accelerated aging tests (salt crystallization, freeze–thaw, wetting, and thermal shock) to assess the effects of weathering factors on degradation of limestones under natural conditions.

The application of hydrophobic coating to building stone makes the surface water-repellent and protect surfaces from weathering environmental conditions (Wheeler 2005). Unfortunately, few studies have been carried out on thermal shock behavior of surface treatments for protecting building stones and andesite up to date. Frascá and Yamamoto (2006) studied aging tests of the Brazilian granitic rocks by thermal shock, action of atmospheric pollutants (SO<sub>2</sub>), salt mist, and salt crystallization tests. Ugur (2014) studied the physical weathering of welded tuff by freezing–thawing and thermal shock cycles tests. Huhta et al. (2016) developed a new method to measure thermal shock of natural stones, and they have determined the capacity of the rock to resist thermal shock with rapid temperature changes. Wang et al. (2016) showed induced change of physical and mechanical properties of red sandstone from 10, 20, 30, and 40 artificial thermal shock cycles. Wang et al. (2016b) experimentally investigated TS and F–T effects on red sandstone before and after artificial tests. They showed the significant reduction of physical and mechanical properties of red sandstone after F–T or TS tests.

Laboratory-accelerated weathering tests are a basic method to investigate extreme weather conditions. By definition, “accelerated alteration tests” use conditions different from those found in the field such as special environmental chambers and instruments that speed up the weathering process (Alves et al. 2017). To study the effects of accelerated weathering tests on some physical and mechanical characteristics of stone, İncehisar andesites were selected. In this work, the changes in andesite properties upon accelerated weathering tests by salt crystallization, cyclic freezing–thawing, and thermal shock tests were studied to provide the necessary information for understanding and assessing the deterioration and its durability. These experiments were carried out in hydrophobic

treated and untreated andesites. After the accelerated weathering tests, the resistance against weathering of dry weight change calculations was assessed, and the change of rock strength and ultrasonic wave velocity measurements were calculated.

## Material and Methods

### Materials

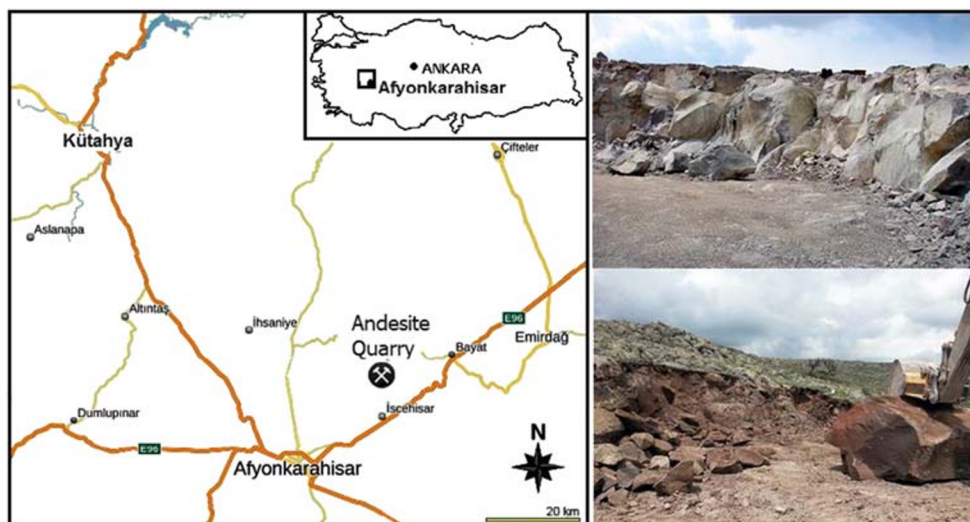
Andesite in this work was sampled from quarry in Ağın Mountains, located at the northern part of İncehisar (Afyonkarahisar, Turkey) (Fig. 1). Andesite is occasionally pinkish to reddish in color, depending on alteration. Hand specimens of the andesitic rock are mainly fine grained and pinkish to reddish in color. Average size of phenocrysts visible to the naked eye is 1–2 mm, occasionally more than 2 mm. It is not possible to identify fine minerals by naked eye due to the mineral sizes in the rock. Andesite has a massive structure and pores smaller than 1 mm. There were no differences in the tested samples that will affect the test results.

The andesite samples used in this study were made into cubic specimens, 50 × 50 × 50 mm for physical, mechanical, and accelerated weathering tests. The specimen sizes met the requirements of TS EN suggested methods. Salty water solution was prepared with the Merck quality sodium sulfate (Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O) for the salt crystallization experiments. In the experiments, Teknosil brand was used as a commercial water repellent which is siloxane- and solvent-based protection for natural stones. All the samples were washed and dried, and then water-repellent chemical was applied as a coating with a brush to the surface. The samples were used in experiments after standing for 36 h to ensure polymerization.

### Methods

The andesite samples were firstly examined under the polarizing microscope for a petrographic analysis using a Nikon Eclipse 2V100POL microscope. Chemical analysis was performed using a Rigaku/ZSX Primus II X-ray fluorescence spectrometer. To identify the morphologies and chemical compositions of the mineral phases present in the andesite, the samples were covered with carbon film, and scanning electron microscope (SEM–EDX) analysis was performed using a LEO 1430-VP microscope. X-ray diffraction (XRD) analyses were performed using a Shimadzu XRD-6000 device, determine the phase compositions of powdered andesite. And the diffraction patterns were obtained from 5 to 70° 2θ. Pore size and pore volume distribution were obtained by mercury porosimetry using a Micromeritics AutoPore IV 9500. Ultrasound pulse velocity (UPV) was measured using an Ele Pundit plus ultrasonic tester.

**Fig. 1** Location map of the tested andesite and photos of the quarry



It was then subjected to a physical and mechanical characterization, in order to determine the following properties: bulk and apparent density, water absorption, total and open porosity, ultrasound pulse velocity, uniaxial compressive strength, as well as accelerated deterioration tests, salt crystallization, cyclic freezing–thawing, and thermal shock. Physical and mechanical properties of the tested andesite were typically assessed through testing in accordance with the following TS EN procedures. (Table 1).

## Accelerated weathering experiments

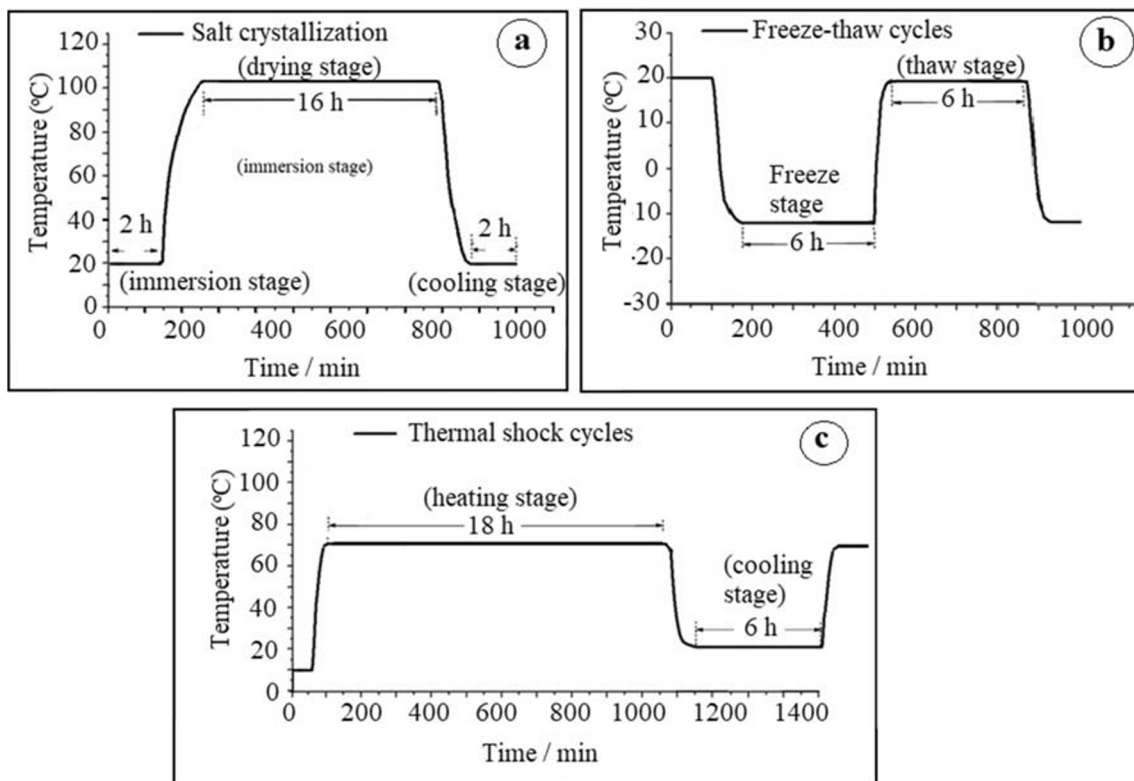
### Salt crystallization weathering

The hydrophobic treated and untreated andesite samples were artificially weathered by the salt crystallization test which was carried out by total immersion in a solution of sodium sulfate decahydrate ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) according to the standard test TS EN 12370 (Fig. 2a). In this test, six samples ( $50 \times 50 \times 50$ -mm cube) were used. The cycles were occurred in three stages. In the first stage (immersion stage), the clean and dried samples were placed in a

container (there is a minimum of 10 mm between samples and at least 20 mm between the samples and the sides of the container) and covered with the sodium sulfate solution ( $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) to a depth of  $(8 \pm 2)$  mm above the top of the sample and the container covered to reduce evaporation. The samples were then left to soak for 2 h at  $(20 \pm 0.5)$  °C. In the second stage (drying stage), after immersion, the samples were removed from the solution and dried in an oven at  $105 \pm 5$  °C for a period of 16 h. In the third stage (cooling stage), the samples were left in the oven for at least 16 h, and then they are cooled at room temperature for  $(2.0 \pm 0.5)$  h before re-soaking in fresh sodium sulfate solution. The cycle of operation was carried out 15 times in all. After the 15th cycle, the specimens were removed from the oven and stored for  $(24 \pm 1)$  h in water at  $(23 \pm 5)$  °C. Finally, they were washed thoroughly with flowing water. The specimens were weighed after drying to constant mass if they were sufficiently coherent. The weight of each test sample was determined before measurements and after each cycle, and the resulting weight loss, ultrasound pulse velocity (UPV), and uniaxial compression strengths (UCS) were determined.

