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The Effect of Freeze-thaw on Mechanical and Ultrasonic Properties of Hungarian Oolitic Stones

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Abstract
The freeze-thaw process is a weathering phenomenon that occurs in rocks and stones when water enters small cracks and pores within the stone. When the temperature drops below freezing, the water inside the rock expands as it freezes, exerting pressure on the rock from within. In this study the effect of freeze thaw on mechanical parameter uniaxial compression strength (UCS) and ultrasonic pulse velocity (UPV) of 47 oolitic samples were considered. At first, capillary absorption for cylindrical and prismatic oolitic samples were measured and then freeze-thaw experiment with 23 cycles were applied to the samples. UPV of samples before and after freeze-thaw was analysed by two ultrasonic devices including Geotron and Pundit. It was observed that freeze thaw decreases primary velocity (Vp) and secondary velocity (Vs) by around 20 and 7% respectively. However, average UCS of 1.6 MPa indicates strength decrease of samples, which represent negative effect of pore expansion due to freeze thaw on strength parameter of samples.

Keywords
freeze-thaw, oolitic limestone, UCS, ultrasonic pulse velocity, capillary

1 Introduction

Cultural and historical buildings in Hungary like Hungarian Parliament Building, St. Stephan's Basilica and Citadella are constructed with stones (limestone) that are affected by various atmospheric conditions, either directly or indirectly, depending on the climate and season. The exposure to these conditions causes partial or complete deterioration of the stones, which makes it difficult for the buildings to maintain their historical significance for a long time. One of the significant factors is the freeze-thaw (F-T) process. This process occurs when water penetrates the building stones through capillarity, leading to weathering. When the temperature drops below 0 °C, the water in the pores and tiny cracks of the stones freezes, causing them to expand in volume and exert pressure on the stones. This cycle is more prevalent in areas with frequent temperature fluctuations above and below freezing and leads to undesirable weathering of the building stones [1]. The freeze-thaw test is used to identify the damage to stones caused by repeated freezing and thawing [2].

Different types of deterioration can occur during freeze-thaw cycles. Several studies, such as those by Amirkiyaei et al. [3], Uğur and Toklu [4], Cárdenes et al. [5], Fener and İnce [1], Prikryl et al. [6], Mutlutürk et al. [7], Karaca et al. [8], Akin and Ozsan [9], Bayram [10], Martínez-Martínez et al. [10], Ghobadi and Torabi-Kaveh [11] have focused on the impact of freeze-thaw cycles on the physical and mechanical properties of various types of rocks. Fener and İnce [1] studied how the engineering and textural properties of Sille andesite change during various freeze-thaw cycles. They observed that extent of the impact varied depending on factors such as the state of the foundation insulation and its size and location on the wall. As the number of freeze-thaw cycles increased, there was an increase in porosity, and a decrease in uniaxial compressive strength, point load strength, and Brazilian tensile strength. Heidari et al. [12] examined potential alterations in the physical and mechanical features of the stone caused by freeze-thaw and salt crystallization aging experiments. The study found that the mechanical properties of the stone decreased significantly after undergoing freeze-thaw and salt crystallization tests. The durability of the stone is mainly controlled by intrinsic factors such as calcite veins and stylolites, and the freeze-thaw process is the primary factor in the weathering of Anahita

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Temple stone. The Uniaxial Compressive Strength (UCS) is a crucial mechanical property used to assess the suitability of natural stones for outdoor use, particularly in colder regions as in colder areas stones undergo excessive cycles of freezing and thawing throughout the year [2]. Bayram [2] studied prediction of percentage of UCS that will be decreased after freeze-thaw process and presented a statistical model for that purpose. Amirkiyaei et al. [3] developed a model to predict UCS of building stones after freeze-thaw process for carbonate building stones. Uğur and Toklu [4] considered damage caused by laboratory freeze-thaw cycles on porous natural stones by evaluating changes in certain physico-mechanical (such as apparent porosity, P-wave velocity, colorimetric values, water vapor transmission rate, and point load strength) and thermal (thermal conductivity) properties. For that purpose, they considered travertine, limestone (biocalcarenite), trachyandesite and tuff stone (ignimbrite). The findings show that these stones have varying levels of resistance to damage from the freezing-thawing process.

