REPUCLIC OF TURKEY
MUGLA SITKI KOÇMAN UNIVERSITY
GRADUATE SCHOOL OF
NATURAL AND APPLIED SCIENCES

DEPARTMENT OF MINING ENGINEERING

INFLUENCE OF THERMAL DAMAGE ON PHYSICO-MECHANICAL PROPERTIES OF CARBONATE ROCKS: POROSITY, HARDNESS, UCS AND ULTRASONIC WAVE EVOLUTIONS

MASTER OF SCIENCE

ELİF AKGÜL

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APPROVAL OF THE THESIS

The thesis submitted by **ELİF AKGÜL** with the title of **"INFLUENCE OF THERMAL DAMAGE ON PHYSICO-MECHANICAL PROPERTIES OF CARBONATE ROCKS: POROSITY, HARDNESS, UCS AND ULTRASONIC WAVE EVOLUTIONS"** has been unanimously accepted by the jury members on the date, 2019 to fulfill the requirements fort he degree of Master of Science in the Department of Mining Engineering.

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I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conducts, I have fully cited and referenced all metarial and results that are not original to this work.

Elif AKGÜL

30/05/2019

ÖZET

KARBONATLI KAYAÇLARIN FİZİKSEL-MEKANİKSEL ÖZELLİKLERİ ÜZERİNDE TERMAL ŞOK İŞLEMİYLE YIPRANMANIN ETKİSİNİN İNCELENMESİ: POROZİTE, SERTLİK, TEK EKSENLİ BASINÇ DAYANIMI VE *P*- DALGA ÖLÇÜMLERİ

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Bu çalışmada, Mugla yöresinde bulunan farklı karbonat kayacı olan mermer türlerinden alınan örnekler üzerinde 100°C'de ısınma-soğuma (termal şok) ve 25, 100, 200, 300, 400 ve 500°C'de yüksek sıcaklığa maruz kalma deneyleri yapılmıştır. Örnekler 2 farklı kategoriye ayrılarak ilk deney setinde 5 farklı mermer örneği üzerinde öncelikle termal şok deneyleri yapılmıştır. Daha sonra, aynı tür mermer örnekleri 25, 100, 200, 300, 400, 500, 600, 700 ve 800°C sıcaklıklara kadar ısıtılmışlardır. İkinci deney setinde 3 farklı mermer örneği kullanılmıştır. Bu örnekler 25, 100, 200, 300, 400 ve 500°C sıcaklığa maruz bırakılmışlardır. Sıcaklık işlemlerinin uygulanmasından sonra, örnekler üzerinde Shore sertliği, *P*-dalga hızı, gözeneklilik, birim hacim ağırlığı ve tek eksenli basınç (TEB) dayanımı deneyleri yapılmıştır.

Termal şok deneyinden sonra yapılan ölçümlerde örneklerin yoğunluk, *P*-dalga hızı, Shore sertliği ve tek eksenli basınç dayanımlarında azalma gözlenirken, toplam porozite değerlerinde artış gözlenmiştir. 800°C sıcaklığa ısıtılan örneklerin ağırlık, *P*-dalga ve Shore sertliği değerlerindeki düşüşler ise termal şok deneyinin etkisinden çok daha fazla olmuştur. İkinci deney setinden sonra yapılan ölçümlerde ise örneklerin ağırlık, *P*-dalga hızı ve Shore sertliği değerlerinde azalma görülürken, toplam porozite değerleri artmıştır.

Yapılan deneyler sonucunda yüksek sıcaklığın mermer örneklerinin üzerinde, sadece fiziksel bozunmadan dolayı oluşan etkiler incelenmiş ve değerlendirilmiştir.

Anahtar Kelimeler: Mermer, Termal Şok, Termal Hasar, Isınma-Soğuma

ABSTRACT

INFLUENCE OF THERMAL DAMAGE ON PHYSICO-MECHANICAL PROPERTIES OF CARBONATE ROCKS: POROSITY, HARDNESS, UCS AND ULTRASONIC WAVE EVOLUTIONS

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Master of Science (M.Sc.)

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In this study, heating and cooling (Thermal shock) tests were conducted at temperature of 100°C and thermal damage tests were carried out at temperatures of 25, 100, 200, 300, 400, 500, 600, 700 and 800°C on different carbonate rocks sampled from various marble types quarried in Mugla Region. The samples were divided into 2 different categories: On the first set of experiments, thermal shock tests were performed on 5 different marble types. The samples of same type of marble were then heated to the temperature steps of 25, 100, 200, 300, 400, 500, 600, 700 and 800°C for thermal damage. On the second set of experiments, the samples of three different marble types were tested. The samples were heated to the temperature steps of 25, 100, 200, 300, 400 and 500°C for thermal damage. Shore hardness, *P*-wave velocity and the weight loss tests were conducted following each temperature step. Porosity, density and uniaxial compressive strength (UCS) tests were implemented following the final temperature step of 500°C.

In the end of thermal shock tests, density, *P*-wave velocity, Shore hardness and uniaxial compressive strength values seemed to decrease and total porosity values tended to increase. In the end of thermal damage tests at temperature of 800°C; the decreases in weight, *P*-wave and Shore hardness values were seemed to be much higher than that of thermal shock tests. In the end of second set of experiments, *P*-wave velocity, Shore hardness and the weight were decreased and the total porosity was increased.

In the end of experimental works, effects of physical weathering solely on marble samples due to high temperatures were investigated and assessed.

Keywords: Marble, Thermal Shock, Thermal Damage, Heating-Cooling

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LIST OF ABBREVIATIONS

SH	Shore Hardness
UCS	Uniaxial Compressive Strength

1. INTRODUCTION

Rocks contain many micro cracks and holes in their structures. In addition, heating and cooling process can lead to numerous micro cracks in the internal structure of the rock. The development and coalescence of the cracks is caused by the deterioration of rock formation and the change of physical and mechanical parameters. Thermal damage of the rock has long been investigated by many researchers and so far, many studies have been reported on this subject.

This thesis is an experimental investigation on thermal damage and thermal shock characteristics of marble and underlying mechanism. Laboratory tests have been conducted on two different experimental sets. All the samples were collected from Mugla region, to investigate index properties of deteriorated rocks due to physical weathering. On the first set of experiments, 90x90x90 mm^3 cubic samples of five different marbles were used on thermal shock and thermal damage experiments. Rock samples were heated to specific temperature levels of 25, 100, 200, 300, 400, 500, 600, 700 and 800°C for thermal damage experiments. Thermal shock action for 45 cycles was simulated at the temperature of 105°C, by complying with the standard suggested by TS EN 14066. On the second set of experiments, three different marbles were used on thermal damage experiment. 50x50x50, 90x90x90 and 150x150x150 mm^3 cubic rock samples were heated to a specific temperature levels of 25, 100, 200, 300, 400 and 500°C. Density, porosity, uniaxial compressive strength, P-wave velocity and Shore hardness tests were conducted on the samples to determine the evolutions in the values under thermal shock end thermal damage condition.

2. LITERATURE RESEARCH

2.1. Marble

Marble is known as the type of rock composed by metamorphism of sedimentary carbonate rocks, mostly of limestone or dolomite type. The metamorphism causes the original carbonate mineral grains to change and re-crystallize. The resulting marble rock is typically from a series of carbonate crystals which interlock with each other. Structures of primary sedimentary textures and original carbonate rocks (protolith) are typically destroyed or modified.

The result of metamorphism of a very pure (silicate-poor) limestone or dolomite protolite is formed by the formation of pure white marble. The characteristic veins and curves of many types of colored marbles originate from various types of minerals, such as clay, sand, silt or iron oxides, which are usually found in layers or grains within the limestone. Green coloring is generally the result of serpentine originating from magnesium-rich limestone or silica dosed dolostone. With the intense pressure and temperature of the metamorphism, these various impurities are mobilized and recrystallized.

Commercial definition of marble is, any type of stone (sedimentary, magmatic and metamorphic) that can give square block in accordance with commercial standards, can be cut, polished or surface treated and stone properties (material properties) conforming to the coating stone. According to this definition, limestone, travertine, sandstone, such as sediment; gneiss, marble, metamorphic like quartzite; granite, syenite, serpentine, andesite, basalt, such as magmatic stones are also called marble. Marble is generally used for sculpture and as a building material (MTA).

2.2. Heating and Cooling

Rocks have many natural micro cracks and holes in their internal structures and have a porous media. Heating and cooling also causes a large number of micro cracks inside the rock (Liu and Xu, 2013). Internal rock structure deteriorates with the increase and coalescence of cracks and this phenomenon changes the physical and mechanical parameters. High temperature cause thermal damage inside the rock and it has been investigated in many studies. Dougill et al. (1976) are the first to incorporate the damage mechanism into the investigation of rock material. Then, in the studies conducted by Dragon and Mroz (1979) and other scholars, continuous rock and concrete damage was investigated based on the concept and method of damage mechanics and continuum medium mechanics models were established. Many studies have been reported on rock damage up to now. Alm (1985) studied the mechanical properties of heated granite samples and discussed the increase of microcrack; Lau and Jackson (1995) investigated the changing law and failure criterion of elastic modulus, Poisson's ratio and compressive strength of granite versus temperature under the condition of low confining pressures; Homandetienne and Houpert (1989) determined the length, width, shape and density changes by applying heat treatment to Senones and Remirement granite samples with the highest temperature up to 600°C and then investigated the qualitative and quantitative effect of microstructural damage on mechanical properties; Wang and Bonner (1989) have used the acoustic emission technique to systematically examine the evolution and mechanism of thermal cracking development in granite samples found in western America; Liu et al. (2001) investigated the change laws of the main mechanical parameters of granite at high temperature (20-600°C); Du et al. (2004), studied the change in peak strain, peak strength, stress–strain, and Poisson's ratio during the thermal damage of granites after experiments at different high temperatures. In addition, it was shown in other studies that the high temperature effect could damage the internal structure of the rock (Brotons et al. 2013; Ferrero and Marini 2001).

2.3. Some Studies About Thermal Shock and Thermal Damage – Literature Research

Hudec (1998)'s study outlined the quantitative relationships between physical rock outlined above and their response to rapid weathering and engineering tests.

Texture of the marble samples, which were collected from six different places, were investigated in a study by Siegesmund, Weiss and Tschegg (2000). The textures with different strength and type were selected and investigated. The directional dependence of the experimentally determined dilatation coefficient was clearly controlled by the texture, and therefore this is predictable from tissue measurements. It had been observed that there was a residual strain on some samples from the first cycle of the high temperature process. However, the residual stress was not completely dependent on the type and strength of the texture. Also, basically, thermal dilatation coefficient and directional dependence of residual tension are large in marbles with strong texture. The marble sample with a different texture used in the study showed a greater residual strain parallel to the maximum dilatation direction than parallel to the minimum dilatation direction. However, another type of marble with a weak texture exhibited a uniform crack formation. It should be known, that by taking only the texture into consideration, no definitive clue can be obtained for large or small residual strains and their directional dependence. There are also marbles with strong or weak texture and having no residual stress. Two different types of marble with a strong texture and a weak texture showed no residual stress. These samples were characterized by a fabric with an irregular grain shape geometry and their grain size was obviously different. Therefore, in the study, a comprehensive approach had been made by evaluating the quality and durability of a marble as a building stone.

Zeisig, Siegfried and Weiss (2002) studied eighteen different types of marble. They were selected experimentally to determine the effect of heating and cooling in the temperature range of 20°C to 85°C. Three different cycles were performed at 40°C, 60°C and 85°C. The composition of marbles differed from calcitic to dolomitic. While the average grain size ranges from 50 μm up to 3 mm and they have different structure in terms of grain boundary geometry. After the heating and cooling procedure they classified the marbles in three different types: Type I showed by an

isotropic thermal expansion (α) and large isotropic residual strain (permanent length changes); Type II was characterized an anisotropic α and no or small isotropic residual strains; while Type III exhibited an anisotropic α and anisotropic residual strain. Most samples had deteriorated due to thermal treatment, and this could not be explained without taking account of the rock fabrics. In this study it was determined that thermally induced microcracks caused a residual stress after the heat treatment and thus the quality of the rock deteriorated, however, it has been found that the fabric cannot be reduced to one or a few parameters (eg only grain size, grain shape, etc.), and the thermal degradation of a marble was determined by the interaction of all fabric parameters.

Malaga-Starzec et al. (2002) examined the porosity changes of a calcitic and a pure dolomitic rock, depending on the temperature changes for the two types of marble. Samples were exposed to increasing temperatures between 40 and 200°C. The results showed that the interparticle adhesion began between 40 and 50°C. Some important differences in the temperature response for these two types of marble were distinguished. In summer, the temperature of 40 to 60°C is easily reached on building surfaces in most European countries. As a result of this study, it was observed that the granular adhesion process for some marble types could start at temperatures between 40 and 50°C. The calcitic marble analyzed showed more sensitivity to temperature changes than dolomitic marble. Changes in surface area and average pore size were found to be highly variable depending on various factors such as the original pore structure, crystallographic and mineralogical properties of the marble types and temperature variability.

Ruedrich et al. (2002) studied the mechanisms of weathering in marbles and the control of the mineralogical composition and the rock fabric in order to optimize stone consolidation. They compared the behavior of weathered and consolidated marbles to verify whether consolidation affected the thermal behavior of marbles. For the research, four marbles with different fabrics (eg texture, grain size, grain boundary geometry, etc.) and different weather conditions were selected. And three consolidation approaches have been selected: a solved polymethyl-methacrylate ($PMMA_{sol}$) dissolved in xylenes, a polysilicic acid ester (PSAE) and a total impregnation with a monomer methyl-methacrylate ($PMMA_{poly}$). Measurements of

the porosity and effective pore size distribution proved a strong modification of the pore space by consolidation. Both PMMA approaches exhibited a re-establishment of cohesion which could be defined by ultrasonic velocity values. By reaching the respective glass transition temperatures of $PMMA_{sol}$ and $PMMA_{poly}$, a strong modification of thermal action happened. The PSAE consolidated marbles exhibited only minor changes of dilatation, but due to its low bonding effected no-significant cohesion between the crystals occurs.

Mutluturk, Altindag and Turk (2004) experimented on different rock types and tested effects of freezing and thawing, and heating and cooling. Rock samples lose their integrity under these cyclic temperature changes and the more frequent and severe these cycles are, the higher the loss of integrity. And, the values of progressive disintegration were not the same for different rock types. In this study, a mathematical model defining the process of loss of integrity was presented if a rock was exposed to recurrent cycles. The model suggests a first order process and provided meaningful parameters for the integrity characteristic of the rocks. The validity of the model was determined in an experimental laboratory study on 10 different rock types. The model offers many meaningful parameters for rock durability or rock disintegration which could be used profitably for engineering evaluations. Another result of this study is that rock types do not provide any clue for rock durability under repetitive freezing-thaw and heating-cooling cycles.

Yavuz et al. (2006) studied on 12 different carbonate rocks. They carried out freeze-thaw and thermal shock experiments for 20 cycles by using standard procedures, and the index properties of the rocks deteriorated by physical decomposition were investigated. Index properties, Schmidt hardness, P-wave velocity and uniaxial compressive strength were defined for three series of rock samples. It was determined that the index properties of the rock samples with the thermal shock and freeze-thaw decreased at varying levels according to the initial rates. A model equation which predicted the index properties of rocks due to thermal shock and freeze thaw process had been developed by multiple regression analysis. This model showed decreasing of the index property of a rock that had deteriorated depending on the initial properties for both thermal shock and freeze-thaw operations and on the porosity of the rock for a given index characteristic. Model was confirmed by

statistical analysis. The final model equation could precisely give notice a feature of a deteriorated carbonate rock depending on heating process, and initial index property and porosity.

Chaki et al. (2008) examined the characterization of porosity and the parameters of total damage in thermally cracked granite rock, in order to evaluate, transport properties and mechanical strength were defined and measured respectively. Samples were heated to 600°C under a certain pressure. The classification was made by measuring gas permeability, velocity, porosity and ultrasonic pulse velocity. This work showed the strong effect of thermal damage on physical properties, and indicated obviously the potential of the previous methods in connection with this type of damage. They showed that the porosity evaluation only informed about open porosity, which was distributed and presented at rock surface, whereas the gas permeability evaluation characterized the connected porosity. As for the ultrasonic pulse velocity distribution, it was sensitive to overall damage in the material. Even so, they noticed that the three methods were similarly and complementary defined the rock behavior in each stage of heating process; it was shown that there was a good consistency between them.

Takarli et al. (2008) investigated the effects of temperature on the physical properties and mechanical behavior of two different granite rock samples by applying thermal shock and freeze thaw experiments. Relating to the physical properties, ultrasonic pulse velocity, open porosity, and permeability were determined in fresh condition and after each 25 freeze thaw cycles (between -20 and +20°C). described the granite samples and the measurement methods used to define the microstructural changes and the results on the mechanical behavior. The permeability and the ultrasonic pulse velocity evolutions were carried out before and after each 25 freeze thaw cycles (20°C/+20°C). The porosity measurement was determined only at the beginning and at the end of the test (300 freeze thaw cycles). The study of the effect of the microstructural varieties on the mechanical behavior was accomplished by measuring: Young modulus, permeability change, deformations, ultimate strength, and acoustic emission (AE) in uniaxial compressive experiment.

Yavuz et al. (2010) investigated the effect of thermal damage on the physical properties of five carbonate rocks. The experiments were managed on three

limestones and two marbles, mostly composed of calcite but with different grain sizes, porosities, structural and textural characteristics. These samples cut into cubic shape and were slowly heated to a specific temperature level of 100, 200, 300, 400 and 500°C, and slowly cooled down to room temperature without causing thermal shock in order to examine the effect of heating temperature on physical properties such as effective porosity, bulk density, microstructure and the ultrasonic pulse velocity. Microscopic analysis from thin sections exhibited that damage in rocks at high temperatures was induced in different intensity depending on porosity, grain size, textural and structural characteristics. Color variations were also seen in porous limestones due to organic material. According with the degree of calcite dilatation depending on heating temperature and in turn new microcrack occurrence, separation along intragrain and/or intergrain boundaries and widening of existing cracks, the ultrasonic pulse velocity decreased to various levels of the initial value, while porosity increased. Microscopic investigations and the ultrasonic pulse velocity evaluations showed that compaction of rock structure up to 150°C consisted and induced calcite dilation had no significant damage effect on the rock sample. Compaction of rock structure led to a rise in the ultrasonic pulse velocity and weak reducing in porosity. Most of the damage happened within 24 hours of heating time and further heating processes brought relatively slight changes in physical properties. Damage intensity was well described with the ultrasonic pulse velocity and effective porosity rates depending on temperature increase.

