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Analysis of the alterations in porosity features of some natural stones due to thermal effect

I. Ugur *, N. Sengun, S. Demirdag, R. Altindag

Mining Engineering Department, Suleyman Demirel University, 32260 Isparta, Turkey

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ABSTRACT

Three types of commercially available carbonate rocks were used in the study to determine the effect of thermal treatment in the range from 100 °C to 500 °C on porosity features in terms of two different approaches such as pore shape factor and quality index values. The ratio of the ultrasonic velocity measurements before and after water saturation was used to differentiate porosity of pores from porosity of cracks under varying temperatures. It was found that, pores in Burdur Beige and Usak White are in the form of cracks, which are situated through inner structure. On the other hand, pores in Patara Limestone are in the form of porosity with lower pore shape factor values. Quality index calculation is another approach based on the comparison of the measured and theoretical ultrasonic velocity values. When the rocks were subjected to higher temperatures, internal stress was developed, crack lengths and numbers were increased and finally the higher pore shape factor and lower quality index values were obtained. This situation was proven by the higher water absorption values for all the stone types with the higher pore shape factor and lower quality index values depend on the noticeable increase in effective porosity values.

1. Introduction

Many building materials, such as natural stones and many artificial construction materials (bricks, mortar, concrete, etc.) are generally composed of a certain volume of empty space in the form of pores, cavities and cracks of various shapes and sizes [1]. These empty spaces are primary concern of the material since their volume, size and distribution affect their behavior under various types of weathering phenomena in aggressive environments (acid rain-related attack and dissolution, salt crystallization, and freeze–thaw cycles, etc.) when used for structural and ornamental purposes [2]. Since the water is the main weathering agent, open pores and micro-cracks have considerable effects on the fluid storage and circulation capacity within the building material inevitably favoring its deterioration and lowering the mechanical resistance [3].

The deterioration is a function of the rock properties and the external outdoor conditions [4]. Rocks, can be defined as solids composed of different types of minerals joined to one another in a tight fabric. So, it can be assumed that the mineralogical composition and the texture are the two most important properties of rocks. Thermal behavior of the stone material especially for carbonate stones is strongly connected to the mineralogical composition and the texture, which is one of the most serious causes of degradation [5–7].

There are several significant studies about the decomposition of the elements that form the structure, such as the effect of high temperature and fire on concrete [8,9], high performance concrete [10,11], lightweight concrete [12,13], mortar [14–16], steel [17,18], clay bricks [19] and engineered stone [20]. The natural weathering and behavior of natural stones have been intensively studied recently but few attempts have been made to understand the behavior of these stones at high temperatures. Studies on the effect of temperature on natural stones are usually based on sandstone [21–24] and granite [25–27]. Researchers have made some experimental studies on the effect of high temperatures around 500 °C in the oven on rock weathering in laboratory conditions in terms of some petro-physical and chemical properties [28–30]. Since extreme high temperatures such as fire have the potential to cause the new crack formation, the knowledge of the variations in types of empty spaces is an important parameter for the evaluation of its behavior in contact with water and therefore predicting their real behavior under weathering conditions.

The primary objective of this work was to assess the alterations in cavities in terms of both porosity of pore and porosity of crack structure due to degradation of three carbonate rocks under the

* Corresponding author. Tel.: +90 2462111310; fax: +90 2462370859.
E-mail address: ibrahimugur@sdu.edu.tr (I. Ugur).

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effect of high temperatures in the range from 100 °C to 500 °C. For this purpose, two different approaches such as pore shape factor (PSF) and quality index (QI) values were used based on P-wave velocity (*Vp*) measurements before and after water saturation.

2. Experimental work

2.1. Sample description

Even though there is a wide range of natural building stone types, the study focused on three types of natural carbonate stones selected based on their different textural features. The samples of the fresh stone types were purchased from the working quarries and extraction of the test specimens was performed by cutting operation using diamond saws to obtain cubic samples having edge dimension of 7 cm from the fresh blocks in the laboratory. Through the testing procedure, totally 12 samples belong to three different stone types and so 4 samples of each were used during the physical and thermal tests without changing the samples to take more accurate results and to avoid natural irregularities of the samples. Since the exact orientation of the layers in the specimens for the selected rocks are not clear and almost isotropic structure, orientation was not taken into consideration during the testing procedure. The general descriptions of the rocks used in the analyses are summarized in Table 1.