The study utilized 47 samples of Hungarian oolitic limestone, with initial measurements of ultrasonic pulse velocity taken using both Pundit and Geotron devices. Water capillary absorption was analyzed for 24 samples to measure pore connection and water suction in porous samples. The samples were then subjected to 23 cycles of freeze-thaw process.

2 Materials and methods

This research utilized a total of 47 samples of oolitic limestone, comprising 24 cylindrical samples and 23 prism samples, selected from those that remained intact and met the criteria (dimensions and flat surface) for acceptability among all prepared samples.

Stones and aggregates used for testing were gathered from the Sóskút quarry, situated about 38 kilometers southwest of Budapest. This type of limestone, prevalent in Central Europe, stands out as a widely utilized building material. Recognized for its high porosity and ease of manipulation, it emerged as a pivotal construction resource during the 18th and 19th centuries. This era witnessed the creation of iconic structures crafted from this stone, including notable landmarks like the House of Parliament and Opera House in Budapest, the Citadella, Saint Stephan's Dom in Vienna, and the Castle in Bratislava. This is a limestone composed of fine bioclasts. Its main components include miliolid foraminifera, small shell fragments, and micritic ooids. The microstructure classifies it as a bioclastic ooid packstone. Pores are partly filled with micritic and microsparitic cement, but intraparticle porosity prevails. Another lithotype from the Sóskút quarry (indicated by SM) is a typical ooid-grainstone. Its micro-fabric consists mainly of well-rounded or partially rounded ooids and micro-oncoids. Pores are partially filled with sparitic cement and partially remain open. The carbonate grains exhibit good sorting, with average grain sizes ranging from 0.3 to 0.4 mm. [13]. Ultrasonic pulse velocity has been measured before and after freeze-thaw process. The direct transmission method was used, where transducers were placed on opposite sides of the rock sample and a low-frequency wave was propagated from one transducer to the other. Two types of ultrasonic devices, the Pundit and Geotron, were used and their results were compared. The Geotron was able to measure S wave velocity in addition to P wave velocity, which the Pundit could not. The study used a modified version of the post-processing method developed by Benavente et al. [14] for Geotron signals, which was further modified for the purpose of this study [15].

2.1 Test methodology

The Pundit (Portable Ultrasonic Non-destructive Digital Indicating Tester) is a user-friendly device. It automatically calculates the propagation time of P waves. The transducers operate at a frequency of 50 kHz, and Plasticine serves as the coupling agent to bridge the gap between the transducers and the sample. For Geotron measurements, UP-SW transducers with an 80 kHz frequency are utilized. These transducers are designed for propagating ultrasonic waves to ascertain elastic material parameters. They aid in pinpointing the arrival of the S wave, particularly beneficial as longitudinal waves experience significant attenuation. Before and after salt crystallisation cycles, P and S wave velocities are measured five times on the same sample. The first onset of the P wave and S wave is identified using selected amplitudes of 50mV and 2V, respectively. Manual evaluation of results is conducted using Benavente et al. [14] method for Geotron [16]. Benavente et al. [14] suggested using a band-pass filter to diminish the influence of the P wave background signal in identifying the initial occurrence of the S wave.

2.2 Freeze-thaw test

For freeze thaw test, the European Norm Standard EN 12371 [17] was used as a reference. The process involved drying the stone samples and then saturating them in distilled water at 20 °C. The saturated samples were then subjected to a freeze chamber at −20 to −22 °C for 6 hours, followed by immersion in water for 6 hours. This cycle was repeated multiple times, and the weight of the specimens was measured before and after each freeze cycle. This test was conducted 25 freeze-thaw cycles on specimens [18]. In this study, samples were destroyed after 23 cycles.

2.3 Water adsorption test

Following the protocol outlined in reference [19], the water saturation test was conducted on all specimens under atmospheric pressure conditions. Initially, the stones were subjected to drying at 105 °C for 48 hours. Subsequently, they were partially immersed in water, reaching half of their height. At this stage, the samples were weighed, and ultrasonic pulse velocities were recorded every minute for the first 10 minutes, then at 15 and 30 minutes. After an hour, water was added until it reached three-quarters of the sample height, and weight and ultrasonic pulse velocity measurements were repeated. After two hours, water was further added until the specimens were fully submerged to a depth of 25 ± 5 mm, and measurements were taken again. This process was repeated every 24 ± 2 hours.