Sygała et al. (2013) studied the current situation of information regarding the investigation of the effect of high temperature on changes of geomechanical properties of rocks. Based on data from previous works, the form of stress-strain properties that described the procedure of the destruction of rock samples as a result of load impact under uniaxial compression in a testing machine, were studied. The results from the investigations on changes in the basic strength and elasticity parameters of rocks, such as Young's modulus and the compressive strength were compared. Fundamentally, it was seen that temperature has an important effect on the change of geomechanical properties of rocks. The basic of these changes also depended on other efficients (apart from temperature) which were the porosity, density and the mineral composition of rock. The investigations exhibited that changes in the rock by heating it at various temperatures and then uniaxially loading

it in a testing machine, were different for different rock types. Most of the important tests that cause changes in the rates of the strength parameters of the analyzed rocks appeared in the temperature range of 400 to 600°C.

Demirdag (2013) studied application of cement filling method in travertine and the effects of thermal shock and freeze–thaw cycles on the rock structure were experimentally examined. Unfilled and filled travertines were compared based on the technical data of rock parameters pre- and post-thermal shock and freeze–thaw cycles. These travertine samples were cut into the form of 40cm x 40cm x 2cm. The travertine tiles were prepared and according to related standards they analyzed in terms of unit volume weight, open porosity and point load strength index evaluations. According to experimental results, it was attepmted to compare the filled and unfilled samples' properties such as physical and mechanical parameters of rock at the end of each period of thermal shock and freeze–thaw for 10, 20, 30, 40 and 50 cycles. The results exhibited remarkable effects of rock weathering level on mechanical resistance of filled travertine than unfilled travertine after freeze–thaw and thermal shock cycles. Freeze–thaw tests were seen to have more destructive effect on the porosity than thermal shock.

Sassoni and Franzoni (2013) examined a new methodology to artificially deteriorate stone samples by heating, using the anisotropic thermal deformation of calcite crystals, had recently been suggested. In this work, the heating impacts on a variety of lithotypes were estimated and the influence of porosity in defining the actual heating effectiveness was specifically examined. One marble and four limestones, having comparable calcite amounts but very different porosity, were heated at 400°C for 1 hour. A systematic comparison between porosity, pore size distribution, water absorption, sorptivity and ultrasonic pulse velocity of unheated and heated samples was accomplished. The results of the investigation showed that the initial stone porosity showed a very important role, as the modifications in microstructural, physical and mechanical properties were way less pronounced for increasing porosity. Heating was thus confirmed as a very promising artificial deterioration method, whose effectiveness in producing alterations that suitably resembled those actually experienced in the field depends on the initial porosity of the stone to be treated.

Brotóns et al. (2013) discussed the results from experiments which were performed in order to study the effect of high temperatures in the physical and mechanical properties of a calcarenite. Samples were heated at different temperatures between 105-600°C. Non-destructive experiments (porosity and ultrasonic wave propagation) and destructive experiments (slake durability and uniaxial compressive strength test) were carried out on the samples. Also, the experiments were performed under different conditions (i.e. water cooled and air cooled) in order to examine the effect of the fire off method. The results showed that uniaxial compressive strength and elastic parameters (i.e. elastic modulus and Poisson's ratio), decrease as the temperature increases for the tested range of temperatures. A reduction of the uniaxial compressive strength up to 35% and 50% was observed in water cooled and air cooled samples respectively when the samples were heated to 600°C. Regarding the Young's modulus, a fall over 75% and 78% in air-cooled and water-cooled samples respectively was observed. Poisson's ratio also declined up to 44% and 68% with the temperature in air-cooled and water-cooled samples respectively. Slake durability index exhibited a reduction with temperature. Other physical properties, closely related with the mechanical properties of the stone, are porosity, attenuation and the ultrasonic wave velocities of in the rock. All evidence significantly changed with temperature.

Ugur et al. (2014) studied on three types of carbonate rocks to establish the effect of thermal treatment between 100-500°C on porosity characteristic in terms of two different approaches such as pore shape factor and quality index rates. 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 seen that, pores in Burdur Beige and Usak White are in the form of cracks, which were situated through inner structure. Also, pores in Patara Limestone were in the form of porosity with lower pore shape factor rates. Quality index evalution was another approach based on the comparison of the calculated and theoretical ultrasonic velocity values. When the rocks were subjected to higher temperatures, internal stress was increased, crack lengths and numbers were developed and finally the higher pore shape factor and lower quality index rates were obtained. It was proven by the higher water absorption rates for all the stone types with the higher

pore shape factor and lower quality index rates depend on the noticeable development in effective porosity rates.

A series of unconfined compressive strength tests was conducted on granite samples by Shaoet al. (2014). Samples with three different grain sizes (fine grained, medium grained and coarse grained) were first heated to four different temperatures (200°C, 400°C, 600°C and 800°C) and then allowed to cool down before examining at two different cooling stages, slow cooling by keeping the samples in the atmosphere and rapid cooling by immersing the heated samples in a water bath. Fine grained granite did not exhibit visible macro-scale thermal cracks after cooling treatment and medium grained granite exhibited thermal cracks for the samples heated to 800°C temperature under both cooling conditions of which the rapidly-cooled sample had failed only by the cooling process. Coarse grained granite samples, heated to 400°C temperature and above, displayed thermal cracks after rapid cooling and the samples heated to 800°C temperature had failed only by the rapid cooling. Besides that, a consistent colour change with rising temperature was observed for all samples where the color had changed from white and grey to reddish from the pre-heated temperature of 200°C to 800°C, irrespective of the cooling condition.

Zhang, Sun, Hao and Wang (2016) studied an experimental investigation on the thermal damage features of limestone and underlying mechanism. Cylindrical rock samples were heated to a specific temperature level of 25, 100, 200, 300, 400, 500, 600, 700, 800, and 900°C. Then the thermal damage evolution equation was determined based on the experimental results, and the characteristics of thermal damage were investigated. Possible mechanisms for the observed thermo-physical and mechanical response were discussed. The results show that with the increase of temperature in the experimented range of temperature, the ultrasonic pulse velocity, peak compressive strength and elastic modulus decreased, but the peak strain increased; the damage factors developed faster between 200–600°C; the increase of high temperature induced cracks conformed to the dislocation theory; the decomposition of magnesium carbonate and dolomite was the main reaction in the experimented temperature range.

Peng, Rong, Cai, Yao and Zhou (2016) studied the physical and mechanical behaviors of a thermal-damaged coarse marble in uniaxial compression experiments.

Samples were heated to 200, 400, and 600°C and then cooled down to room temperature (25°C) for analyzing. When the samples were heated to high temperatures, their colour changed significantly and many microcracks were generated in the samples. As the applied temperature increased, Young's modulus, uniaxial compressive strength, and the longitudinal wave velocity decreased gradually and the peak strain that corresponded to the peak strength increased. With the increase of temperature, the non-linearity in the initial deformation stage was enhanced and the stress–strain behavior changed from brittle to ductile. The complete stress–strain curves of the thermal-damaged coarse marble were then simulated using a phenomenological model. It was found that the simulated stress–strain curves were in good agreement with the experiment results.

3. EXPERIMENTS

3.1. Sample Preparation

All samples were collected from different places in Mugla Province, western Turkey, and appeared in different colors. These samples were cut into 90x90x90 mm^3 samples for the first set of experiments (thermal shock and 25-800°C high temperature experiments) and 50x50x50 mm^3, 90x90x90 mm^3 and 150x150x150 mm^3 samples for thermal damage (25-500°C high temperature) experiments.

All the experiments were carried out in Rock Mechanics and Natural Stones Research Laboratories of the Department of Mining Engineering at Mugla Sıtkı Koçman University.

3.2. Test Procedure

3.2.1. Thermal shock

The methodology used to conduct the thermal shock test obeyed the Turkish standard, namely TS EN 14066, to verify the impact of an abrupt change in temperature on fore-mentioned properties of rocks. As suggested by the standard, the samples were placed inside an oven heated to 105°C for 18 hours. Then, they were immersed in water at a temperature of 20°C for 6 hours. Those two stages were consisted of: immersion, during which time the samples remained immersed in the water for 6 hours; and heating, the samples were placed inside an oven heated to 105°C for 18 hours. These two stages are considered to be one full cycle. Five different rock samples were selected. And then, 45 cycles implemented at a temperature of 105°C. The weight of samples, *P*-wave velocity and Shore hardness values were determined following every five cycles after the immersion. At the end of the 45th cycle, the samples were dried in the oven at a temperature of 105°C and were then cooled down to room temperature of 20°C.

The oven used in the experimental work is shown in the Figures 3.1. and 3.2.

Figure 3.1. Oven

Figure 3.2. Oven

3.2.2. Thermal damage

In the first set of experiments, nine different temperature levels (i.e., 25, 100, 200, 300, 400, 500, 600, 700, 800°C, respectively) were applied on five different limestone samples to determine the effects thermal damage on rock samples. Experiments were carried out to determine weight, density, porosity, uniaxial compressive strength, *P*-wave velocity and Shore Hardness values of the samples prior to and post-heating processes Thermal treatment process was consisted of three stages: (1) samples were heated in a high-temperature furnace at the rate of 5°C/minute until the targeted temperature was reached; (2) each specimen was kept at its designated temperature for about 2 hours before the power was automatically cut off; and (3) the samples were allowed to cool down naturally to room temperature.

In the second set of experiments, six different temperature levels (i.e., 25, 100, 200, 300, 400, 500°C, respectively) were applied on three different limestone samples samples which were cut into the dimensions of 50x50x50 mm^3, 90x90x90 mm^3 and 150x150x150 mm^3. In this set, maximum temperature was increased to 500°C, owing to the fact that at higher temperatures the samples were seen to disintegrate.

High temperature furnace used in the experimental work is shown in Figures 3.3. and 3.4.

Figure 3.3. High Temperature Furnace

Figure 3.4. High Temperature Furnace

3.2.3. Pulse velocities

An ultrasonic pulse velocity test is nondestructive test to check the quality of concrete and natural rocks. In this experiment, the strength and quality of intact rock are assessed by measuring the velocity of ultrasonic pulse passing through a natural rock material. This experiment is conducted by passing a pulse of ultrasonic wave through rock samples to be examined and measuring the time taken by pulse to get through the sample. Higher velocities indicate good quality and continuity of the material, while slower velocities may indicate that rock sample may contain many cracks or voids, etc.

Ultrasonic wave (pulse) testing device is shown in Figure 3.5.

Ultrasonic testing equipment includes a pulse generation circuit, consisting of electronic circuit for generating pulses and a transducer for transforming electronic pulse into mechanical pulse having an oscillation frequency in range of 40 kHz to 50 kHz, and a pulse reception circuit that receives the signal.

Figure 3.5. PROCEQ Ultrasonic Pulse Velocity Tester

Pulse velocity is measured by a simple formula:

$$Pulse\ Velocity = \frac{\text{Width of structure}}{\text{Time taken by pulse to go through}}$$

Calculation of the propagation velocities of the compression and shear waves, Vp and Vs respectively, as follows:

$$V_P = L_P/T_P$$

$$V_S = L_S/T_S$$

where:

V = pulse-propagation velocity, in./s (or m/s),

L = pulse-travel distance, in. (or m),

T = effective pulse-travel time (measured time minus zero-time correction), s,

and subscripts ‘p’ and ‘s’ denote the compression wave and shear wave, respectively.

3.2.4. Shore hardness

Shore hardness (SH) has been approved as a proper and nondestructive technique in determining the hardness of rocks and widely used in rock mechanics since it can be correlated with other mechanical properties of weak rocks, such as uniaxial compressive strength (UCS).

The concept of rock integrity includes both the hardness and the structural wholeness of the rock. Therefore, different parameters can be used as proxies for rock integrity. Shore hardness (SH) has been used as the measure of rock integrity.

SH test is a non-destructive way to compare the hardness values of the rock samples. To measure SH values, Proceq Equotip Portable Hardness Tester was used (Figure 3.6.).

Figure 3.6. Proceq Equotip Portable Hardness Tester

Figure 3.7. The Uniaxial Compressive Strenght Testing Device

3.2.5. The uniaxial compressive strength (UCS)

Uniaxial compressive strength (UCS) is one of the most important mechanical properties of rocks and is widely used in different engineering related experiments to determine the stability of structures under load. Determination of the UCS demands the presence of high quality rock samples which can not always be provided due to existence of natural weaknesses such as cracks, fractures, foliations etc. in natural rock.

The uniaxial compressive strength of rock samples was determined in compliance with ASTM Standard (D7012 – 14) and tests were carried out on cubical block samples having an edge length of 90 mm.

The uniaxial compressive strength testing device is shown in Figure 3.7.

3.2.6. Porosity

Porosity is one of the basic physical properties of rocks. Porosity influences the internal surface area per unit material volume and this in turn, defines the transport properties and strength of the material. A comprehensive analysis of porosity can provide valuable information in order to define whether a given type of rock is susceptible to thermal stress or not (Martin et. al., 1996). Carbonate rocks, in particular, exhibit wide range of porosities. The effective porosity and bulk density of rock samples were evaluated using saturation and buoyancy techniques, as suggested by ISRM (1981) and TSE (TSE 699). The method uses Archimedes principle and gives accurate results. In order to prevent air trapping in the pores, one-fourth height of the samples was filled with water at 1 hour intervals. Then, samples were left in water for a period of 48 hours with periodic agitation. Later, the samples were transferred underwater to a basket in an immersion bath and their saturated-submerged weights were measured. Then, the surface of the samples was dried with a moist cloth and their saturated surface dry weights were measured outside the water. Bulk sample volumes were found from weight differences between saturated-surface-dry weight and saturated-submerged weight. The dry mass of samples was determined after oven drying at a temperature of 105°C for a period of at least 24 hours. The effective pore volumes were determined from weight difference between saturated-surface-dry weight and dry sample weight. The bulk density of samples was calculated by dividing the dry weight of samples to the bulk volumes; whereas, the effective porosity was found by the ratio of pore volume to bulk sample volume. Grain density of rocks was determined following the procedures recommended by ISRM (1981) and TSE (TSE 699).

Porosity can be classified into different types such as absolute or total porosity, open porosity, and effective or connected porosity. The total porosity is simply the fractional volume of all void space inside a porous material. While the open porosity, considers only the proportion of voids that are communicated with the outside of the sample. The effective or connected porosity is the volume fraction of pore spaces that are fully interconnected between two opposite end faces and allowing the fluid

flow through the material. This last porosity is classically quantified by permeability measurement.

4. EXPERIMENTAL RESULTS

4.1. The First Set of Experiments

In the first set of the experiments, samples were dimensioned into 90x90x90 mm^3 blocks and were shown in Figures 4.1. – 4.5. Thermal shock tests were performed in 45 cycles. Weight, Shore hardness and ultrasonic pulse velocity experiments were conducted every five cycles from the tenth cycle. The results of the thermal shock tests are given in Table 4.1. On the other hand, samples of the same kind of rocks were exposed to high temperatures gradually. They were heated to 25, 100, 200, 300, 400, 500, 600, 700 and 800°C in the furnace to observe the thermal damage which are displayed in Table 4.2.

Figure 4.1. Thermal shock – Sample A1

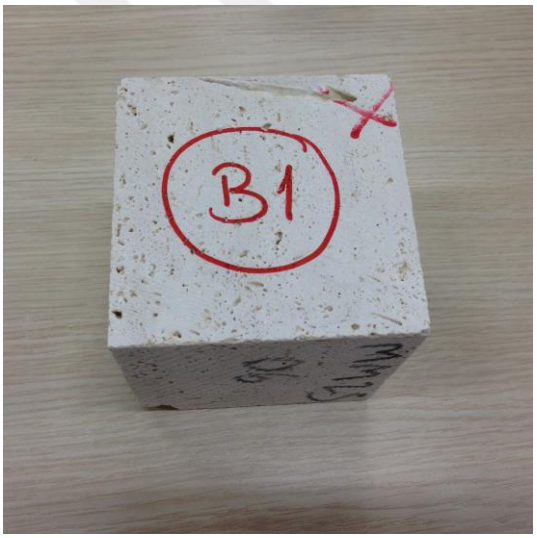

Figure 4.2. Thermal shock – Sample B1

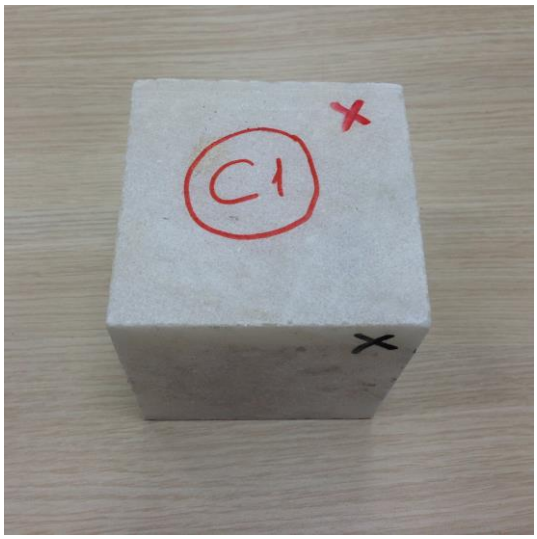

Figure 4.3. Thermal shock – Sample C1

Figure 4.4. Thermal shock – Sample D1

Figure 4.5. Thermal shock – Sample E1

Table 4.1. The experimental result of samples on thermal shock test

SAMPLE	Temperature	Cycle	Weigth (gr)	Shore Hardness (SH)			*P*- wave velocity (m/s)
				1st Side	2nd Side	Average	
A1	105°C	0	1629.37	49,17	50,17	49,67	4032
		1	1624.90	50,76	51,68	51,22	4032
		10	1624.13	46,80	53,32	51,56	3874
		15	1623.14	49,37	48,21	49,79	3829
		20	1622.49	49,15	53,41	51,28	3846
		25	1622.40	52,97	50,36	51,66	3614
		30	1621.78	51,44	51,02	51,23	3071
		35	1621.63	48,77	52,67	50,72	3030
		40	1621.22	48,72	49,23	48,98	2777
		45	1621.11	47,70	52,38	50,04	3000
B1	105°C	0	1725.53	50,11	48,66	49,38	5952
		1	1722.13	47,21	47,17	47,19	5491
		10	1722.06	46,62	47,99	47,30	5214
		15	1721.73	47,69	48,02	47,86	5172
		20	1721.69	47,44	46,42	46,93	5056
		25	1721.61	47,48	46,64	47,06	5172
		30	1721.59	46,65	47,37	47,01	3703
		35	1720.75	47,72	47,03	47,38	3673
		40	1720.64	46,90	48,82	47,86	3643
		45	1720.48	47,54	46,62	47,08	3600
C1	105°C	0	2008.86	46,53	47,73	47,13	3861
		1	2008.15	46,24	46,40	46,32	3836
		10	2008.11	45,89	46,97	46,43	3803
		15	2007.80	46,78	46,97	46,88	3600
		20	2007.70	45,00	44,05	44,52	2990
		25	2007.65	45,48	43,83	44,66	3040
		30	2006.93	45,00	45,31	45,16	2307
		35	2006.54	44,86	46,62	45,74	2261
		40	2006.39	45,46	45,19	45,32	2107
		45	2006.18	46,19	44,36	45,28	2004
D1	105°C	0	1958.75	48,83	50,20	49,52	5616
		1	1957.96	47,60	47,61	47,60	4062
		10	1957.72	47,67	47,78	47,72	3628
		15	1957.61	48,51	48,36	48,44	3629
		20	1957.45	49,25	48,29	48,77	3448
		25	1957.42	44,29	47,40	45,84	3422
		30	1956.84	49,57	50,23	49,40	2795
		35	1956.44	47,82	50,25	49,04	2608
		40	1956.28	49,70	47,60	48,65	2686
		45	1956.11	48,27	50,29	49,28	2472
E1	105°C	0	1971.82	46,03	45,71	45,87	6804
		1	1970.00	45,58	45,17	45,38	5356
		10	1970.00	47,54	45,93	46,74	4706
		15	1969.64	47,00	48,54	47,77	3529
		20	1969.41	46,05	45,65	45,85	3020
		25	1969.11	44,10	46,33	45,22	3180
		30	1968.76	46,29	44,26	45,28	2331
		35	1968.51	47,40	45,15	46,28	2313
		40	1968.30	46,37	44,95	45,66	2356
		45	1968.06	46,96	47,19	47,08	2255

Experimental results of thermal shock tests are listed in Table 4.1., in which cycle ‘0’ indicates original sample and cycle ‘1’ indicates dried sample at 105°C. When Table 4.1. is examined, it can be noticed that there is no significant decreases in the sample weights as the number of cycles increased. When Shore hardness values are examined, it can be seen that there is no regular and significant increases or decreases in the values of Shore hardness. When *P*-wave velocity values are examined, it can be seen that *P*-wave velocities decreased as the number of cycles increased.

