The Difference in the Mechanical Properties of the Limestone and Sandstone Rocks at Different Temperatures

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July 1, 2024

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Abstract

A series of experiments were carried out to measure the influence of the heat and the saturation on the strength of the 2 types of sedimentary rocks (Limestone and Sandstone) at temperatures ranging between normal room temperature 22°C and 750°C using the Uniaxial compressive strength test and the Brazilian test after, and also the same process for the saturation group in the water. The drilling and cutting process took place in the first step, then the samples were distributed into several groups, each group faced a specific temperature, after that, exposure of the samples to the heat using the oven to reach the maximum temperature that was needed, the strength of each sample was measured according to the UCS and Brazilian Test. The results show that till 450°C both rocks resisted the temperature, but the limestone was much stronger than the sandstone, after increasing the temperature more than 450°C, the strength was strongly decreased. For the saturation group, the water harms the rocks, especially the limestone, and the decreasing strength is proportional to the density and the ultrasonic wave velocity.

Because limestone and sandstone rocks are materials, the question is: Do their physical and mechanical properties change with high temperature?

In this paper, we are going to answer this question for the limestone and sandstone rocks from room temperature to 750°C.

Keywords: Sandstone-Limestone-Samples-Density-Ultrasonic wave velocity-Weight-UCS and Brazilian test.

1. Introduction

In the controlled environment of a laboratory, the effect of high temperatures on sandstone and limestone rocks takes center stage as scientists endeavor to understand the complex reactions of these geological materials to thermal stress. By subjecting these rocks to elevated temperatures under carefully controlled conditions, researchers gain insights into the thermal behaviors, mineral alterations, and structural changes that unfold within a miniature representation of geological processes. This laboratory exploration allows us to simulate and understand how sandstone and limestone react to heat, providing valuable knowledge that contributes to our comprehension of both natural geological phenomena and potential uses across a range of scientific and industrial domains.

Minerals and rocks can be found all over the place! They are present in our daily lives, and they assist us in the development of new technologies.

Kompaníková et al. (2014) extensively documented modifications observed in two distinct types of sandstone under varying extreme temperatures. The alterations in the behavior of sandstones exposed to high temperatures were linked to both the stability of their mineral composition and porosity. The primary factor determining the suitability of rocks in high-temperature environments was identified as their thermal characteristics. Pribnow et al. (1996) conducted a study on the thermal conductivity of rocks saturated with water in the KTB Pilot Hole, spanning temperatures from 25°C to 300°C. The results indicated that the thermal conductivity of water-saturated amphibolite and gneiss decreased with increasing temperature, by approximately 40% and 20%, respectively. In contrast, sandstone has been utilized as a construction material for centuries due to its ability to withstand diverse environmental conditions (Hajpál and Török, 2004).

The changes in sandstone properties under high temperatures have been investigated both in the field and laboratory. Typically, common characteristics observed in rocks subjected to high temperatures include a decline in structural integrity and changes in visual properties. Numerous studies have focused on examining transformations in mineral phases during exposure to fire, particularly the phases of fire decay, which are associated with chemical modifications.

There have been numerous studies on cooling high-temperature rocks using different methods, but few have explored dry ice cooling. Rapid cooling of dry ice is expected to enhance rock porosity, improving both porosity and permeability (Savage, 2016).

The initial insights into the impact of thermal stresses and thermal fatigue can be traced back to Bartlett (1832), who conducted experiments aimed at determining the thermal expansion and contraction of rocks utilized in construction. During high temperatures, new minerals may form, and initial ones may disappear. One prevalent mineral alteration in sandstones exposed to fire is the disintegration of kaolinite and smectite structures.

The disintegration of the kaolinite structure occurs at temperatures near 550 ℃, and complete disappearance is noted around 579 ℃. In contrast, smectite structures exhibit greater stability compared to kaolinite and can still be detected at temperatures as high as 900 ℃. However, they start losing hydroxyl radicals as early as 553 ℃. Prominent consequences of exposure to fire include the development of cracks, soot deposition (especially in stones with iron), and significant influences on future deterioration patterns.

Hence, studying the damage effects and variations in thermo-physical properties of common rocks under temperature changes, from freezing to ultra-high temperatures, provides valuable insights. Numerous studies have examined the effects of high temperatures on various rock types, focusing on changes in surface characteristics, microstructure, mass loss, bulk density, effective porosity, gas permeability, and P-wave velocity. Some specifically delve into the behavior of sandstones under extreme temperatures, attributing changes to mineral composition and porosity, ultimately determining the rocks' utility in high-temperature conditions.

In this paper, our emphasis is on examining the changes in the physical and mechanical properties of limestone and sandstone as the temperature increases from ambient temperature (22 ℃) up to 750 ℃. So, let's turn up the heat and see the captivating story that unfolds in the heated world of rocks.

Experimental tests – Equipment’s - Equations

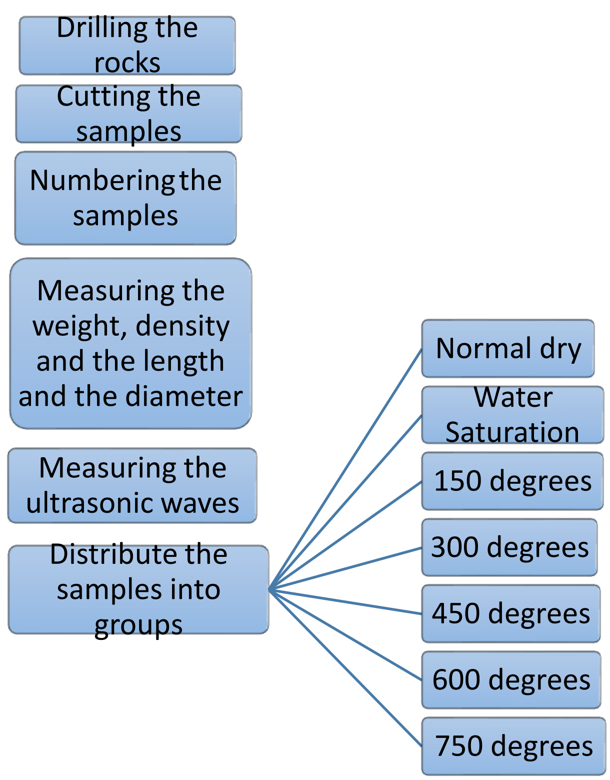
The laboratory tests were carried out on 136 pieces of regularly shaped limestone and sandstone cylindrical samples.
Each type of rock has 68 samples (34 samples for the UCS Test +34 samples for the Brazilian Test)

The average diameter of the specimens was 29.4 mm for the limestone rock and 29mm for the sandstone rock.
The specimens were cut from the core drillings and prepared for thermal treatment and laboratory measurements.

7 thermal groups were made, at 22°C (room temperature, not tempered)
150°C, 300°C,450°C,600°C, and 750°C, respectively, and fully saturated in water.
The thermal treatment was performed in a Carbolite ABA 7/35 electric oven.

The heating rate was set to 20°C /min for generating a homogeneous thermal field and was linearly increased to 150°C, 300°C, 450°C, 600°C, and 750°C and kept for 4 hours.
The cooling was 5°C /min until room temperature was reached and were tested at room temperature.

