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## ON THE VARIATION IN SEVERAL ROCK PROPERTIES DUE TO MAGNESIUM SULFATE WEATHERING TESTS – A CASE STUDY FOR LIMESTONES

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### ABSTRACT

Contributions to the behavior of rock materials under various conditions provide a practical knowledge about issues relating the performance and long-term serviceability of rocks. In this study, various limestones with varying textural features were investigated in terms of their resistance against magnesium sulfate weathering tests. For this purpose, initial physico-mechanical properties of limestones were determined. Then, rock materials were subjected to magnesium sulfate weathering tests (up to 20 cycles) and the variation in physico-mechanical properties were determined for each rock type.

As a result of laboratory tests, compared to initial rock properties, effective porosity ( $n_e$ , %) increased in the range of 3% – 14% and 12% – 35% after 10<sup>th</sup> and 20<sup>th</sup> magnesium sulfate weathering cycles, respectively. Uniaxial compressive strength of rocks (UCS, MPa) decreased by 9% – 24% after 10<sup>th</sup> cycles and by 32% – 58% after 20<sup>th</sup> cycles. Brazilian tensile strength of rocks (BTS, MPa) decreased in the range of 7% – 19% and 20% – 49% after 10<sup>th</sup> and 20<sup>th</sup> cycles, respectively. Similar to the variations in UCS and BTS, Tangential Young Modulus ( $E_{ti}$ , GPa) also decreased at a rate of 13% – 28% and 23% – 64% after 10<sup>th</sup> and 20<sup>th</sup> cycles, respectively. However, the values of Tangential Poisson's Ratio ( $\nu_{ti}$ ) fluctuated with progressive accelerated weathering cycles, which could be linked to varying axial and lateral strain rates at 50% of UCS values for the limestones investigated. Furthermore, the variation in crack initiation stress  $\sigma_{CI}$  (MPa) due to progressive magnesium sulfate tests were also evaluated considering two strain-based methods and the findings showed that  $\sigma_{CI}$  of limestones slowly decreased with increasing weathering test cycles.

It could be claimed that cyclic magnesium sulfate tests performed on rock materials would be beneficial for assessing the long-term serviceability of rocks. In this context, mud-supported limestones seem to have a greater resistance against magnesium sulfate weathering tests compared to the grain-supported ones. However, the number of samples should be increased in order to achieve a comprehensive understanding about the degradation processes of limestones.

**Keywords:** Limestone, Magnesium sulfate weathering test, Strength, Deformability, Stress-Strain

## INTRODUCTION

Geotechnical characterization of rock materials is one of the principle parts in assessing their durability especially for building stones. Owing to the physical, chemical and biological processes, the weathering degree in rocks increases which is dependent upon several external factors such as the usage area, meteorological features of the environment and the elapsed time throughout its usage. The resistance of rocks against various environmental conditions is mainly measured by both field measurements and accelerated weathering tests in laboratory [1, 2]. Excluding the mechanical impacts, most destructive environmental factors for rocks could be declared the drying – wetting, freezing – thawing, heating – cooling and salt crystallization processes. Of these environmental factors, effects of freezing – thawing and salt crystallization become much important in terms of the durability of building stones and monuments. Effects of freezing – thawing cycles in nature are directly attempted to represent by experiments where freezing – thawing cabinets are used [3] whereas it is indirectly simulated by magnesium sulfate soundness tests in laboratory [4].

Heidari et al. [5] conducted an extensive laboratory investigation about the disintegration process of limestones and concluded that strength properties such as uniaxial compressive strength (UCS, MPa), Brazilian tensile strength (BTS, MPa) decrease whereas effective porosity ( $n_e$ , %) increase with progressive freezing – thawing and salt crystallization processes. As mentioned in the above-mentioned study, UCS of limestones decreased approximately by 29% after 16 cycles of magnesium sulfate tests. Momeni et al. [6] concluded that, after 90 cycles of magnesium sulfate tests, UCS of granitic rocks decreased at a rate of 22% – 26%. Furthermore, the researchers claimed that higher values of  $n_e$  resulted in a higher disintegration for granitic rocks under the influence of sodium and magnesium sulfate solutions. Köken et al. [7] stated that UCS of basaltic rocks decreased in the range of 23% – 28% after 40 cycles of magnesium sulfate tests.