Table 4.2. The experimental result of the samples subjected to thermal damage tests

SAMPLE	Temperature (°C)	Weigth (gr)	SH	*P*-wave velocity (m/s)
A2	0	1578,59	52,13	4035
	25	1578,29	56,15	3994
	100	1577,44	55,81	3964
	200	1575,60	53,21	3829
	300	1574,78	50,43	3543
	400	1573,66	49,53	2506
	500	1571,95	47,65	2036
	600	1568,75	46,04	1645
	700	1558,44	42,22	1491
	800	1512,84	0,00	1339
B2	0	1760,74	47,19	5882
	25	1760,64	48,27	5487
	100	1760,62	46,43	5325
	200	1758,51	46,88	4500
	300	1758,20	47,54	4326
	400	1757,93	46,07	3734
	500	1757,13	47,28	3422
	600	1755,59	44,19	2064
	700	1747,87	45,22	1711
	800	1720,14	0,00	1543
C2	0	2012,54	44,20	3571
	25	2012,40	46,01	2970
	100	2012,32	45,83	2694
	200	2012,26	45,20	2500
	300	2011,81	0,00	1898
	400	2011,37	0,00	1446
	500	2011,00	0,00	1216
	600	2010,45	0,00	1038
	700	2005,45	0,00	907
	800	0,00	0,00	380
D2	0	2173,89	49,03	6122
	25	2173,77	50,17	5806
	100	2173,87	49,27	5172
	200	2173,37	47,29	3409
	300	2172,70	46,64	3030
	400	2172,28	47,10	2542
	500	2171,84	46,91	2153
	600	2171,17	46,39	1778
	700	2163,19	44,71	1463
	800	2130,00	0,00	1410
E2	0	2000,44	45,58	6122
	25	2000,23	45,84	5806
	100	1999,55	46,52	4891
	200	1995,86	43,61	2735
	300	1992,24	43,70	2349
	400	1989,82	0,00	1927
	500	1987,80	0,00	1581
	600	1987,20	0,00	1201
	700	1981,34	0,00	1070
	800	1944,91	0,00	334

Experimental results of thermal damage tests are exhibited in Table 4.2.

When the weights of the samples are examined, there is a general decrease with the increase in temperature, however, above 600°C, the weight loss increased, and after heating to 800°C, a significant decrease can be observed in the weights of the samples. The most important reason for this, is that the samples tend to disintegrate especially when they are exposed to 700°C. Above the temperature of 800°C, the samples were observed to disintegrate. At this stage, the sample C2 was completely dispersed.

When Shore hardness data were examined, it became difficult to measure the hardness values owing to the disintegration of the samples as the temperature increased. Particularly, it became impossible to take Shore hardness values on the samples C2 (at 300°C temperarture) and E2 (at 400°C temperature). As the temperature is increased, ultrasonic pulse velocity values were significantly decreased. This proves that that high temperature has a significant influence on physical and mechanical properties of rocks.

Figures 4.6., 4.7. and 4.8. show the variation in average weight, *P*-wave velocity and Shore hardness with the cycle numbers of thermal shock test.

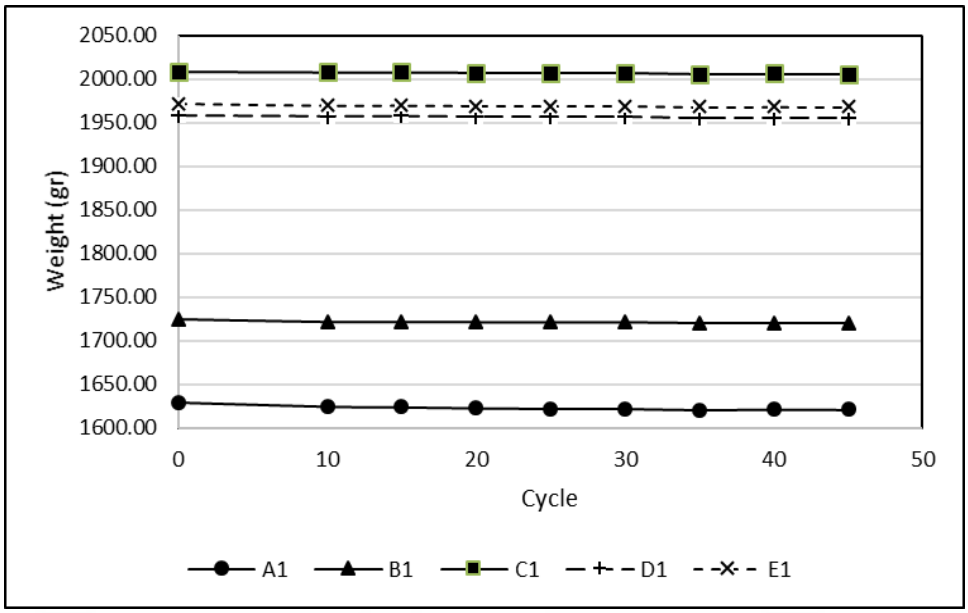

Figure 4.6. Variation in weight with thermal shock cycle

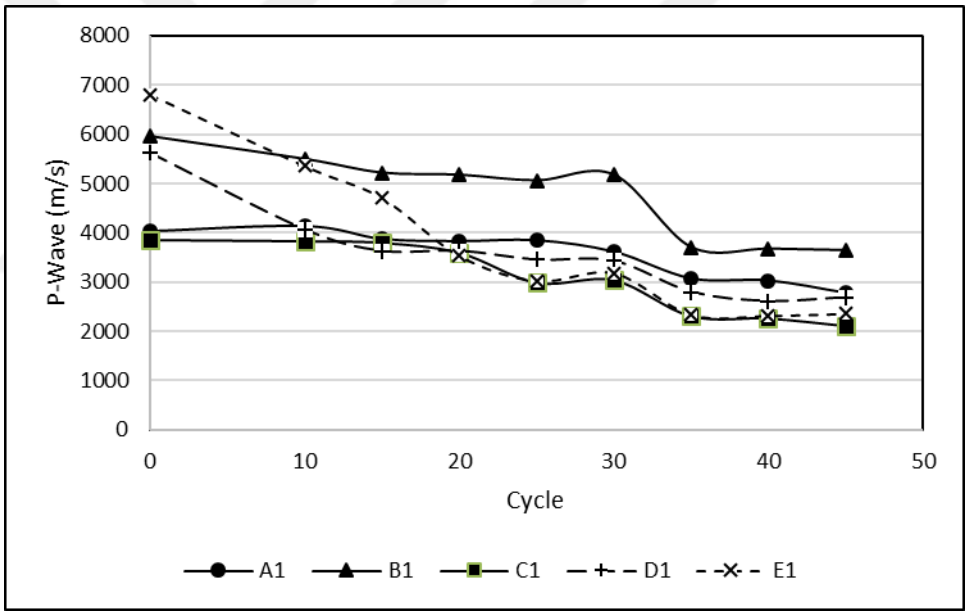

Figure 4.7. Variation in *P*-wave velocity with thermal shock cycle

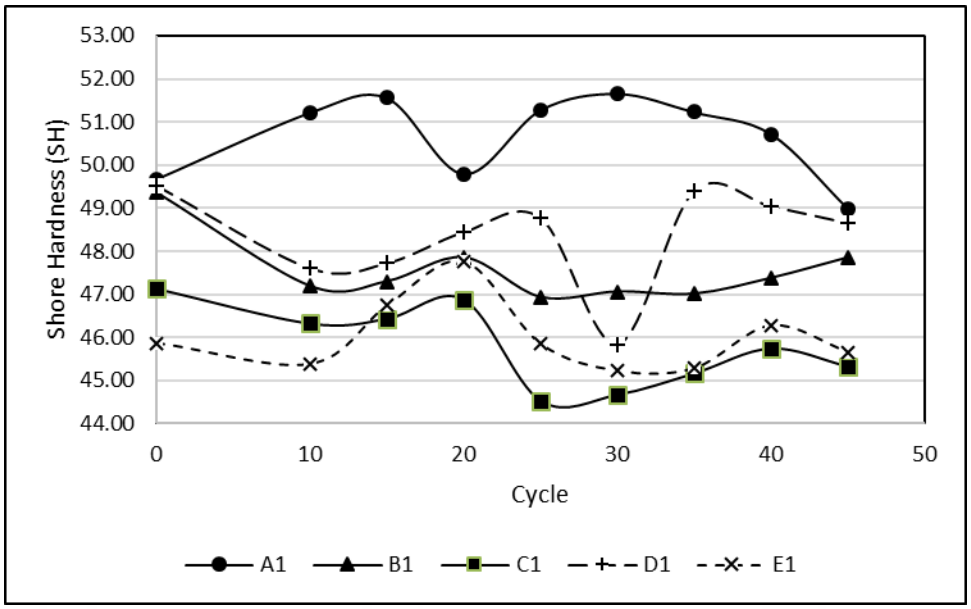

Figure 4.8. Variation in Shore hardness with thermal shock cycle

The variations in the values of average weight, *P*-wave velocity and Shore hardness as the temperarture increases during the thermal damage tests are given in Figures 4.9., 4.10. and 4.11., which proved that high temperatures have significant effect on *P*-wave velocity and Shore hardness values.

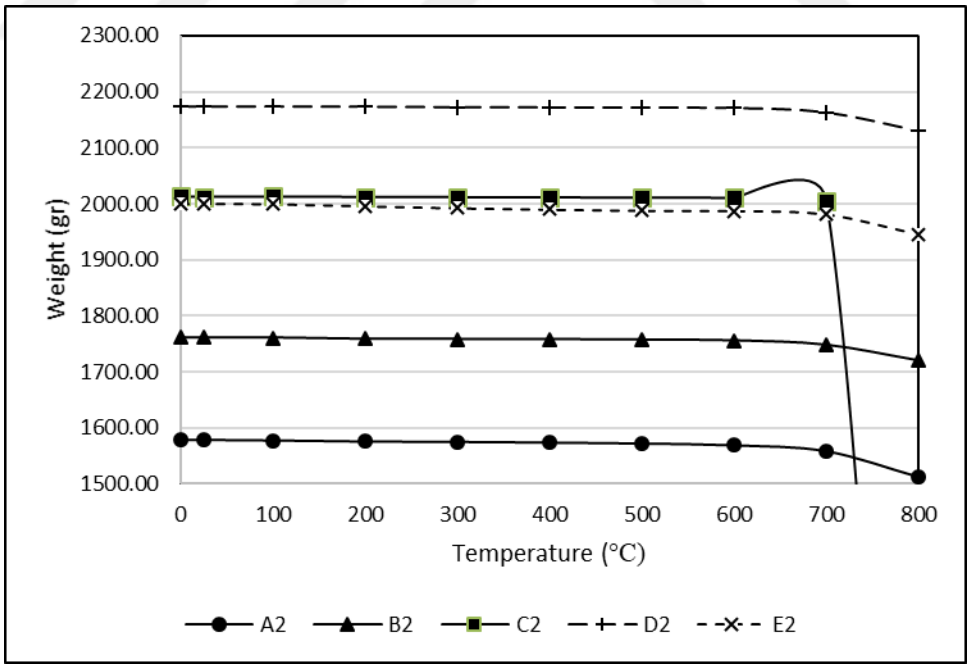

Figure 4.9. Variations in weight as the temperature increases

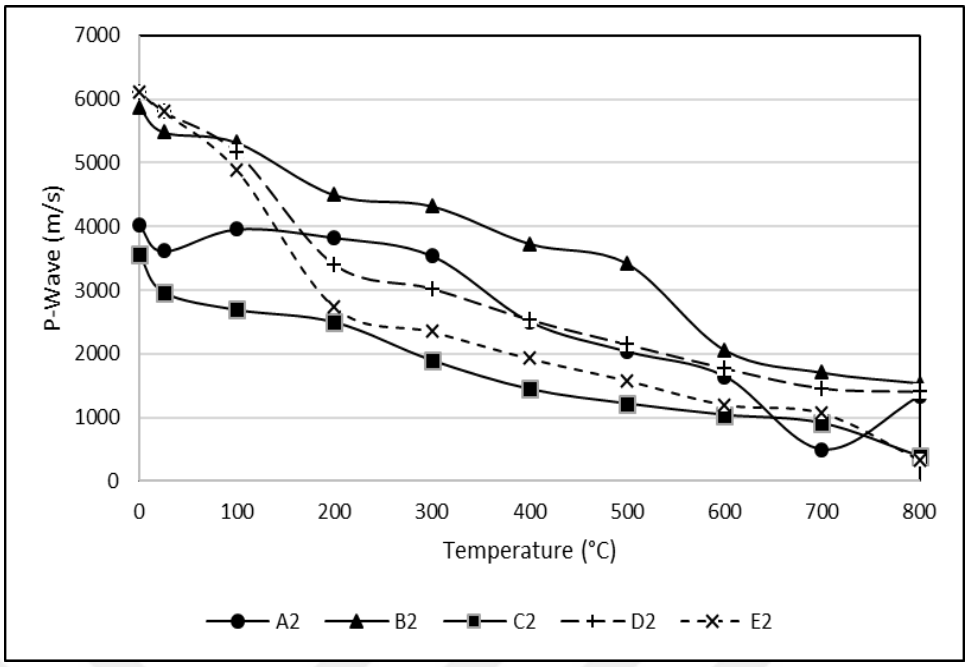

Figure 4.10. Variations in *P*-wave velocity as the temperature increases

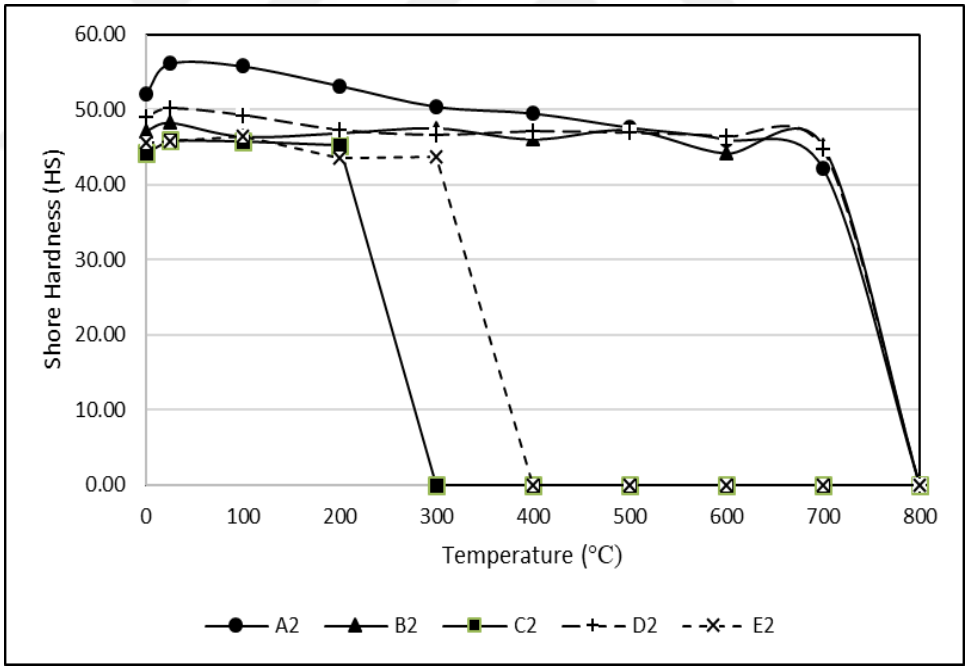

Figure 4.11. Variations in Shore hardness as the temperature increases

4.2. The Second Set of Experiments

Three different limestone samples were dimensioned into 5x5x5 cm^3, 9x9x9 cm^3 and 15x15x15 cm^3 blocks. Then, they were heated to the steps of temperatures of 25, 100, 200, 300, 400, and 500°C in the furnace. Weight, Shore hardness and ultrasonic pulse velocity experiments were conducted folowing each temperature step.

The samples are shown in Figures 4.12. – 4.79., original (at 0°C) and after heating process (at 500°C).