The electric oven's built-in digital temperature gauge confirmed the heating and cooling rates.
The temperature-related changes in the samples are visible to the naked eye.



Flowchart 1: Experimental Methodology

Firstly, all these samples were tested in dry conditions and their **mass, density, and ultrasonic wave velocity,** were tested before the heating.

Secondly, the heating test was operated for the groups, until each group reached its predetermined temperature.

Thirdly, the mass loss, the density, and the ultrasonic wave velocity (P-wave) were measured after the cooling of the samples.

a. The weight of the sample was measured by using the balance equipment and according to the following formula the mass loss can be calculated:

$$\Delta \boldsymbol{m} = \frac{\boldsymbol{m - mT}}{\boldsymbol{m}} \qquad \textbf{(1)}$$

m: Dry mass before high temperature

m_T: Dry mass after high temperature.

b. The density can be calculated from the tested weight and volume of samples according to the following equation:

$$\boldsymbol{\rho} = \frac{\boldsymbol{m}}{\boldsymbol{V}} \qquad (2)$$

m: sample mass

ρ: density of the sample

v: Volume of the sample

c. The ultrasonic wave velocity was measured by an *acoustic wave instrument type: RS-ST01C* these sound waves bounce back images, which reveal key characteristics of a material's properties. The images created by ultrasonic testing can indicate cracks, weld grooves, and fractures, as well as point out the material thickness and moving components.

d. The heating test is the process of heating the samples with different high temperatures in the oven which has different types as shown in Table (1)

Carbolite ABA 7/35 electric oven, 25-750, the heating rate was set to 20°C, The cooling was 5°C /min until room temperature was reached, and were tested on room temperature.

Furnace type	Range of temperature ℃	The heating rate ℃/min	The cooling rate ℃/min
Carbolite ABA 7/35 electric oven	25-750	20	5

Table1: The characteristics of the oven

Finally, the **UCS and the tensile strength** test were tested in each group.

e. The UCS test was conducted according to *haft-Zugmessgerat type F20D easy* **(<u>Christoph Franzen et.al 2023</u>)**, a *CMT5305 electronic universal testing machine* **(<u>Guansheng Han et.al 2019</u>)**, an *RMT150C hydraulic servo testing machine* **(<u>Pin Wang et.al 2023</u>)** and *AS4133.4.2.2-2013 testing machine* **(<u>Savani Vidana Pathiranagei et.al 2021</u>)**

The UCS and the Brazilian test machine comprise a pair of metallic plates. The upper plate, affixed to a load measuring device, remains stationary, while the lower plate is positioned on the loading ram. A computer is connected to the apparatus to record and document the forces applied to the samples.
The UCS test involved the preparation of cylindrical core specimens, each with a height of 60 mm and a diameter of 30 mm, and for the Brazilian test, the specimens were used with 30 mm height and 30 mm diameter. Following the heat treatment, the rock specimens were subjected to compression loading at a constant displacement rate of 0.1 mm/min. After positioning the sample on the lower plate, the computer is configured with the test parameters, and the apparatus is activated. Upon the sample's failure, the machine is halted. Afterwards, data is collected and analyzed.
The computer shows the stress-strain curve for each sample during the UCS test until the failure, in this case, the curve indicates the maximum force that each sample can resist. The following formula assists us in computing the Uniaxial compressive strength of each sample:

Figure 1: Uniaxial compressive test

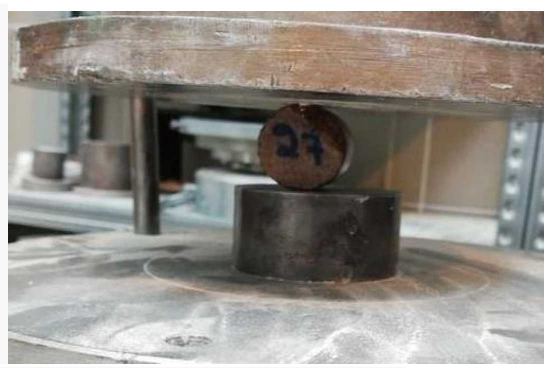


Figure 2: Tensile strength Test

$$\sigma c = \frac{F}{A} \qquad (5)$$

σc: UCS Strength of the sample.

F: Maximum force applied during the UCS test

A: Area of the sample $A = \frac{d^2}{4}$

d: diameter of the sample

The following formula is used to determine the tensile strength of the sample, taking into account the applied load and the diameter of the specimen undergoing the Brazilian test. The tensile strength formula after the Brazilian test is typically expressed as:

$$\boldsymbol{\sigma T = \frac{2 * P}{\pi * D}} \qquad (6)$$

σT: Tensile strength of the specimen.