Above-mentioned studies showed that rock strength properties decrease with increasing magnesium sulfate test cycles. However, the strength reduction rates due to magnesium sulfate tests are changeable for different rock lithologies. In this study, the variation in several rock properties of limestones due to magnesium sulfate tests was investigated. For this purpose, initial physico-mechanical properties of each rock type were determined. Then, cylindrical core samples were subjected to magnesium sulfate weathering tests (up to 20 cycles) and the variation in physico-mechanical properties were determined. The rate of increase (ROI) and decrease (ROD) observed in considered rock properties were stated and discussed.

## MATERIALS AND METHODS

Representative rock blocks of six different limestones were obtained from rock quarries located in various parts of Turkey. Cylindrical core samples with a diameter of  $54.0 \pm 0.02$  mm were obtained from these rock blocks. Following that, they were cut considering the geometrical instructions of related testing methods. Physical and mechanical properties of rocks were determined in accordance with the suggested methods by International Society of Rock Mechanics (ISRM) [8]. The physical properties considered in this study are dry unit weight ( $\gamma_d$ , kN/m<sup>3</sup>) and effective porosity ( $n_e$ , %).

Mechanical properties include uniaxial compressive strength (UCS, MPa), Brazilian tensile strength (BTS, MPa), Tangential Young Modulus ( $E_{ti}$ , GPa) and Tangential Poisson's Ratio ( $\nu_{ti}$ ). Mineralogical and textural properties of limestones were also determined by thin sections observations and investigated limestones were identified in accordance with the classification of Dunham [9].

Saturated magnesium sulfate solutions were prepared at  $21 \pm 1$  °C by using magnesium sulfate heptahydrate salt ( $MgSO_4 \cdot 7H_2O$ ) for the investigation of variations in selected rock properties, Smooth-cut core samples were placed into the saturated magnesium sulfate solution for 16 – 18 hours. Then, they were taken out of the solution and left to drain for 4 – 6 hours. Following that, core samples were placed in a drying-oven at  $105 \pm 2$  °C for 24 hours. The entire process described above was acknowledged as one cycle of magnesium sulfate weathering test ( $N_{ft-m} = 1$ ), which approximately took 2 days. Core samples were subjected to 20 cycles (i.e.  $N_{ft-m} = 20$ ) of magnesium sulfate tests in total. During the determination of above-mentioned rock properties, each test to determine selected rock properties was repeated at least five times and average values of related rock properties were presented.

## LABORATORY STUDIES

Six types of limestones with different textural properties were used in this study. Laboratory studies were divided into three parts. In the first part, physico-mechanical properties of each limestone were determined using core samples prepared (Fig 1a). Physical properties of limestones determined in this study were dry unit weight ( $\gamma_d$ ,  $kN/m^3$ ) and effective porosity ( $n_e$ , %) whereas mechanical properties were uniaxial compressive strength (UCS, MPa), Brazilian tensile strength (BTS, MPa), Tangential Young Modulus ( $E_{ti}$ , GPa) and Tangential Poisson's ratio ( $\nu_{ti}$ ). Physico-mechanical properties were determined in accordance with the suggested methods by ISRM [8].

