

**DURABILITY OF CONSOLIDATED POROUS LIMESTONES, A
LABORATORY TESTING APPROACH**

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Abstract

Three types of porous limestone were tested under laboratory conditions to assess the durability of various consolidation trials. Fine-, medium and coarse-grained types from Sóskút quarry were used. Cylindrical samples were consolidated by saturation. In the tests three consolidants were applied: one type of silica-acid-ester, an aliphatic uretan resin and a polymethyl methacrylate. The durability was tested on consolidated cylindrical specimens by accelerated weathering tests in the form of freeze-thaw cycles. Physical parameters such as density, total porosity, pore-size distribution, ultrasonic pulse velocity, Duroskop rebound values and indirect tensile strength tests (Brazilian test) were measured on natural, consolidated, and freeze-thaw affected consolidated samples. The durability was calculated by comparing test results of consolidated and freeze-thaw subjected consolidated samples. The indirect tensile strength tests were proved to model the changes in strength. Our analyses have shown that the tested consolidants have different penetration depth and different effect on strength of porous limestone. The durability against freeze-thaw of consolidated limestone types depends on micro-fabric (pore-size distribution) but cannot be directly assessed from the indirect tensile strength of the test specimens.

Keywords: porous limestone, stone consolidants, durability, tensile strength

1. Introduction

Porous limestones were commonly used as dimension stone in the 19th century in Central Europe especially in Austria and Hungary. Famous monuments such as St. Stephan's Dom in Vienna or the Parliament building and the Citadel in Budapest were constructed from these types of stones (Török et al. 2004). This type of limestone is very sensitive for weathering processes, which affect not only the aesthetic appearance of stones but also cause structural damage (Török 2002). Stone consolidation is aimed to slow down the rapid deterioration and/or strengthen the already weathered stones. Previous consolidation trials include testing of various carbonate stones (Wheeler et al. 2000, Alvarez de Buergo & Fort 2002, Lukaszewicz 2004, Ahmed et al. 2006, Ferrera Pinto & Delgado Rodriguez 2012). In this study three different consolidants (silicic acid ester, aliphatic uretan resin, Paraloid B72) were tested on three porous limestone types under laboratory conditions. The tests aimed to clarify how the different consolidating agents change the properties of porous limestone and how modify the durability of stones. Pápay & Török 2008 showed the first part of the experiments, which examined the effect of consolidation on air dry samples. The density, ultrasonic pulse velocity and

tensile strength were measured on the three limestone types prior and after the consolidation in order to understand the physical changes caused by the consolidants. Pápay & Török 2008 found that the treatment by silicic acid ester had the best efficiency.

2. Materials and methods

The Miocene limestones from Sós-kút were used in the tests, which are also known as oolitic limestone. From fine-, medium- and coarse-grained limestone blocks cylindrical specimens were drilled with diameter and height of 5 cm. The experiments were carried out according to the recommendations of the previous Hungarian standards and new EU norms at Rock analytical laboratory of Budapest University of Technology and Economics, Department of Construction Materials and Engineering Geology.

The cylindrical specimens were divided into analytical groups on the basis of non-destructive testing methods such as density (MSZ EN 1936:2000), ultrasonic pulse velocity (MSZ EN 14579:2005) according to MSZ 18282-4. In each testing group an equalized set of samples were grouped, namely to minimize the standard deviation of fabric related variations, such as density and ultrasonic pulse velocity. The tested consolidants and their properties are listed in Table 1. The specimens were treated under laboratory conditions at normal atmospheric pressure. The apparent density was determined by water saturation under atmospheric pressure. The tensile strength was determined by indirect tensile test (Brazilian test). In the course of Brazilian tensile strength test (MSZ 18285-2:1979), the specimens are loaded by parallel plates. Freeze-thaw cycles were carried out according to previous Hungarian Standard 18289/2-78 therefore specimens were saturated fully in water and frozen at -20 °C for 6 hours. The freeze-thaw cycles were continued until the first cracks appeared on specimens. The tested consolidants and their properties are detailed in Table 1.