P: maximum applied load during the test

D: Diameter of the cylindrical specimen

_5._ Results

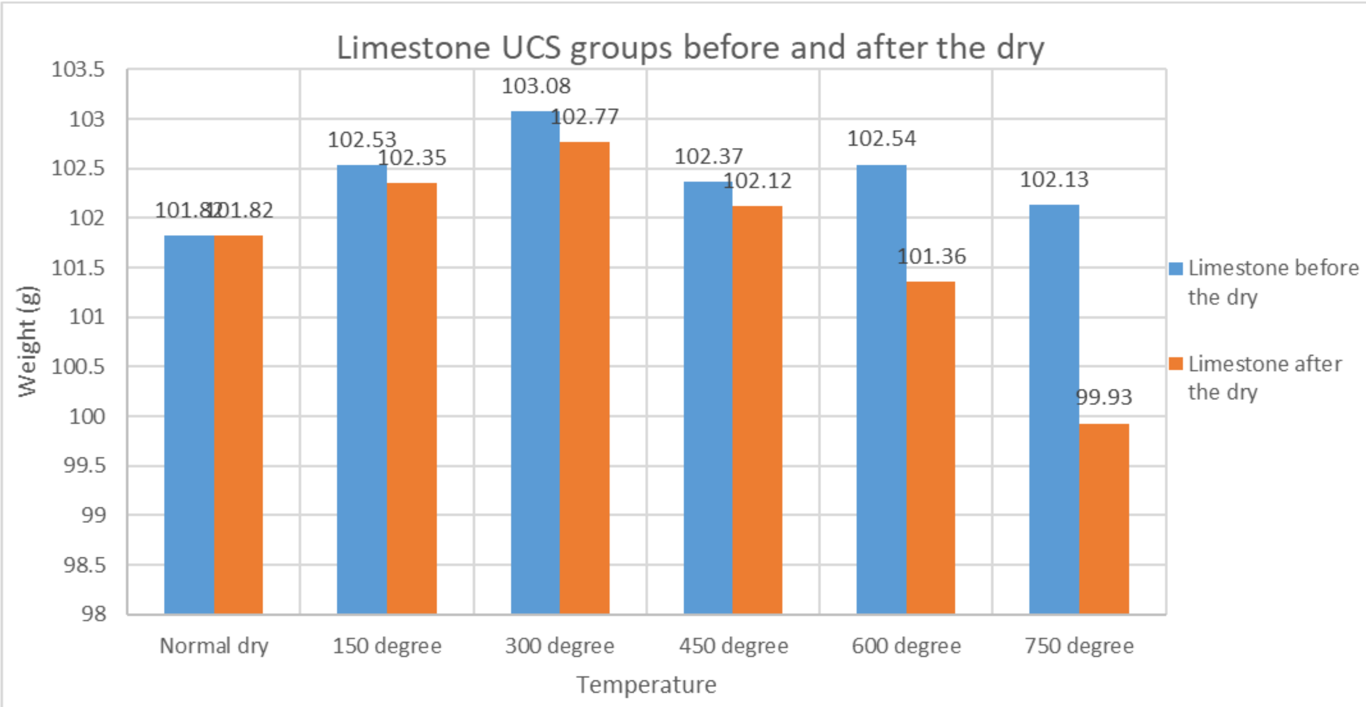


Figure 3: The effect of the temperature on the mass loss for Limestone rock

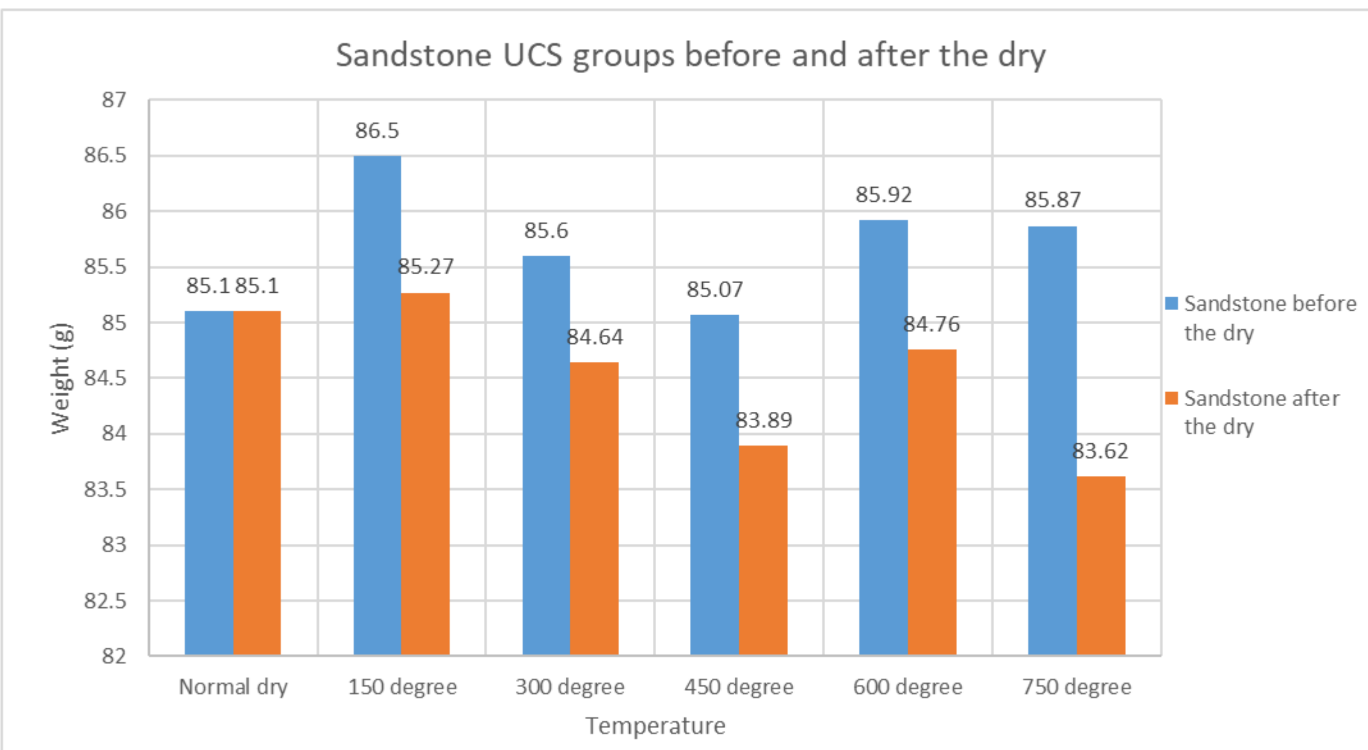


Figure 4: The effect of the temperature on the mass loss for Sandstone rock

Figures 3 and 4 present compelling insights into how high temperatures affect the weight of limestone and sandstone rocks. Initially, as temperatures rise, there is a discernible increase in weight observed in both sandstone and limestone samples. For sandstone, this weight gain is noticeable up to 150°C, while for limestone, it extends up to 300°C. This increase in weight can be attributed to the evaporation of free water present within the rocks. Specifically, the weight of sandstone rose from 85.1g to 85.27g, and for limestone, it increased from 101.82g to 102.77g.

However, beyond these temperature thresholds, an intriguing trend emerges. As the temperature continues to climb, the weight of the samples begins to decline. This decrease is particularly pronounced as temperatures reach their peak at 750°C. At this point, the average weight of sandstone drops to 83.62g, while limestone registers an average weight of 99.93g.

This reduction in weight can be attributed to the complete evaporation of water trapped within the rock samples. Furthermore, the emergence of micro-cracks becomes evident after the 300°C mark. These micro-cracks initially develop due to the channels forming between the pores as water evaporates. With the continued increase in temperature, these micro-cracks propagate and evolve into larger cracks. Consequently, the presence of numerous pores and cracks contributes significantly to the overall reduction in weight observed in the rock samples.

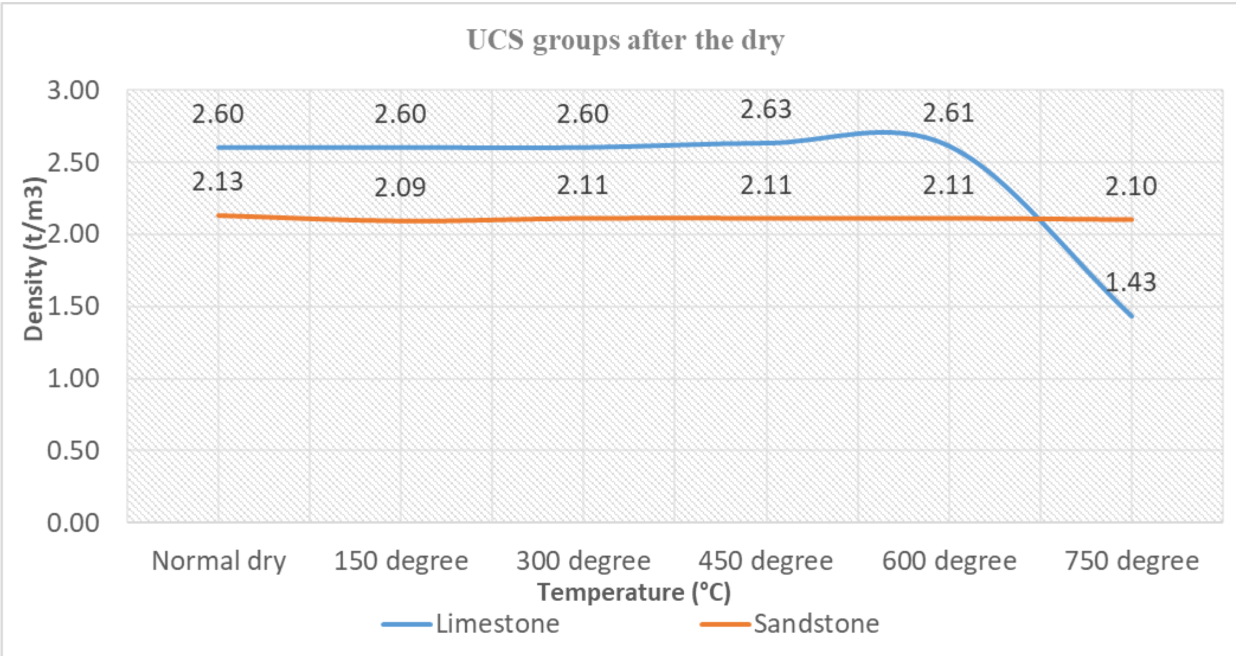


Figure 5: The effect of the temperature on the density for limestone and Sandstone rocks