3 Results

Based on Fig. 1, it can be claimed that frost resistance (frost resistance is the property that a material can withstand several freeze-thaw cycles without being destroyed) for oolitic limestone samples is fluctuating between 4 and 7% saturated mass loss rate after 23 freeze-thaw cycle. The correlation between frost resistance and mass loss was negative which means with increasing mass loss, frost resistance decreased. Comparing Figs. 1 and 2, it can be concluded that shape of samples affects mass loss rate and frost resistance of samples and cylindrical samples had higher durability to frost with less than 8% mass loss rate, while Prismatic ones, experienced more than 14% mass loss rate. Within a small number of cycles,

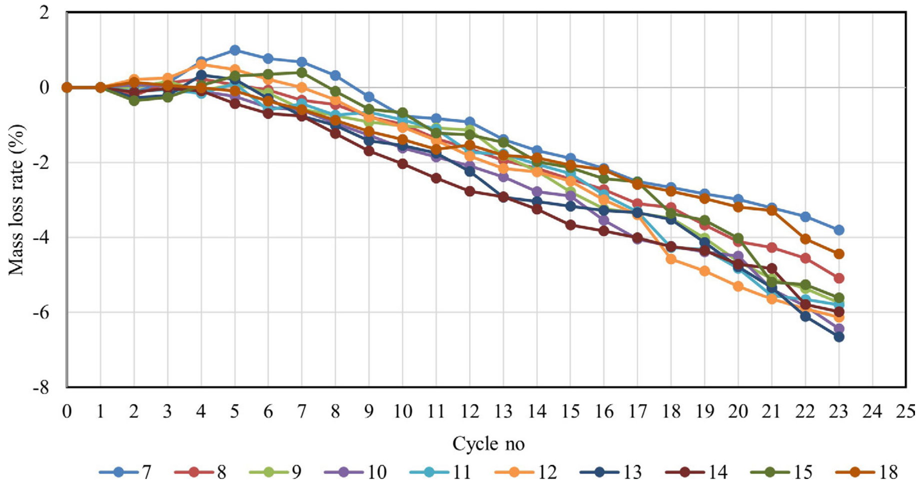


Fig. 1 Average mass loss rate for cylindrical samples

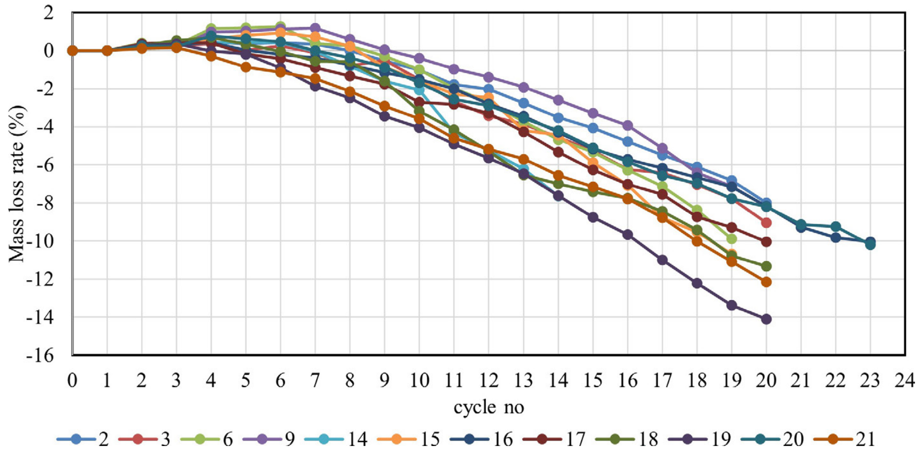


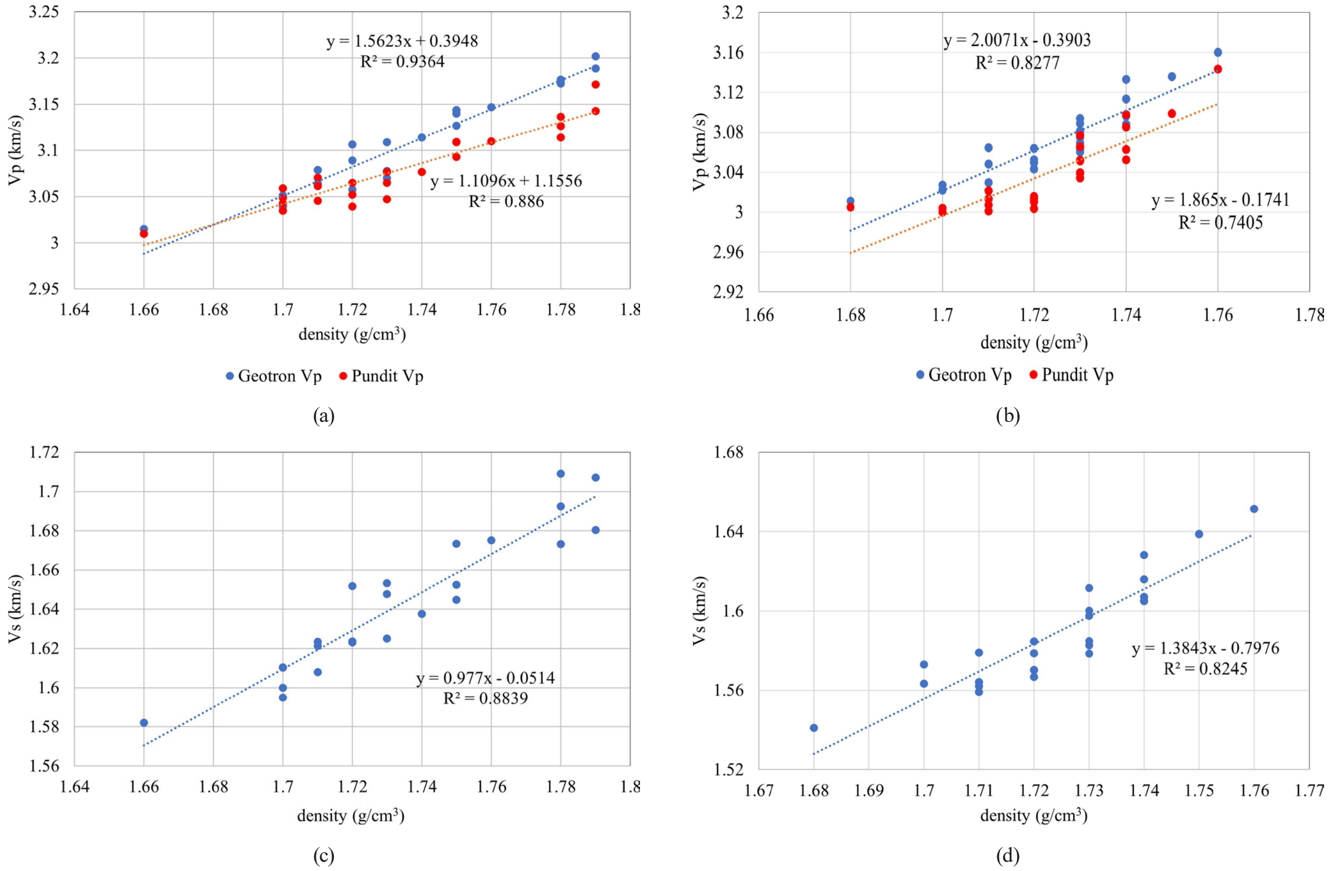
Fig. 2 Average mass loss rate for Prismatic samples

these microcracks due to freeze-thaw can rapidly develop into macro-cracks, leading to the disintegration or breakage of the sample. In this study, after 4 cycles, changes were visible in the microscopic structure of the andesite, with minerals possessing cleavage being more affected than non-discontinuous minerals. These changes cause increase in porosity. The porosity for cylindrical samples fluctuates between 32.73% to 36.04% with average 34.94% and for prismatic ones the highest porosity is 36.96% and lowest one is 34.3% with average 35.37%. Although porosity differential for both sample types is not immense, it can be concluded those samples with higher porosity are more prone to damage induced due to freeze thaw.

According to the information provided in Fig. 3, as the density of both cylindrical and prismatic samples increased, the velocity of P-waves (Vp) and S-waves (Vs) also increased. However, it is important to note that the data for cylindrical samples appeared to have less variation compared to prismatic samples. Therefore, it can be concluded that using the Geotron device would lead to more accurate results compared to the Pundit device. This is likely due to the Geotron device being more precise in measuring the velocity of waves in the samples, resulting in less variability in the data obtained as R square value for Geotron device is higher than Pundit device in Fig. 3 which indicate more precise result for Geotron than Pundit device and this difference for cylindrical samples is more obvious than prism samples with comparing Fig. 3 (a) and (b).

