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Laboratory Study of Weight Loss and Ultrasonic Wave Propagation Velocity in Conventional Concrete Under High Temperature, Along with Validation by SEM and XRD Analysis

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ABSTRACT

In this laboratory study, a mixed concrete design containing Portland cement type 2 with a grade of 500 kg/m³ was made. Weight loss and ultrasonic pulse velocity (UPV) tests were performed on concrete samples at a curing age of 90 days at room temperature (21 °C) and under a temperature of 600 °C. In order to further investigate and verify the results, scanning electron microscopy (SEM) and X-ray diffraction (XRD) analyses were performed on concrete samples at a curing age of 90 days. The results of the tests in this study were evaluated together and compared and analysed with the results of other researchers. Applying high heat to concrete samples caused a decrease in results. In this regard, in the weight loss test of concrete samples, the weight of the concrete sample decreased from 2378 to 2225 grams, which was a decrease of 72.6%, and in the UPV test, the speed of ultrasonic waves decreased from 6179 to 3411 m/s, which was a decrease of 79.44%. At the end of this study, the results of SEM and XRD analysis at room temperature and under high temperature, while coordinating with each other, overlapped with the results of the weight loss test and the ultrasonic wave velocity test in concrete samples.

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1. Introduction

In order to develop national security and passive defense in the field of civil engineering infrastructure, a lot of laboratory work has been carried out to produce structural concrete for strategic and sensitive centers of the country [1-3]. The strength of reinforced concrete structures of these centers plays an important role in reducing destruction and human injuries caused by enemy

defense operations. Improving the strength of concrete can be achieved through the type of materials used in its mixture composition [4-10]. One of the methods for evaluating the durability of concrete against various physical factors is to examine changes in the density of concrete in different temperature ranges. Concretes with low durability against heat lose more water in the pores and interlayer capillary pores in hydrated gels, therefore experiencing greater weight loss. UPV testing is one of the

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types of non-destructive tests for measuring the homogeneity and strength of concrete. In various studies, this test has been used to qualitatively evaluate the strength of concrete [2-5,7]. Research has shown that with increasing curing age in concrete, the UPV improvement process is divided into three parts based on the time-rate curve. In the first part, called the stationary period, UPV is very small. In the second part, with the development of the polymerization process (hydration or polymerization), UPV increases rapidly until, with the emergence of a solid network (final setting), the rate of increase decreases, which is characterized as the third part [11].

Portland cement is one of the major types of cements that is most widely used in the construction and production of ordinary concrete. Portland cement is a type of hydraulic cement that mainly consists of CaO , SiO_2 , Al_2O_3 and Fe_2O_3 , these oxides are mainly present in the concrete in a bonded form [12]. In ordinary concrete paste containing Portland cement, calcium silicate-hydrate (C-S-H) gel is formed from silicon groups [13]. This gel constitutes a high volume (between 50 and 60%) of the Portland cement paste. Many models have been proposed to describe the exact structure of C-S-H. According to the Powers-Bronwar model, this structure is in the form of layers with a large surface area. The specific surface area of C-S-H is estimated to be about 100 to 700 m^2/g [14]. The strength of this structure is attributed to van der Waals forces. In the Feldman-Serda model, the C-S-H structure is represented as a series of irregular layers that are randomly placed together to create interlayer spaces with various sizes and shapes, ranging from 5 to 25 angstroms [4]. The C-S-H gel is a factor in improving the strength of the hardened cement paste matrix structure. One of the methods for improving the results of the aforementioned tests (weight reduction and UPV) in conventional concrete is to increase the cement content in the concrete. Research has shown that by increasing the cement content from 275 to 400 kg/m^3 in conventional concrete, the density of the concrete increases [15]. By increasing the density of the concrete, the mechanical properties and durability of the concrete will improve [1,6].