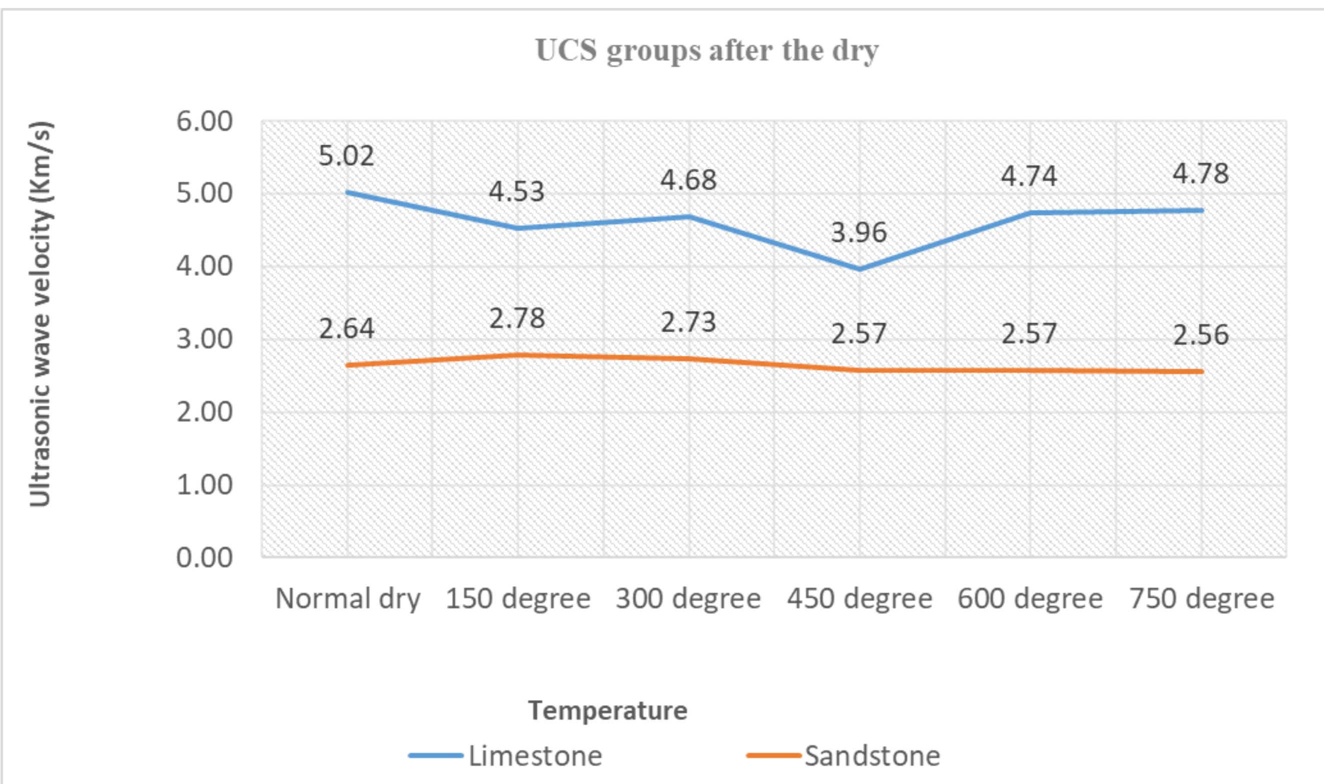


Figure 6: The effect of the temperature on the USV for Limestone and Sandstone rocks

According to Figure 5, the density of the limestone is higher than the sandstone in different high temperatures and this is because the limestone is formed from calcite or calcium carbonate ($CaCO_3$), while the sandstone is formed of quartz (SiO_2) or feldspar with other minerals, and the calcium carbonate generally has a high density than the quartz or feldspar.

Plus, sandstone has a lot of pores inside it and it is filled with air or water which decreases its density, in opposite to the limestone which has fewer and smaller pores making it denser.

As we can see the density of both sandstone and limestone rocks slightly decreased when we increased the temperature but after 600 °C, the density of limestone strongly decreased from 2.61 t/m^3 to 1.43 t/m^3 and this is because when the limestone is heated to temperatures above 600 °C, it undergoes a chemical decomposition reaction:

$CaCO_3 \rightarrow CaO + CO_2$

In this reaction, limestone (solid calcium carbonate) decomposes into quicklime (solid calcium oxide) and carbon dioxide gas. The decrease in the density is because the volume of the solid limestone decreases as it decomposes into smaller volumes of solid calcium oxide and gaseous carbon dioxide. The overall mass remains the same but, the volume increases due to the gas being released. This leads to a decrease in the density of the limestone.

Additionally, the quicklime (calcium oxide) formed from the decomposition of limestone has a different crystal structure compared to limestone, which can also contribute to the change in density.

The minerals that make up sandstone, particularly quartz, and feldspar, are known for their high melting points and thermal stability. They generally remain solid and maintain their crystal structures at high temperatures, as a result, the density of sandstone remains relatively constant within this temperature range.

Figure 6 shows us the effect of the high temperatures on the ultrasonic wave velocity for the limestone and sandstone rocks, The USV has a little change when the temperature increased from the normal temperature to 300 °C proportionally to the density and because in this range of temperatures, the structural and bonding water immigrated from inside to outside the rock sample but the density and the USV still almost constant, after 300 °C, the water evaporated and the micro-cracks started to make a channel between the pores until the macro-cracks appeared by increasing the temperature more and more.

The USV of the limestone at 450 °C decreased from 4.68 km/s to 3.96 km/s because this temperature causes thermal expansion and induces stress within the limestone rock. This stress can lead to microcracking or other structural changes, which in turn can affect the propagation of ultrasonic waves and result in a decrease in velocity.

Also, the thermal decomposition of limestone can result in changes in its elastic properties, such as stiffness and density. These changes can influence the velocity of ultrasonic waves traveling through the rock.