2.2. Thermal treatment and testing procedure

The specimens were gradually heated in an oven at atmospheric pressure with a rate of 10–12 °C/min for 5 different temperatures (100, 200, 300, 400 and 500 °C) to induce damage in the stone matrix for 3 h. This rate of heating was used to ensure that cracking events result only from the temperature effect. The selection of the temperatures around 100 °C was aimed to imitate the conditions that could be found on natural stone claddings outdoors during summertime. Other temperatures were applied for the observation of the stone materials under extreme conditions such as fire and heat release after the deposition of radioactive wastes. Cooling treatment of the rock samples was performed in the oven by the rate of 1–2 °C/min, while the oven is totally enclosed. So, cooling of the samples may be obtained slowly and it took approximately 24 h to bring normal room conditions without being under the influence of thermal shock. The samples were tested to determine the ultrasound propagation before and after the thermal treatment for each temperature under dry and water saturated conditions.

2.3. Physical properties of rock

Physical properties of the untreated and thermally treated rock samples were determined for each temperature according to relevant standards described in the literature.

2.3.1. Porosity and bulk density

The total porosity (P_t (%)) and bulk density (d_b (g/cm^3)) of rock samples were determined using saturation and buoyancy techniques, as recommended by ISRM [31] and TSE [32]. After drying the all samples to constant mass (M_1) (at 60 °C), they were placed in a water bath during 48 h and periodically agitated to eliminate the air contained in the pores. Later, the saturated samples were then weighed in water with a scale having 0.01 g accuracy (hydrostatic weighing: M_2) and in air (M_3). The apparent porosity, (P_a (%)) is expressed as the ratio of the volume of the pores accessible to water to the bulk volume of the sample (Eq. (1)).

$$P_a = \frac{M_3 - M_1}{M_3 - M_2} * 100, \quad (\%) \tag{1}$$

The bulk density was determined as the ratio of the dry mass to the bulk volume of the sample (Eq. (2)).

$$d_b = \frac{M_1}{M_3 - M_2}, \quad (\mathrm{g/cm^3}) \tag{2}$$

The real density (d_r (g/cm^3)) corresponds to the ratio of the mass to the impermeable volume of the sample. In this case, the weight of the pycnometer filled only with water was established. The dry, pre-weighed 0.2 mm grain size crushed rock sample was then added to the pycnometer and the rest was filled with water. The real densities of the rock samples were then calculated from the known density of the water; the weight of the pycnometer filled only with water (M_{pw}); the weight of the pycnometer containing both sample and water (M_{psw}); and the weight of the sample (M_s) (Eq. (3)).

$$d_r = \frac{M_s}{M_{psw} - M_{pw}}, \quad (\mathrm{g/cm^3}) \tag{3}$$

Finally, effective porosity was calculated from Eq. (4).

$$P_e = \left(1 - \frac{d_b}{d_r}\right) * 100, \quad (\%) \tag{4}$$

2.3.2. P-wave velocity

In this study, *Vp* was used as the P-waves are more stable and may give more accurate information about many of the properties of the material such as porosity, the degree of degradation, the homogeneity of the material, the presence of discontinuities (joints, fractures) in a non-destructive way. P-wave transmission method by Pundit Plus tester consists of placing two piezoelectric transducers (a pulser and a receiver) on two opposite and parallel sides of the samples. The travel time is measured through the exact distance between the transducers and the wave propagation velocity was calculated. A special water based transmission gel was used to achieve good coupling between the transducers and to obtain reusability of the same samples by washing to avoid heterogeneity of the samples which may negatively affect the test results. Since the velocity is generally influenced by the temperature and water content, the conditions were tried to be kept constant during the P-wave velocity measurements. P-wave velocity measurements

Table 1
The general descriptions of the rocks.

Rock name	Rock type	Genesis	Petrographic description
Patara Lymra (PL)	Limestone	Sedimentary	It is heterogeneous in mineralogical composition but homogeneous in structure and texture with common pores. It is composed of mainly intraclasts with micritic calcite. There are some clay matrix and scarce organic material
Burdur Beige (BB)	Limestone	Sedimentary	Sparitic limestone. It is homogeneous in mineralogical composition with mainly moderate crystalline calcite, heterogeneous in structure, contain opaque minerals and too many micro-cracks
Uşak White (UW)	Marble	Metamorphic	Granoblastic texture. It is homogeneous in composition, texture and structure. Mineralogical composition is almost coarse–moderate crystalline calcite and small amount of opaque minerals