Estimating the durability of building stones against Salt crystallization: considering the physical properties and strength characteristics

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Abstract
Salt crystallization is one of the most important weathering agents and may limit the durability of building stones. Salt crystallization induces stresses inside the pores of stones. Consequently, stone durability is closely related to its physical properties and strength. The purpose of this study was to propose a statistical model for estimating stone durability against salt crystallization considering both the physical properties and strength of the stones -utilizing multiple regression. For this purpose, 14 samples of building stones were selected and their mineralogical characteristic, physical properties, and strength (density, porosity, water absorption, uniaxial compressive strength, and Brazilian tensile strength) were determined. Then, the salt crystallization test at a sodium sulfate solution of up to 20 cycles was carried out, and the Dry Weight Loss (DWL) of samples was measured. The durability of each sample was assessed by the percentage of weight loss after the salt crystallization test. The relationships between stone durability and the physical properties and strength of the stones- using simple and multiple regression analyses- were investigated. Moreover, statistical models for estimating stone durability were proposed. These models show that stone durability can be estimated accurately by considering both the physical properties and strength characteristics.

Keywords: *Durability, Physical properties, Salt crystallization, Sodium sulfate, Statistical models. Strength.*

Introduction

Salts crystallization in porous construction materials has been considered as one of the important weathering processes contributing to the decay of masonry, cement, and mortar in a range of environments (Goudie & Viles, 1997; Novak & Colville, 1989). In buildings, highways, and civil engineering works, salt weathering has been connected, in many cases, to the crystallization of soluble salts released from Portland cement (Ritchie, 1955). Among those salts, sodium sulfate is considered a very dangerous contributor to stone decay because of its strong crystallization pressure (cultrone & Sebastian, 2007). Sodium sulfate is responsible for significant decay rates in porous building materials that are apparently caused by the generation of high crystallization and hydration pressures (Evans, 1970; Winkler, 1970; Winkler & Singer, 1972). Its destructive nature has made it the salt of choice to perform accelerated decay tests to estimate the durability of building materials.

Many experimental studies have been carried out to assess stone decay rates produced by salt crystallization (e.g., Tsui *et al.,* 2003; Benavente *et al.,* 2004; Ulusoy, 2007; Yu & Oguchi, 2010; Urosevic *et al*., 2010; Angeli *et al.,* 2010; Molina *et al.,* 2011; Yavuz, 2012). Linking the durability of porous building stones to their physical and strength characteristic, including: porosity, water absorption, water flow, pore structure, uniaxial compressive strength, tensile strength and flexural strength, has been a long-term aim that has generated great interest in many fields such as engineering geology, material science, architecture, and earth sciences. Salt weathering of soluble salts is one of the most important decay processes that affects the durability of porous building stones. A number of factors influence salt crystallization's decaying the porous building stones, including (1) environmental controls, (2) the nature of the salt, (3) physical properties of the stone, and (4) strength characteristics, i.e., the material's resistance to crystallization pressure.

The aim of this study was to propose a statistical model for estimating the stone's durability against salt crystallization, using both the physical properties and strength characteristics, using multiple regression analysis.

Materials and Methods

To carry out the research, the researchers selected 14 samples of building stones from factories around Tehran and quarries of Iran (Fig. 1). The name, type and stratigraphic unit of the rocks are given in Table 1. These stones are marketed and used as building materials and are highly homogeneous not only in the hand specimen, but also in the quarry. During sampling, the stone types with no bedding planes were selected to eliminate any anisotropy effects on the measurement. The mineral composition and textural properties of the samples were studied by means of optical microscope. The physical properties (dry and saturated density, porosity, water absorption, and specific gravity) and strength characteristics (uniaxial compressive strength, and Brazilian tensile strength) of each sample were determined. A salt crystallization test of up to 20 cycles was carried out on sodium sulfates, and the dry weight loss (DWL) of the samples was measured.