7.4.1 ***Plot UCS (average value) of Limestone and Sandstone in the function of Temperature:***

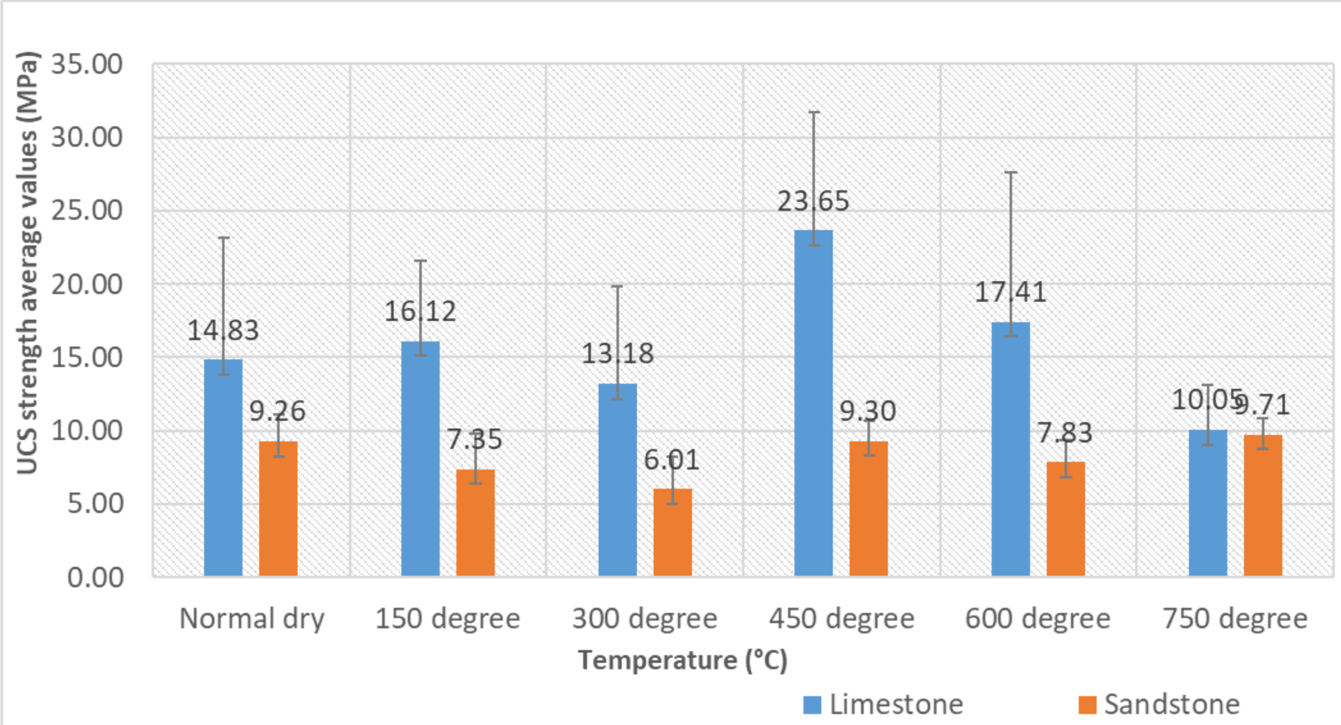


Figure 7: The effect of the temperature on the UCS test

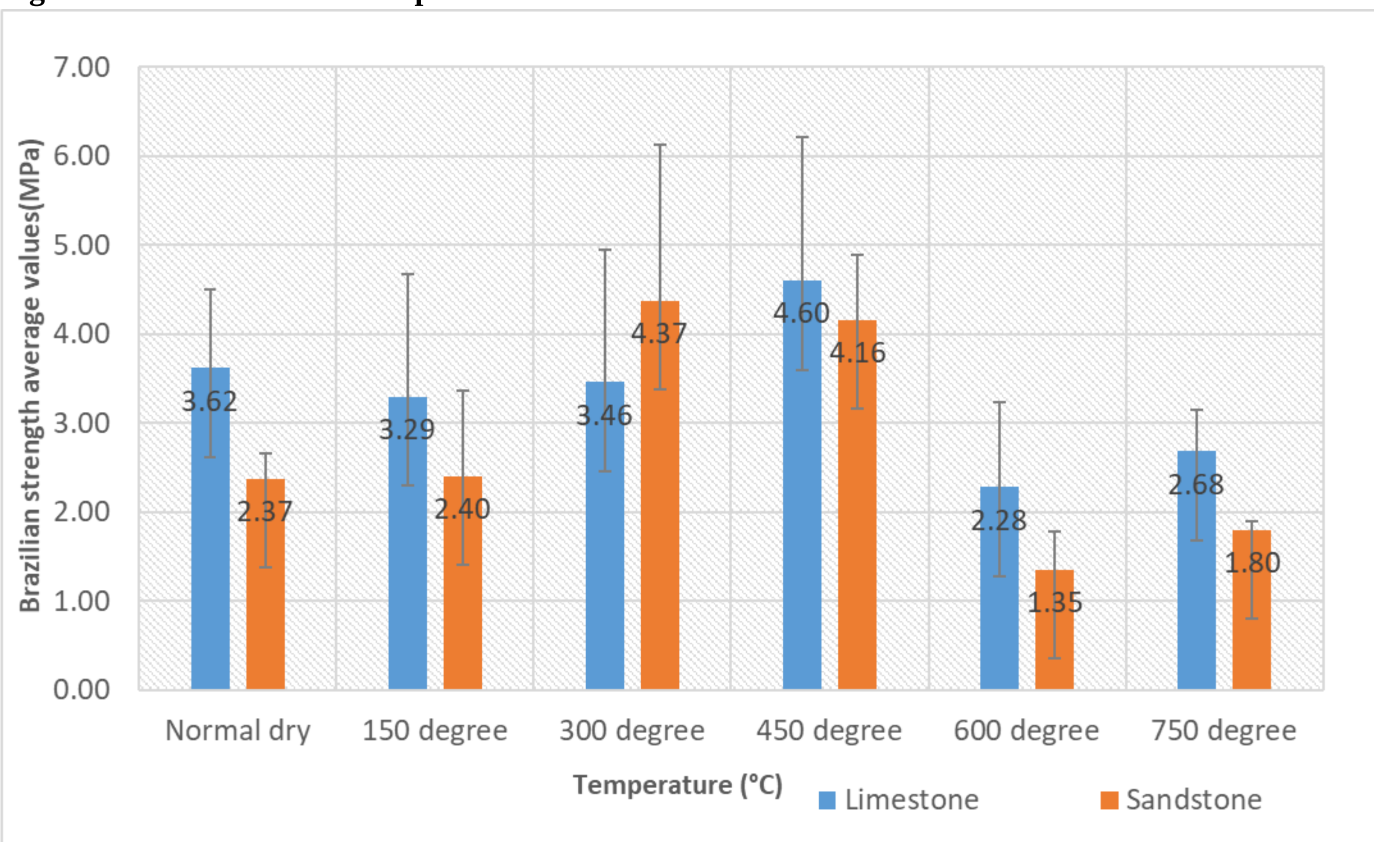


Figure 8: The effect of the temperature on the tensile test

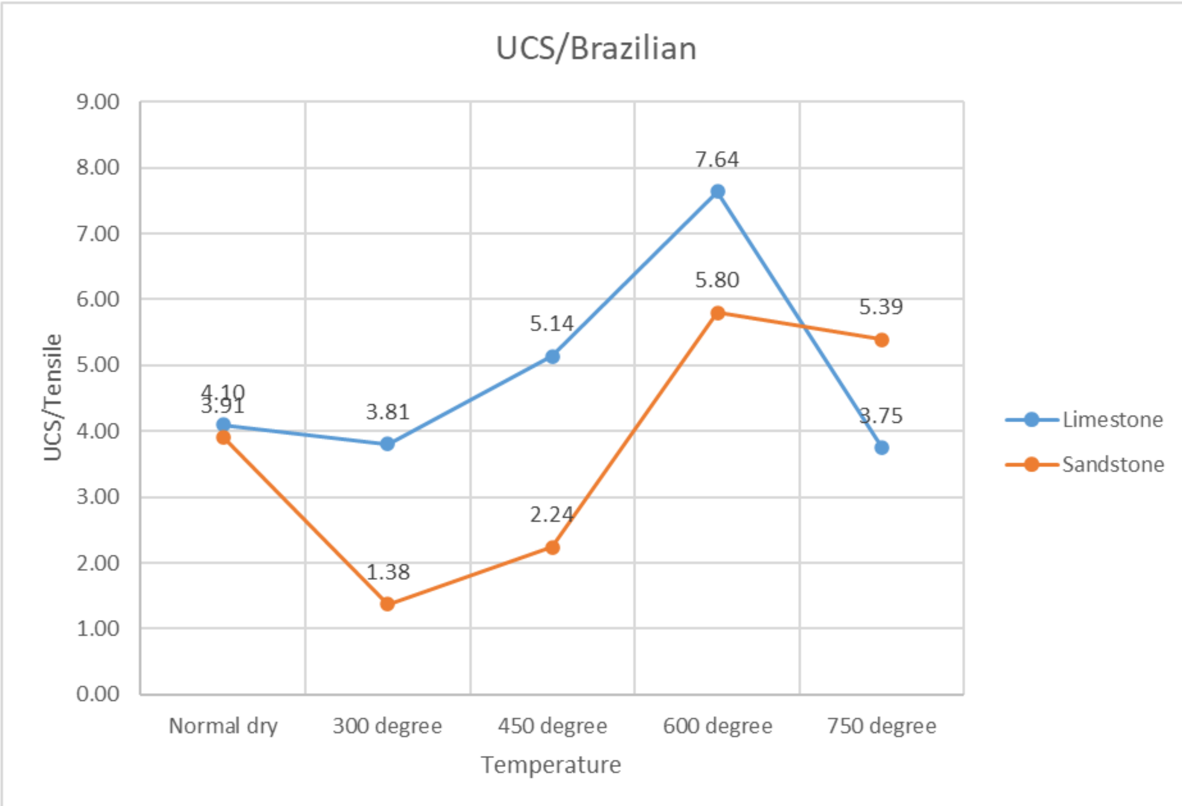


Figure 9: The effect of the temperature on the compression and tensile ratio

Figures 7 and 8 show us the effect of the high temperature on the strength of the limestone and sandstone rock for the compression and tensile strength, and figure 12 concludes the results of the temperature impact on the UCS and tensile strength ratio for both rocks.

As we can see in figure 9 by increasing the temperature from the 22 °C to 300 °C the ratio of UCS/T decreased slightly for the limestone rock from 4.10 to 3.81, but for the sandstone it decreased strongly from 3.91 to 1.38, as limestone typically possesses lower porosity than sandstone because its constituent calcium carbonate tends to form tightly packed grains due to its crystalline structure, resulting in greater density and reduced pore space, and the cementing minerals in limestone, such as calcite or dolomite, can fill in pore spaces during diagenesis, further reducing porosity. Sandstone, on the other hand, consists of loosely packed grains of quartz, feldspar, and other minerals. These grains may have irregular shapes and sizes, leaving more space between them, which contributes to higher porosity.
According to their constituents, pores, and the results of the strength ratio from normal temperature till 300°C, we can conclude that the sandstone is weaker and has high porosity than the limestone.