**Table 1** Physical, mechanical, and accelerated weathering tests and related standards of the andesite

Tests	Sample size (mm)	Standards
Density ( $\text{kg/m}^3$ )	$50 \times 50 \times 50$	TS EN 1936 (2010)
Absorption by weight (%)	$50 \times 50 \times 50$	TS EN 13755 (2009)
Total porosity (%)	$50 \times 50 \times 50$	TS EN 1936 (2010)
Ultrasound pulse velocity (km/s)	$300 \times 50 \times 50$	TS EN 14579 (2006)
Uniaxial compressive strength (MPa)	$50 \times 50 \times 50$	TS EN 1926 (2007)
Freeze–thaw processes	$50 \times 50 \times 50$	TS EN 12371 (2011)
Thermal shock	$300 \times 50 \times 50$	TS EN 14066 (2015)
Salt crystallization	$50 \times 50 \times 50$	TS EN 12370 (2001)



**Fig. 2** The generalized temperature–time schematic diagrams of the salt crystallization test experimental procedure of TS EN 12370 (a), freeze–thaw cycle experimental procedure of TS EN 12371 (b), and thermal shock cycles experimental procedure of TS EN 14066 (c)

### Cyclic freezing–thawing weathering

Cyclic freezing–thawing test was carried out for hydrophobic treated and untreated andesite on specimens of cubic shape (50 mm) in order to study their resistance to decay experimentally and determine the weight loss, ultrasound pulse velocity (UPV), and uniaxial compression strengths (UCS). The cycles of freezing–thawing test were occurred in three stages according to the standard test TS EN 12371 (Fig. 2b). In the first stage (drying and immersion stage), the andesite samples were dried in an oven at 107 °C for 48 h, and then all the samples were saturated by water immersion for a period of 48 h. In the second stage (freezing step), all the samples were frozen for 6 h after the temperature of the test chamber reached – 12 °C. In the third stage (thawing stage), all samples were taken out from the freezing process, and they were put into water at 20 °C for 6 h. Each cycle consisted of a 6-h freezing period in air followed by a 6-h thawing period during which the specimens were immersed in water. The andesite samples after 56 freezing–thawing cycles were dried in an oven at 70 °C for constant weight and then cooled to room temperature. Weight of each test sample was determined before measurements, and after each cycle, and the resulting weight loss, ultrasound pulse

velocity (UPV), and uniaxial compression strengths (UCS) were determined.

### Thermal shock weathering

Thermal shock test was carried out for the hydrophobic treated and untreated andesite on specimens of prismatic shape (300 × 50 × 50 mm) according to TS EN 14066 (Fig. 2c). First, all the samples were placed in an oven at 70 °C and dried to constant weight. Then, all samples were taken from oven, and they were heated in the thermal shock cabin at 70 °C for 18 h. Finally, all samples were rapidly immersed in distilled water at 20 °C for 6 h. When thermal shock test was up to the 20 cycles, the samples were dried in an oven for constant weight. At the end of 20 cycles, the weight loss, ultrasound pulse velocity (UPV), and uniaxial compression strengths (UCS) were determined.

## Experimental investigation and results

### Chemical analysis

Two representative andesites were analyzed for major elements by an XRF method (Table 2). Percentages of significant major element oxides including SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, TiO<sub>2</sub>, Na<sub>2</sub>O, and K<sub>2</sub>O in the andesite samples are

**Table 2** Chemical composition of the tested andesite samples

Oxides (wt%)	Sample 1	Sample 2
Na <sub>2</sub> O	3.32	3.26
MgO	2.50	2.39
Al <sub>2</sub> O <sub>3</sub>	16.00	15.90
SiO <sub>2</sub>	57.00	57.50
P <sub>2</sub> O <sub>5</sub>	0.99	0.95
SO <sub>3</sub>	0.03	0.02
K <sub>2</sub> O	7.02	6.95
CaO	5.31	5.26
TiO <sub>2</sub>	1.05	1.06
Cr <sub>2</sub> O <sub>3</sub>	0.03	0.03
MnO	0.09	0.14
Fe <sub>2</sub> O <sub>3</sub>	4.77	4.86
ZnO	0.07	0.01
SrO	0.11	0.11
ZrO <sub>2</sub>	0.05	0.06
LOI	1.49	1.51

showed in Table 2. The andesites show a restricted range in major element composition, SiO<sub>2</sub> contents range from 57.00 to 57.50%, and the other components increase or decrease similarly.

Igneous rocks are classified according to mineralogical or chemical parameters. A chemical classification of total alkaline to silicate (TAS) method is applied on volcanic rocks if these parameters cannot be determined. For this purpose, chemical parameters of silica (SiO<sub>2</sub>) weight percent and total alkalis (Na<sub>2</sub>O + K<sub>2</sub>O) wt% were used (Le Bas and Streckeisen 1991). The silica (SiO<sub>2</sub>) and total alkalis (Na<sub>2</sub>O + K<sub>2</sub>O) weight percent data are also plotted in a TAS diagram (Fig. 3). It can be seen that the andesitic rocks were plot in the fields of trachyandesite.

## Petrography and mineralogy

### Polarizing optical microscope analysis

Smaller pores than 1 mm were observed on the andesite surface (Fig. 4a). SEM was used for detecting the minerals and other micro rock properties of the tested andesite. The micropores (5–30 µm) from the surface of andesite were determined by microanalysis of SEM images. It was observed that the essential minerals of andesite were plagioclase (Fig. 4b).

Polarizing optical microscope analysis is applied as a standard method for the quantification of the mineral composition of the stones. Stones are petrographically classified according to their mineral composition on this method. Furthermore, porosity, grain size, mineral alteration, dissolution, or decomposition due to weathering is described (Fitzner 2016).

Andesite samples are porphyritic rocks with phenocrysts dominantly of plagioclase and lesser amounts of biotite and hornblende in a glassy groundmass with microlites of plagioclase and contain abundant iron oxides (Fig. 4c, d). Altered hornblende and biotite minerals distributed randomly inside and some small volcanic glass fragments and cavities were also observed.

### X-ray diffraction analysis

X-ray diffraction (XRD) is the most efficient method and non-destructive tool for identifying and quantifying the mineralogy of crystalline compounds in building stones. Representative samples of the andesite were selected for X-ray diffraction (XRD) analyses. In andesite sample, the predominant phase is oligoclase, and the second in intensity is sanidine although peaks with low intensity correspond to andesine, tridymite, montmorillonite, and biotite (Fig. 5). In the XRD graph, it can be found that clay minerals are represented by montmorillonite as a product of a volcanic glass and feldspar minerals.

### Pore size distribution

Mercury porosimetry represents the most frequently applied indirect measuring method for porosity studies. With this method – contrary to all other measuring procedures – almost the whole range of pore sizes occurring in stones can be studied. According to the principle of mercury intrusion in dependence on pressure, pores with radii in the range of 0.0019 to 200 µm can be measured. Based on proportionality between pressure for mercury intrusion and the dimension of the pores, the measuring results allow the calculation of total porosity, densities, median pore radius, pore size distribution, and pore surface (Fitzner 2016). Pore size range is described by Klopfer (1985) as micropores (< 0.1 µm), capillary pores (0.1–1 mm), and macropores (> 1 mm).

Pore size distribution of andesite was determined using the mercury intrusion porosimetry (MIP) technique. The mercury intrusion data obtained from the MIP tests conducted on the sample of fresh andesite are presented in Table 3. According to mercury porosimetry results, median pore diameter (volume), median pore diameter (area), and average pore diameter (4 V/A) represented are 2.39, 0.01, and 0.03 µm, respectively. The cumulative curves attained by MIP show the incremental amount of mercury, and the accumulative amount of mercury at a certain pore diameter reveals the characteristics of pore diameter distribution, the continuity, and the uniformity of pore structures, respectively. The pore surface area shows the size of pore structures in a certain range of pore diameter. The data of pore structures attained during the process of mercury extrusion reveal the characteristics of pore diameter distribution, especially the amount of semi-closed pore in sample. The pore surface area shows the amount of semi-

Fig. 3 TAS diagram of the andesite samples. Total alkali versus SiO<sub>2</sub> classification plot after Le Bas et al. (1986)