Table 1. Properties of the applied consolidants

Consolidant	Diluting agent	Effective substance	Density [g/m ³]
Silicic acid ester	ready to use	ca. 20 m%	0,79
Aliphatic uretan resin	white spirit	50 m%	0,93
Paraloid B72	nitro-thinner	4 m%	0,85

Three different porous limestone types were used for the tests: fine-, medium- and coarse-grained ones. The fine grained variety has a microfabric of pelloidal wackestone. It contains micritic peloids and some forams. Intergranular pores dominate however small amount of intragranular pores also occur. The matrix is characterized by micritic

calcite with patches of microsparitic calcite. Majority of pores are within the range of 0,1 μm and 1 μm , but pores of 10 μm in size were also detected (Fig 1a). The water absorption rate is 16 m%; while the measured total porosity is 24 m%. The medium-grained limestone is characterised by well to moderately rounded calcitic ooids, micro-ocoids and visible but evenly scattered small pores (Fig 1b). Its microfabric is oolitic grainstone with few sand sized quartz grains in the nuclei of ooids (Török 2002). The coarse-grained limestone has a bioclastic ooidal grainstone micro-fabric (Török et al. 2007). The most common bioclasts are gastropods (*Cerithium* sp.). Large mouldic pores (up to 1 cm-size) are irregularly scattered within the stone (Fig. 1c.).



Figure 1. Fine-grained (a), medium-grained (b) and coarse-grained (c) limestone

3. Changes in physical properties

The measured parameters of treated and untreated test groups are listed in Table 3 for fine-grained, in Table 4 for medium-grained and in Table 5 for coarse-grained limestone. Tables show the average values of 3 or 4 specimens and the standard deviations are also listed in brackets.

Ultrasonic pulse velocity data show that the ultrasonic sound pulse velocity increased due to treatment and decreased after freeze-thaw cycles with no exception; for all the three limestones and for the three consolidating agents. For fine-grained limestone there was no significant change in ultrasonic pulse velocity. The largest decrease in ultrasonic pulse velocity was measured on silicic acid ester treated coarse-grained specimens (32,09%). Nearly the same change was observed for silicic acid ester and Paraloid B72 treated medium-grained test groups (-21,25% and -25,57%). Tensile strength values show the same trend as ultrasonic pulse velocity values for medium-grained and coarse-grained limestone. The change in tensile strength of treated air dry vs. treated frozen samples was the greatest for Paraloid B72 treated medium-grained limestone and silicic acid ester treated coarse-grained limestone (-57,9% and -58,1%). For silicic acid ester treated medium-grained and for Paraloid B72 treated coarse-grained limestone 34,1% and 39,5% decrease were observed. Aliphatic uretan resin caused less decrease 9,4 % for medium-grained and 6,25 % for coarse-grained limestone. Untreated samples show no damage in its structure, the tensile strength values are nearly the same. For fine-grained limestone increase in tensile strength was observed for frozen Paraloid B72 treated specimens (26,9%). Smaller change was measured for silicic acid ester

(+7,6%) and aliphatic uretan resin (-10,2%) treated test groups. The changes in tensile strength do not correspond to surface strength changes. There was no significant change in surface strength values except medium grained Paraloid B72 treated, coarse-grained untreated and silicic acid ester treated samples (-20% and -22,2%).

Before performance of freeze-thaw cycle the water absorption rate was measured for the three consolidating agents and for the reference test group. Fig. 2, Fig. 3 and Fig. 4 show the result of water absorption in 20 days period. The water absorption rates and their standard deviations are listed in Table 6.

Table 3. Physical properties of fine-grained limestone (ρ : density before/after treatment; v : ultrasonic pulse velocity before/after treatment, standard deviations of strength tests are given in brackets)

Test group	ρ [kg/m ³]	v [km/s]	Tensile strength [MPa]	Tensile strength after frost [MPa]	Surface strength [-]	Surface strength after frost [-]
untreated	1848/ 2060	2.27/ 2.24	2.09 (0.37)	1.92 (0.57)	24	22
silicic acid ester	2004/ 2060	2.70/ 2.59	3.43 (1.25)	3,69 (1.03)	24	24
Paraloid B72	1888/ 1991	2.58/ 2.39	2.68 (0.52)	3,4 (1.20)	25	26
aliphatic uretan resin	1924/ 1923	2.55/ 2.32	2.92 (0.29)	2,64 (0.12)	29	29