On the other hand, by increasing the cement content in the concrete and keeping the water-to-cement ratio low, the mechanical properties of the concrete can be increased, and the poor performance of the concrete in these conditions can be compensated by adding a superplasticizer [16]. As the cement content of concrete increases, the volume of C-S-H gel produced in the chemical process increases. This gel increases the density and strength of concrete by filling the pores and bonding the bonds, especially in the interfacial transition zones (ITZ), the interface between the paste and the aggregates. However, the high consumption of cement has always been accompanied by environmental concerns. In this regard, research shows that cement

factories are responsible for the emission of about 5% of the total carbon dioxide entering the Earth's atmosphere [17]. In the microstructure of ordinary concrete containing Portland cement, hydrated calcium silicate (C-S-H) known as tobermorite gel accounts for 50 to 60 percent, calcium hydroxide ($\text{Ca}(\text{OH})_2$) or portlandite accounts for 20 to 25 percent, and hydrated calcium aluminum silicate (C-A-S-H) or ettringite gel accounts for 15 to 20 percent of the cement paste volume, with the remainder being non-hydrated clinker grains [18]. The effect of increasing temperature on the hardened hydrated cement paste depends on various factors, including the degree of polymerization and the moisture content of the cement paste. A well-hydrated and saturated cement paste contains large amounts of capillary water and free water in excess of surface water. The collapse of the concrete structure occurs when the rate of increase in the pressure of the vapor gas inside the material exceeds the rate of decrease in pressure due to the release of vapor into the atmosphere. When the temperature in the concrete reaches 300 °C, the interlayer water in the hydrated calcium silicate gel and some of the water that is chemically combined and results from C-S-H and sulfoaluminate hydrates are lost. The subsequent dehydration of the cement paste due to the decomposition of calcium hydroxide (hydrated lime with the chemical formula $\text{Ca}(\text{OH})_2$) begins in the temperature range of 600 °C, but a temperature of about 900 °C is required for complete decomposition of C-S-H [14]. Hertz reported that the C-S-H structure decomposes at 600 °C and is destroyed at 800 °C [19]. Applying high temperatures to concrete causes a decrease in the results of weight loss tests [1,6] and UPV [3-5] in concrete. Weight loss of conventional concrete samples under high temperatures has also been reported in other studies [20]. Using SEM and XRD image analysis is an effective way to evaluate the microstructure and the extent of chemical changes in the hardened cement matrix structure [3,4].

By examining the results of these tests, it is possible to have a good understanding of the performance of concrete in tests on the evaluation of mechanical properties and durability of concrete at room temperature and high temperatures. The investigation of mechanical, durability, microstructural and chemical properties in conventional concrete with high grade (500 kg/m^3) of Portland cement at room temperature and under high temperatures is proposed in this article as an innovative design that can be used in structures requiring high mechanical properties and durability.

2. Sample Construction and Laboratory Program

2.1. Consumable Materials

In this study, Portland cement type 2 with chemical and physical characteristics according to Tables 1 and 2 was used. This cement is a product of Gilan Sabz Cement Industries Company (Dilman) and is produced under the EN 197-1 standard. The water used to prepare lime water and make the mixture design in the present study is from the drinking water of Lahijan city, this water has a pH in the range of 6.5 to 7.5 and a density of 1000 kg/m³. According to Section 9-10-4-2 and 9-10-4-3 of the fourth edition of the National Building Regulations of Iran, water that is drinkable, has no distinct taste or odor, and is clean and clear can be used in concrete without testing, unless previous records indicate that this water is unsuitable for concrete [21].

The aggregate grading curve used is within the ASTM C33 standard range, as shown in Figure 1. The aggregates used were supplied from sand and gravel factories in Lahijan city and were cleaned to remove organic impurities. Some of the characteristics of the fine and coarse aggregates used in this study were determined based on Table 3. The ideal aggregate grading is of particular importance because the aggregates with the highest density and the least amount of pores are the most economical materials used in concrete construction, which, if of appropriate quality, will require the least amount of cement paste [18]. The superplasticizer used in this study is a fourth-generation normal polycarboxylate-based product from Durochem Middle East Company under the trade name Flowcem R700. This material is used to compensate for the poor efficiency and maintain the fluidity of the mortar mixture due to the high cement content in concrete. Some characteristics of normal polycarboxylate superlubricants are presented in Table 4.

Table 1.