Figure 4.12. Pre-thermal damage–A1-15 (at 0°C)

Figure 4.13. Post-thermal damage–A1-15 (at 500°C)

Figure 4.14. Pre-thermal damage–A2-15 (at 0°C)

Figure 4.15. Post-thermal damage–A2-15 (at 500°C)

Figure 4.16. Pre-thermal damage–A3-15 (at 0°C)

Figure 4.17. Post-thermal damage–A3-15 (at 500°C)

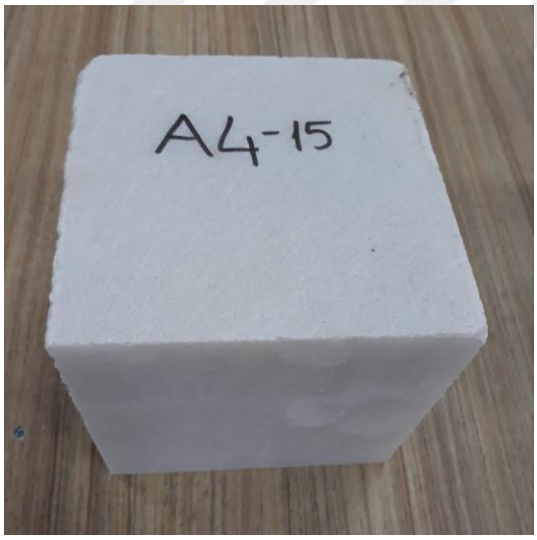

Figure 4.18. Pre-thermal damage–A4-15 (at 0°C)

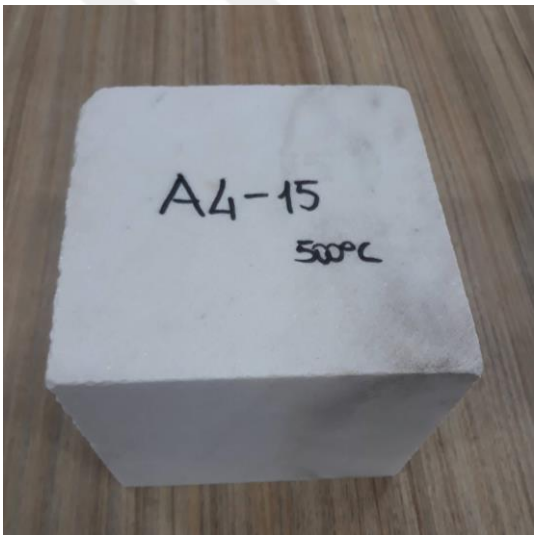

Figure 4.19. Post-thermal damage–A3-15 (at 500°C)

Figure 4.20. Pre-thermal damage–B1-15 (at 0°C)

Figure 4.21. Post-thermal damage–B1-15 (at 500°C)

Figure 4.22. Pre-thermal damage–B2-15 (at 0°C)

Figure 4.23. Post-thermal damage–B2-15 (at 500°C)

Figure 4.24. Pre-thermal damage–B3-15 (at 0°C)

Figure 4.25. Post-thermal damage–B3-15 (at 500°C)

Figure 4.26. Pre-thermal damage–B4-15 (at 0°C)

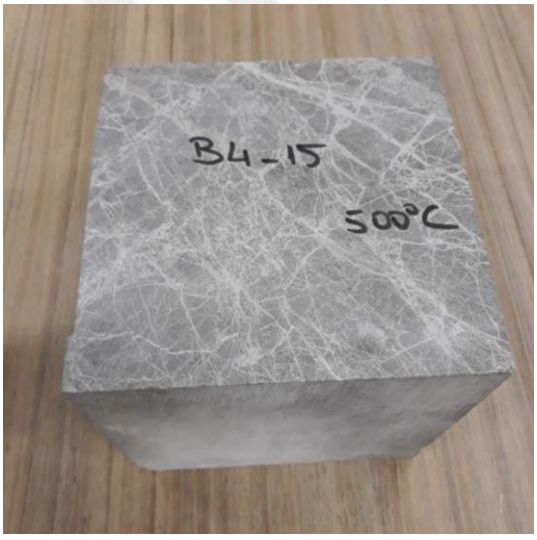

Figure 4.27. Post-thermal damage–B4-15 (at 500°C)

Figure 4.28. Pre-thermal damage–C1-15 (at 0°C)

Figure 4.29. Post-thermal damage–C1-15 (at 500°C)

Figure 4.30. Pre-thermal damage–C2-15 (at 0°C)

Figure 4.31. Post-thermal damage–C2-15 (at 500°C)

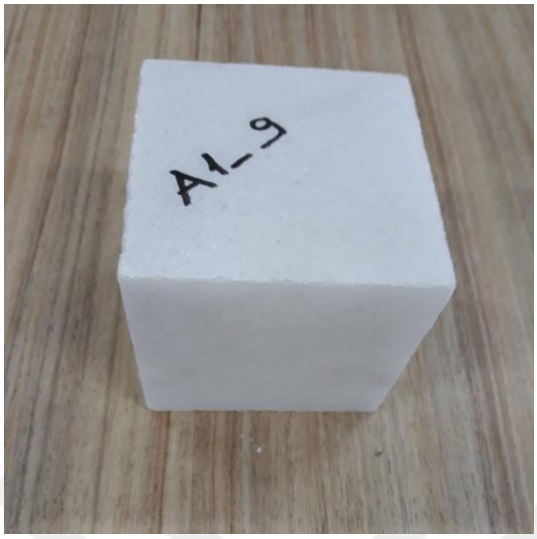

Figure 4.32. Pre-thermal damage–A1-9 (at 0°C)

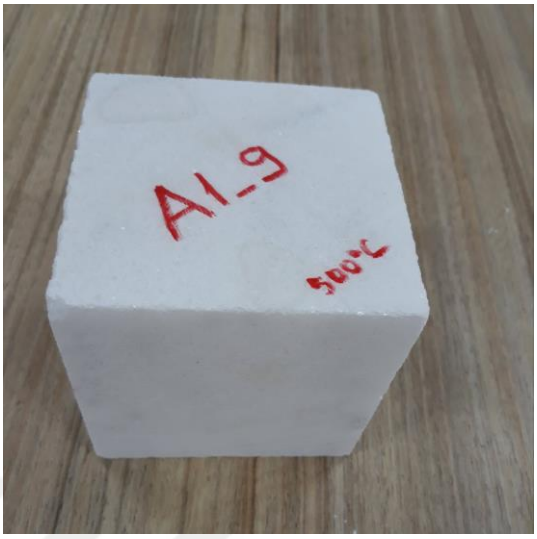

Figure 4.33. Post-thermal damage–A1-9 (at 500°C)

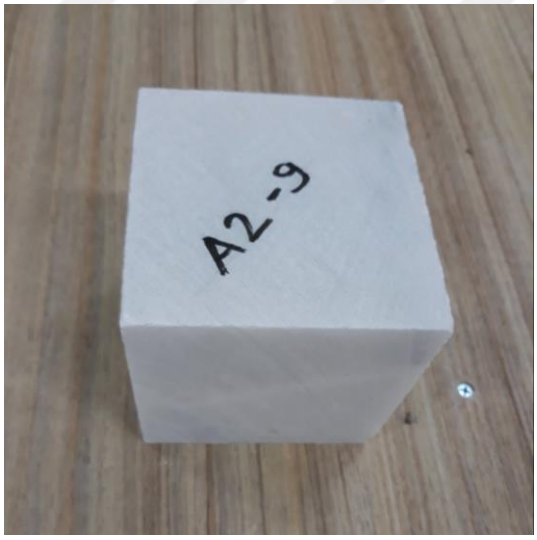

Figure 4.34. Pre-thermal damage–A2-9 (at 0°C)

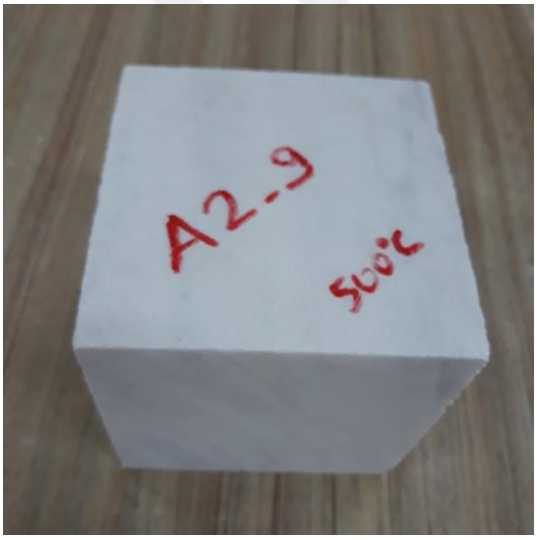

Figure 4.35. Post-thermal damage–A2-9 (at 500°C)

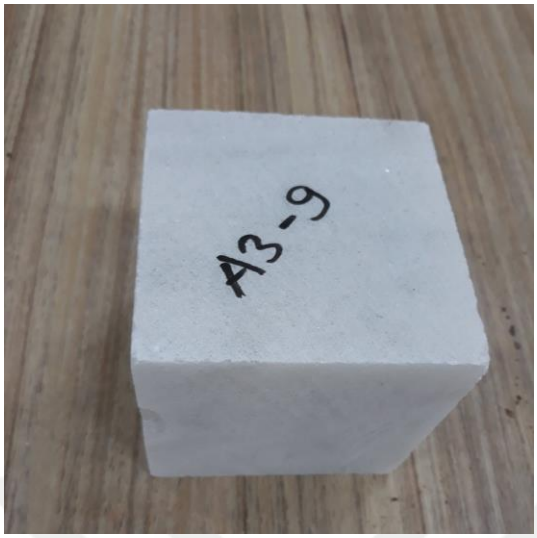

Figure 4.36. Pre-thermal damage–A3-9 (at 0°C)

Figure 4.37. Post-thermal damage–A3-9 (at 500°C)

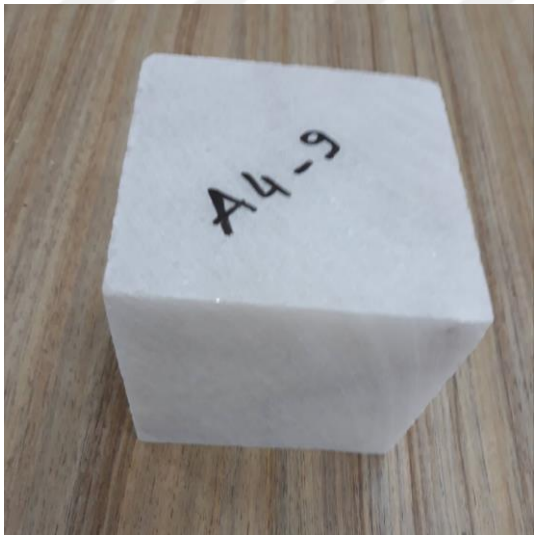

Figure 4.38. Pre-thermal damage–A4-9 (at 0°C)

Figure 4.39. Post-thermal damage–A4-9 (at 500°C)

Figure 4.40. Pre-thermal damage–B1-9 (at 0°C)

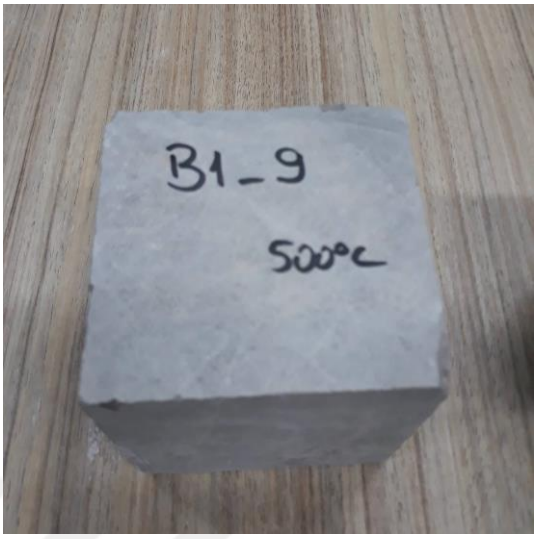

Figure 4.41. Post-thermal damage–B1-9 (at 500°C)

Figure 4.42. Pre-thermal damage–B2-9 (at 0°C)

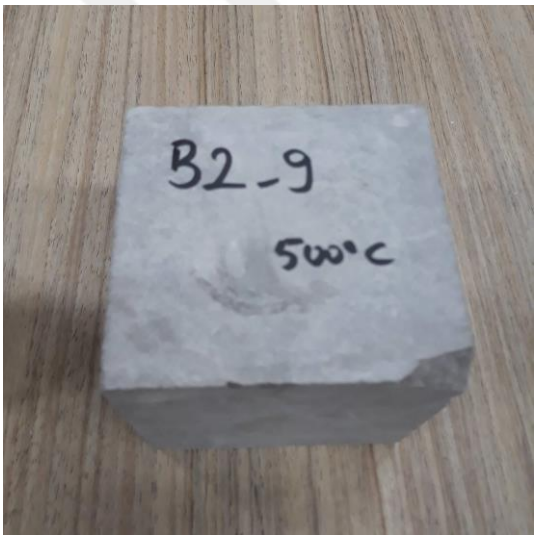

Figure 4.43. Post-thermal damage–B2-9 (at 500°C)

Figure 4.44. Pre-thermal damage–B3-9 (at 0°C)

Figure 4.45. Post-thermal damage–B3-9 (at 500°C)

Figure 4.46. Pre-thermal damage–B4-9 (at 0°C)

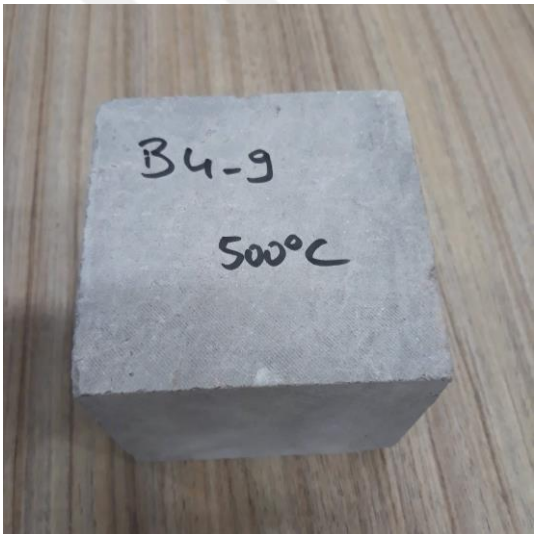

Figure 4.47. Post-thermal damage–B4-9 (at 500°C)

Figure 4.48. Pre-thermal damage–C1-9 (at 0°C)

Figure 4.49. Post-thermal damage–C1-9 (at 500°C)

Figure 4.50. Pre-thermal damage–C2-9 (at 0°C)

Figure 4.51. Post-thermal damage–C2-9 (at 500°C)

Figure 4.52. Pre-thermal damage–C3-9 (at 0°C)

Figure 4.53. Post-thermal damage–C3-9 (at 500°C)

Figure 4.54. Pre-thermal damage–C4-9 (at 0°C)

Figure 4.55. Post-thermal damage–C4-9 (at 500°C)

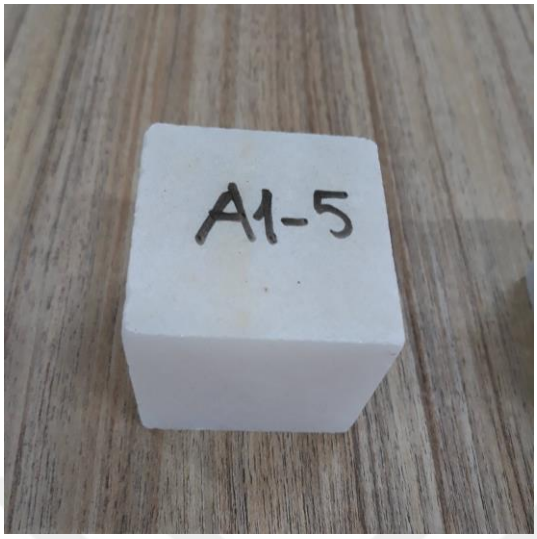

Figure 4.56. Pre-thermal damage–A1-5 (at 0°C)

Figure 4.57. Post-thermal damage–A1-5 (at 500°C)

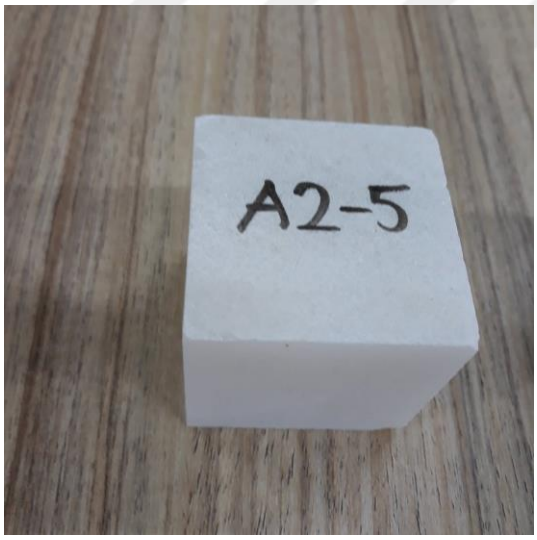

Figure 4.58. Pre-thermal damage–A2-5 (at 0°C)

Figure 4.59. Post-thermal damage–A2-5 (at 500°C)

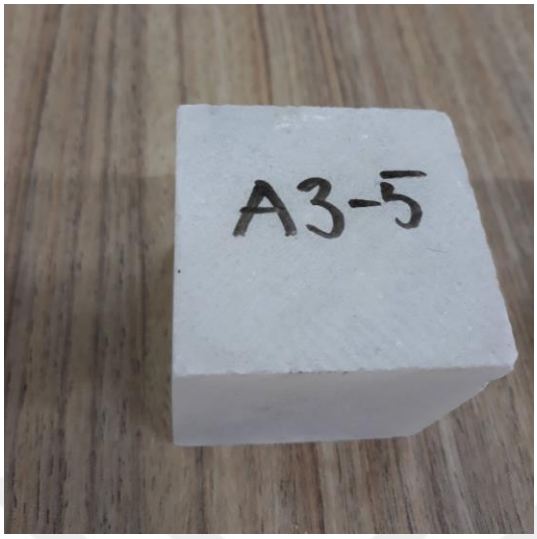

Figure 4.60. Pre-thermal damage–A3-5 (at 0°C)

Figure 4.61. Post-thermal damage–A3-5 (at 500°C)

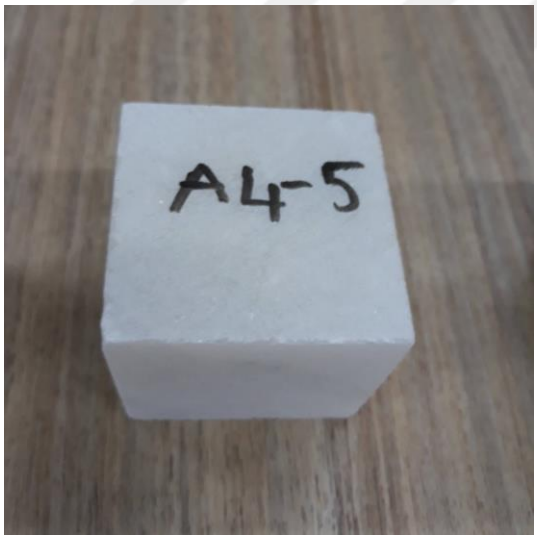

Figure 4.62. Pre-thermal damage–A4-5 (at 0°C)

Figure 4.63. Post-thermal damage–A4-5 (at 500°C)