After 300°C the ratio increased with both rocks till the maximum value at 600°C to reach 7.64 for limestone rock and 5.80 for sandstone rock. The strength increases for both rocks due to their minerals and their expansion under high temperature, when sandstone or limestone undergoes expansion, the grains within the rock may shift or compact. This compression of grains can result in the pores between them becoming smaller or being partially or fully filled. Consequently, the overall porosity of the rock decreases as a result of this reduction in pore space.

Around 600°C the quartz inside the sandstone has a phase transition known as the α-β transition. This phase transition is accompanied by changes in the crystal structure of quartz. In the α-phase, quartz has a trigonal crystal structure, while in the β-phase, the crystal structure changes to hexagonal. This transition can result in changes in various physical properties of quartz, including its thermal expansion coefficient, density, and elastic properties.

After 600°C, The strength of limestone strongly decreased from 7.64 to the almost half of it 3.75, and for the sandstone the strength slightly decreased from 5.80 to 5.39.

Because, above 600 °C, calcite decomposes into lime (calcium oxide) and carbon dioxide gas. This decomposition weakens the limestone structure, as the cohesive bonds holding the rock together are disrupted and the thermal stresses can exceed the strength of the rock, causing microcracks to initiate and propagate. As these cracks propagate, they further weaken the rock structure, making it more susceptible to failure.

7.5. ***<u>Plot the saturation results:</u>***

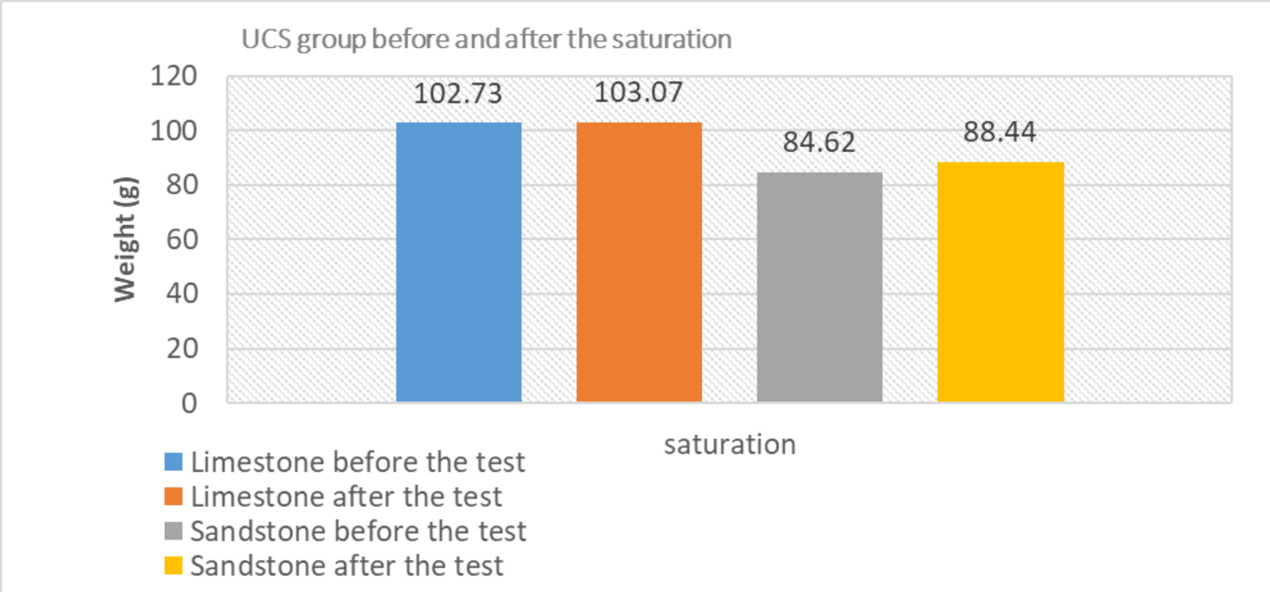


Figure 10: The effect of the saturation on the weight

Figure 10 illustrates the impact of saturation on the weight of limestone and sandstone rocks. It is evident that the weight of the limestone experienced a slight increase from 102.73 to 103.07g after saturation. In contrast, the weight of the sandstone significantly increased from 84.62 to 88.44g following saturation.