Table 4. Physical properties of medium-grained limestone (ρ : density before/after treatment; v : ultrasonic pulse velocity before/after treatment)

Test group	ρ [kg/m ³]	v [km/s]	Tensile strength [MPa]	Tensile strength after frost [MPa]	Surface strength [-]	Surface strength after frost [-]
untreated	1684/ 1705	2.32/ 2.10	0.91 (0.13)	0.90 (0.17)	5	5
silicic acid ester	1720/ 1717	2.40/ 1.89	1.32 (0.13)	0.87 (0.26)	6	6
Paraloid B72	1649/ 1655	2.19/ 1.63	0.95 (0.18)	0.40 (0.10)	10	8
aliphatic uretan resin	1725/ 1722	2.54/ 2.28	1.7 (0.15)	1.54 (0.14)	12	11

Table 5. Physical properties of coarse-grained limestone (ρ : density before/after treatment; v : ultrasonic pulse velocity before/after treatment)

Test group	ρ [kg/m ³]	v [km/s]	Tensile strength [MPa]	Tensile strength after frost [MPa]	Surface strength [-]	Surface strength after frost [-]
untreated	1568/ 1594	1.95/ 1.84	0.53 (0.15)	0.66 (0.11)	9	7
silicic acid ester	1596/ 1602	2.15/ 1.46	0.74 (0.12)	0.31 (0.02)	9	7
Paraloid B72	1609/ 1600	2.12/ 1.73	0.81 (0.16)	0.49 (0.22)	9	10
aliphatic uretan resin	1590/ 1607	2.24/ 2.22	1.60 (0.13)	1.50 (0.21)	13	13