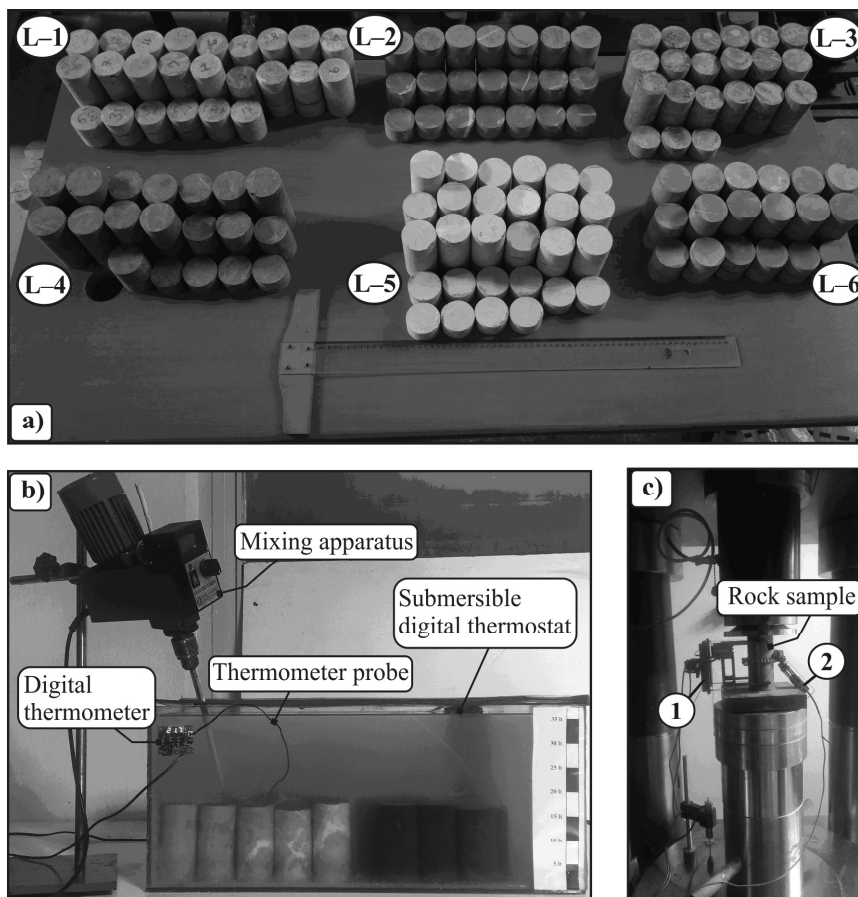
Second part of the laboratory studies covers the mineralogical and textural characterizations of limestones determined by thin section observations. Thin sections were prepared for each limestone and they were analyzed under a polarized microscope. In the last part of the laboratory studies, core samples were subjected to magnesium sulfate tests (Fig 1b) up to 20 cycles ( $N_{ft-m} = 20$ ) and the variations in several physical and mechanical properties were investigated. During UCS tests, axial and lateral deformations were measured using linear variable differential transformers (LVDTs) (Fig 1c).

Static-elastic constants (i.e.  $E_{ti}$  and  $\nu_{ti}$ ) of limestones were determined using stress-strain curves of each core sample. Axial ( $\varepsilon_z$ ), lateral ( $\varepsilon_l$ ) and volumetric deformations ( $\Delta = 2\varepsilon_l + \varepsilon_z$ ) were measured using LVDTs and  $E_{ti}$  (GPa) and  $\nu_{ti}$  were determined by the following equations:

$$E_{ti} = \frac{\sigma_z}{\varepsilon_z} \quad (1)$$

$$\nu_{ti} = -\frac{\varepsilon_l}{\varepsilon_z} \quad (2)$$

where:  $\varepsilon_l$  and  $\varepsilon_z$  are lateral and axial deformations, which correspond to the axial stress levels of 50% of UCS, respectively (i.e. values of  $\varepsilon_l$  and  $\varepsilon_z$  correspond to  $\sigma_z \approx 0.5UCS$ ).



LVDT sensors; sensor 1 used to measure axial deformations, sensor 2 used to measure lateral deformations.

Fig 1. Some of the laboratory equipments and materials used in the study a) Core samples b) Magnesium sulfate test c) Uniaxial compressive strength (UCS) test

As a result of thin section observations, limestones were characterized in terms of their textural properties (Table 1). Consequently, the texture of limestones varied from mud-supported to grain-supported types according to Dunham [9]. Moreover, weathering grade of limestones was found to be between unweathered ( $W_1$ ) and moderately weathered ( $W_3$ ) types.

The variation in stress-strain curves of limestones due to progressive magnesium sulfate cycles are given in Fig 2a. These curves, some of which were illustrated in Fig 2a, were used for the determination of crack initiation stress ( $\sigma_{CI}$ , MPa). For this purpose, two quantitative strain-based methods were adopted in this study.

The implementation of these methods to determine  $\sigma_{CI}$  is given in Fig 2b and Fig 2c. Fig 2b shows the implementation of the volumetric stress response (VSR) method, which was proposed by Pengfei et al. [10]. On the other hand, the implementation of lateral strain response (LSR) method by Nicksiar and Martin [11] is given in Fig 2c. Crack initiation stress for each limestone was determined by averaging values (i.e.  $\sigma_{CI(VSR)}$  and  $\sigma_{CI(LSR)}$  values) obtained from VSR and LSR approaches.

Table 1. Mineralogical and textural properties of the investigated limestones.