Figure 4.64. Pre-thermal damage–B1-5 (at 0°C)

Figure 4.65. Post-thermal damage–B1-5 (at 500°C)

Figure 4.66. Pre-thermal damage–B2-5 (at 0°C)

Figure 4.67. Post-thermal damage–B2-5 (at 500°C)

Figure 4.68. Pre-thermal damage–B3-5 (at 0°C)

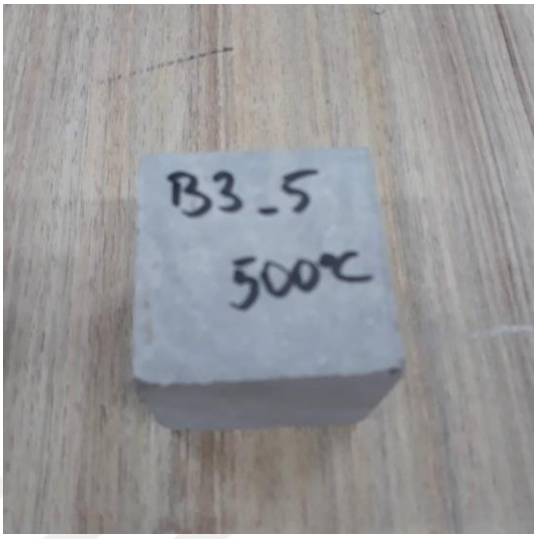

Figure 4.69. Post-thermal damage–B3-5 (at 500°C)

Figure 4.70. Pre-thermal damage–B4-5 (at 0°C)

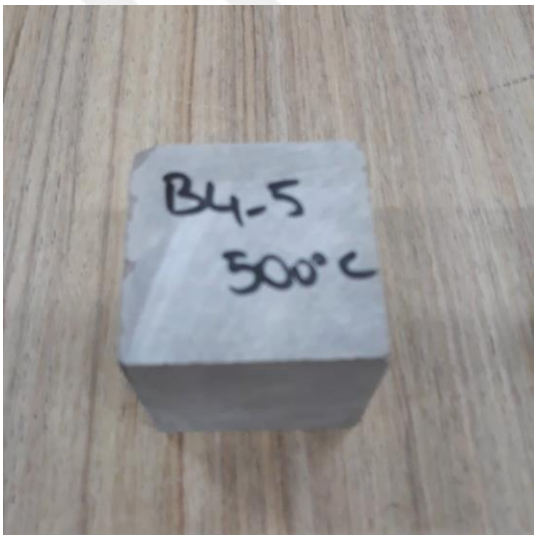

Figure 4.71. Post-thermal damage–B4-5 (at 500°C)

Figure 4.72. Pre-thermal damage–C1-5 (at 0°C)

Figure 4.73. Post-thermal damage–C1-5 (at 500°C)

Figure 4.74. Pre-thermal damage–C2-5 (at 0°C)

Figure 4.75. Post-thermal damage–C2-5 (at 500°C)

Figure 4.76. Pre-thermal damage–C3-5 (at 0°C)

Figure 4.77. Post-thermal damage–C3-5 (at 500°C)

Figure 4.78. Pre-thermal damage–C4-5 (at 0°C)

Figure 4.79. Post-thermal damage–C4-5 (at 500°C)

The thermal damage tests were carried out at temperatures steps of 25, 100, 200, 300, 400 and 500°C. Each sample was gradually heated to the step of temperatures. Weight, Shore hardness and ultrasonic pulse velocity tests were carried out at each temperature step. Tables 4.3. – 4 .11. display the results of these tests of three different cubic limestone samples.

Table 4.3. The experimental results of the tests for samples (A1-15, A2-15, A3-15, A4-15) at different temperature steps

Sample	Temperature Steps (°C)	Weight (gr)	*P*-Wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A1-15	0	8393	7142	53.7	10	4.26	60.4	47.5	12.9
	25	8393	7142	52.1	10	4.93	61.9	45.4	16.5
	100	8392.5	5639	48.3	10	3.72	54.7	43	11.7
	200	8391	4322	46.7	10	3.56	56	43.4	12.6
	300	8390	3875	47.6	10	2.9	51.4	44.1	7.3
	400	8389	3198	45.2	10	2.55	48.8	42.4	7.4
	500	8387	2459	45.4	10	1.3	47.6	43.8	3.8
	600	8383.5	1203	43	2	0.64	43.4	42.5	0.9
A2-15	25	8615.5	7075	49.1	10	2.12	51.5	45.9	5.6
	100	8616.5	5000	45	10	1.16	47.5	43.8	3.7
	200	8615	3588	46.1	10	2.76	50.9	42.7	8.2
	300	8614	2640	44.2	5	1.76	47.2	42.7	4.5
	400	8613.5	1986	0	0	0	0	0	0
	500	8613	1453	0	0	0	0	0	0
A3-15	25	8127.5	7462	53.1	10	3.49	57.4	46.2	11.2
	100	8127.5	6122	51.7	10	4.67	60.2	45.3	14.9
	200	8126.5	4601	48.1	10	3.31	54.2	44.9	9.3
	300	8125.5	3989	47.4	11	4.11	56.7	42.4	14.3
	400	8125.5	3000	47.9	10	3.86	54	42.9	11.1
	500	8125	2402	45.6	9	2.92	50.3	42.6	7.7
A4-15	25	9104.5	6276	47.3	10	2.71	50.8	42.6	8.2
	100	9104.5	4854	45.9	10	1.79	48.8	43.4	5.4
	200	9103	3355	45.5	10	2.17	48.4	42.5	5.9
	300	9103	3006	44.9	10	2.83	51.5	42.5	9
	400	9102.5	1421	0	0	0	0	0	0
	500	9102	1813	0	0	0	0	0	0

Table 4.4. The experimental results of the tests for samples (A1-9, A2-9, A3-9, A4-9) at different temperature steps

Sample	Temperature Steps (°C)	Weight (gr)	*P*-Wave velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A1-9	25	1972.5	6164	48.7	10	4.52	58.1	43.6	14.5
	100	1972	3781	44.6	10	2.13	50.3	42.6	7.7
	200	1972,88	2670	44	5	1.44	45.7	42.4	3.3
	300	1970.76	1918	0	0	0	0	0	0
	400	1970.52	1461	0	0	0	0	0	0
	500	1970.26	984	0	0	0	0	0	0
A2-9	25	1968	6870	49.1	10	3.53	53.6	44.8	8.8
	100	1967.5	5263	49.8	10	3.19	55.7	46.9	8.8
	200	1966.28	4522	48.7	10	3.91	55	42.6	12.4
	300	1966.1	3913	48	9	2.34	51.7	44.8	6.9
	400	1965.91	3180	44.5	8	1.76	48.1	42.7	5.4
	500	1965.5	2521	0	0	0	0	0	0
A3-9	25	1979	6617	47.9	10	3.71	53.9	42.6	11.3
	100	1979	4390	46.5	10	2.98	51.4	43.9	7.5
	200	1977.46	3202	44.8	5	1.71	47.6	43.4	4.2
	300	1977.23	2412	0	0	0	0	0	0
	400	1977.12	1836	0	0	0	0	0	0
	500	1976.78	1200	0	0	0	0	0	0
A4-9	25	1953	6617	46.1	10	2.89	52.9	43.1	9.8
	100	1952.5	4186	46.4	10	3.39	53	42.5	10.5
	200	1951.4	2990	44.5	5	1.01	45.7	43.3	2.4
	300	1951.22	2017	0	0	0	0	0	0
	400	1950.97	1428	0	0	0	0	0	0
	500	1950.64	954	0	0	0	0	0	0

Table 4.5. The experimental results of the tests for samples (A1-5, A2-5, A3-5, A4-5) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A1-5	25	349.57	7142	45.5	10	3.23	51.6	42.6	9
	100	349.55	5434	46.7	10	2.78	51.5	42.7	8.8
	200	349.53	4273	44.4	10	2.24	49.9	42.6	7.3
	300	349.54	3521	46.9	8	3.5	52.1	42.5	9.6
	400	349.51	2293	45.3	3	2.95	48.7	43.2	5.5
	500	349.48	1858	0	0	0	0	0	0
A2-5	25	350.83	6944	47.1	10	3.42	53.7	42.8	10.9
	100	349.55	6097	46.3	10	3.02	51	43.2	7.8
	200	350.78	4807	46.9	10	3.81	53	43	10
	300	350.76	3731	44.4	10	2.1	48.2	42.5	5.7
	400	350.75	2824	0	0	0	0	0	0
	500	350.72	2118	0	0	0	0	0	0
A3-5	25	346.38	6493	46.2	10	4.53	57	42.5	14.5
	100	346.35	5882	44.3	10	2.29	49.6	42.4	7.2
	200	346.32	4132	46.2	10	1.85	49.2	43.5	5.7
	300	346.29	3311	46.5	10	2.31	50	44.2	5.8
	400	346.26	2577	44.7	5	1.23	46.2	43	3.2
	500	346.23	1945	0	0	0	0	0	0
A4-5	25	351.24	6493	45.7	10	1.55	47.4	42.6	4.8
	100	351.21	5434	45.3	10	3.06	52.2	42.9	9.3
	200	351.18	4132	45.5	40	2.43	51.5	42.4	9.1
	300	351.17	3378	44.2	8	1.14	45.9	42.6	3.3
	400	351.15	2450	44.2	3	2.17	46.7	42.6	4.1
	500	351.12	1779	0	0	0	0	0	0

Table 4.6. The experimental results of the tests for samples (B1-15, B2-15, B3-15, B4-15) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
B1-15	0	8629.5	4347	57.2	10	5.05	62.9	45.9	17
	25	8629.5	4373	59.9	10	4.29	66.8	53.9	12.9
	100	8628.5	4573	57	10	6.21	65.5	44.4	21.1
	200	8622.5	4285	55.8	10	5.6	66	44.9	21.1
	300	8618.5	3685	56.9	10	3.65	62.5	51.3	11.2
	400	8610.5	3157	51	10	4.82	60.3	45.9	14.4
	500	8606.5	2767	49.9	10	3.35	55.3	44.2	11.1
	600	8535	1965	46.6	10	2.01	50	42.9	7.1
B2-15	25	8499	4385	56	10	4.41	60.9	45.9	15
	100	8498	4360	58	10	6.67	66.8	46.9	19.9
	200	8492	3722	57.5	10	3.81	63.2	51	12.2
	300	8488	3048	55.7	10	5.89	64.8	45.8	19
	400	8485.5	2912	55.1	10	2.56	61.4	52.7	8.7
	500	8476.5	1712	48.8	10	5.37	58.7	43.1	15.6
B3-15	25	8466.5	4054	53.1	10	3.49	57.4	46.2	11.2
	100	8465.5	3750	52.4	10	3.66	57.2	43.7	13.5
	200	8460.5	3260	53.3	10	3.73	58.7	46.2	12.5
	300	8457.5	2595	54	10	3.71	60.7	49.6	11.1
	400	8454	1547	53.6	10	4.36	59.7	46.2	13.5
	500	8449.5	700	48.3	10	2.97	54	44.4	9.6
B4-15	25	8588.5	4373	55	10	7.3	65.6	45.2	20.4
	100	8587	4545	51.1	10	6.75	63.7	42.9	20
	200	8582.5	4178	51.6	10	5.17	61.3	46.7	14.6
	300	8578.5	3267	55.3	10	5.05	66.1	49	17.1
	400	8574.5	2443	48.9	10	3.1	54.7	43.7	11
	500	8567.5	1712	48.9	10	2.92	53.7	44.7	9

Table 4.7. The experimental results of the tests for samples (B1-9, B2-9, B3-9, B4-9) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
B1-9	25	2057.16	4433	59.6	10	5.55	69.5	48.1	21.4
	100	2057.14	4326	58.8	10	5.32	67.6	51.7	15.9
	200	2057.09	4147	58.4	10	4.03	63.2	51.1	12.1
	300	2056.81	3734	60.4	10	6.2	67.7	46.6	21.1
	400	2056.41	3797	59.6	10	2.44	62.9	54.6	8.3
	500	2055.52	3643	58.4	10	4	63.7	52.8	10.9
B2-9	25	2048.17	4433	61.8	10	5.42	69.1	53.4	15.7
	100	2048.11	4147	63.1	10	4.37	68.2	54.9	13.3
	200	2048.07	3734	60.5	10	4.44	69.4	55	14.4
	300	2047.74	3114	58.4	10	4.66	65.3	51.4	13.9
	400	2047.31	3180	57.4	10	5.23	64.6	50.5	14.1
	500	2046.08	3092	55	10	2.89	59.8	50.1	9.7
B3-9	25	2041.04	3643	56.7	10	5.77	66.3	46	20.3
	100	2040.96	3797	56.1	10	6.7	64.7	44	20.7
	200	2040.92	3272	57.8	10	4.49	63.2	47	16.2
	300	2040.7	2719	54.6	10	4.26	60.5	46.4	14.1
	400	2040.38	2980	53.5	10	4.07	60.3	48.7	11.6
	500	2039.62	2960	54	10	2.47	59.4	50.9	8.5
B4-9	25	2065.64	3600	58.8	10	3.15	64.9	53.7	11.2
	100	2065.55	3734	59.4	10	3.58	65.4	55.1	10.3
	200	2065.51	3308	57	10	4.61	66.6	51.3	15.3
	300	2065.37	2803	58.5	10	4.01	63.5	51.9	11.6
	400	2065.17	2571	53.9	10	5.93	61.7	43.9	17.8
	500	2064.43	2362	53.9	10	2.77	58.7	49	9.7

Table 4.8. The experimental results of the tests for samples (B1-5, B2-5, B3-5, B4-5) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
B1-5	25	355.94	5102	52	10	4.34	60.3	45.2	15.1
	100	355.93	4854	59.9	10	3.39	66	55.4	10.6
	200	355.92	4672	60.3	10	3.1	63.9	52.9	11
	300	355.87	3246	60.3	10	2.3	64	57.6	6.4
	400	355.78	4310	56.8	10	2.57	61.6	53.9	7.7
	500	355.61	3472	54.8	10	2.63	58.6	49.4	9.2
B2-5	25	352.36	4854	56.2	10	2.95	61.1	50.2	10.9
	100	352.35	4629	57.4	10	5	63.4	47.1	16.3
	200	352.35	4424	58.1	10	4.97	65.8	48.5	17.3
	300	352.31	4201	57.6	10	4.33	66.6	52.7	13.9
	400	352.26	4065	54.2	10	5.44	64.2	45.2	19
	500	352.11	3906	53.5	10	5.95	65.4	44.7	20.7
B3-5	25	353.65	4807	59.6	10	2.57	64.3	55.4	8.9
	100	353.64	4629	60.2	10	1.86	63.9	58	5.9
	200	353.63	4424	61	10	1.97	65.4	58.9	6.5
	300	353.57	3906	60.9	10	2.56	64.4	57.1	7.3
	400	353.48	4098	59.8	10	4.07	64.9	50.9	14
	500	353.31	3623	56.6	10	2.34	60.2	54.3	5.9
B4-5	25	355.29	4629	57.1	10	5.86	67.6	48.3	19.3
	100	355.29	4424	55.5	10	4.82	61.1	46.1	15
	200	355.28	4237	58.1	10	4.91	66.2	52.1	14.1
	300	355.23	3623	56.1	10	3.39	59.6	50.3	9.3
	400	355.18	3875	54.5	10	4.9	63.3	46.2	17.1
	500	355.04	3597	54.8	10	2.91	59.6	50.7	8.9

Table 4.9. The experimental results of the tests for samples (C1-15, C2-15) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
C1-15	25	8590.5	6024	61.2	10	4.65	67.4	51.1	16.3
	100	8589	5952	58.7	10	7.37	70.2	44.1	26.1
	200	8583	5791	61.6	10	7.04	70.8	47.3	23.5
	300	8579	5136	59	10	3.99	65.2	52.5	12.7
	400	8575.5	3370	57.4	10	6.83	64.4	43.5	20.9
	500	8564	2307	53.3	11	5.02	59.1	42.4	16.7
C2-15	25	9044	6250	65.6	10	3.69	71.5	57.7	13.8
	100	9043.5	6224	60.4	10	7.6	71.8	50.1	21.7
	200	9037.5	6097	61.7	10	6.85	66.9	47	19.9
	300	9033	5660	60.8	10	7.12	68.3	45.1	23.2
	400	9026	2358	57.5	10	6.72	66.3	46.6	19.7
	500	9011	478	50.5	10	4.62	59.8	44.3	15.5

Table 4.10. The experimental results of the tests for samples (C1-9, C2-9, C3-9, C4-9) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
C1-9	25	1681.73	6081	55.8	10	5.51	64.7	46.7	18
	100	1681.48	6081	53.6	10	6.27	59	42.5	16.5
	200	1680.73	5806	54.1	10	7.05	69.2	45	24.2
	300	1680.43	5487	59.7	10	3.32	66.3	55.3	11
	400	1679.86	4663	52.9	10	5.42	62.1	47.3	14.8
	500	1677.88	3157	52.2	10	5.64	59.3	43	16.3
C2-9	25	1758.62	6081	52.7	10	6.68	65.8	42.8	22.9
	100	1758.3	6081	55.2	10	7.99	72.2	44.1	28.1
	200	1757.13	5732	54.9	10	8.68	69.4	43.7	25.7
	300	1756.67	5590	56.6	10	5.64	64.8	50.3	14.5
	400	1756.14	5172	54	10	4.73	60.8	47	13.8
	500	1754.03	3673	47.6	10	6.03	59	42.7	16.3
C3-9	25	1748.03	6040	56.8	10	5.72	64.1	43.3	20.8
	100	1747.61	6040	51.8	10	5.02	62.2	45.4	16.8
	200	1746.55	5769	55	10	7.18	65	46	19
	300	1746.08	5421	54.5	10	4.81	63.3	47.4	15.9
	400	1745.44	4736	54.8	10	5.69	63.5	46	17.5
	500	1743.11	3345	52.2	10	4.49	58.2	44	14.2
C4-9	25	1822.66	6040	56.3	10	4.52	62	46.3	15.7
	100	1822.37	6040	54.9	10	5.76	63.7	45.6	18.1
	200	1820.97	5769	55.7	10	7.53	66.1	44.6	21.5
	300	1820.39	5421	55.1	10	3.51	59.3	47.7	11.6
	400	1819.7	4761	56.2	10	4.87	64.1	47.8	16.3
	500	1817.42	3284	52.4	10	4.43	59.9	47.1	12.8

Table 4.11. The experimental results of the tests for samples (C1-5, C2-5, C3-5, C4-5) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
C1-5	25	308.97	5747	46.8	10	3.54	52.1	42.8	9.3
	100	308.92	5747	46	10	2.59	50.4	42.5	7.9
	200	308.85	5208	45.7	10	3.23	53	42.5	10.5
	300	308.81	4504	48.5	10	2.86	52.5	45.4	7.1
	400	308.69	3816	48.8	10	3.94	56.6	44.4	12.2
	500	308.4	2551	47.2	10	3.51	54.6	43.1	11.5
C2-5	25	310.36	5813	46.5	10	2.24	49.4	42.6	6.8
	100	310.28	5813	48	10	3.34	51.9	43.2	8.7
	200	310.2	4950	50	10	4.13	57	44.2	12.8
	300	310.17	4310	49.6	10	3.09	54.7	43.6	11.1
	400	310.06	3424	48.3	10	4.1	54.7	42.7	12
	500	309.72	2358	47.2	10	3.3	53	43.1	9.9
C3-5	25	312.87	6097	47.3	11	3.26	51.5	42.4	9.1
	100	312.81	6097	48.6	10	3.21	53.2	42.9	10.3
	200	312.72	5434	47.9	10	3.03	52.2	43	9.2
	300	312.68	5208	51.7	10	3.78	56	45.9	10.1
	400	312.54	4166	47.6	10	3.97	55	43.7	11.3
	500	312.17	2808	47.4	10	3.24	51.9	42.6	9.3
C4-5	25	311.93	5747	46.8	10	2.79	50.6	42.7	7.9
	100	311.89	5747	45.8	10	2.6	52.3	43.6	8.7
	200	311.84	5434	48.1	10	2.83	52.6	43.5	9.1
	300	311.8	4950	50.2	10	2.5	54.9	46.7	8.2
	400	311.64	4098	48.7	10	2.45	52.3	45.5	6.8
	500	311.19	2873	46.7	10	2.52	49.5	42.5	7

Tables 4.12., 4.13. and 4.14. summarizes the average values of weight, *P*-wave velocity and Shore hardness values of each type of sample.