The results of water saturation tests for cylindrical samples (No 11,12 & 13) are presented in Figs. 4–6. These chosen samples were better consistency in terms of dimension. The plots can be segmented into two distinct regions. The initial region, spanning up to 25 $\sqrt{t}\,(\text{sec})$, displays a rapid and linear increase in both water absorption and water content with respect to the square root of time. The second region, extending from 25 $\sqrt{t}\,(\text{sec})$ to 130 $\sqrt{t}\,(\text{sec})$, exhibits a gradual trend and a smoother slope in comparison to the preceding phase. Notably, the slope of the plots varies across all samples, with a higher density corresponding to a lower slope in the first phase and reduced water absorption.

Based on Tables 1 and 2, freeze-thaw decreased UPV values for both cylindrical and prism samples. It is worth noting that 19 samples were chosen as these samples have



(a) (b) (c) (d)

Fig. 3 Ultrasonic wave velocity vs. density: (a) Vp for Cylindrical samples, (b) Vp for Prism samples, (c) Vs for Cylindrical samples, (d) Vs for Prism samples

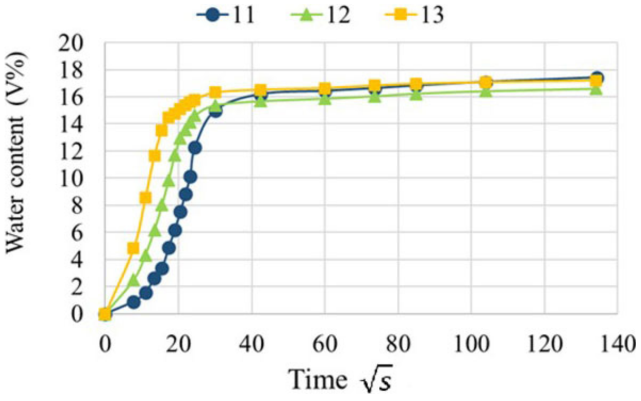


Fig. 4 Water content (V%) vs. time

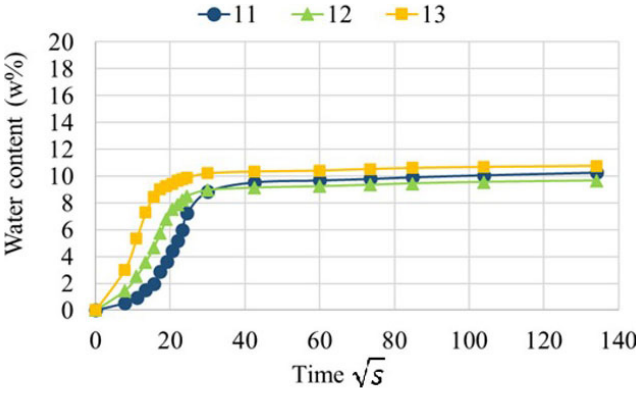


Fig. 5 Water content (w%) vs. time

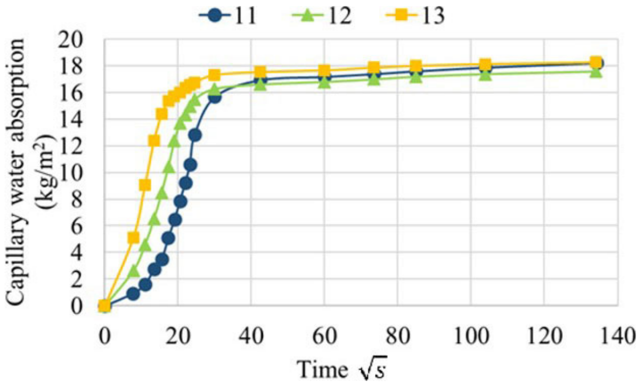


Fig. 6 Capillary water absorption (kg/m^2) vs. time

not been deteriorated. Vp differences due to freeze-thaw was between 18 to 21 %, while Vs experienced only 7–8 % rises. It can be concluded that Vp can be more applicable to recognition of freeze-thaw effect. Based on Fig. 7, P wave velocity will be remained the approximately the same when volumetric water content increase to 15%, while after 15% increasing, P wave velocity increased around 12%.