Rock type	Description	Texture	Mineralogical composition	Weathering Grade
L-1	Wackestone <sup>(I)</sup>	Mud-supported limestone with oolites.	Micro crystalline calcite (80%), skeletal and non skeletal fragments (18%) <sup>(III)</sup> , Siderite (2%)	W <sub>1</sub> – W <sub>2</sub> <sup>(III)</sup>
L-2	Grainstone	Grain-supported limestone with bearing dolomite. Densely veined with sparry calcite.	Micro crystalline calcite (24%), well-crystallized calcite (58%), skeletal and non skeletal fragments (6%), dolomite (10%), glauconite (2%).	W <sub>1</sub>
L-3	Mudstone	Mud-supported limestone with very fine-grained micro crystalline type. Randomly veined with sparry calcite in various thickness.	Micro crystalline calcite (90%), skeletal and non skeletal fragments (10%)	W <sub>1</sub>
L-4	Packstone	Grain-supported limestone with oolites. No calcite veins.	Micro crystalline calcite (60%), well-crystallized calcite (4%), skeletal and non skeletal fragments (25%), Siderite (7%), glauconite (2%), Opaque minerals (2%)	W <sub>1</sub> – W <sub>2</sub>
L-5	Mudstone	Mud-supported limestone. Densely veined with sparry calcite.	Micro crystalline calcite (90%), well-crystallized calcite (3%), skeletal and non skeletal fragments (7%)	W <sub>1</sub>
L-6	Packstone	Grain-supported limestone with fossiliferous micritic carbonate mud. No calcite veins.	Micro crystalline calcite (60%), well-crystallized calcite (5%), skeletal and non skeletal fragments (18%), Siderite (5%), glauconite (2%), Opaque minerals (10%)	W <sub>2</sub> – W <sub>3</sub>

**Note: (I)** Limestones were described according to Dunham [9]. **(II)** Skeletal and non skeletal fragments include fossils (e.g. foraminifers) oolites and pisolites etc. **(III)** Weathering grades of limestones were stated according to ISRM [8]. W<sub>1</sub>: Unweathered W<sub>2</sub>: Slightly weathered and W<sub>3</sub>: Moderately weathered.

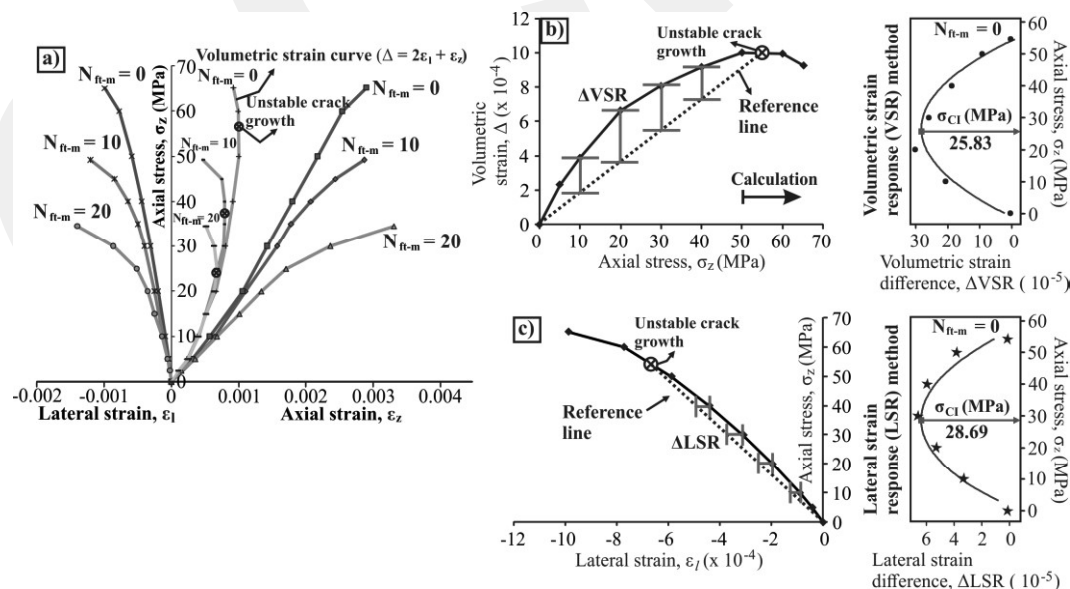


Fig 2. Determination of crack initiation stress a) Typical stress-strain curves of a limestone (L-1) b) Implementation of VSR method c) Implementation of LSR method.

Laboratory test results are given in Table 2. It was acquired that the values of  $\gamma_d$ , UCS, BTS and  $E_{ti}$  decreased whereas  $n_e$  of limestones increased with increasing  $N_{ft-m}$  in magnesium sulfate tests. Similar to the variations in the above-mentioned parameters,  $\sigma_{CI}$  also decreased with progressive magnesium sulfate tests.