Table 4.12. The experimental results of the tests for samples (A-15, B-15, C-15) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A-15	25	8669.0	6988.8	49.8	40	3.31	61.9	42.6	10.4
	100	8669.0	5403.8	47.4	40	2.84	60.2	43	8.9
	200	8667.7	3966.5	46.4	40	2.95	56.0	42.5	9.0
	300	8667.1	3377.5	45.8	36	2.90	56.7	42.4	8.8
	400	8666.6	2401.3	18.6	20	1.60	54.0	0	4.6
	500	8665.8	2031.8	18.2	19	1.06	50.3	0	2.9
B-15	25	8554.4	4296.3	55.8	40	4.87	66.8	45.2	14.9
	100	8553.2	4307.0	53.9	40	5.82	66.8	42.9	18.6
	200	8548.0	3861.3	54.0	40	4.58	66.0	44.9	15.1
	300	8544.2	3148.8	55.4	40	4.58	66.1	45.8	14.6
	400	8539.8	2514.8	51.5	40	3.71	61.4	43.7	11.9
	500	8533.5	1722.8	49.0	40	3.65	58.7	43.1	11.3
C-15	25	8817.3	6137.0	63.4	20	4.17	71.5	51.1	15.1
	100	8816.3	6088.0	59.6	20	7.49	71.8	44.1	23.9
	200	8810.3	5944.0	61.7	20	6.95	70.8	47	21.7
	300	8806.0	5398.0	59.9	20	5.56	68.3	45.1	18.0
	400	8800.8	2864.0	57.5	20	6.78	66.3	43.5	20.3
	500	8787.5	1392.5	51.9	21	4.82	59.8	42.4	16.1

Table 4.12. displays experimental results of the samples A-15, B-15 and C-15, dimensioned to15x15x15 cm^3.

A negligibly slight decrease (0.037%) in the weight was observed in sample A-15. As the temperature increases, relatively higher losses in weights of the samples were observed for the samples B-15 (0.24%) and C-15 (0.34%).

P-wave velocities of the same samples were also evaluated. Results indicate that the highest decrease in *P*-wave velocity was noted in sample C-15 (77%). Samples A-15 and B-15 yielded lower decreases in *P*-wave velocity values to be 71% and 60%, respectively.

When Shore hardness values were scrutunized, a wide difference was noticed in the results of sample A-15 and the samples B-15 and C-15. Shore hardness value of sample A-15 was reduced by 63% at 500°C temperature while the reduction in samples B-15 and C-15 was determined to be 12% and 18% at the same temperature, respectively. This can be explained by more destructive degradation in sample A-15 as the temperature increases and difficulty encountered during the acquisition of SH values.

Table 4.13. The experimental results of the tests for samples (A-9, B-9, C-9) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A-9	25	1965.1	6567.0	47.6	40	3.66	58.1	42.6	11.1
	100	1964.7	4405.0	46.7	40	2.92	55.7	42.5	8.6
	200	1963.6	3346.0	45.3	25	2.02	55.0	42.4	5.6
	300	1963.3	2565.0	9.6	9	0.59	51.7	0	1.7
	400	1963.1	1976.3	8.9	8	0.44	48.1	0	1.4
	500	1962.8	1414.8	0.0	0	0.00	0.0	0	0.0
B-9	25	2055.5	4027.3	59.1	40	4.97	69.5	46	17.2
	100	2055.5	4001.0	59.4	40	4.99	68.2	44	15.1
	200	2055.4	3615.3	58.1	40	4.39	69.4	47	14.5
	300	2055.2	3092.5	58.1	40	4.78	67.7	46.4	15.2
	400	2054.9	3132.0	55.7	40	4.42	64.6	43.9	13.0
	500	2054.0	3014.3	55.0	40	3.03	63.7	49	9.7
C-9	25	1766.7	6060.5	55.6	40	5.61	65.8	42.8	19.4
	100	1766.4	6060.5	54.1	40	6.26	72.2	42.5	19.9
	200	1765.3	5769.0	55.1	40	7.61	69.4	43.7	22.6
	300	1764.8	5479.8	56.2	40	4.32	66.3	47.4	13.3
	400	1764.2	4833.0	54.8	40	5.18	64.1	46	15.6
	500	1762.0	3364.8	51.4	40	5.15	59.9	42.7	14.9

Table 4.13. displays the results of experiments for the samples A-9, B-9 and C-9, dimensioned to 9x9x9 cm^3 and also shows the effect of sample size on weight, *P*-wave velocity and Shore hardness.

The largest weight loss was observed in sample C-9 (0.27%). The reductions in weight were relatively smaller for samples A-9 (0.11%) and B-9 (0.073%).

In terms of the variations in *P*-wave velocities, reductions were recorded as 78.5%, 44.5% and 25.2% for the samples A-9, C-9 and B-9, respectively.

When Shore hardness values were evaluated for different steps of temperatures, for sample A-9, reliable Shore hardness values were recorded up to the temperature of 200°C and the reduction in Shore hardness was determined to be 4.8%. Owing to the disintegration and degradation of the sample, it was not possible to obtain any SH values for the temperaturfes above 200°C. Reductions in samples B-9 and C-9 were measured to be 6.9% and 7.6%, respectively.

Table 4.14. The experimental results of the tests for samples (A-5, B-5, C-5) at different temperature steps

Sample	Temperature (°C)	Weight (gr)	*P*-wave Velocity (m/s)	Shore Hardness (SH)					
				x	n	s	max	min	R
A-5	25	349.9	6768.0	46.0	40	3.18	57.0	42.5	9.8
	100	349.6	5711.8	45.6	40	2.79	52.2	42.4	8.3
	200	349.8	4336.0	45.7	70	2.58	53.0	42.4	8.0
	300	349.8	3485.3	45.2	36	2.26	52.1	42.5	6.1
	400	349.8	2536.0	35.7	11	1.59	48.7	0	3.2
	500	349.7	1925.0	0.0	0	0.00	0.0	0	0.0
B-5	25	354.5	4848.0	56.4	40	3.93	67.6	45.2	13.6
	100	354.5	4634.0	57.7	40	3.77	66.0	46.1	12.0
	200	354.5	4439.3	59.1	40	3.74	66.2	48.5	12.2
	300	354.4	3744.0	58.2	40	3.15	66.6	50.3	9.2
	400	354.4	4087.0	56.0	40	4.25	64.9	45.2	14.5
	500	354.2	3649.5	54.9	40	3.46	65.4	44.7	11.2
C-5	25	311.2	5851.0	46.8	41	2.96	52.1	42.4	8.3
	100	311.2	5851.0	46.8	40	2.94	53.2	42.5	8.9
	200	311.1	5256.5	48.0	40	3.31	57.0	42.5	10.4
	300	311.1	4743.0	50.0	40	3.06	56.0	43.6	9.1
	400	310.9	3876.0	48.4	40	3.62	56.6	42.7	10.6
	500	310.5	2647.5	47.0	40	3.14	54.6	42.5	9.4

In Table 4.14., experimental results for samples A-5, B-5 and C-5 are displayed. Also, the effect of sample size on weight, *P*-wave velocity and Shore hardness can be seen.

The largest weight loss was observed in sample C-5 (0.22%). The reductions in weight were relatively smaller for samples A-5 (0.057%) and B-5 (0.085%).

In terms of the variations in *P*-wave velocities, reductions were recorded as 71%, 54.8% and 24.0% for the samples A-5, C-5 and B-5, respectively.

When Shore hardness values were evaluated for different steps of temperatures, for sample A-5, reliable Shore hardness values can be recorded up to the temperature of 300°C and the reduction in Shore hardness value was determined to be 1.74%. Increasing degradation and disintegration in the sample above 300°C, it was possible to obtain SH values for the temperaturfes up to 300°C. SH was seen to increase by 4.3% in sample C-5 and decrease by 2.7% in sample B-5.

4.2.1. Effect of temperature on sample weight loss

It has been given in figure, there is very little change on weight of the samples while temperature is increasing. Influence of increasing temperature on variation of sample weight is illustrated in Figures 4.80. – 4.82. As can be seen in the Figures that increase in temperature did not induce any significant weight loss for all the samples of different dimensions.

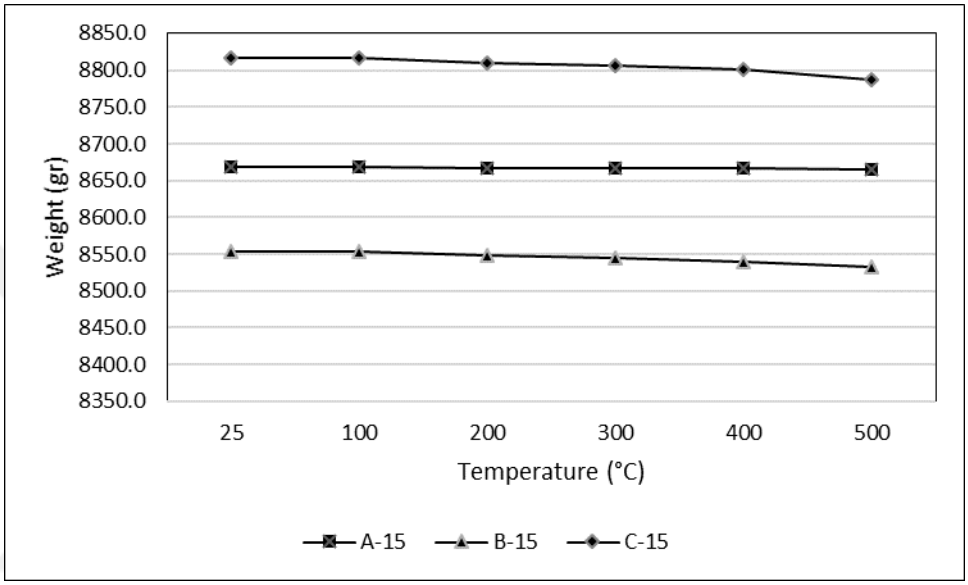

Figure 4.80. Variations in weight loss as the temperature increases for samples A-15, B-15 and C-15.

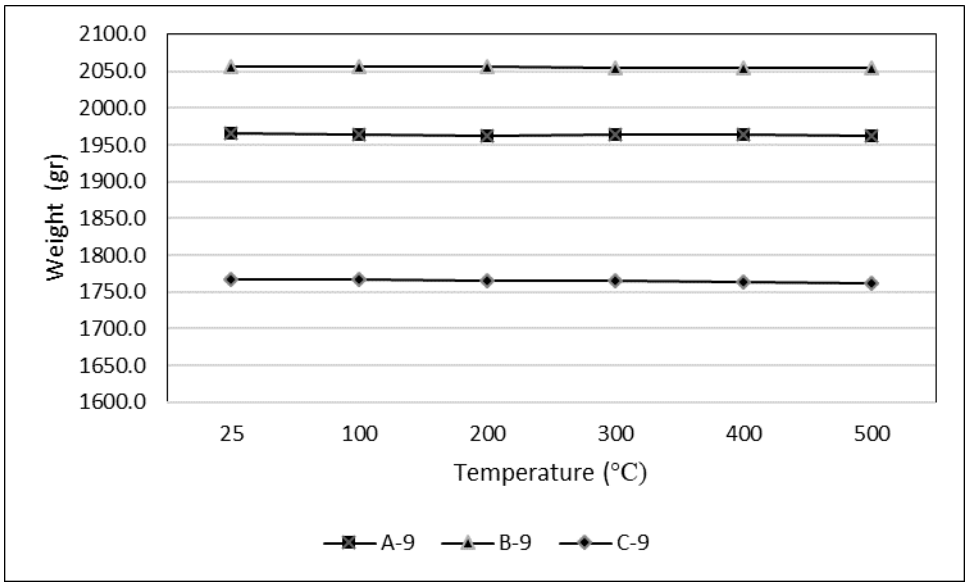

Figure 4.81 Variations in weight loss as the temperature increases for samples A-9, B-9 and C-9

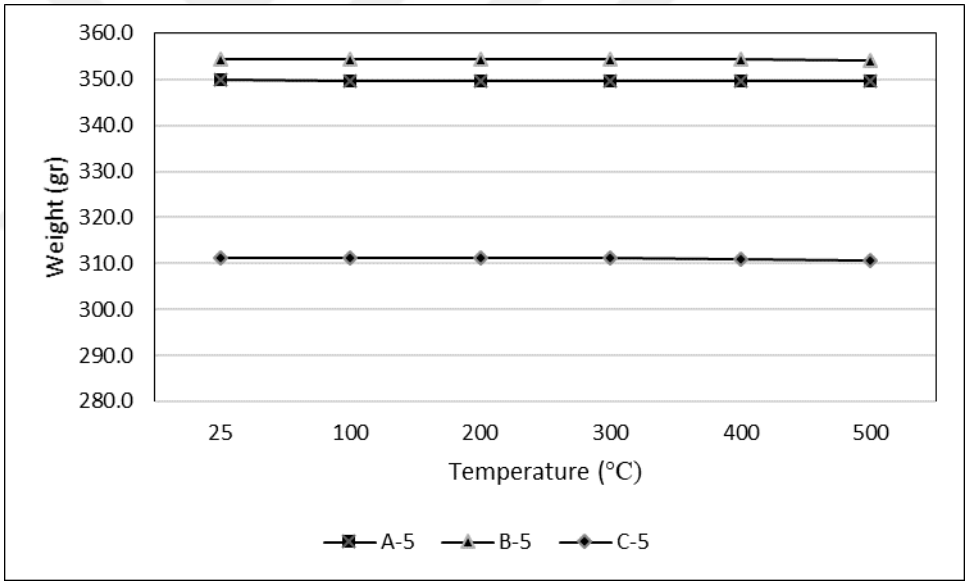

Figure 4.82. Variations in weight loss as the temperature increases for samples A-5, B-5 and C-5

4.2.2. Effect of sample size on weight loss

Figures 4.83. – 4.85. exhibit the values of weight losses for three different limestone samples of different sizes. It can be noticed that increases in temperature did not affect the weights of the samples significantly.

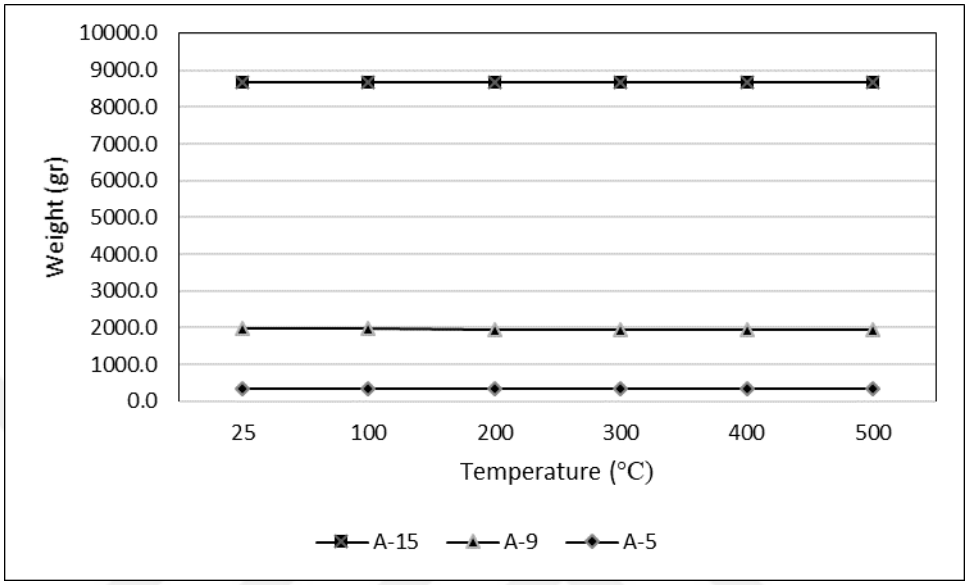

Figure 4.83. Variations in weight loss as the temperature increases for samples A-15, A-9 and A-5

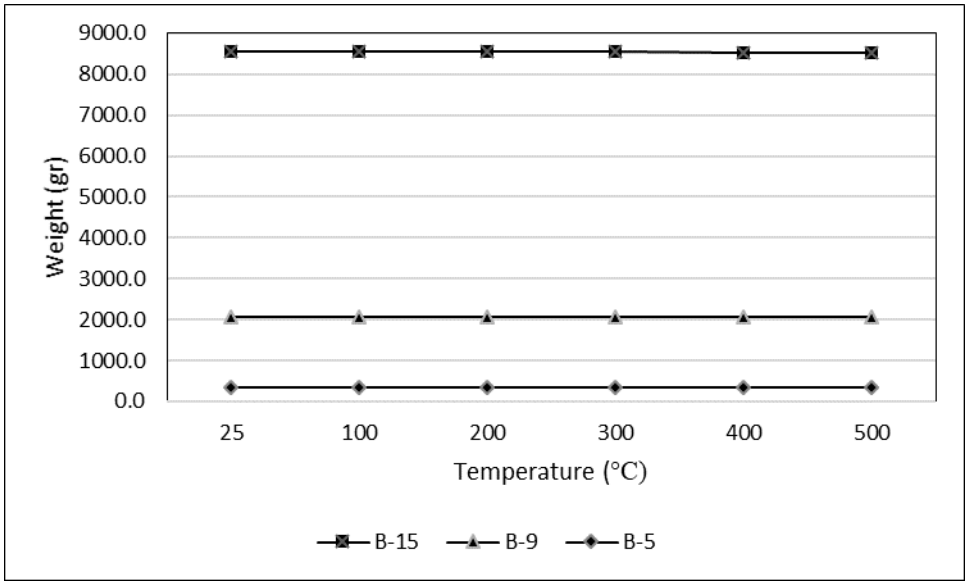

Figure 4.84. Variations in weight loss as the temperature increases for samples B-15, B-9 and B-5