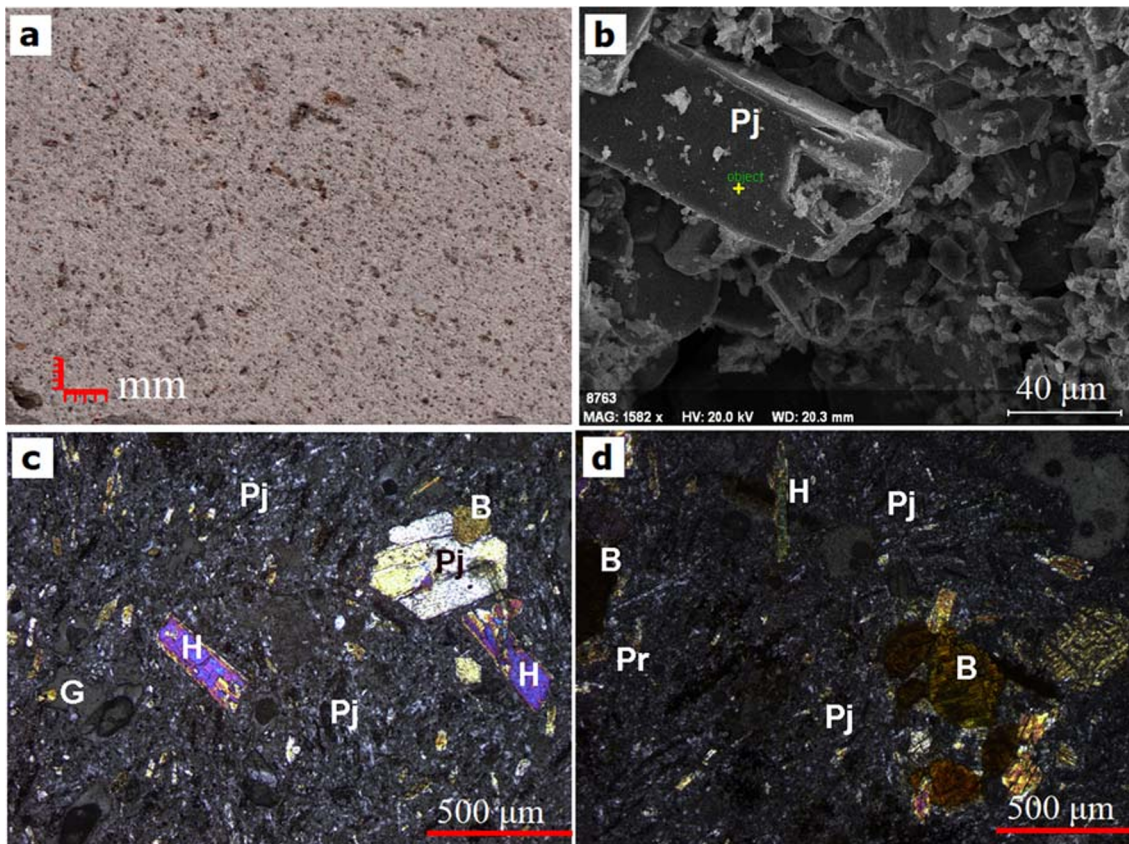
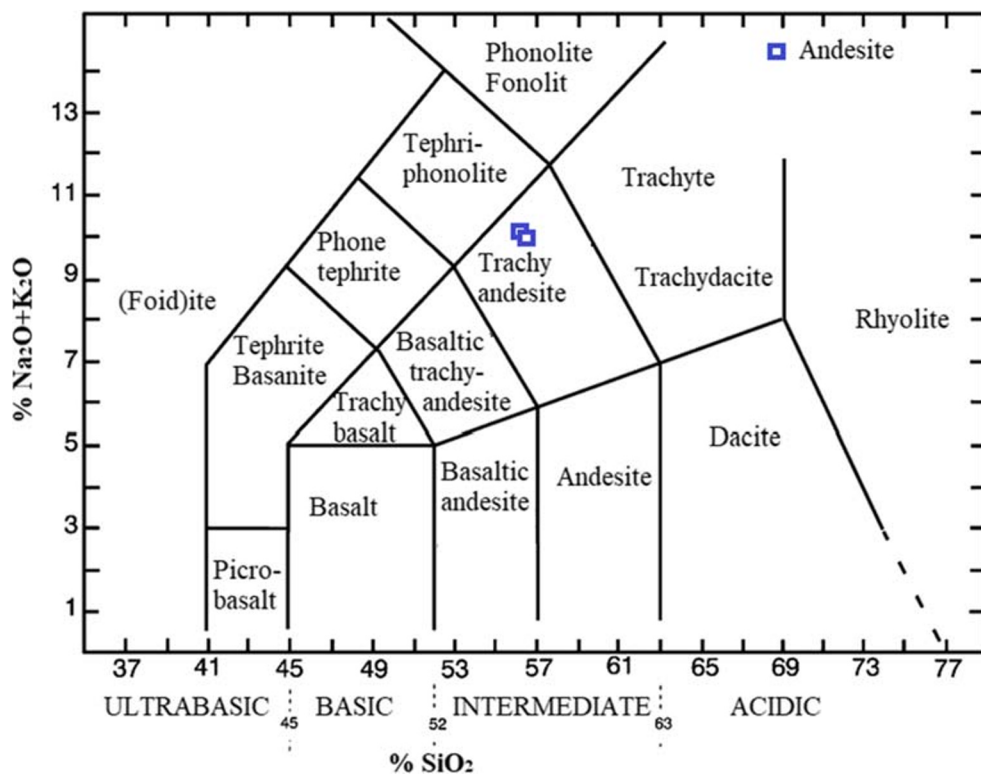
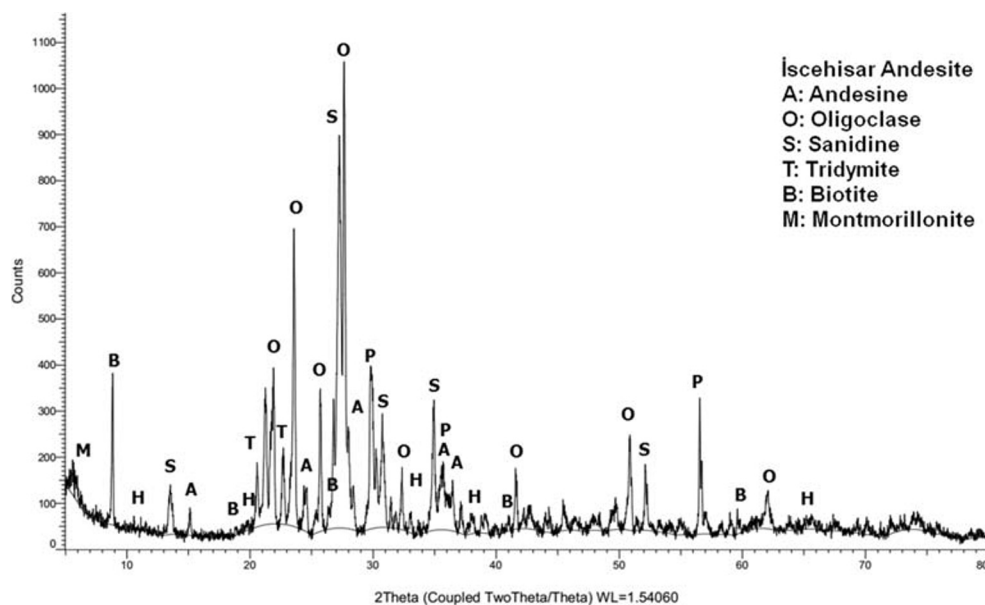


Fig. 4 Macroscopic view of andesite (a), SEM images of void spaces and plagioclase minerals of microstructure of andesite (b) and polarizing optical microscope images of thin sections of andesite (c, d). (Pj) plagioclase, (B) biotite, (H) hornblende, (G) glass, and (P) pore

**Fig. 5** XRD pattern of andesite samples



closed pore (Wensheng et al. 2005). Figure 6 shows the differential porosimetric curves for tested andesite, measured by mercury intrusion porosimetry (MIP). It is seen that the andesite sample has a pore diameter distribution between 0.003 and 100  $\mu\text{m}$ . The pore size distribution of andesite is intense between 0.01 and 10  $\mu\text{m}$ . The salt solutions which can be transported with the water by dissolution are mostly contained in the material through capillary pores. For this reason, pore distribution between 0.1 and 1 mm is significant. In this case, it can be said that the andesites have a small pore diameter distribution which can infiltrate the salt solutions.

### Physical and mechanical properties

Weathering effects on the physical and mechanical properties of natural stones of buildings cause durability problems. These properties are studied using the laboratory test methods used for investigation in the andesite samples. Laboratory tests were carried out to determine physical and mechanical

properties including density, water absorption, porosity, ultrasonic velocity, and uniaxial compressive strength in accordance with the relevant standards (Table 4). The percentage of water absorption ability of the andesite by weight was 2.44%. The uniaxial compressive strength of the andesite samples recorded was 70 MPa.

### Results of accelerated weathering experiments of the hydrophobic treated and untreated andesites

The durability of the hydrophobic treated and untreated andesites was investigated after salt crystallization, freezing–thawing, and thermal shock tests by the weight loss, ultrasound pulse velocity (UPV), and uniaxial compression strengths (UCS). The results obtained on the treated and untreated andesite samples in the accelerated weathering experiments are showed in Table 5.