Table 1: Name, type and stratigraphic unit of the samples under study

Rock code	Rock type	Rock class	Stratigraphic position	Time-Stratigraphic unit
1	Rhyolite	Igneous	Taknar Rhyolite Extrusive	Precambrian
2	Ignimbrite	Igneous	Zarigan Granite Intrusion	Precambrian
3	Granite-I	Igneous	Mashhad Granite Intrusion	Carboniferous
4	Granite-II	Igneous	Shahkuh Granite Intrusion	Middle Jurassic
5	Dacite	Igneous	Zarigan Granite Intrusion	Precambrian
6	Travertine-I	Sedimentary	Old Travertine	Quaternary
7	Travertine-II	Sedimentary	Old Travertine	Quaternary
8	Limestone I	Sedimentary	Gaveh Rud Basin (Sanandaj Shales)	Paleocene - Eocene
9	Dolomitic Limestone	Sedimentary	Sibzar Formation	Middle Devonian
10	Limestone -II	Sedimentary	Bahram Formation	Late Devonian
11	Vitric tuff	Sedimentary	Karaj Formation	Eocene
12	Marble-I	Metamorphic	Khabar Complex	Devonian
13	Marble-II	Metamorphic	Khabar Complex	Devonian
14	Amphibolite	Metamorphic	Deh Salam Metamorphosis Complex	Late Triassic – Middle Jurassic

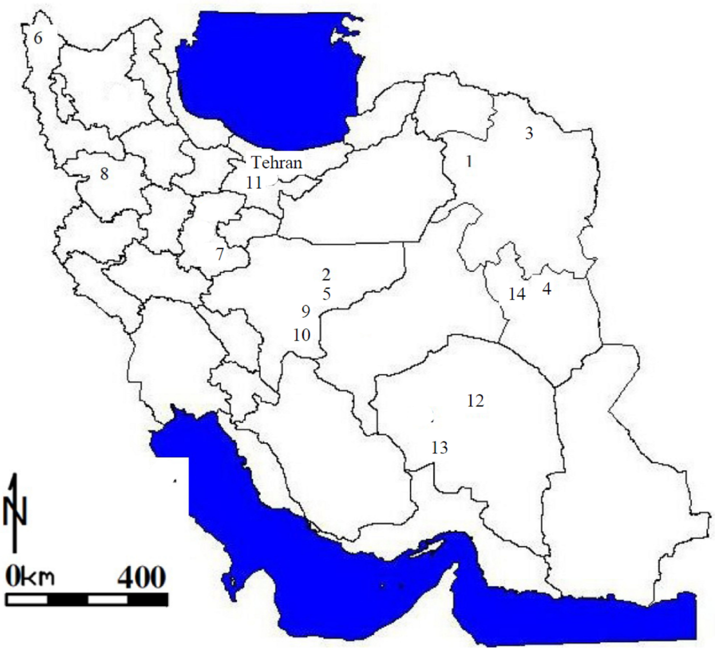

Figure 1: The location of sampling (1: Rhyolite, 2: Ignimbrite, 3: Granite-I, 4: Granite-II, 5: Dacite, 6: Travertine-I, 7: Travertine-II, 8: Limestone-I, 9: Dolomitic Limestone, 10: Limestone-II, 11: Vitric tuff, 12: Marble-I, 13: Marble-II, 14: Amphibolite

Mineralogical and textural properties

Mineralogical and textural properties studies not only provide information on the mineralogical composition and provenance of the rock origin, but also they provide an important tool for assessing its durability and resistance against weathering agents. Optical microscope was used to determine the mineralogical composition and textural properties of the samples. Fig. 2 shows the thin sections of the samples. Mineralogical composition and textural properties for samples are given in Table 2.

Table 2: Type, class, mineralogical composition and textural properties of the samples under study