Table 2. Laboratory test results.

$N_{ft-m} = 0$									
Rock type	$\gamma_d$ (kN/m <sup>3</sup> )	$n_e$ (%)	BTS (MPa)	UCS (MPa)	$E_{ti}$ (GPa)	$\nu_{ti}$	$\sigma_{CI(LSR)}$ (MPa)	$\sigma_{CI(VSR)}$ (MPa)	$\sigma_{CI-Mean}$ (MPa)
L-1	25.87	0.61	6.89	65.19	21.67	0.23	28.69	25.83	27.26
L-2	25.80	0.25	10.25	95.01	69.72	0.17	39.43	42.04	40.74
L-3	25.41	0.39	7.62	87.04	37.16	0.21	33.82	35.86	34.84
L-4	25.18	0.55	7.03	61.55	30.65	0.22	26.53	24.77	25.65
L-5	25.80	0.26	6.34	67.08	39.40	0.19	29.37	28.17	28.77
L-6	25.26	0.78	5.71	47.39	20.11	0.26	18.54	19.25	18.89
$N_{ft-m} = 10$									
Rock type	$\gamma_d$ (kN/m <sup>3</sup> )	$n_e$ (%)	BTS (MPa)	UCS (MPa)	$E_{ti}$ (GPa)	$\nu_{ti}$	$\sigma_{CI(LSR)}$ (MPa)	$\sigma_{CI(VSR)}$ (MPa)	$\sigma_{CI-Mean}$ (MPa)
L-1	25.35	0.65	6.15	49.20	18.64	0.22	18.75	20.56	19.66
L-2	25.56	0.26	8.19	86.46	60.20	0.16	32.57	36.09	34.33
L-3	25.16	0.43	7.08	72.18	29.52	0.19	26.87	29.12	28.00
L-4	24.91	0.60	6.34	50.28	24.07	0.23	20.05	18.77	19.41
L-5	25.44	0.28	5.78	55.39	35.14	0.19	19.48	18.95	19.22
L-6	24.41	0.89	4.57	35.69	14.43	0.28	14.19	12.56	13.38
$N_{ft-m} = 20$									
Rock type	$\gamma_d$ (kN/m <sup>3</sup> )	$n_e$ (%)	BTS (MPa)	UCS (MPa)	$E_{ti}$ (GPa)	$\nu_{ti}$	$\sigma_{CI(LSR)}$ (MPa)	$\sigma_{CI(VSR)}$ (MPa)	$\sigma_{CI-Mean}$ (MPa)
L-1	24.18	0.71	4.65	34.58	14.90	0.23	12.09	13.03	12.56
L-2	24.66	0.28	6.05	63.93	49.53	0.17	21.83	24.35	23.09
L-3	24.60	0.46	5.14	58.23	25.66	0.22	22.64	24.66	23.65
L-4	24.51	0.69	3.96	34.55	14.06	0.23	12.05	12.40	12.23
L-5	24.97	0.31	5.05	44.26	30.04	0.21	17.50	16.90	17.20
L-6	23.15	1.06	2.87	19.55	7.07	0.31	6.81	6.43	6.62

**Note:** Results obtained from core samples treated by magnesium sulfate tests in different number of cycles (e.g.  $N_{ft-m} = 10$ ).

## RESULTS AND DISCUSSION

Laboratory test results showed that the durabilities of limestones subjected to magnesium sulfate tests considerably decrease. Compared to the initial rock properties, the variations in selected rock properties due to progressive magnesium sulfate tests are listed in Table 3. Accordingly,  $\gamma_d$  of rocks were found to be the variable with presenting relatively minimal changes, whereas UCS seemed to be the most effected rock property under the influence of magnesium sulfate weathering tests.

It was observed that the mean value of  $n_e$  compared to the ones after 10 and 20 cycles of magnesium sulfate tests increased at an average rate of 8% (ROI = 3 – 14%) and 20% (ROI = 12 – 35%), respectively.

Table 3. Variations (in percentage) in several rock properties of limestones due to progressive magnesium sulfate tests.