**Table 3** Mercury porosimetry pore data of fresh andesite sample

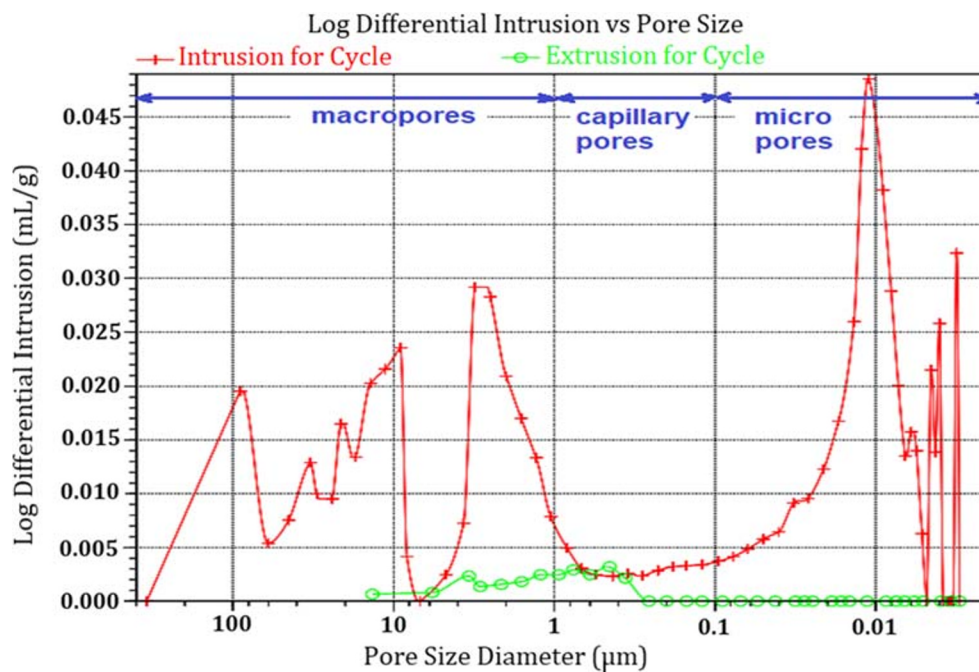
	Fresh andesite
Total intrusion volume (mL/g)	0.06
Median pore diameter (area) ( $\mu\text{m}$ )	0.01
Average pore diameter (4 V/A) ( $\mu\text{m}$ )	0.03
Median pore diameter (Volume) ( $\mu\text{m}$ )	2.39
Bulk density at 0.52 psia (g/mL)	1.99
Apparent (skeletal) density (g/mL)	2.27
Total pore area ( $\text{m}^2/\text{g}$ )	8.89
Porosity (%)	12.44
Stem volume used (%)	23

### Results of salt crystallization experiment

To help document the deterioration of the specimens and the alteration in their visual aspect, visual observations were made during each cycle. Additionally, photographs of the specimens were taken at the end of each drying stage to document the damage caused during the previous cycle. The macro view of the results obtained for  $\text{Na}_2\text{SO}_4$  crystallization tests for the untreated and hydrophobic treated andesite samples is shown by photos in Fig. 7, representing the effect of the sulfate solution through the exposed surface of the andesite samples. The changes on the surface of the andesites and the beginning of



Fig. 6 Pore size distribution of the tested andesite



the degradation in the salt crystallization experiment can be seen in these images.

Congruent with the insignificant change in the visual appearance of the hydrophobic treated and untreated andesite samples, little to no loss of material was observed either in the wet or drying phase of each cycle. Small amounts of fine-sized particles were observed in the bottom of the immersion containers after some cycles. Large quantities of efflorescence were never observed after (or during) the drying stage. Most of the efflorescence formed was noticed within the first few cycles, when some small crystals were observed attached to the surface. As efflorescence salt crystallizes outside the sample, it did not occur to induce deterioration.

**The variation of dry weight loss due to salt crystallization**

Dry weight loss (DWL) is the most commonly used parameter to evaluate salt crystallization damage in building stones in accelerated weathering tests (Yu and Oguchi, 2010). The salt

Table 4 Physical and mechanical properties of andesite samples

Tests	Andesite		
	min	max	mean
Density (real) (kg/m <sup>3</sup> )	2777.30	2787.20	2781.70
Water absorption by weight (%)	1.97	3.78	2.44
Open porosity (%)	3.44	6.04	4.74
Total porosity (%)	18.32	23.49	19.73
Ultrasonic velocity (km/s)	3.84	4.31	4.16
Uniaxial compressive strength (MPa)	53.05	96.75	70.00

crystallization test of up to 15 cycles was carried out on sodium sulfates (Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O), and the dry weight loss (DWL) of the untreated and hydrophobic treated andesite samples was measured. The dry weight loss was calculated for the hydrophobic treated and untreated andesite samples after washing with water and drying to constant mass by comparing the before and after test. The results obtained are given in Fig. 8.

The salt crystallization test results indicated that the untreated andesite had a dry weight loss of 0.80%, while the hydrophobic treated andesite had an increase of 0.24%. Generally, it is said that tested andesite is slightly affected from salt crystallization. Already within each cycle, the mass losses due to salt crystallization were very minute. Dry weight loss (DWL) occurs when salt crystallization pressure exceeds the stone cohesion limit. At the same time, when salt crystallization pressure is not enough to exceed the cohesion limit, there is no disintegration and weight loss, although other deteriorations such as enlargement of preexisting pores and fissures may appeared. As a matter of fact, this situation is manifested by the decrease in the uniaxial compression strengths (UCS) and ultrasound pulse velocity (UPV) values.

**The variation of ultrasound pulse velocity values due to salt crystallization**

In the past, many studies have attempted to relate the ultrasonic pulse velocity (UPV) to various stone properties such as mechanical strength and porosity parameters. UPV test can also be used to assess the level of weathering or deterioration in stones. UPV testing was used to investigate the damage in the hydrophobic treated and untreated andesite samples. The ultrasound

**Table 5** The results obtained on the treated and untreated andesite samples in the accelerated weathering experiments

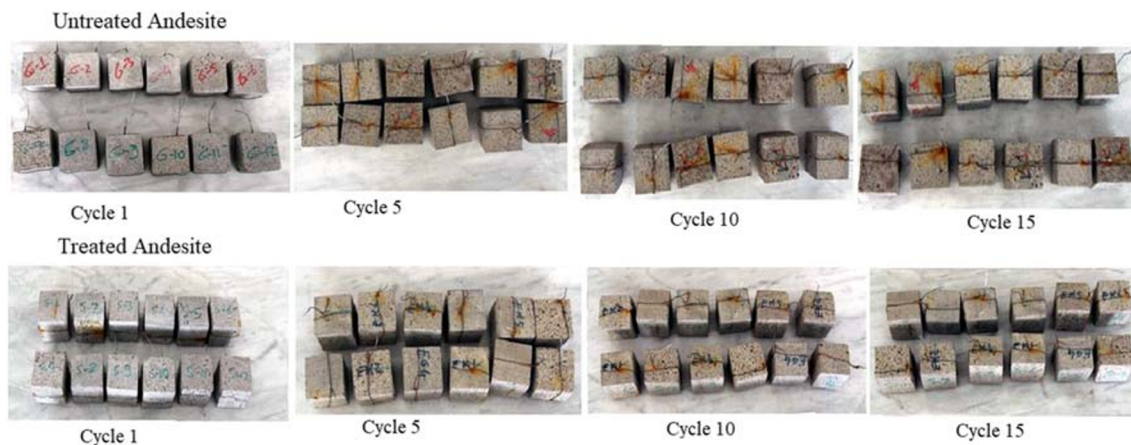
	Andesite samples		Salt crystallization test	Freezing–thawing test	Thermal shock test
Dry weight loss (DWL)	Untreated		0.82	0.11	0.23
	Treated		0.12	0.06	0.09
Ultrasound pulse velocity (UPV)	Untreated	Before	4.22	4.15	4.15
		After	4.15	4.03	4.13
	Treated	Before	4.24	4.16	4.16
		After	4.15	4.10	4.16
Uniaxial compression strengths (UCS)	Untreated	Before	70.00	69.58	
		After	53.00	56.68	
	Treated	Before	92.00	75.00	
		After	67.00	64.76	
Flexural strength (FS)	Untreated				14.87
	Treated				14.76
	Treated				15.48
Treated					15.43

pulse velocity is affected by many properties. These properties include stone mineralogy, physical properties (porosity, pore space, density), mechanical properties (strength), and degree of deterioration (Ahmad et al. 2009). Many of these properties can be affected due to the damage induced by salt crystallization cycles. Measuring the UPV after the salt crystallization tests can help provide insight into the degree of deterioration inflicted.

The velocity of longitudinal ultrasonic waves in the untreated and hydrophobic treated andesite samples was higher than that after the salt crystallization test (Fig. 9). The decrease of ultrasound pulse velocity in the untreated andesite samples after the salt crystallization test was 1.69%, and the hydrophobic treated andesite samples showed a decrease of 2.17%. After the salt crystallization test, all the samples showed a decrease in ultrasound pulse velocity due to no widening of micropores and fissures.

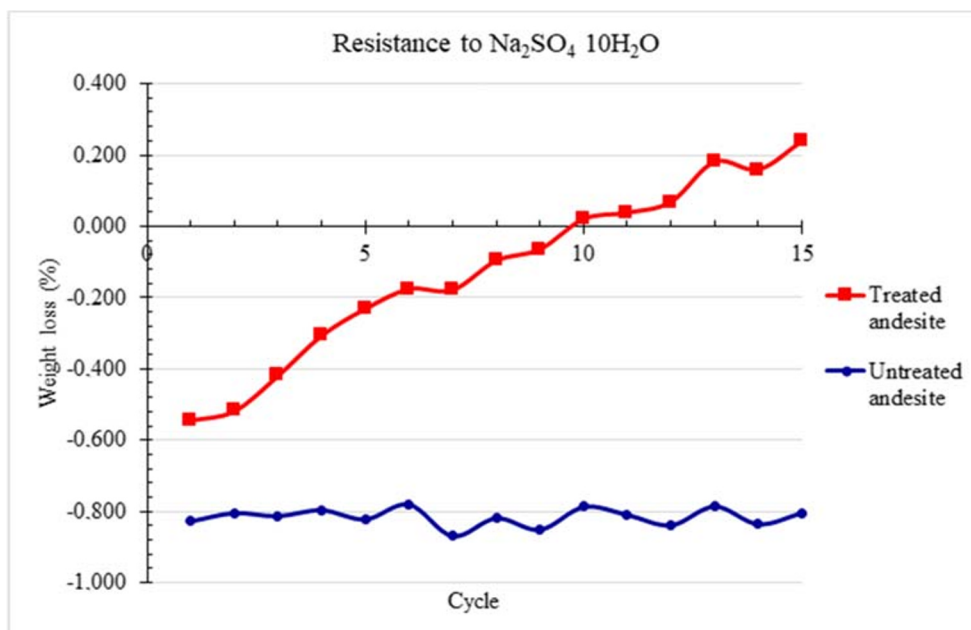
### The variation of the uniaxial compression strength values due to salt crystallization

The level of damage that salt crystallization can cause depends on several factors such as the environmental conditions and, of course, the material properties, namely, the porous structure. The uniaxial compression strengths (UCS) of the untreated and hydrophobic treated andesite samples before and after the salt crystallization tests are shown in Fig. 10. The untreated and hydrophobic treated andesite samples showed a decrease of the uniaxial compression strengths after the salt crystallization tests. The obtained results suggest that the variation because of the enlargement of preexisting pores and fissures after salt crystallization in pores influences the mechanical behavior of the untreated and hydrophobic treated andesite samples. The decrease of the uniaxial compression strengths (UCS) in untreated andesite samples after the salt crystallization test was 32.08%, and the hydrophobic treated andesite samples showed a decrease of 37.31%.