Based on Fig. 8, average UCS is 1.6 MPa. Compare to dry uniaxial compressive strength that is 2.51 MPa, more than 36% decrease in UCS was recorded. Please note that all samples in Fig. 8 experienced 23 cycle of freeze-thaw test and only prepared for UCS test and out of 10 samples only these 6 samples were remained for UCS test.

4 Discussion

In this study it was observed, where with freeze-thaw process, porosity of samples raised due to expansion of ice crystals in pores. Fener & Ince [1] also indicate that freeze-thaw process has a positive effect on porosity and there is a correlation between the number of freeze-thaw cycles and the porosity of andesite, with a linear relationship observed (R2: 0.88). As the number of freeze-thaw cycles increased, there was a corresponding increase in the porosity of the samples.

Based on Fig. 3, for both cylindrical and prism samples with increasing density, Vp and Vs raised. The same result was also observed by other researchers [16, 20–22]. This conclusion was then same for two different type of devices including Geotron and Pundit. The linear relationship between Vp and Vs was also observed. It is worth nothing that Pundit device only measure Vp. Based on Grune et al. [23] studies, Stones can be classified based on their capillary absorption capacity. Stones with low capillary absorption take up only 0.5 kg/m^2 s$_{0.5}$ of water, those with medium absorption can range from 0.5 to 3.0 kg/m^2 s$_{0.5}$, while stones with strong water absorption can take up as much as 3.0 kg/m^2 s$_{0.5}$ of water. So according to Fig. 6, limestones samples in this study can classify as stones with strong water absorption. Capillary water absorption is between 17 and 18 kg/cm^2 and initial suction of water for all samples presented the same trend. Strong water suction and final water absorption can prove good connection between pores in oolitic limestone. Celik [24] also proved that high water capillary can refer to effective and high pore connection for stones.

Based on Tables 1 and 2, it can be concluded that Vp can be more applicable to recognition of freeze-thaw effect. The same trend was observed by Heidari et al. [12] where the main reason for dropping Vp is increasing porosity with freeze-thaw cycles. The same trend was observed by Celik et al. [24], when they considered travertine samples and recorded more than 27% rise due to freeze thaw. Based on Amirkiyae et al. [3] apart from type of stones, for those ones with higher porosity, percentage loss of ultrasonic pulse velocity is higher and can reach 23% loss for stone with 10.22% porosity. In this study total reduction of 36% for UCS was observed and clearly oolitic limestone cannot

Table 1 Ultrasonic pulse velocity before and after 23 freeze-thaw cycles for cylindrical samples

SampleNo	Vp Pundit (km/s)			Vp Geotron (km/s)			Vs Geotron (km/s)		
	Dry	FT*	Reduction %	Dry	FT*	Reduction %	Dry	FT*	Reduction %
7	3.049	2.477	18.76	3.040	2.551	16.09	1.595	1.482	7.08
8	3.043	2.434	20.01	3.035	2.532	16.57	1.610	1.468	8.82
9	3.059	2.456	19.71	3.052	2.476	18.87	1.610	1.501	6.77
10	3.093	2.361	23.67	3.127	2.422	22.55	1.645	1.532	6.87
11	3.171	2.688	15.23	3.202	2.668	16.68	1.707	1.587	7.03
12	3.047	2.399	21.27	3.070	2.471	19.51	1.625	1.475	9.23
13	3.039	2.472	18.66	3.089	2.512	18.68	1.623	1.519	6.41
14	3.126	2.542	18.68	3.177	2.618	17.60	1.692	1.563	7.62
15	3.061	2.532	17.28	3.064	2.508	18.15	1.621	1.508	6.97
16	3.109	2.355	24.25	3.140	2.426	22.74	1.652	1.502	9.08
17	3.110	2.411	22.48	3.147	2.483	21.10	1.675	1.522	9.13

Table 2 Ultrasonic pulse velocity before and after 23 freeze-thaw cycles for prism samples