Chemical Characteristics of Portland Cement Type II

L.O.I	MAX 1/5
C ₃ A	5.5-7.5
I.R	MAX 0/7
Na ₂ O+0.658K ₂ O	MAX 0.6
SO ₃	2-2.3
MgO	MAX 4/5
Cao	42-43
Fe ₂ O ₃	3.5-3.8
Al ₂ O ₃	4.5-4.8
SiO ₂	21-22
Cl	MAX 0/003

2.2. Mix Design and Curing Concrete

In this laboratory study, the mixture design recommended by ACI 211.1-89 was used to make regular concrete. In this regard, the mixture design of concrete samples in this study was prepared and adjusted based on Table 5. In order to make concrete samples, dry materials (aggregate and cement) were first poured into a rotating mixer and the mixing process lasted for 2 minutes, then

water and superplasticizer were added to the mixture and the mixing of the materials continued for another 2 minutes. Finally, the prepared concrete mixture was poured into pre-lubricated molds in three stages and in each stage, the concrete mixture was compacted with 25 rod blows. After the first 24 hours of concrete pouring and the samples were kept in a dry environment and at room temperature (21 °C), the samples were separated from the mold and kept in lime water at room temperature until the test.

Table 2.

Physical Characteristics of Portland Cement Type II

Density (kg/m ³)	3250
Specific Surface Area (cm ² /g)	3000-3200
Initial Setting (min)	115< <130
Secondary Setting (min)	190< <205

Table 3.

Aggregate Specifications

Concrete Aggregates	Gravel	Sand
Minimum Diameter	4.75 (mm)	75 (μm)
Maximum Diameter (mm)	19	4.75
Modulus of Elasticity (mm)	5.7	2.85
Density (kg/m ³)	2750	2650
Water Absorption (%)	2.2	2.9

Table 4.

Superplasticizer Characteristics

Chemical Formula	Normal Polycarboxylate
Physical Condition	Liquid
Color	Light Brown
Density (kg/m ³)	1100
Consumption Standard	ASTM C494
pH	About 7

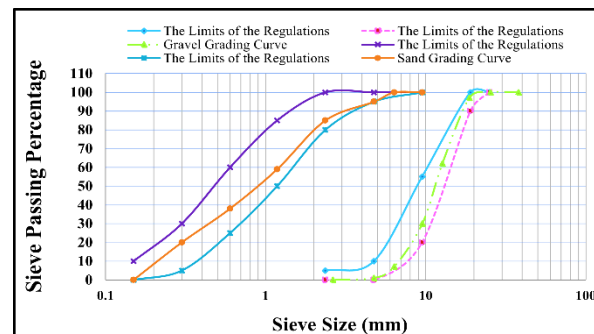


Fig. 1. Aggregate Curve

Table 5.

Specifications of Concrete Mix Design

Materials	kg/m ³	%
Cement	500	20.02
Water	225	9.01
Gravel	1000	40.04
Sand	765	30.63
Superplasticizer	7	0.0028
- Curing Conditions After Demolding = In the water		
- Density of Concrete Mix = 2497 kg/m ³		
- W/C = 45 %		

2.3. Test Methods and Standards

The weight loss test of concrete at a curing age of 90 days at room temperature and under high heat was carried