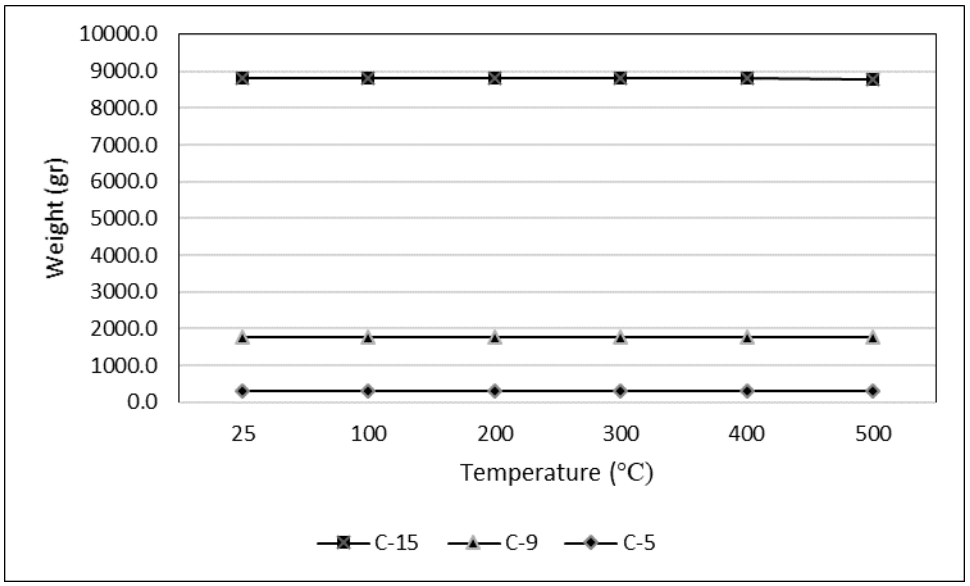

Figure 4.85. Variations in weight loss as the temperature increases for samples C-15, C-9 and C-5

4.2.3. Effect of temperature on *P*-wave velocity

Variation in *P*-wave velocities as the temperature increases are shown in Figure 4.86. – 4.88. *P*-wave velocities seem to decrease significantly up to 70% for the cubic samples with edge length of 15 cm, as the temperature increases. The ratios of decreases were found to be 49% for cubic samples with edge length of 9 cm and 50% for cubic samples with edge length of 5 cm.

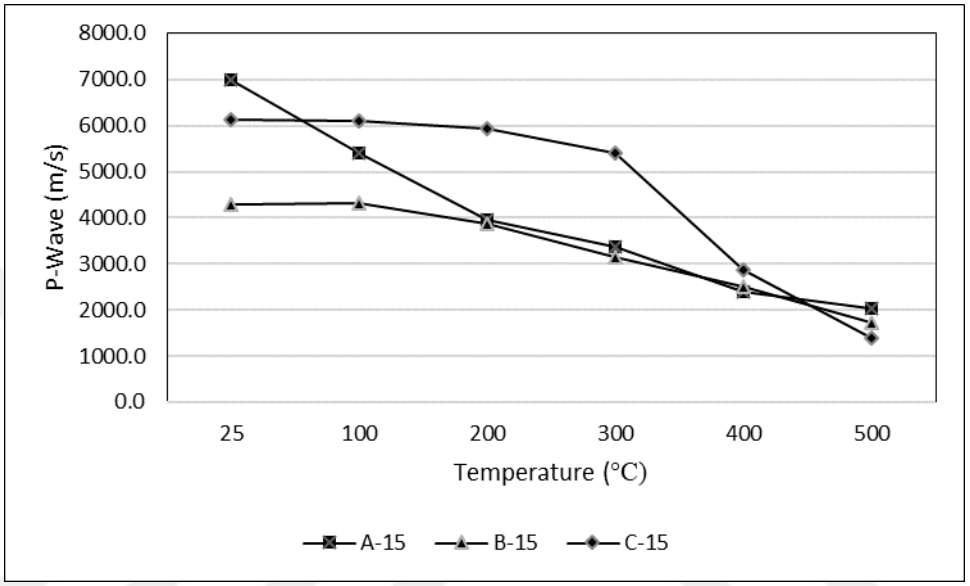

Figure 4.86. Variations in *P*-wave velocities as the temperature increases for samples A-15, B-15 and C-15

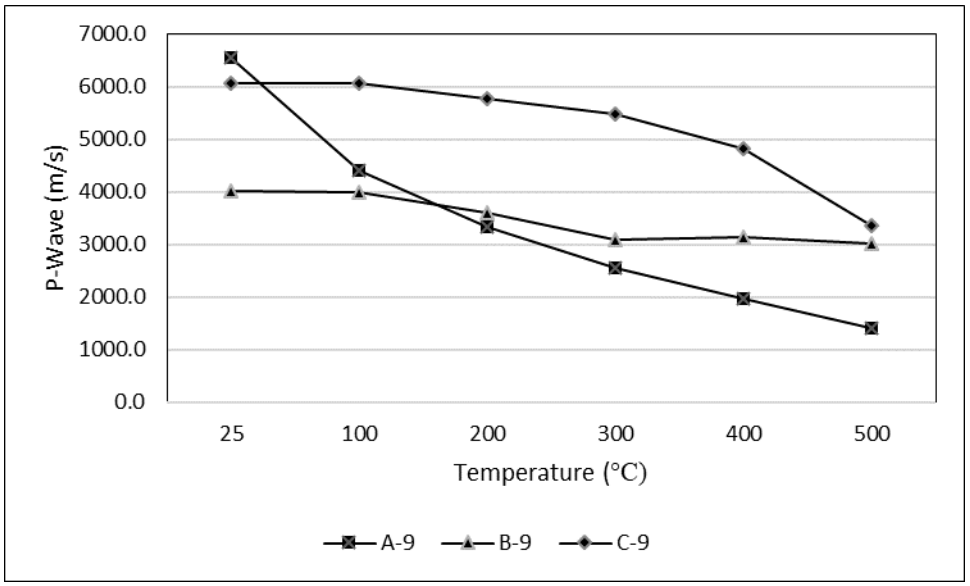

Figure 4.87. Variations in *P*-wave velocities as the temperature increases for samples A-9, B-9 and C-9

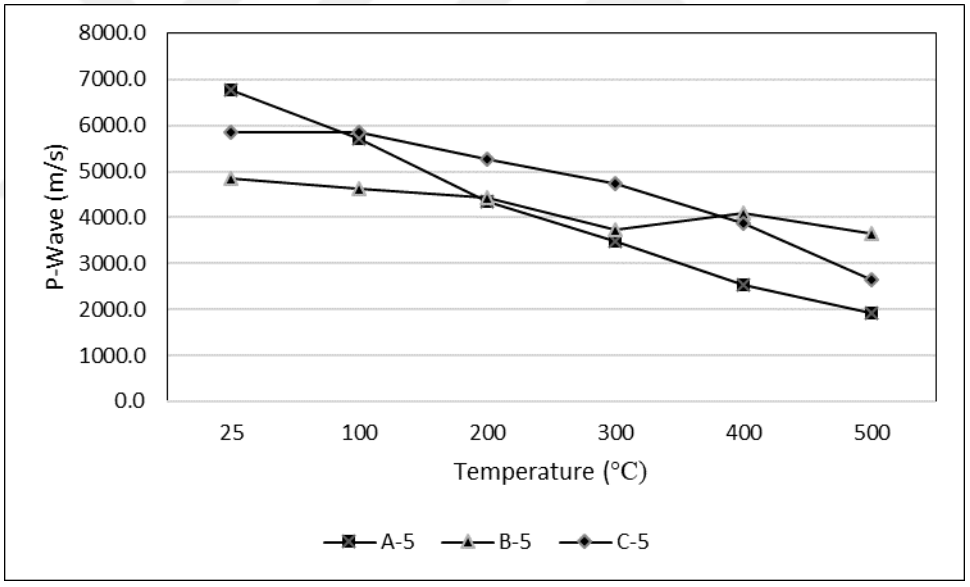

Figure 4.88. Variations in *P*-wave velocities as the temperature increases for samples A-5, B-5 and C-5

4.2.4. Effect of sample size on *P*-wave velocity

Figures 4.89. – 4.91. present the effect of sample size on *P*-wave velocity values of three different limestone samples. In Figure 4.89., a uniform decreasing trend between temperature and *P*-wave velocity is observed up to 500°C for samples A. For samples B and C, *P*-wave velocity values are seen to decrease at different rates depending on the sample size as the temperatures increases (Figures 4.90. and 4.91.).

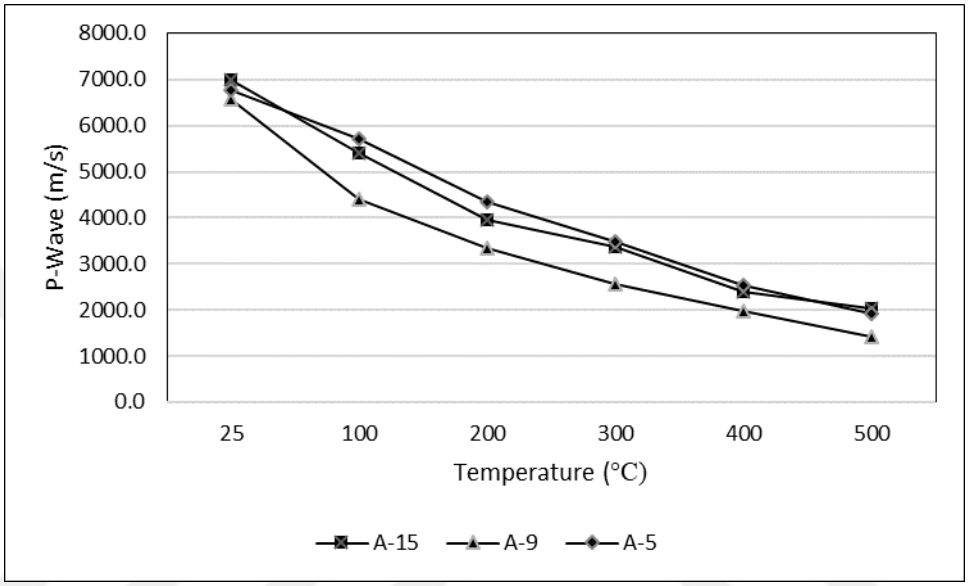

Figure 4.89. Variations in *P*-Wave velocity as the temperature increases for samples A-15, A-9 and A-5

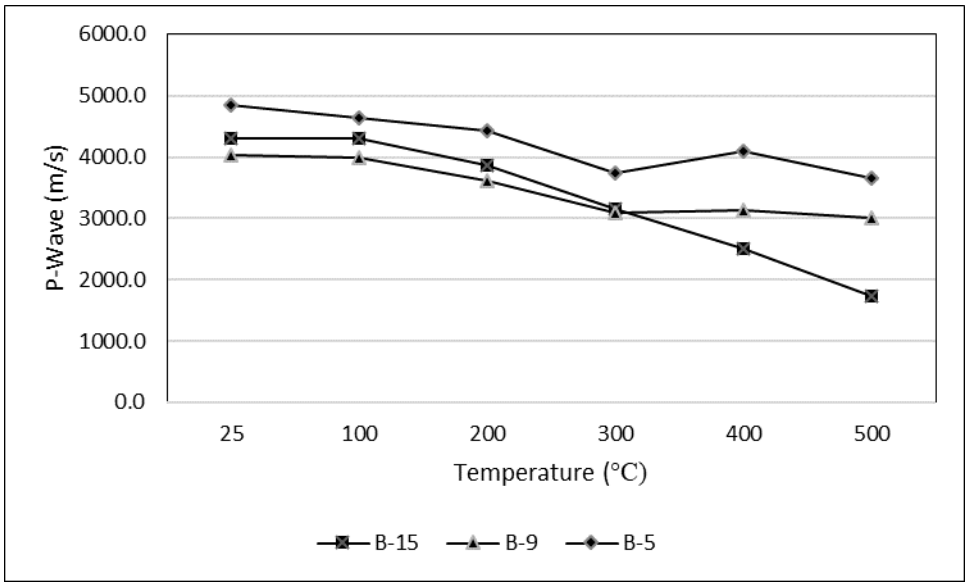

Figure 4.90. Variations in *P*-Wave velocity as the temperature increases for samples B-15, B-9 and B-5

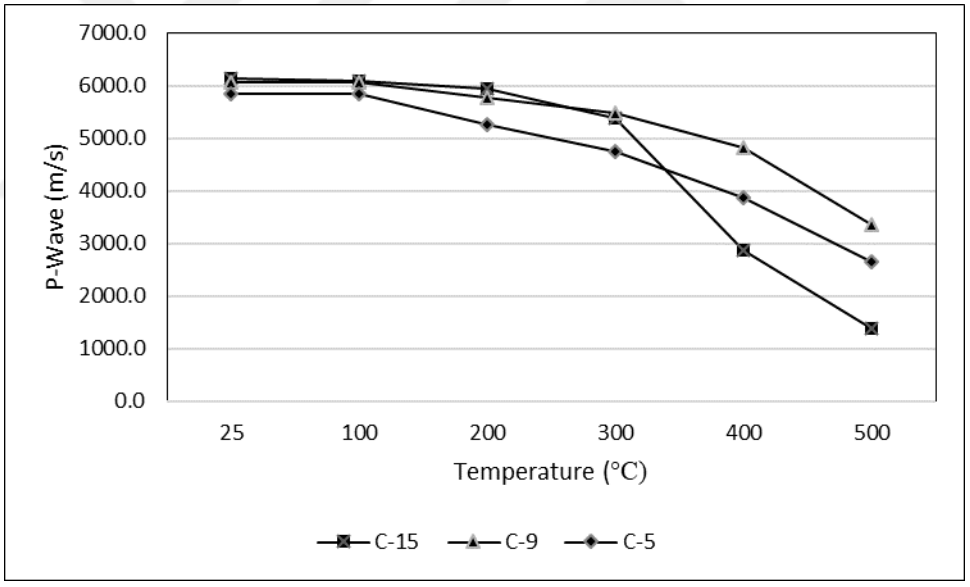

Figure 4.91. Variations in *P*-Wave velocity as the temperature increases for samples C-15, C-9 and C-5

4.2.5. Effect of temperature on Shore hardness values

Variations in Shore hardness values with the increasing temperature are shown in Figures 4.92. – 4.94. Especially the samples of Mugla White marble (sample A) demonstrated a significant drop on SH values at the temperatures above 200°C - 300°C, because of the fact that Muğla White marble tended to disintegrate beyond such temperatures and it became so difficult to acquire SH data on samples A. For the samples B-9 and C-9, a slight decrease of 6-8% in SH values were observed. For the samples B-5, the decrease in SH value was near 3%. However, for sample C-5, a slight increase of 4% in SH value was noted up to the temperature of 200°C and a slight decrease of 4% was observed above the temperature of 200°C (Figure 4.94.).

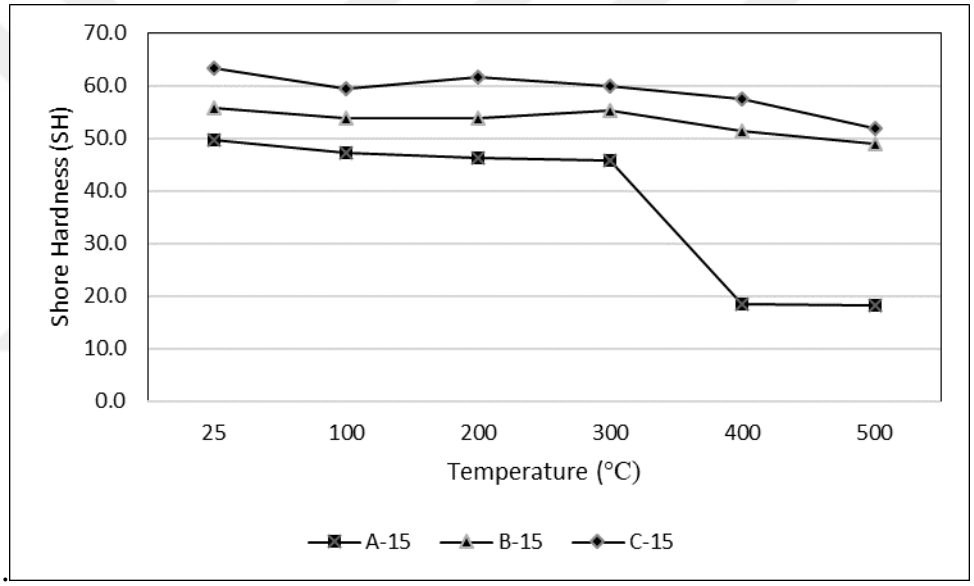

Figure 4.92. Variations in Shore hardness as the temperature increases for samples A-15, B-15 and C-15

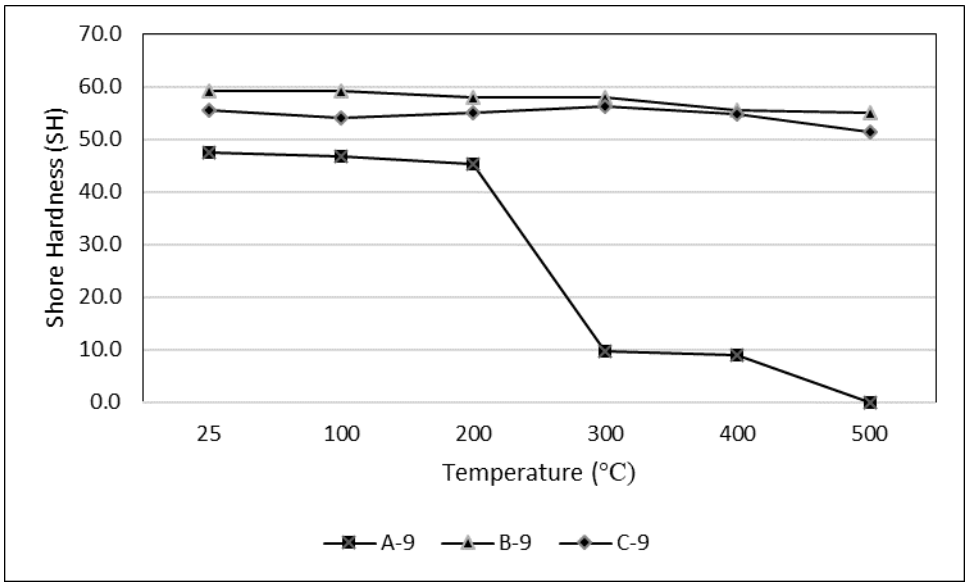

Figure 4.93. Variations in Shore hardness as the temperature increases for samples A-9, B-9 and C-9