SampleNo	Vp Pundit (km/s)			Vp Geotron (km/s)			Vs Geotron (km/s)		
	Dry	FT*	Reduction %	Dry	FT*	Reduction %	Dry	FT*	Reduction %
2	3.034	2.420	20.24	3.089	2.436	21.14	1.612	1.500	6.95
9	3.066	2.513	18.04	3.081	2.531	17.85	1.598	1.511	5.44
16	3.001	2.309	23.06	3.030	2.336	22.90	1.559	1.472	5.58
17	3.099	2.446	21.07	3.136	2.476	21.05	1.639	1.546	5.67
18	3.013	2.423	19.58	3.014	2.446	18.85	1.562	1.477	5.44
19	3.004	2.440	18.77	3.064	2.461	19.68	1.579	1.489	5.70
20	3.040	2.421	20.36	3.094	2.496	19.33	1.598	1.483	7.20
21	3.105	2.377	23.45	3.098	2.391	22.82	1.617	1.516	6.25

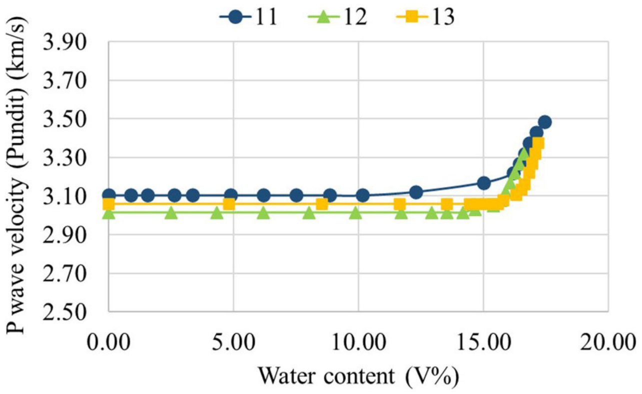


Fig. 7 Capillary water absorption (kg/m^2) vs. time

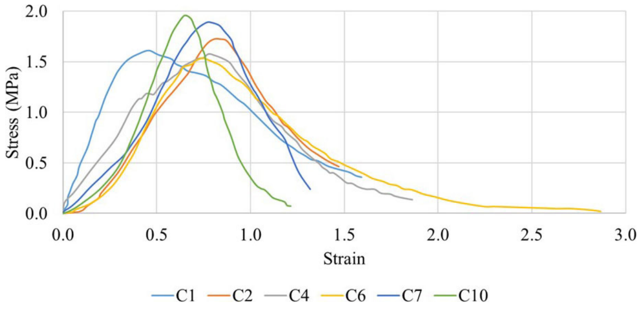


Fig. 8 UCS of oolitic limestone after 23 freeze-thaw cycle

stand cold weather. Heidari et al. [12] observed a significant decrease of mechanical properties after freeze-thaw process. Based on Bayram [10] if strength percentage loss exceeds 20%, the sample is unsuitable for use in cold regions. The Negative effect of freeze-thaw process on UCS was proven by other researchers [3, 12, 24]. Based on Celik et al. [24] the formation of microcracks may contribute to the observed reduction in UCS.

5 Conclusions

The Vp decreased for all studied stones in this research, with an increasing number of freeze-thaw cycles. The decreasing trend of Vp with an increase in the number of freeze-thaw cycles indicates the creation and extension of freeze-thaw-induced microcracks and pores.

Direct effect of increasing porosity due to freeze-thaw cycles can be observed on Vp and Vs changes. In this study, Vp dropped down by 28 to 20% while Vs experienced decreasing of 7 to 8%.

There is a negative correlation between the uniaxial compressive strength and the number of freeze-thaw cycles, resulting in a decrease in strength values. Building materials should not have a uniaxial compression strength percentage loss exceeding 20%, or they will be unsuitable for use in cold regions. In this study, UCS reduction percentage was more than 36%, which is indicate these types of stones are not suitable for cold areas.

The decrease in mechanical strength due to the process indicated a negative impact on the durability of the oolitic stones, particularly for those with higher pore connection and porosity. The decrease of 18 to 21% for Vp and 7–8% for Vs indicated that the freezing of water between pores facilitated easier wave transition in the samples. Overall, the results showed a negative impact of the freeze-thaw process on mechanical parameters of oolitic limestone such as UCS.

As a suggestion for future work, the effect of freeze-thaw on combination of oolitic limestone and mortar can be investigated both experimentally and numerically.

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