out in accordance with the ISO834 standard on cubic samples with dimensions of 10×10×10. In this standard, the temperature applied to concrete samples is recommended to be up to 1000 °C and the duration of heat application is one hour [1,6]. The samples were first weighed at room temperature, then the samples were exposed to a temperature of 600 °C for 1 hour in an oven, this action causes water to evaporate from the capillary spaces and possible pores in the concrete, at the end of the heating time and after the temperature of the samples reached room temperature, the samples were re-weighed and the average weight loss of the samples was entered as the final result. UPV test in concrete was measured under ASTM C597 standard on cubic samples with dimensions of 10×10×10 cm at room temperature and high temperature, with a Dundee type device with a vibration frequency of 55 kHz and an accuracy of 0.1 and ±2% of the time interval of the waves. In this regard, at a curing age of 90 days, the surface dimensions of the samples that were in contact with the mold bottom were measured by calipers, then a suitable acoustic connection was achieved between the concrete surface (smooth surfaces that were in contact with the mold faces) and the transducer surface to remove air pockets through Vaseline on the surface of the samples. After determining the time of wave passage through the concrete sample, using the relationships in the mentioned standard, UPV was obtained in meters per second. XRD analysis of concrete at a curing age of 90 days under room temperature and high temperature was performed using an XRD device with a Philips PW1730 model. In this regard, crushed samples taken from the center of the concrete sample were placed inside the device and during the test, a diffraction diagram of concrete crystals was prepared. The data obtained from X-ray diffraction is in the form of photon intensity in terms of detector angle 2θ, which is presented as a list of peak locations and their intensities on the graphs. SEM analysis was performed at a curing age of 90 days at room temperature and under high temperature using a scanning electron microscope with the FEI Quanta200 model. In this regard, the crushed concrete sample was placed in the device and images were recorded at the desired magnification and then microstructurally examined. Before performing high-temperature tests (600 °C) at a curing age of 90 days, according to ISO834 standard, concrete samples were placed in a furnace at a temperature of 600 °C for 1 hour, then the samples remained in the furnace for another 1 hour to avoid the effect of thermal shock. After the samples were removed from the furnace, the samples were kept at room temperature for 24 hours to reach temperature equilibrium. The use of this standard in other research on high-temperature tests in concrete has been reported [3,4,5,22].

3. Laboratory Results

3.1. Weight Loss Test Results

The results of the weight loss test of concrete samples at a curing age of 90 days at room temperature and under a temperature of 600 °C are shown in Figure 2. In this regard, the weight of the concrete sample has experienced a 6.81% decrease from 2387 to 2225 grams. Considering the passage of 90 days of concrete curing age, a significant part of the polymerization process has been completed in the concrete. In this regard, most of the cement binder and hydrated filler particles such as calcium hydroxide have participated in the chemical process, and the result is the production of a large volume of hydrated calcium silicate gel (C-S-H) in the concrete structure, which, while filling the pores and cavities, has caused a bond in the ITZ between the aggregates and the cement paste, and in this way has increased the compaction, density and strength of the concrete. These characteristics cause the resulting concrete to have less weight loss against the applied heat [1,6]. Researchers have reported that the decrease in strength is mainly attributed to the decomposition of calcium hydroxide, and this phenomenon usually occurs in the temperature range between 450 and 500 °C [23,24]. Therefore, in high-strength concrete, greater strength occurs in this temperature range. Heating up to 100 °C causes water to evaporate from the pores and capillary spaces in the concrete structure and intensifies the porosity in the concrete. At temperatures above 100 °C, due to the exit of water from the concrete structures and the start of the polymerization process, the sample shrinks and cracks form due to drying caused by the evaporation of water in the concrete. According to the study of other researchers, when the vapor pressure reaches its maximum, the dense structure of concrete with low permeability is unable to control the thermal stresses, and this causes thermal cracks to appear on the surface of the sample due to shrinkage. In concrete, this phenomenon is called the "vapor effect" [25].

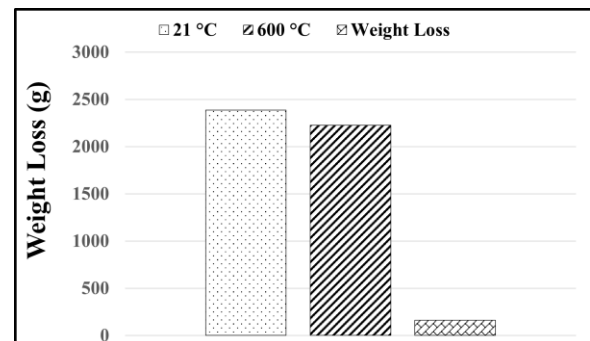


Fig. 2. Results of Weight Loss Test of Concrete Sample

Also, the evaporation of water from the concrete structure is accompanied by weight loss, which can cause thermal cracks due to shrinkage. The results of XRD and SEM analysis in this study confirm the results of the weight loss test.