Rock type	$\gamma_d$ (kN/m <sup>3</sup> )		$n_e$ (%)		BTS (MPa)		UCS (MPa)		$E_{ti}$ (GPa)		$v_{ti}$	
L-1	-2.01(*)	-6.53(**)	6.55	16.39	-10.74	-32.51	-24.53	-46.96	-13.98	-31.24	-3.88	-0.86
L-2	-0.93	-4.41	4.00	12.00	-20.09	-40.97	-9.00	-32.71	-13.65	-28.96	-4.09	1.75
L-3	-0.98	-3.19	10.25	17.95	-7.08	-32.55	-17.07	-33.10	-20.56	-30.95	-8.01	4.71
L-4	-1.07	-2.66	9.09	25.45	-9.81	-43.67	-18.31	-43.87	-21.47	-54.12	5.38	4.93
L-5	-1.39	-3.21	3.84	15.38	-8.83	-20.35	-17.43	-34.02	-10.81	-23.76	-2.54	9.14
L-6	-3.36	-8.35	14.10	35.89	-19.96	-49.74	-24.69	-58.75	-28.24	-64.84	7.22	20.15

**Note:** Negative and positive values indicate the rate of decrease (ROD) and the rate of increase (ROI) observed in rock properties, respectively. Values given in left (\*) and right (\*\*) columns indicate the variations in related rock properties determined after 10 and 20 cycles, respectively.

Mechanical rock properties such as BTS and UCS decreased at average rates of 36% (ROD = 20 – 49%) and 42% (ROD = 32 – 58%) after 20 cycles, respectively. The ROD in  $E_{ti}$  was found to be in the range of 13 – 28% and 23 – 64% for 10 and 20 cycles, respectively. However, the variation in  $v_{ti}$  values were changeable for investigated limestones. For instance, compared to the initial mean value of  $v_{ti}$  for grain-supported limestones (i.e. L-2), it decreased (ROD  $\approx$  4%) after 10 cycles but slightly increased (ROI  $\approx$  2%) after 20 cycles. For mud-supported limestones (i.e. L-1 and L-4),  $v_{ti}$  of L-1 decreased (ROD  $\approx$  4%) whereas  $v_{ti}$  of L-4 increased (ROI after  $\approx$  5%) after 10 cycles of magnesium sulfate tests. Generally, it could be claimed that  $v_{ti}$  of limestones slightly increase with progressive magnesium sulfate tests. However, the trend of axial and lateral strain rates was non-linear and therefore the variation in  $v_{ti}$  values of limestones fluctuated when axial stresses were around to be  $\sigma_z \approx 0.5UCS$ .

Focusing on the variations in stress-strain curves (e.g. Fig 2a) due to progressive magnesium sulfate tests, it was shown that, axial and lateral deformations non-linearly increase with increasing  $N_{ft-m}$  in magnesium sulfate tests. The stress levels when the unstable crack growth (where  $N_{ft-m} = 0$ ) began, were found to be in the range of  $\sigma_z \geq 0.76 - 0.90$  of the UCS for this study.

With progressive magnesium sulfate tests, crack propagation stresses occurred earlier than the above-mentioned stress levels. In parallel with this statement,  $\sigma_{CI}$  of limestones also decreased slowly. In general, crack initiation stress ( $\sigma_{CI}$ , MPa) levels of non-treated limestones varied between 0.40 – 0.42 of the UCS. With progressive magnesium sulfate tests,  $\sigma_{CI}$  values were found to be between 0.38 – 0.39 and 0.37 – 0.38 of the UCS after 10 and 20 cycles, respectively.

The findings obtained from this study reveal that the durability of limestones decreases with increasing  $N_{ft-m}$  in magnesium sulfate tests. Particularly, mud-supported limestones (i.e. L-3 and L-5) seem to have a greater resistance against magnesium sulfate weathering tests compared to the grain-supported ones (i.e. L-4 and L-6) for this study. However, this finding is only valid for the present study and therefore, number of samples should be increased to achieve a comprehensive understanding about the degradation processes of limestones.

## CONCLUSIONS

The present study covers a comprehensive laboratory investigation on the resistance of limestones against magnesium sulfate weathering tests. Core samples were subjected to saturated magnesium sulfate solution for 20 cycles in total and the variations in several rock properties were determined. The results showed that the durability of limestones decrease with progressive magnesium sulfate tests. The most effected rock property due to magnesium sulfate tests seems to be the UCS of rocks. Compared to the grain-supported ones, mud-supported limestones seem to have a greater resistance against magnesium sulfate tests. Last but not the least, long-term behavior of rocks could be assessed by accelerated weathering tests. As an inductive approach based on comparisons between natural and artificial environmental impacts on rocks, such investigations could also be beneficial for further studies.

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