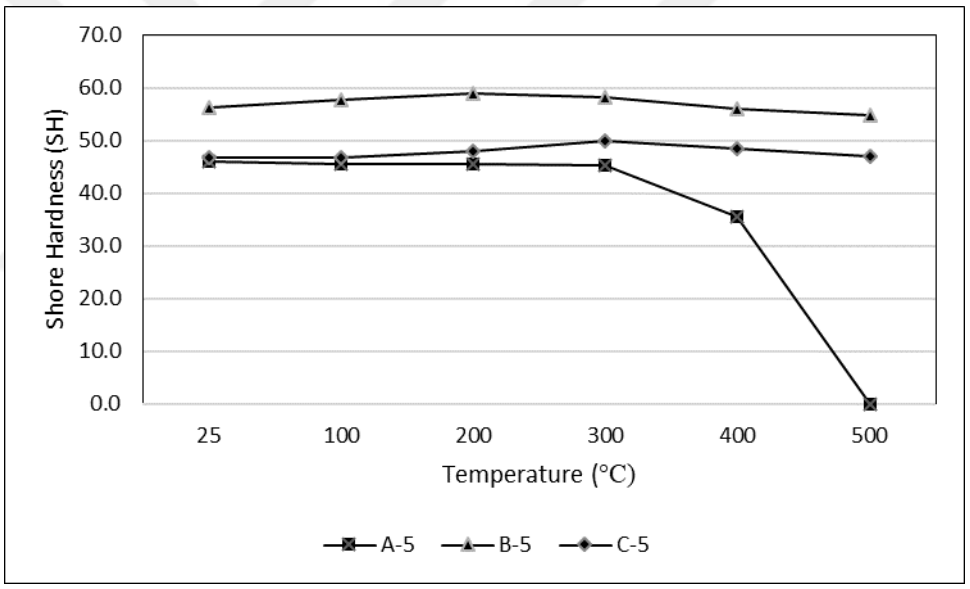

Figure 4.94. Variations in Shore hardness as the temperature increases for samples A-5, B-5 and C-5

4.2.6. Effect of sample size on Shore hardness

Variations in Shore hardness values with sample size are given in Figures 4.95. – 4.97. Similarly, the samples of Mugla White marble (sample A) displayed a significant drop on SH values at the temperatures above 200°C -300°C, because of the same fact that Muğla White marble tended to disintegrate beyond such temperatures and it became so difficult to acquire SH data on samples A.

No significant variations in SH values were determined for the samples B and C, as shown in Figures 4.96. and 4.97.

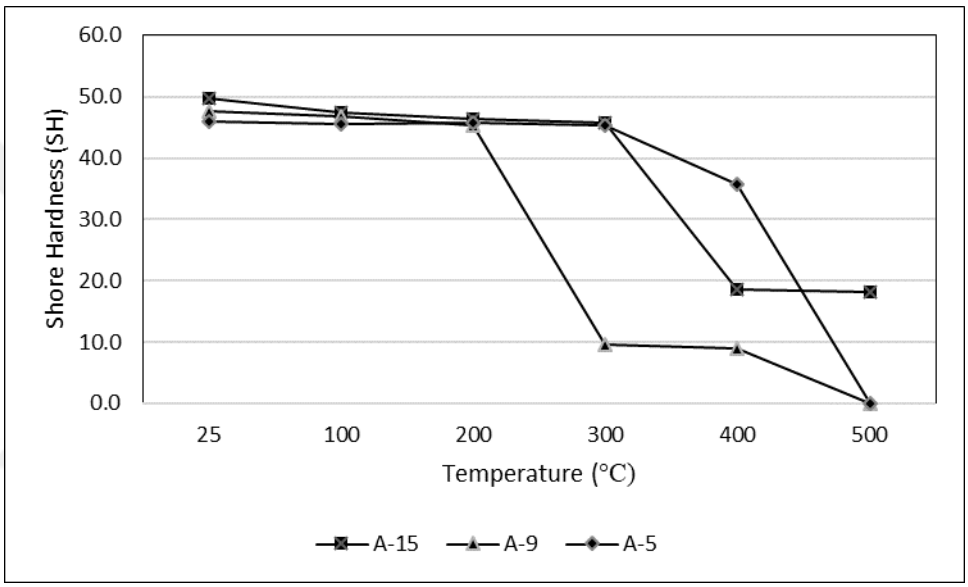

Figure 4.95. Variations in Shore hardness as the temperature increases for samples A-15, A-9 and A-5

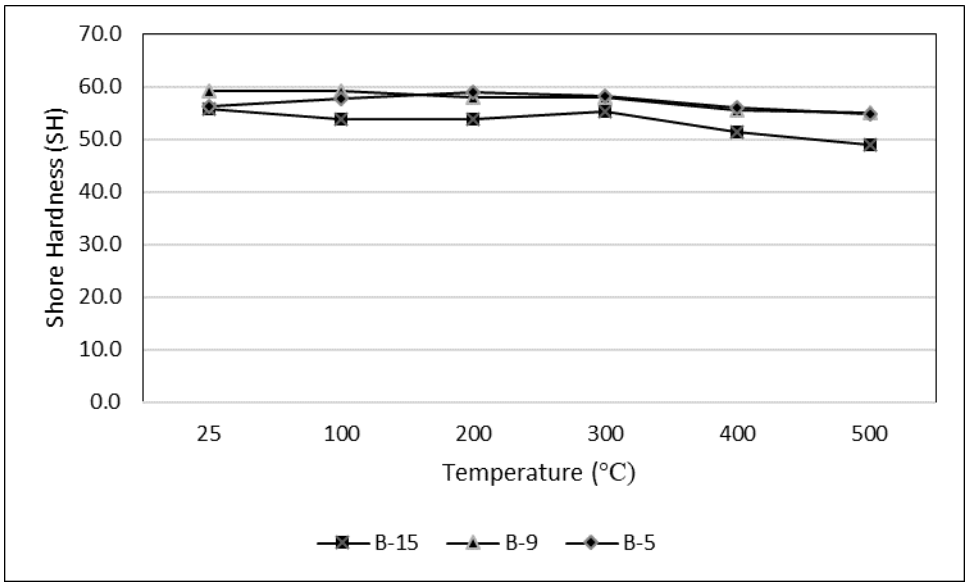

Figure 4.96. Variations in Shore hardness as the temperature increases for samples B-15, B-9 and B-5

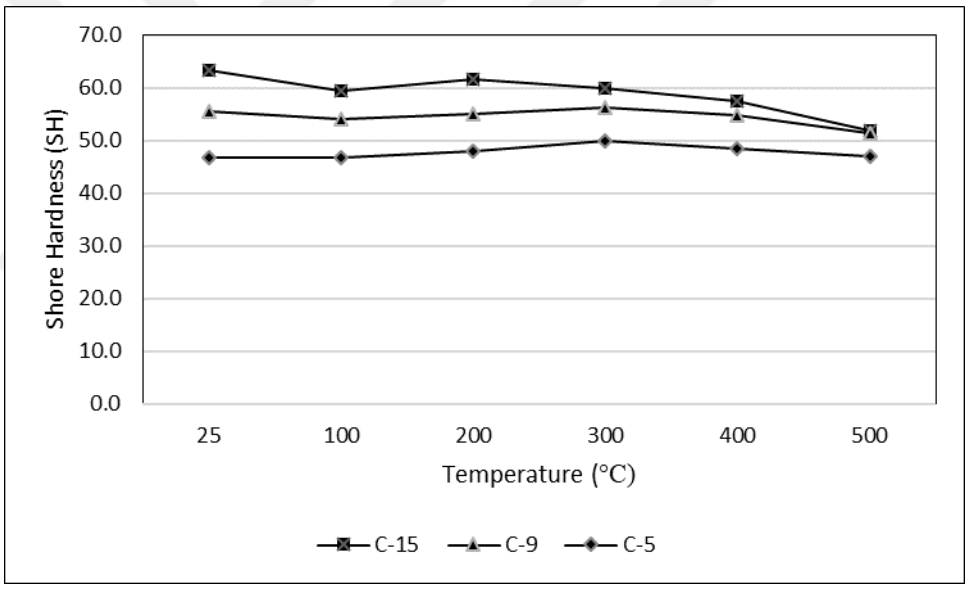

Figure 4.97. Variations in Shore hardness as the temperature increases for samples C-15, C-9 and C-5

4.3. Porosity and UCS Experiments Post Heating Process

For porosity and uniaxial compressive strenght (UCS) tests, cubic samples of 9 cm edge length were selected and were used in the experiments of thermal shock and thermal damage. In Figure 4.98., samples A, B, C, D and E are the original samples which were used in the first experimental set (Thermal shock). Sample A1, B1, C1, D1 and E1 (Figure 4.99.) were heated to 100°C in 45 cycles and thermal shock tests were performed.

Figure 4.100. illustrates the samples A-9, B-9 and C-9, prior to thermal damage test and A1-9, B1-9 and C1-9, following the heating process of 500°C temperature.

Figure 4.98. Original samples (at 0°C)

Figure 4.99. Samples subjected to thermal shock tests (at 100°C)

Figure 4.100. Samples pre- and post-thermal damage tests (A-9, B-9, C-9 at 0°C and A1-9, B1-9, C1-9 at 500°C)

Some of the physical properties of the samples are listed in Table 4.15. When the density values are evaluated, in the end of both thermal shock and thermal damage tests, variations between the densities were in general found to be insignificant. Maximum difference in density values was calculated to be 0.36 for the samples B and B1 in thermal shock tests (Table 4.15.).

Total porosity was seen to increase in all samples in the end of both thermal shock and thermal damage tests. The largest increase was noticed in the samples A and A1 in the thermal shock test (Table 4.15.).

Table 4.15. Physical properties of the samples

Test	Sample	Volume (cm^3)	Weight (gr)	Dry Weight (gr/cm^3)	Saturated Weight (gr/cm^3)	Dry Density (gr/cm^3)	Saturated Density (gr/cm^3)	Natural Density (gr/cm^3)	Pore Volume (cm^3)	Mineral Volume (cm^3)	Mineral Grain Density (gr/cm^3)	Effective Porosity (%)	Void Ratio (%)	Total Porosity (%)
Thermal Shock	**A (0°C)**	729.00	1726.26	1725.13	1765.30	2.3664	2.4215	2.3680	40.170	688.83	2.563	5.51	0.058	7.660
	A1 (105°C)	729.00	1627.81	1624.75	1719.72	2.2287	2.3590	2.2329	94.970	634.03	2.712	13.03	0.150	17.830
	B (0°C)	770.22	2120.44	2120.00	2131.08	2.7524	2.7668	2.7530	11.080	759.14	2.807	1.44	0.015	1.951
	B1 (105°C)	720.90	1723.54	1723.21	1745.28	2.3904	2.4210	2.3908	22.070	698.83	2.497	3.06	0.032	4.287
	C (0°C)	728.91	2015.83	2015.31	2017.48	2.7648	2.7678	2.7655	2.170	726.74	2.776	0.30	0.003	0.405
	C1 (105°C)	728.91	2009.36	2009.16	2012.10	2.7564	2.7604	2.7567	2.940	725.97	2.772	0.40	0.004	0.549
	D (0°C)	736.74	1956.33	1955.67	1970.90	2.6545	2.6752	2.6554	15.230	721.51	2.732	2.07	0.021	2.824
	D2 (105°C)	761.11	1959.66	1959.27	1973.42	2.5742	2.5928	2.5747	14.150	746.96	2.642	1.86	0.019	2.563
	E (0°C)	819.00	2118.87	2112.44	2128.95	2.5793	2.5995	2.5871	16.510	802.49	2.653	2.02	0.021	2.776
	E2 (105°C)	778.60	1971.64	1971.24	1991.68	2.5318	2.5580	2.5323	20.440	758.16	2.627	2.63	0.027	3.625
Thermal Damage	**A-9 (0°C)**	729.00	1997.33	1997.18	1999.93	2.7396	2.7434	2.7398	2.750	726.25	2.754	0.38	0.004	0.514
	A1-9 (105°C)	729.00	1969.88	1969.80	1986.14	2.7021	2.7245	2.7022	16.340	712.66	2.787	2.24	0.023	3.046
	B-9 (0°C)	729.00	2013.05	2030.22	2033.68	2.7849	2.7897	2.7614	3.460	725.54	2.803	0.47	0.005	0.644
	B1-9 (105°C)	729.00	2055.61	2055.37	2070.51	2.8194	2.8402	2.8198	15.140	713.86	2.900	2.08	0.021	2.793
	C-9 (0°C)	729.00	1739.94	1738.33	1766.94	2.3845	2.4238	2.3867	28.610	700.39	2.523	3.92	0.041	5.480
	C1-9 (105°C)	729.00	1677.97	1677.63	1713.42	2.3013	2.3504	2.3017	35.790	693.21	2.472	4.91	0.052	6.896

Table 4.16. The experimental results for UCS

Test	Sample	Max Load (kN)	Pace Rate (kN/s)	Stress (MPa)	Max Load (kg)	Area (cm^2)	Compressive Strength (kg/cm^2)
Thermal Shock	A (0°C)	171.7	0.8	21.198	17,508.52	81	216.15
	A1 (105°C)	163.9	0.8	20.235	16,713.15	81	206.34
	B (0°C)	1342.6	0.8	165.753	136,907.07	81	1690.21
	B1 (105°C)	391.4	0.8	48.321	39,911.68	81	492.74
	C (0°C)	829.2	0.8	102.370	84,554.85	81	1043.89
	C1 (105°C)	144.8	0.8	17.877	14,765.49	81	182.29
	D (0°C)	688.0	0.8	84.938	70,156.46	81	866.13
	D2 (105°C)	563.1	0.8	69.519	57,420.21	81	708.89
	E (0°C)	567.6	0.8	70.074	57,879.08	81	714.56
	E2 (105°C)	469.1	0.8	57.914	47,834.88	81	590.55
Thermal Damage	A-9 (0°C)	520.0	0.8	64.198	53,025.23	81	654.63
	A1-9 (105°C)	352.6	0.8	43.531	35,955.19	81	443.89
	B-9 (0°C)	174.8	0.8	21.580	17,824.64	81	220.06
	B1-9 (105°C)	1268.5	0.8	156.605	129,350.97	81	1596.93
	C-9 (0°C)	236.8	0.8	29.235	24,146.87	81	298.11
	C1-9 (105°C)	302.9	0.8	37.395	30,887.20	81	381.32

When the result obtained from uniaxial compressive strength tests are evaluated, the samples were seen to lose strength in general in the end of both thermal shock and thermal damage tests. However, in thermal damage test, a sharp increase in the strength of sample B-9 was noticed. This may be elucidated by examining evolution of micro-cracks in rock. There exists a large amount of micro-cracks in original rock sample. As the samples are heated to about 100℃ and above, internal mineral particles will start swelling, which will cause the reduction in the length of original cracks and area. Depending on the amount of swelling, they may even close and the number of micro-cracks will be reduced. This process will result in the increase in the value failure strength of rock material.

5. DISCUSSION

In literature, a number of studies have been conducted on the influence of heat treatment on certain physical and mechanical properties of carbonate rocks. The results obtained in this study have proven to be coherent with the outcomes of the studies found in literature.

Demirdag (2013) have implemented thermal shock tests on travertine samples by 50 cycles. He reported increases in porosity values while decreases were noticed in weight, unit volume weight, *P*-wave and point load index values. In this research, increasing heat has caused an increase in porosity as it induced an increase in weight loss and a decrease in unit volume weight.

Yavuz et al. (2006) conducted thermal shock experiments on different carbonate rocks by 20 cycles. In their work, *P*-wave values appeared to decrease. They have tested a total of 12 samples to determine the UCS values of the rocks. Eleven of 12 samples demonstrated a decrease in UCS values. Only one sample displayed an increase in UCS value. In this thesis, only two samples demonstrated an increase in UCS values in thermal damage tests. Also, fluctuation was observed in Shore hardness tests displayed fluctuating results owing to the extreme degradation of some of the rock samples under high temperatures above 300°C.

In this thesis study, samples of carbonate rocks were subjected to very high heats by increasing the temperartures up to 800°C (for thermal damage) to determine evolutions in certain physical and mechanical properties. Also, samples were treated heat to 105°C for thermal shocks under thermal cycles. In the result of thermal stresses, micro or visible macro cracks grew and coalesced or new cracks were formed and induced notable strength losses, decreases in certain physical and mechanical properties of rocks, depending on various factors such as temperature, duration of heating, cooling and some other rock properties like mineralogical content, grain size, porosity, micro cracks before the heating process. Strength loss is usually caused by the variations in strains of different minerals that make cracking at the contacts of minerals and grows through present cracks. By this process, physical

weathering of rock like materials will occur. On the other hand, high temperatures will induce chemical weathering and that must be noted as to why strength losses should ocur in rock like natural materials due to the thermal changes. For chemical weathering, mineralogical content of rock and temperature level will be deciding parameters. In this study, only influence of phsical weathering was rather considered. However, the auther of this thesis is fully aware of the importance of the effect of chemical weathering on the evolution in physical and mechanical properties.

6. CONCLUSIONS

In this thesis, two sets of experimental works were implemented to investigate the influence of temperature variations (heating/cooling) and thermal damage at high temperatures on key physical properties and mechanical behavior of marbles which were collected from the quarries situated in Mugla region.

In the first set of experiments, laboratory tests were carried out on the samples dimensioned from 5 different marble types to investigate the properties of deteriorated rocks due to thermal shock treatment (at 105°C) of 45 cycles and thermal damage tests at high-temperature steps of 25-800°C. It was concluded from the test results that index properties (Weight, *P*-wave velocity and Shore hardness) of heat treated rocks were seen to decrease by varying levels when compared to the original values.

In the second set of experiments, samples prepared from 3 different marble types were utilized in the investigation of the effects of thermal damage at high temperature of 25-500°C. It was found that the index properties such as weight, *P*-wave velocity and Shore hardness of the treated samples decreased by varying levels when compared to the original values. Especially *P*-wave velocity and Shore hardness values were seen to decrease significantly as the temperature increased to high degrees.

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