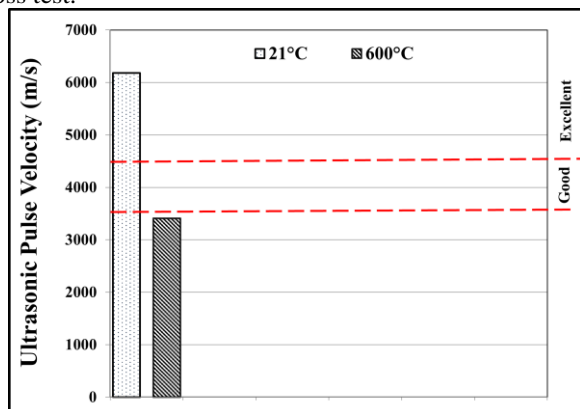


Fig. 3. UPV Test Results

3.2. UPV Test Results

In this study, the results of the UPV test at the 90-day curing age of concrete at room temperature and under high temperature are shown in the graph of Figure 3. Research has shown that the UPV in ordinary concrete at the 90-day curing age has the least changes compared to the ages of 7 and 28 days due to the completion of a large part of the polymerization process [26]. Based on the results of Figure 3, it is observed that at room temperature and 600 °C, the UPV rate has reached 6179 and 3411 m/s, respectively, which shows a 79.44% drop in UPV at high temperature. The IS 13311-1 standard shows the quality range of concrete in the UPV test [27]. The use of this standard for classifying the quality of ultrasonic wave velocity has also been reported in other studies [2-5,7,28]. In this study, at room temperature, the concrete is in the excellent quality range. According to IS 13311-1, as long as the UPV values are in the “excellent” category, it means that the concrete in question does not have large cracks or voids that affect the structural integrity of the specimen [29].

The resistance of concrete to heat depends on various factors such as the chemical composition of the concrete ingredients, temperature, and curing method [4,30]. Heat causes water to evaporate from the pores and cavities in the concrete, and while weakening the polymerization process and destroying the C-S-H gel structure, it will increase the porosity in the concrete [3,5]. The presence of any porosity and cracks in the concrete will lead to a decrease in the UPV in the concrete. Considering the 90-day curing time of the concrete in this study, the participation of the filler and cementitious adhesive particles in the polymerization process has reached its maximum, and this has led to the

production of a high volume of C-S-H gel in the concrete, which has contributed to increasing the compaction and density of the concrete (according to the SEM result in Figure 5) by filling the cracks and improving the bond in the ITZ. The bond in conventional concretes is formed based on polymerization reactions between calcium oxide and silicon dioxide to form hydrated calcium silicate (C-S-H).

3.3. XRD Analysis Results

In this laboratory study, X-ray diffraction (XRD) spectroscopy was used to study and analyze the crystalline structure of materials and to investigate the size of grains and particles in concrete. The results of XRD analysis at a curing age of 90 days at 21 and 600 °C are shown in Figure 4. Based on these results, it is observed that at 21 °C, aluminum phosphate compounds with a maximum peak height of 2670 at an angle of 59.85 degrees, followed by calcium hydroxide with a maximum peak height of 2452 at an angle of 24.41 degrees, titanium oxide with a maximum peak height of 1794 at an angle of 24.29 degrees, calcite with a maximum peak height of 1600 at an angle of 26.45 degrees, and dolomite with a maximum peak height of 671 at an angle of 17.92 degrees, have the highest dispersion. The presence of calcium hydroxide with the highest dispersion in XRD indicates that a large part of this hydrated material has not been able to fully participate in the chemical process, and this could be due to the high cement content in concrete, which increased the polymerization rate and did not allow some cementitious materials to participate chemically. After the removal of some elements in the calcination process at high temperature, compounds such as calcium manganese carbonate with a maximum peak height of 2092 at an angle of 29.47 degrees, carbon with a maximum peak height of 1865 at an angle of 27.9 degrees, hydrated potassium iron manganese aluminum silicate with a maximum peak height of 1712 at an angle of 28.13 degrees, and hydrated calcium aluminum silicate with a maximum peak height of 726 at an angle of 26.42 degrees have the highest dispersion. The application of high heat reduced the height of the peaks due to the presence of compounds, in this regard, the difference in peak height in the XRD graph at 21 °C (with a height of 2092), compared to 600 °C (with a height of 2670), is 21.64%. Research has shown that at high temperatures, CH gel does not convert into calcium carbonates such as Calcite, and as is clear from the results of the table, CH disappears at high temperatures and is actually converted into Carbon and C-A, which is the main cause of concrete weakness at high temperatures [31,32]. Research has shown that after the materials used in the preparation and manufacture of concrete are combined, the chemical reaction process (polymerization) between cementitious