This discrepancy in weight change can be attributed to the higher porosity of sandstone compared to limestone. Sandstone possesses greater pore space, and when saturated, these pores fill with water, resulting in a notable increase in weight.

Conversely, limestone, with lower porosity, shows a relatively minor weight increase post-saturation. The increased weight of the sandstone reflects its ability to absorb and retain more water due to its porous nature, whereas the limited pore space in limestone restricts its water absorption capacity and subsequent weight gain.

Additionally, factors such as grain size, mineral composition, and pore connectivity also contribute to the differential weight changes observed between the two rock types after saturation.

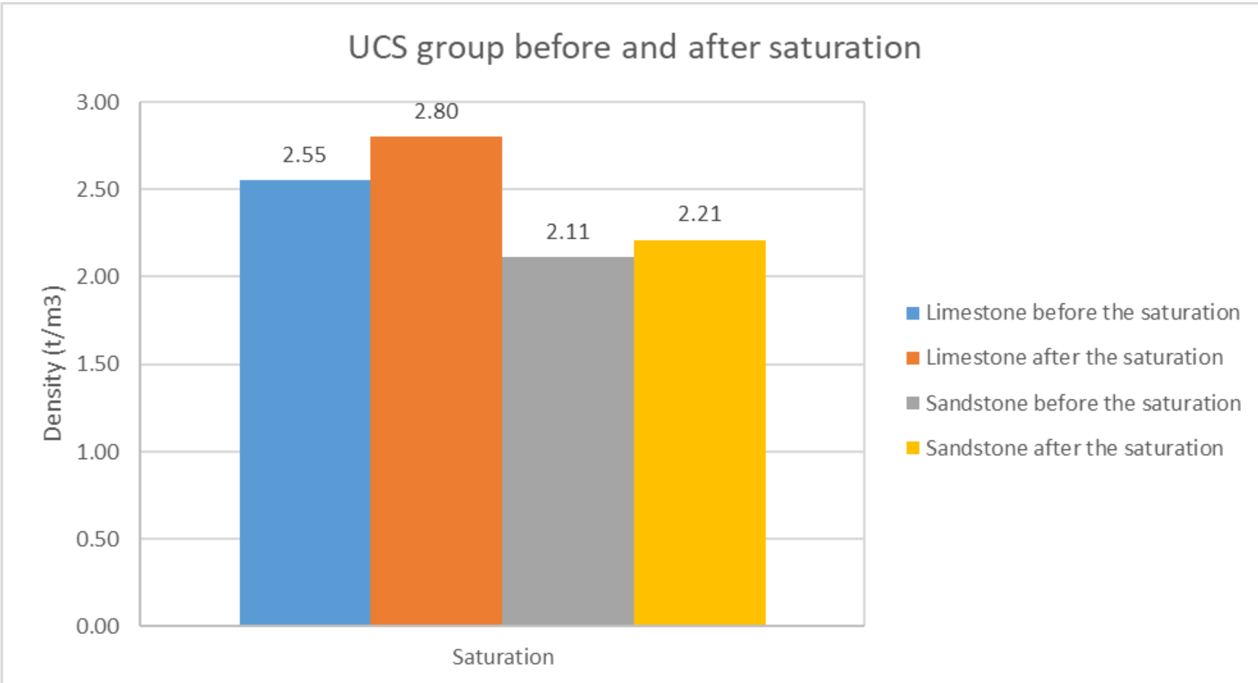


Figure 11: The effect of the saturation on the density

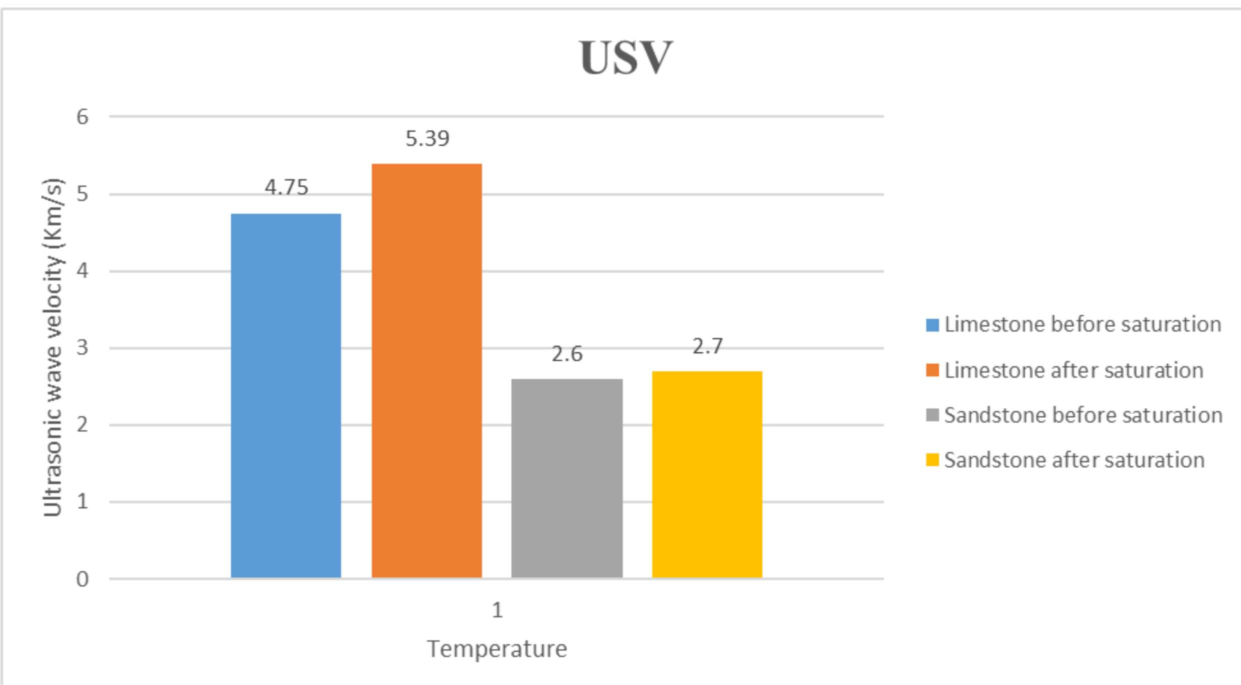


Figure 12: The effect of the saturation on the USV

Figures 11 and 12 show us the effect of the density and the ultrasonic wave velocity after the saturation for limestone and sandstone rocks.

The density and the ultrasonic wave velocity are proportional to each other, a higher density typically corresponds to stronger interatomic bonds and a stiffer material, resulting in faster transmission of ultrasonic waves. This relationship holds true in homogeneous materials with uniform density, where denser packing of atoms leads to higher ultrasonic velocities. However, the presence of porosity or structural irregularities can disrupt this proportional relationship by impeding the transmission of ultrasonic waves.