**Fig. 7** The change in visual appearance of the untreated and hydrophobic treated andesite samples in the salt crystallization tests

**Fig. 8** Dry weight loss values of the untreated and hydrophobic treated andesite samples in the salt crystallization tests

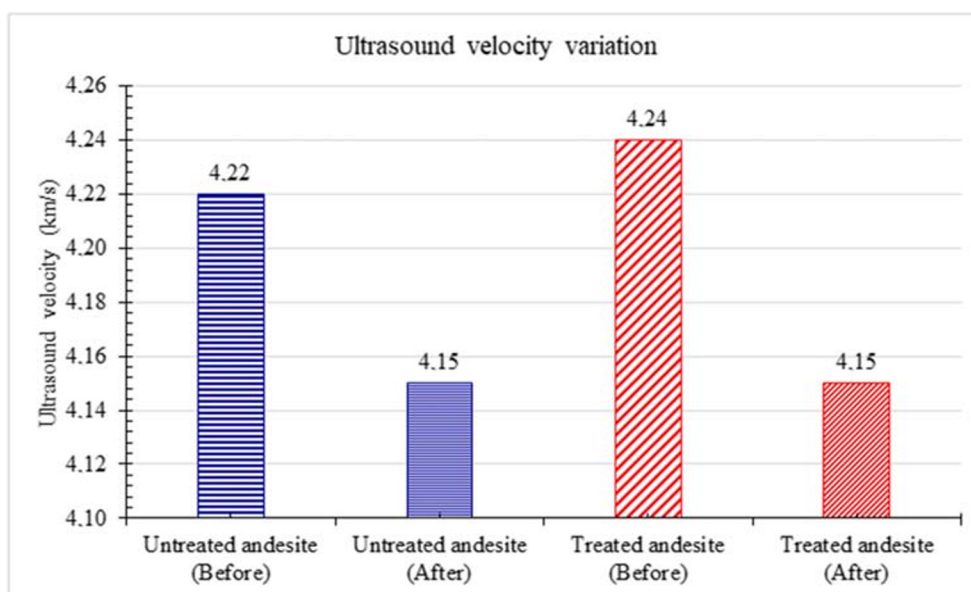


**Microstructural analysis by SEM after the salt crystallization test**

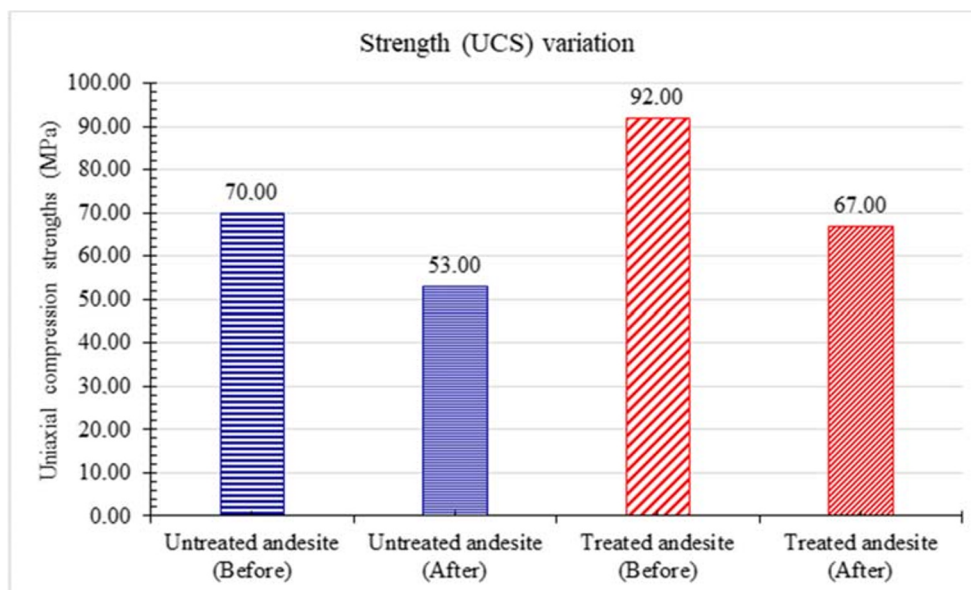
The location on the andesite sample surface of the precipitated salt minerals was analyzed by SEM analysis (scanning electron microscope). Additionally, EDX (Energy Dispersive X-ray) analysis was performed for element characterization. Close-up SEM analysis of the andesite samples showed sodium sulfate crystals in pores and on top of the surface of the stone. This accumulation of salt crystals on the surface is attributed to the lower drying temperature, and therefore slower drying speed allows

the salt solution to accumulate close to the surface. The SEM analysis demonstrated that the salt minerals mainly consisted of mirabilite and thenardite. In the tested andesite, the thenardite minerals were observed usually in small grain and anhedral crystal forms. Mirabilite (Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O) is another sodium sulfate originated mineral, and it is identified as elongated crystal forms. In these SEM micro photos, deteriorations such as cracks and pores reducing the mechanical strength of the andesite were observed. Repeated cycles of the salt crystallization test and dissolution in pores led to this micro fissure development (Fig. 11).

**Fig. 9** The variation of ultrasound pulse velocity (UPV) values of the untreated and hydrophobic treated andesite samples before and after the salt crystallization tests



**Fig. 10** The variation of the uniaxial compression strength (UCS) values of the untreated and hydrophobic treated andesite samples before and after the salt crystallization tests



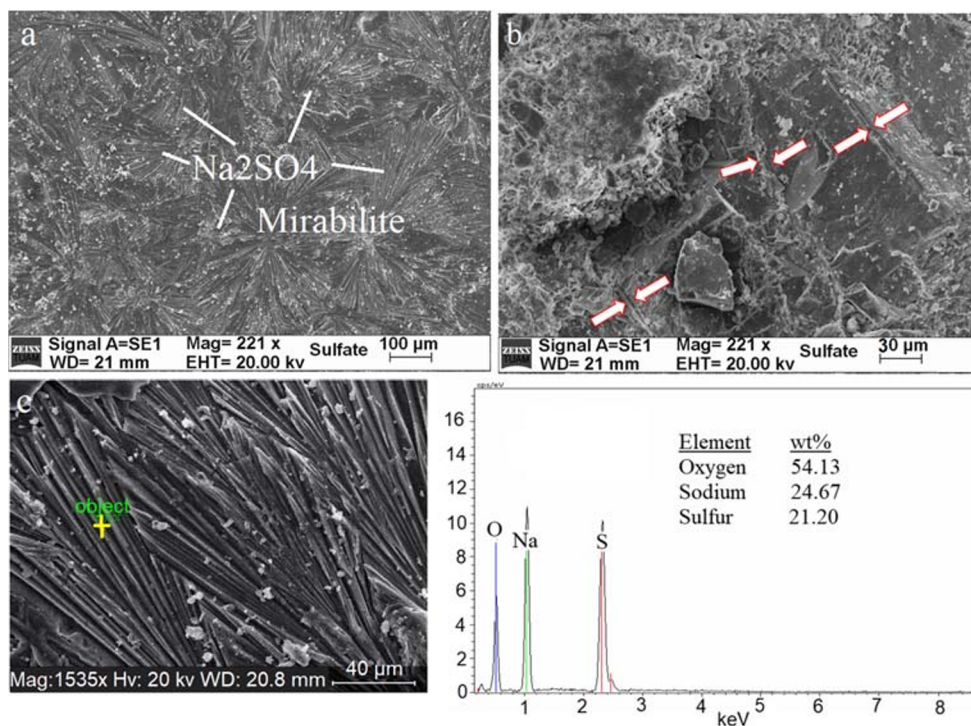
### Results of cyclic freezing–thawing test

Freeze–thaw action is a major physical deterioration process, which may cause a significant change in the strength properties of building stones and a decrease in their resistance. Freezing–thawing cycles are probably responsible for most of the deterioration in stones in the cold seasons. When the temperature decreases below 0 °C, water begins to freeze inside pores of the stones and expands about 9% of the original volume. This