materials and water begins, the speed and extent of formation of hydrated calcium silicate gel (C-S-H), which is the final product of the chemical composition, mainly depends on the properties and ratios of cementitious materials and water [1,2].

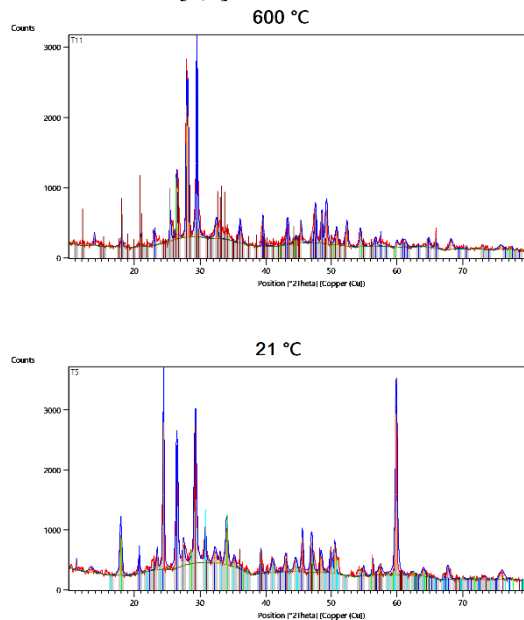


Fig. 4. XRD Analysis Results

3.4. SEM Analysis Results

The results of SEM analysis at a curing age of 90 days at a scale of 5 μm and a magnification of 13,000 times at temperatures of 20 and 600 $^{\circ}\text{C}$ are shown in Figure 5. Based on these images, the microstructure of concrete in all mix designs can be summarized in three basic phases as follows [5,1-7]:

1. The first phase consists of polymerization products including hydrated gels, which are mainly dark in color in the images. After formation and in combination with other concrete components, these gels are known to be the main factor in strengthening the microstructure of concrete by filling pores and cavities, as well as improving the bond in the ITZ and interlayer (in the hydrated gels themselves).
2. The second phase consists of unreacted crystals that are formed as a result of impurities in the raw materials or unreacted particles in the polymerization process and are mainly white in the images.
3. The third phase includes the bonding of cement paste with aggregate in ITZ, as well as the bonding of interlayers in the structure of hydrated gels.

According to SEM images at 21 $^{\circ}\text{C}$, it is clear that the presence of hydrated C-S-H gel in the concrete sample has caused the connection of fracture surfaces, the homogeneity of the hydration product, and the densification of the Portland cement paste structure. Also, this gel, by filling the ITZ space, causes reinforcement at the paste-aggregate boundary or the adhesion zone between aggregate and cement paste (ITZ). The presence of some small crystals, fracture surfaces, and white spherical particles caused by calcium hydroxide crystals that did not participate in the production of hydrated gel in the concrete structure of this design, which can be seen in the images, can be attributed to the high grade of cement used, which leads to the phenomenon of nucleation and agglomeration of particles (due to the high speed of the hydration process). Also, the presence of pores and capillary pores can often be considered the result of water evaporation from the interlayer capillary spaces in the hydrated calcium silicate gel (C-S-H), which contributes to the creation of cracks and shrinkage phenomena due to drying.