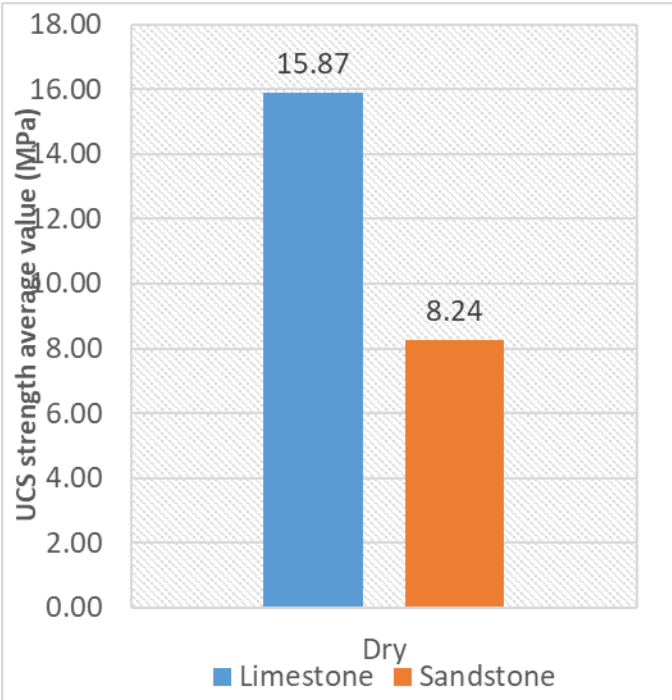
As we can see in the figures 11 and 12, the density and the USV of the limestone increased strongly after the saturation 2.55to 2.80 t/m^3 and 4.75 to 5.39 Km/s respectively.

Limestone typically possesses a denser structure with fewer pore spaces compared to sandstone. When saturated with water, limestone can more effectively fill its remaining pore spaces, leading to a significant increase in density and stiffness.

Additionally, limestone's mineral composition, primarily composed of high-density minerals like calcite or dolomite, contributes to its enhanced response to saturation.

The cementation between mineral grains in limestone is often more pronounced than in sandstone, further reducing porosity and increasing material density.

Conversely, sandstone, with its higher initial porosity and potentially less dense mineral composition, undergoes a comparatively smaller increase in density and ultrasonic wave velocity following saturation from 2.11 to 2.21 t/m^3 for the density and from 2.6 to 2.7 Km/s for USV..



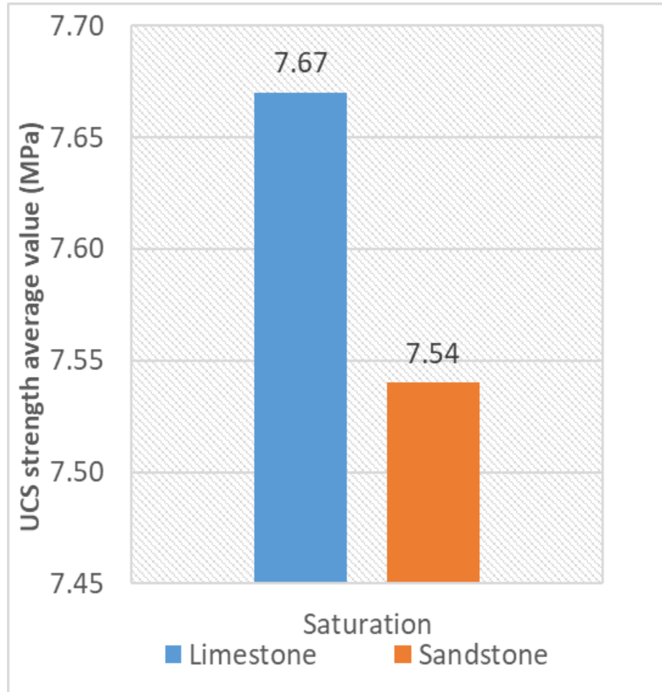


Figure 13 and 14: Changes in Strength from Dry Condition to Saturation in Limestone and Sandstone

Figures 13 and 14 show us the UCS strength for both rocks before and after the saturation, as we can see that after the saturation the strength decreased for both rocks from 15.87 to 7.67 Mpa for limestone rock and from 8.24 to 7.54 Mpa for sandstone rock.

The significant reduction in limestone strength following saturation can be attributed to a complex interaction of various factors. When water permeates the pore spaces of limestone during saturation, it disturbs the bonds between mineral grains, diminishing the cohesive forces. This infiltration not only raises pore pressure within the rock but also aids in the propagation of microcracks, further undermining the limestone's structural integrity.

Additionally, the presence of water alters the mechanical properties of the minerals in the rock, potentially leading to mineral dissolution or modification, which worsens the weakening effect. Furthermore, limestone's relatively limited porosity hampers effective drainage, resulting in water accumulation within its pore network, which exacerbates the adverse effects of saturation on its strength.

In contrast, sandstone's response to saturation is characterized by a more nuanced interplay of factors. While water infiltration into sandstone pore spaces also weakens intergranular bonds and induces microcrack formation, the higher porosity of sandstone allows for more efficient drainage and limits water retention within the rock. This mitigates the extent of pore pressure buildup and reduces the severity of microcrack propagation, resulting in a milder decrease in strength compared to limestone.

Furthermore, the mineral composition and cementation characteristics of sandstone play a significant role in its response to saturation. Sandstone may exhibit greater resilience against saturation-induced weakening due to the presence of cementing materials that enhance intergranular cohesion and resist water infiltration. Additionally, the water occupying sandstone pores may act as a supportive medium, providing additional mechanical stability and partially offsetting the strength loss.

In summary, the pronounced decrease in limestone strength after saturation is primarily attributed to its low porosity, limited drainage capacity, and susceptibility to water-induced microcracking and mineral alteration.

Conversely, sandstone's higher porosity, effective drainage, and resilient mineral composition contribute to a comparatively milder decrease in strength following saturation. The complex interaction of these factors underscores the diverse responses of different rock types to saturation and highlights the importance of considering their inherent properties when assessing their behavior under environmental conditions such as saturation.

6. Conclusion:

In conclusion, the effects of high temperature and saturation on limestone and sandstone rocks manifest in significant alterations across various properties.

High temperature exposure leads to mass loss in both limestone and sandstone, attributed to thermal decomposition and mineral alterations, with limestone exhibiting a more pronounced decrease due to its carbonate-rich composition. This loss in mass correlates with decreased density and ultrasonic wave velocity in both rock types, indicative of structural changes and reduced material stiffness.

Moreover, high temperature significantly decreases the Uniaxial Compressive Strength (UCS) and tensile strength of limestone, reflecting its diminished mechanical integrity.

Conversely, the impact of saturation on mass loss, density, ultrasonic wave velocity, and strength differs between limestone and sandstone. Limestone experiences a substantial decrease in strength after saturation, attributed to water infiltration-induced microcracks and mineral alterations, while sandstone exhibits a lesser decrease in strength due to its higher porosity and drainage capacity.

Additionally, both limestone and sandstone show increases in density and ultrasonic wave velocity post-saturation, with sandstone demonstrating a more modest increase. These findings underscore the complex interplay of temperature and saturation on the mechanical and physical properties of limestone and sandstone, emphasizing the importance of considering their distinct characteristics when assessing their behavior under environmental conditions.

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