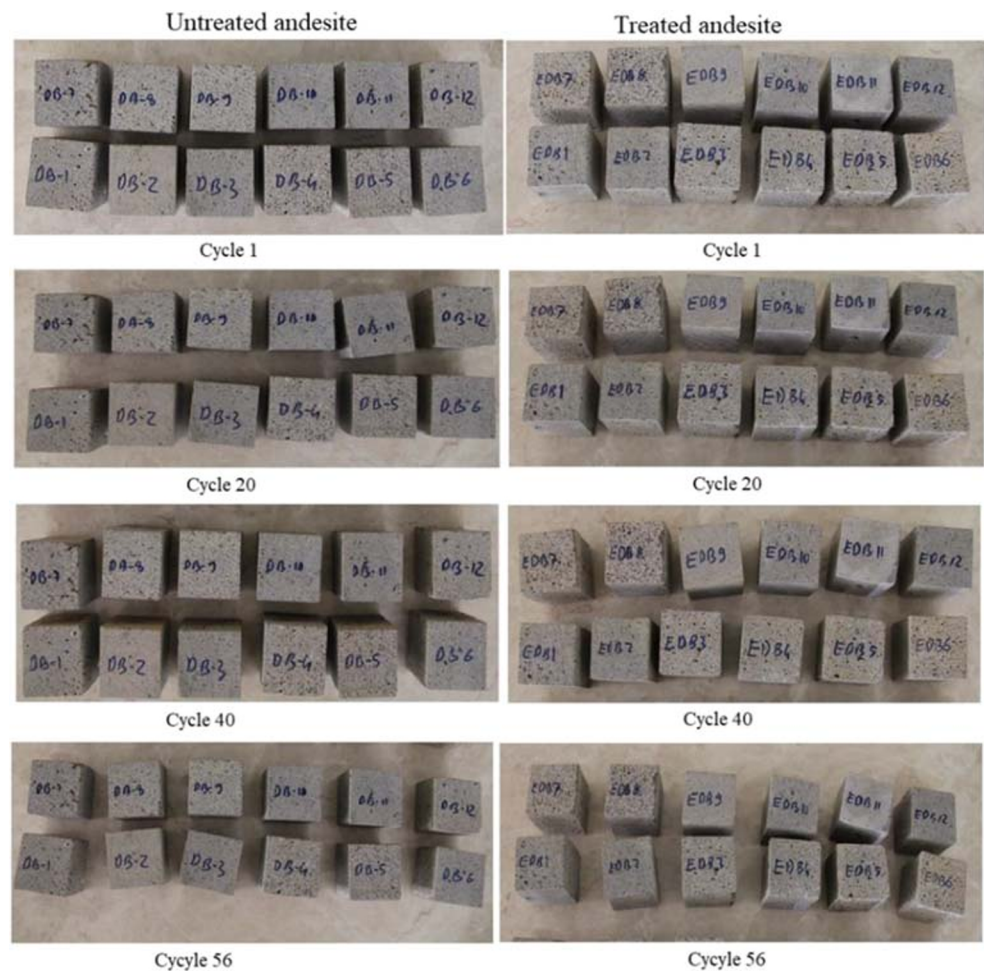
expansion may cause tensile stress, and micro fissure in the micropores may occur (Chen et al., 2004). Annual freeze–thaw cycle over 25 per year may occur in Central Turkey (e.g., in Afyonkarahisar) (Binal et al. 1997).

Cyclic freezing–thawing susceptibility of the untreated and hydrophobic treated andesite samples was studied. The changes of the untreated and hydrophobic treated andesite samples after the freeze–thaw tests were investigated by ultrasonic wave measurements and SEM

**Fig. 11** Scanning electron microscope images of andesite sample after the salt crystallization test. Detail of **a** mirabilite crystals covering the andesite surface, **b** salt crystallization induced micro fissure (red arrow), and **c** SEM images of mirabilite crystals in andesite and EDX analysis of element characterization



**Fig. 12** Macro views of the cyclic freezing–thawing tests compared with the untreated and hydrophobic treated andesite samples



observations, while the strength properties were determined by uniaxial compressive strength tests. Photographs of some untreated and hydrophobic treated andesite samples exposed to freeze–thaw cycles are shown in Fig. 12. These photographs show the untreated and hydrophobic treated andesite samples of 1, 20, 40, and 56 cycles in freeze–thaw test. Changes on the surface of samples and macroscopic color change were examined with a visual during the freezing–thawing test. The edges and corners of the samples were observed to be little deformed. After the freezing–thawing cycles of test, the andesite samples underwent a slight change in color. The variations such as roughness and loss of gloss in andesite samples were observed with the naked eye.

#### The variation of dry weight loss due to freezing–thawing

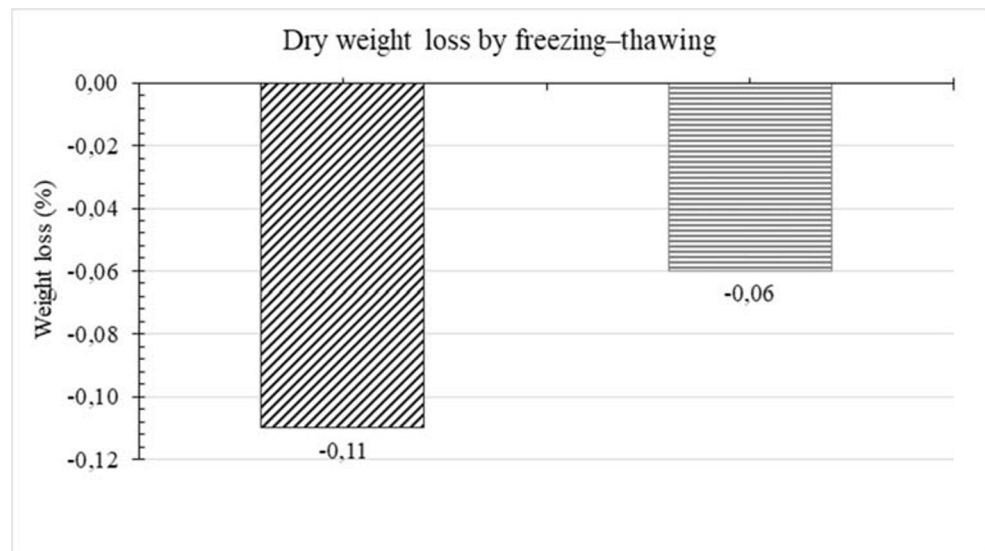
Some porous and weak stones may show cracking or spalling after cycles of freezing and thawing, whereas denser and less porous stones can survive the test visually unaffected. The durability of the building stone is assessed by the magnitude of the weight loss that occurs

after the test. The variation of dry weight loss of the untreated and hydrophobic treated andesite samples throughout freezing–thawing tests was calculated, and the results are given in Fig. 13. The decrease of dry weight loss in the untreated andesite samples after the freezing–thawing test was 0.11%, and the hydrophobic treated andesite samples showed a decrease of 0.06% (Fig. 13). It may be said that change limits of these data are very low, which can support the resistance of the andesite samples against cyclic freeze–thaw deterioration. According to the dry weight loss variation, this parameter was very slowly decreased, and it can be said that dry weight loss was very little affected after 56 cycles of the test.

#### The variation of ultrasound pulse velocity values due to freezing–thawing

Ultrasound pulse velocity of andesite samples was measured after freezing–thawing tests. Based on the results after the freeze–thaw cycles ultrasound pulse velocity (UPV) presented in Fig. 14, the untreated samples show a decrease of 2.89% in

**Fig. 13** The variation of dry weight loss with the freeze–thaw cycles of the untreated and hydrophobic treated andesite samples



ultrasound pulse velocity (UPV), whereas the decrease is 1.44% for the hydrophobic treated samples. The tested andesite samples exhibit a little decrease of ultrasound pulse velocity (UPV) after freezing–thawing tests. Ultrasound pulse velocity (UPV) variation for tested andesite indicates that some physical properties are not sensitive to deterioration and will not be damaged from freeze–thaw effects.

#### The variation of the uniaxial compression strength values due to freezing–thawing

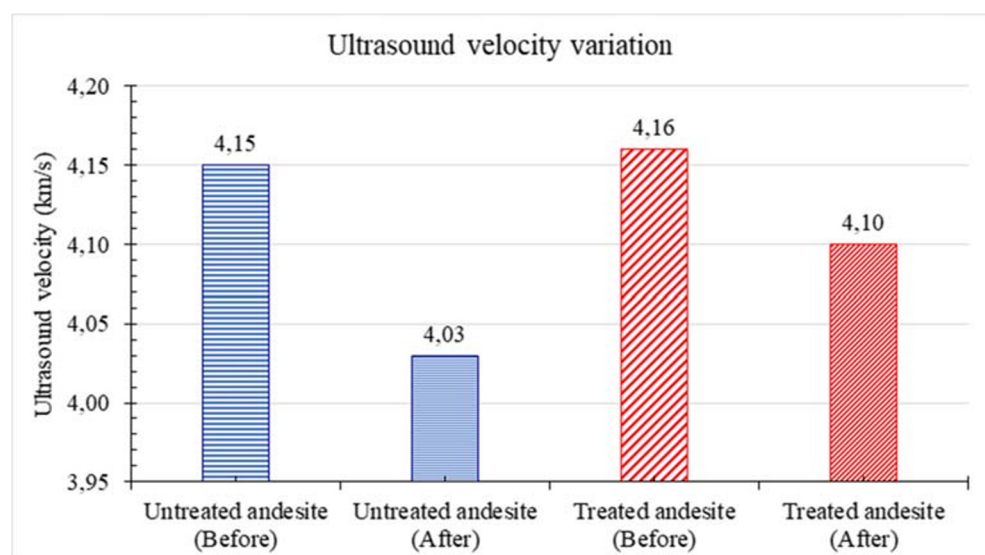
The deterioration mechanism of the freeze–thaw action is concerned with the tensile stress of occurred ice and deterioration in the micropores. This process may appear after numerous freeze–thaw cycles. It is well known that stone durability

decreases depending on increased freeze–thaw cycle. The deterioration mechanism of the cyclic freeze–thaw consists of three stages:

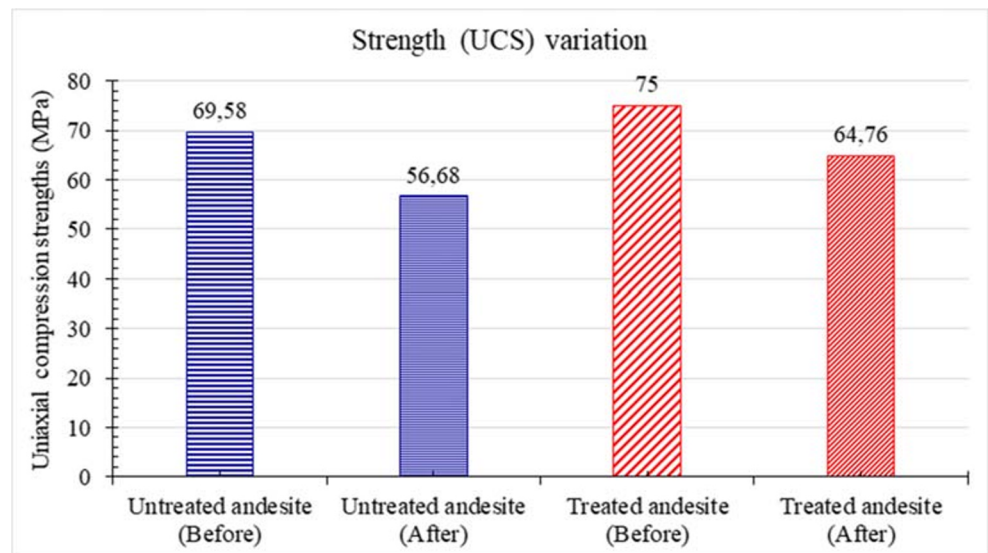
- Start phase (micro fissure formation)
- Uniform increased phase (stable crack propagation)
- Accelerated phase (unstable crack propagation results in sudden deterioration) (Xiao et al. 2010)

Results of the variation of the uniaxial compression strength (UCS) values after 56 cycles are given in Fig. 15. Based on the results after the freeze–thaw cycles uniaxial compression strengths (UCS) presented in Fig. 15, the untreated samples show 18.54% decrease in the uniaxial compression strengths (UCS), whereas the decrease is 13.65% for the

**Fig. 14** The variations of ultrasound pulse velocity (UPV) before and after the freeze–thaw cycles of the untreated and hydrophobic treated andesite samples



**Fig. 15** The variations of the uniaxial compression strengths (UCS) before and after the freeze–thaw cycles of the untreated and hydrophobic treated andesite samples



hydrophobic treated samples. The results show that the freeze–thaw action induced a somewhat decline of the uniaxial compression strengths (UCS) in both the untreated and hydrophobic treated andesite samples.

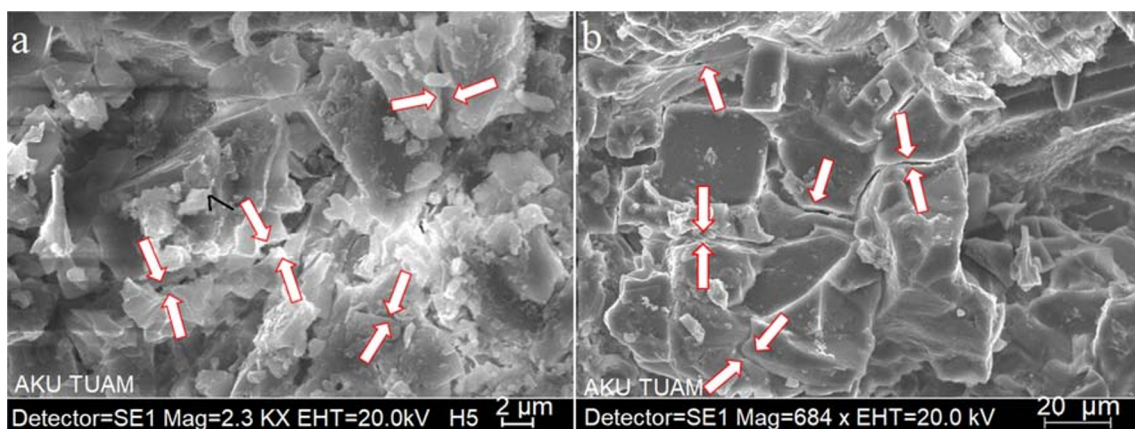
#### Microstructural analysis by SEM after freezing–thawing test

In the SEM studies of the tested andesite samples at the end of the freeze–thaw test, some micro fissures were observed (Fig. 16). In SEM studies, some pore and fragmented grains that were affected by deterioration processes were clearly observed. The come up micro fissures were not extended depending on low numbers of freeze–thaw cycles. As the freeze–thaw cycles increase, the deterioration begins depending on the stress in the pores. Therefore, the macroscopic decay

properties estimated will occur after a large number of freeze–thaw cycles for andesite samples.

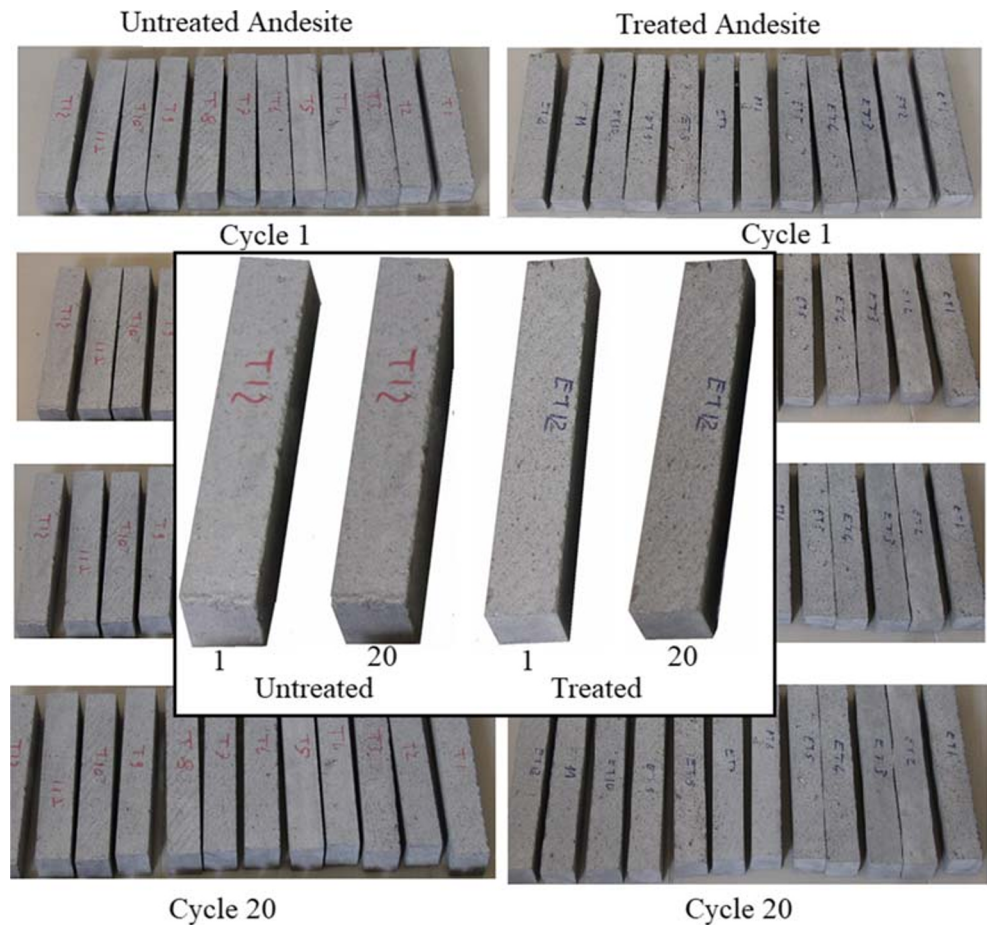
#### Results of thermal shock test

The basic reason for deterioration in thermal shock action is supposed to be because of different thermal expansion and contraction of the inner and outer area of the mineralogical components of the building stone. In some studies, it has been expressed that existence of humidity is a prerequisite for the emergence of thermal shock deterioration. The pore water heated during thermal shock process weakens the stone structure by moving the soluble minerals toward the rock surface (Hale and Shakoor 2003). The thermal shock test was carried out with the objective of examining the deterioration of the andesite samples after cycles of water absorption, heating, and



**Fig. 16** Scanning electron microscope images of andesite sample after the freezing–thawing test. Detail of freeze–thaw induced micro fissure on andesite surfaces after the freeze–thaw cycle. Red arrow indicates micro fractures within the andesite sample

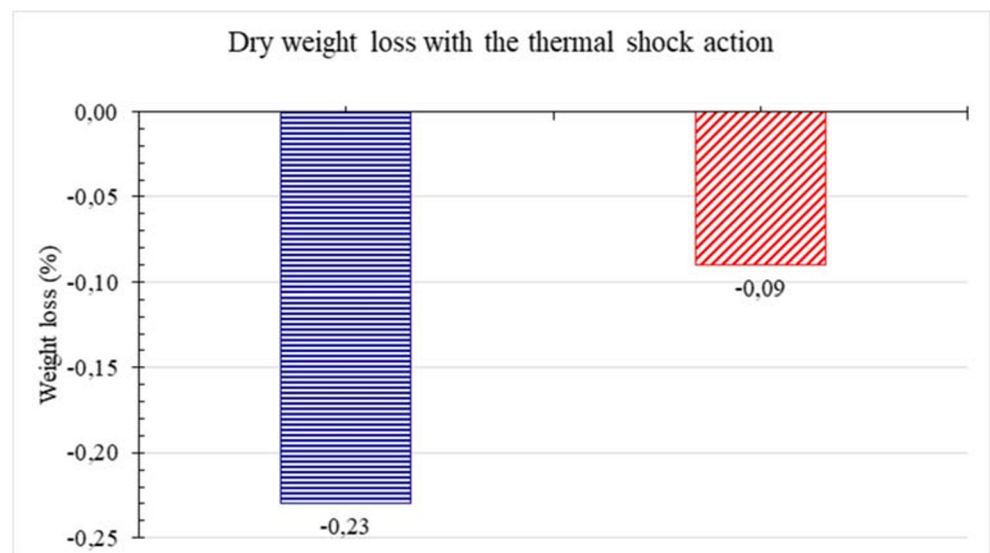
**Fig. 17** Macro views of the thermal shock action tests compared with the untreated and hydrophobic treated andesite samples



cooling action. In this study, the effect of the thermal shock process of andesites on the engineering properties was studied by some parameters such as dry weight loss, ultrasound pulse velocity (UPV), and flexural strength (FS) values after thermal shock cycles.