Research has shown that in the bulk part of Portland cement paste, ions such as calcium, sulfate, hydroxide, and aluminate, which are formed through aerobic dissolution into calcium silicate and calcium aluminate, combine to form ettringite gel (C-A-S-H) and calcium hydroxide ($\text{Ca}(\text{OH})_2$), meaning that ettringite is formed by the reaction of calcium aluminate with calcium sulfate, and as the hydration stage progresses, weak C-S-H crystals and the second generation of crystals formed from calcium hydroxide and ettringite gel begin to fill the empty spaces in the ettringite and calcium hydroxide network (called portlandite), and this operation increases the density, hardness, and ITZ strength of the concrete [18]. Research has shown that the main factor of strength in concrete is hydrated gels resulting from the polymerization process, which increase in strength as the curing age of concrete increases [5,1-7].

Applying high heat (600 $^{\circ}\text{C}$) to the concrete sample has affected the microstructure of the concrete. According to the SEM image at 600 $^{\circ}\text{C}$, the formation of a tree structure, the presence of numerous capillary pores due to the evaporation of water between the capillary spaces in the hydrated calcium silicate gel (C-S-H), and the reduction of the amount of hydrated C-S-H gel (dark areas) following destruction are evident in the concrete sample, which indicates a weakness in the microstructure of the concrete after heating. Previous studies [3-5] show that the C-S-H gel present in the microstructure of concrete containing Portland cement will cause the most breakage in the concrete from a temperature of 450 $^{\circ}\text{C}$ onwards. In the images, the increase in volume and pore size can be attributed to the release of water under heat from capillary pores in the concrete microstructure, and this amount can

also be increased by increasing the water-to-cement (W/C) ratio in the preparation of the concrete mix design, and contribute to the porosity and embrittlement of the concrete structure. According to the SEM images in Figure 5, the hexagonal structure of portlandite changes at a temperature of 500 °C and the C-S-H nanostructure also decomposes [21].

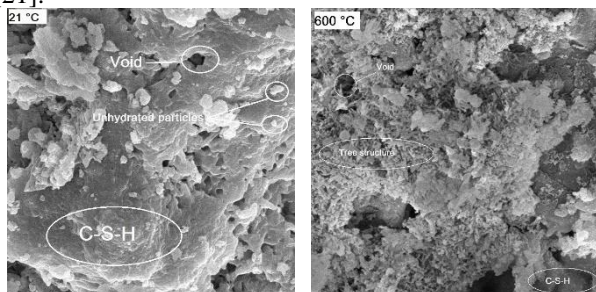


Fig. 5. SEM Analysis Results

4. Conclusion

In this study, the weight loss and UPV were tested at a curing age of 90 days at room temperature and high temperature in ordinary concrete with a cement content of 500 kg/m³. The results were evaluated based on XRD and SEM analysis. The main results of this laboratory study are presented as follows.

1) In the weight loss test of concrete samples, the weight of the concrete sample decreased from 2387 to 2225 grams, which was a decrease of 786.6%. As a result of the increase in temperature in the concrete samples, the results in the weight loss test of concrete were decreased. Similar results were obtained in the research of others [1,6].

2) In the UPV test, the speed of ultrasonic waves decreased from 6179 to 3411 m/s, which was a decrease of 44.79%. As a result, the increase in temperature in the concrete samples caused the results in this test to decrease. Similar results were obtained in the research of others [3,4].

3) In XRD analysis, at a temperature of 21 °C, most of the large peaks occurred in the regions of 16 to 29 and also 60 degrees, and aluminum phosphate compounds, followed by calcium hydroxide, titanium oxide, calcite and dolomite, have the highest dispersion in concrete. However, applying a temperature of 600 °C caused a decrease in the height of the peaks in the resulting graph. In this regard, most of the large peaks occurred in the regions of 26 to 29 degrees, and compounds such as calcium manganese carbonate, carbon, hydrated aluminum manganese iron phosphate silicate and hydrated aluminum calcium silicate have the highest dispersion.

4) In SEM analysis, at a temperature of 21 °C, the tree structure, pores and unhydrated particles are seen in their minimum amount, but after applying heat to the concrete sample at 600 °C, the volume and number of these values have increased. On the other hand, applying heat to the concrete sample has had destructive effects on the hydrated C-S-H gel and structural disintegration is evident in these gels. Similar results were obtained in the research of others [3-5].

5) At the end of this research, the results of SEM and XRD analysis at room temperature and under high temperature, while coordinating with each other, overlapped with the results of other tests.

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