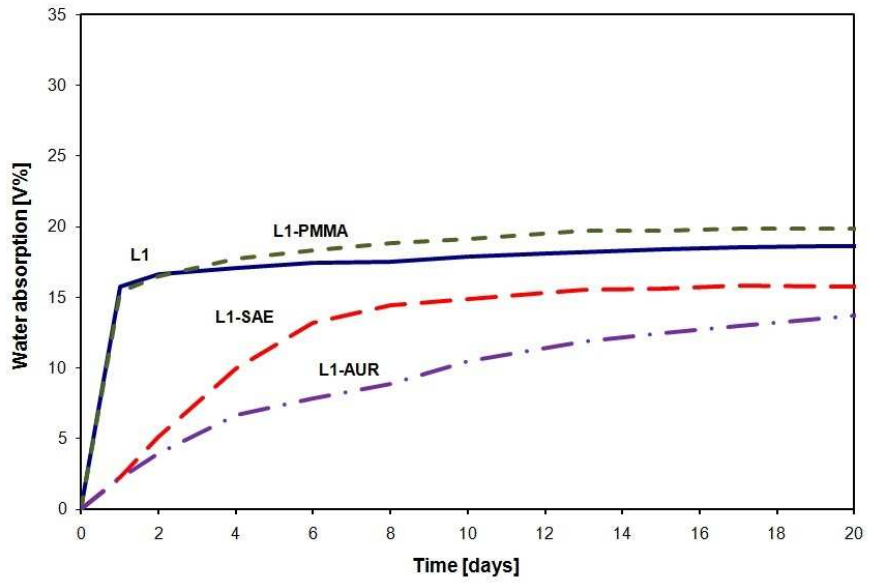


Fig. 2. Water absorption of fine-grained limestone

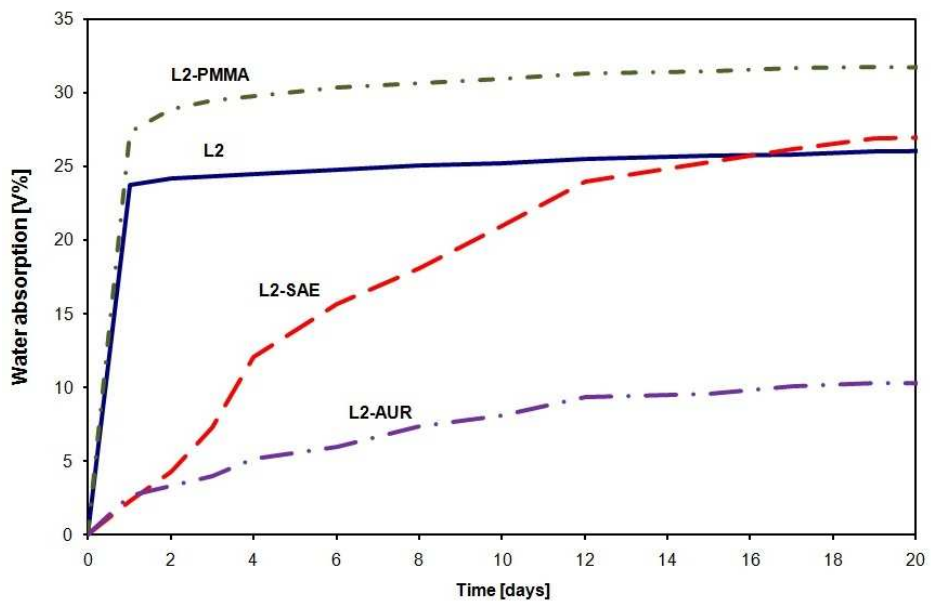


Fig. 3. Water absorption of medium-grained limestone

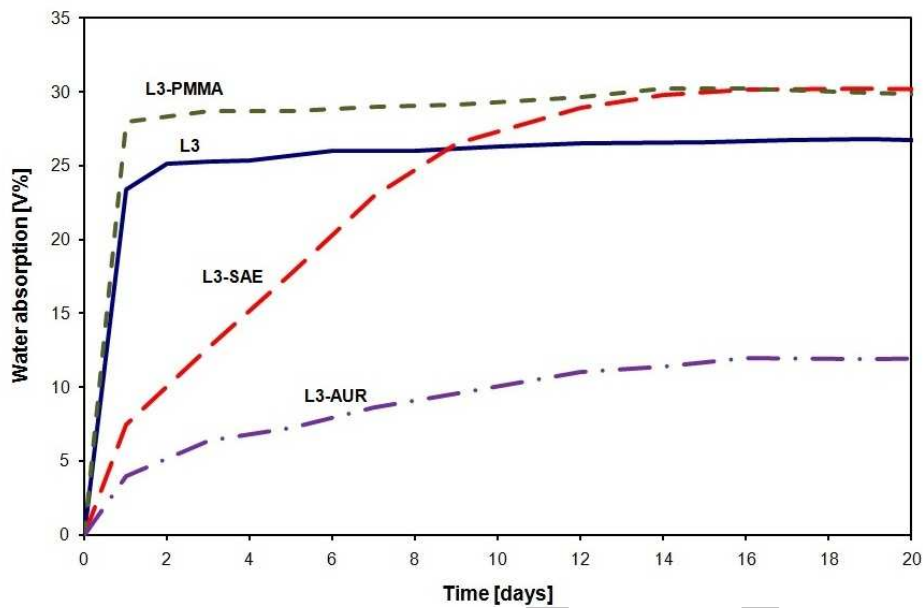


Fig. 4. Water absorption of coarse-grained limestone

Table 6. Water absorption rate of untreated and (treated) samples

Limestone type	air dry	silicic acid ester	Paraloid B72	aliphatic uretan resin
fine-grained	9.79 (3.79)	7.79 (2.88)	10.24 (4.39)	6.7 (1.38)
medium-grained	15.46 (1.51)	15.31 (0.96)	19.19 (0.21)	5.9 (0.48)
coarse grained	16.93 (1.35)	18.94 (0.34)	18.93 (0.89)	6.54 (2.24)

4. Conclusions

The freeze-thaw durability tests of untreated and consolidated limestones have shown that frost resistance of consolidated medium- and coarse-grained limestone samples became smaller. It is in good agreement with the measured increase in water absorption. It is difficult to find a clear and similar trend for the fine-grained limestone. The strength and porosity of treated vs. non-consolidated specimens show high scatter in data, although most consolidated samples have higher strength and less porosity, than that of the natural fine-grained limestone (Table 3 and Table 6). From the non-

destructive tests the Duroscope rebound values are less representative, while the ultrasonic pulse velocities indicate better the effect of consolidation. The treated samples have higher velocities compared to the non treated ones.

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