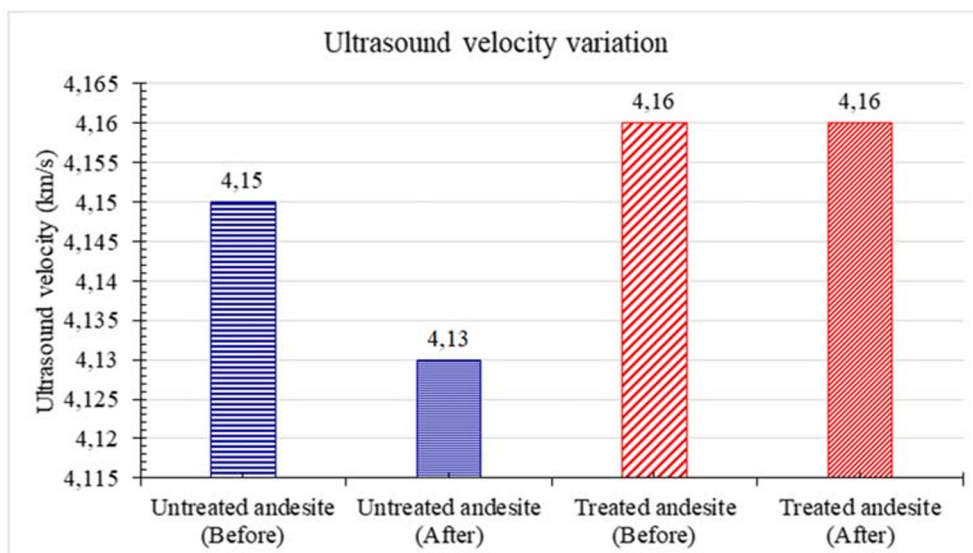
The macroscopic visuals of the untreated and hydrophobic treated andesite samples by the end of 20 cycles of thermal shock test are visible in Fig. 17. The tested andesite samples did not show a significant visual alteration after the thermal shock test. There is not visible

**Fig. 18** Variations of dry weight loss with the thermal shock action of the untreated and hydrophobic treated andesite samples





**Fig. 19** The variations of ultrasound pulse velocity (UPV) before and after the thermal shock action of the untreated and hydrophobic treated andesite samples



granular disintegration, and edge fracture in the tested andesite samples after thermal shock test.

**The variation of dry weight loss due to thermal shock action**

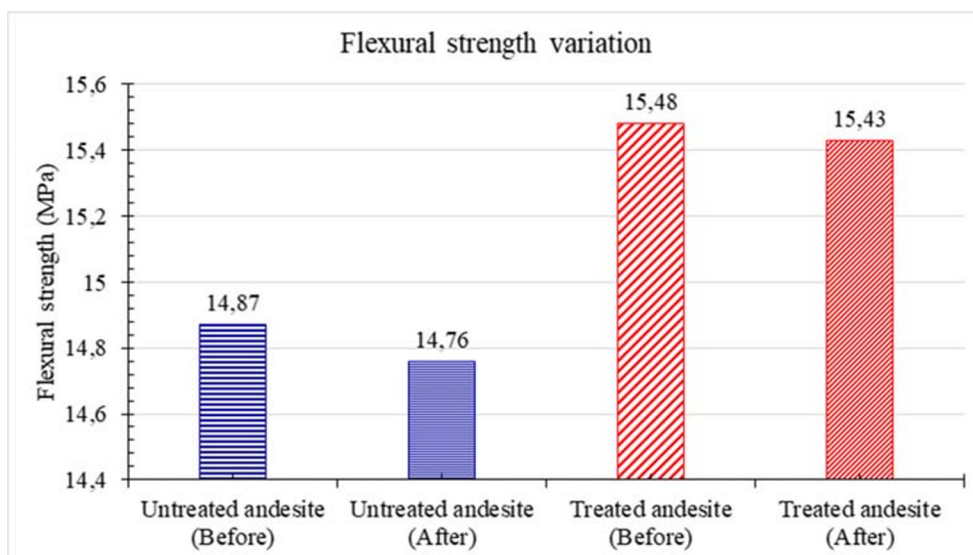
The variation of dry weight loss results obtained from thermal shock tests for the untreated and hydrophobic treated andesite samples is given in Fig. 18. The variation of dry weight loss in the untreated andesite samples was 0.23%, and the treated andesite samples showed a decrease of 0.09%. With the use of hydrophobic chemicals, it has been found that the dry weight loss decreases by more than 60%. The dry weight loss during the thermal shock test is related to both porosity and

fissure. The untreated andesite samples were more affected due to the open porosity they had than those treated samples.

**The variation of ultrasound pulse velocity values due to thermal shock action**

Ultrasound pulse velocity of andesite samples was measured after thermal shock tests. The ultrasound pulse velocity variations data are given in Fig. 19 including before and after the thermal shock action of the untreated and hydrophobic treated andesite samples. Thermal shock tests created new fissures that led to a decline in ultrasound pulse velocity. This was evident in the untreated andesite than the treated ones. It was

**Fig. 20** The variations of flexural strength (FS) before and after the thermal shock action of the untreated and hydrophobic treated andesite samples



seen that the treated andesite samples were less affected than the destructive effect of thermal shock compared with the untreated samples. The decrease of ultrasound pulse velocity values was found to be 0.48% for the untreated samples, and there were no changes for the hydrophobic treated samples.

### The variation of the flexural strength values due to thermal shock action

The variations in flexural strength (FS) after the thermal shock cycling values are given in Fig. 20. Based on the results presented in thermal shock test, the untreated and hydrophobic treated andesite samples showed small decreases in flexural strength (FS) after 20 heating–cooling cycles: 0.74% and 0.32%, respectively. The most plausible explanation of the reduction of ultimate flexural strength after thermal shock cycling that due to the tensile stress occurred micro fissures of the untreated andesite samples.

## Conclusions

The andesite samples for characterization were subject to petrographic analysis, chemical analysis, and laboratory tests to determine the physical and mechanical properties. Physical and mechanical properties of tested andesite were typically assessed through testing in accordance with the TS EN procedures: total and open porosity, bulk density, ultrasound pulse velocity, water absorption, uniaxial compressive strength, salt crystallization, cyclic freezing–thawing, and thermal shock. The pore size distribution of the andesite was measured using a mercury intrusion porosimeter. Andesite sample has a pore diameter distribution between 0.003 and 100  $\mu\text{m}$ . The pore size distribution of andesite is intense between 0.01 and 10  $\mu\text{m}$ .

The atmospheric and environmental conditions are the two important parameters in the deterioration of building stones. Freezing–thawing, salt crystallization, and thermal shock are commonly used accelerated weathering tests performed in laboratory. Andesite is commonly used for internal and external cladding of buildings. In cold climate, in order to determine the most suitable stone for the areas that are internal and external cladding of buildings, the deterioration value after salt crystallization, cyclic freezing–thawing, and thermal shock is investigated. According to the test results, decreases in some physical and mechanical values were found in the samples with weight loss. The salt crystallization test results indicated that the untreated andesite had a dry weight loss of 0.80%, while the hydrophobic treated andesite had an increase of 0.24%. The decrease of dry weight loss in the untreated andesite samples after the freezing–thawing test was 0.11%, and the hydrophobic treated andesite samples showed a decrease of 0.06%. After the thermal shock tests, the variation of

dry weight loss in the untreated andesite samples was 0.23%, and the treated andesite samples showed a decrease of 0.09% from thermal shock tests.

The decrease of the UCS in untreated andesite samples after the salt crystallization test was 32.08%, and the hydrophobic treated andesite samples showed a decrease of 37.31%. The untreated samples after the freezing–thawing test show 18.54% decrease in the UCS, whereas the decrease is 13.65% for the hydrophobic treated samples. The untreated and hydrophobic treated andesite samples showed small decreases in FS after 20 heating–cooling cycles: 0.74% and 0.32%, respectively.

The porous structure of a stone can be described by several parameters such as the open porosity and pore size distribution. The study of these characteristics is fundamental to understanding salty water transport within their network and the impact of salt crystallization. It is the crystallization–dissolution and remobilization of salts that lead to the development of damage.

The uniaxial compression strength and the ultrasound pulse velocity are known to be the best parameters that show weathering in the aging experiments such as salt crystallization, cyclic freeze–thaw, and thermal shock action. It was found that after the freeze–thaw cycles, the ultrasound pulse velocity and uniaxial compressive strength decreased in the untreated andesite samples, and also same values were determined a lesser decrease in treated samples. The deteriorations such as cracks and pores reducing the mechanical strength of the andesite were observed in SEM micro photos. Repeated cycles of the salt crystallization test and dissolution in pores led to micro fissure development. The fact that the micropores do not form enough cracks is believed to play a major role in the low damage that occurs in the aging tests of the tested andesite samples.

Stone buildings have a prominent position in historical and cultural buildings and monuments in all countries. After the aging experiments, it was determined that the andesites used as construction material can function for long years without any deterioration. However, more attention should be paid to its use in humid environments due to high porosity and water absorption properties. The information obtained after the aging experimental studies is very important in new constructions and restoration projects where andesites will be used as a building stone. The slightly degradation of the andesite has been mainly through edges and corners of the samples. According to the results of this study, the tested andesite may be suitable for exterior use in areas subject to frequent freeze–thaw cycles and the crystallization–dissolution of salts. For this reason, it is recommended that this building stone is used for decorative covering in the exterior of buildings.

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