Rock type	Rock class	Mineralogical composition and textural properties
Rhyolite	Igneous	Fine grained texture. Fine grain quartz and Alkali-feldspar are within the vitreous groundmass. Less than 3% muscovite occur in the stone
Ignimbrite	Igneous	Composed mainly rock fragment, quartz, Alkali-feldspar, biotite. Plagioclase is partly chloritized
Granite-I	Igneous	Medium to coarse-grained (2–5 mm) and granular texture. Quartz, feldspar and muscovite are main mineral, plus less amount plagioclase and biotite. feldspar partly altered to sericite
Granite-II	Igneous	Composed mainly of quartz, Alkali-feldspar (microcline) and muscovite. Granular texture. Plagioclase and biotite are minor
Dacite	Igneous	Main minerals observed are Alkali-feldspar and plagioclase that are more or less equidimensional. Groundmass with a high iron content
Travertine-I	Sedimentary	Essential minerals are calcite, occur in a lime mud. Calcites are mainly euhedral and subhedral. The pores partly are filled with iron oxides cement
Travertine-II	Sedimentary	Micritic limestone. Micrite lime mud is highly porous which filled with calcite
Limestone I	Sedimentary	Micritic limestone. Essential minerals are calcite in a lime mud. Less than 4% opaque minerals occur in the stone
Dolomitic Limestone	Sedimentary	Recrystalised limestone. Essential minerals are coarse- grained calcites and medium grained dolomites
Limestone -II	Sedimentary	Essential minerals are calcite in a carbonate matrix. Calcites are fine- grained and euhedral. Less than 10% dolomite occur in the stone
Vitric tuff	Sedimentary	Perlitic texture. Composed manily quartz, rock fragments and plagioclase that are the within the vitreous groundmass
Marble-I	Metamorphic	Consists mainly of fine-grained calcite crystals interlocked with each other. Slightly metamorphosed.
Marble-II	Metamorphic	Formed by entirely coarse calcite crystal that arranged in an interlocking pattern. Slightly to medium metamorphosed
Amphibolite	Metamorphic	Amphibole and plagioclase are main minerals. Amphibole partly altered to chlorite. Garnet, sphene and epidotic can be observed in different dimensions

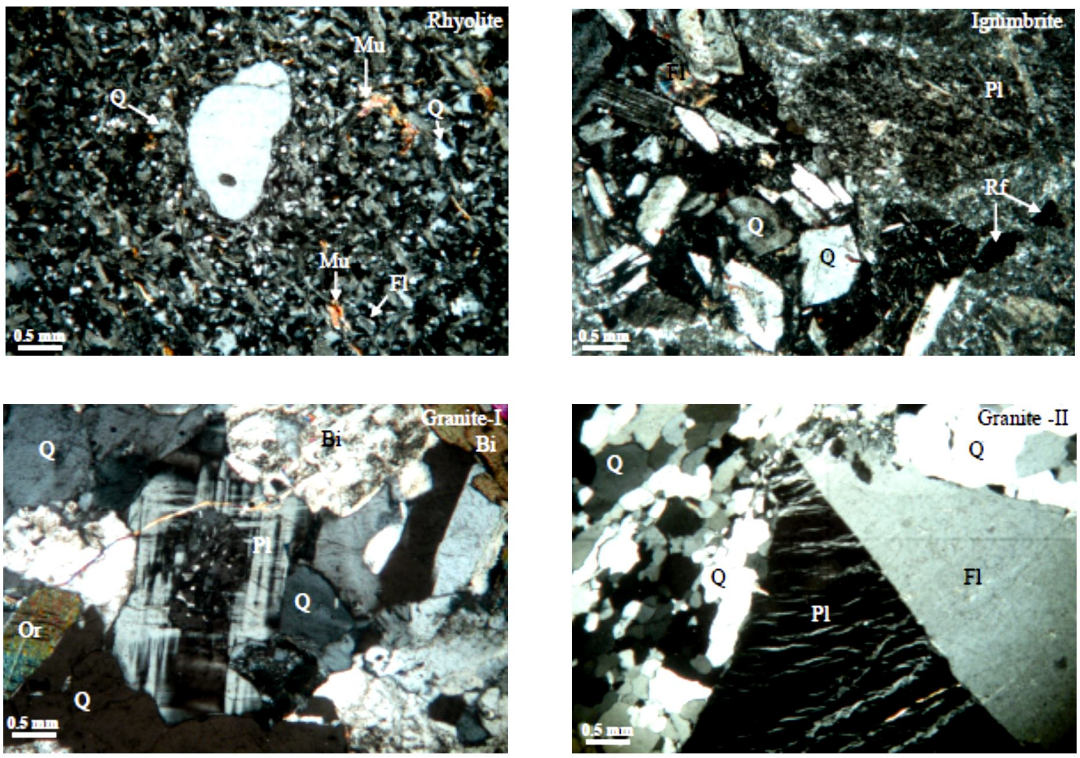

Figure 2: Continued on the next page

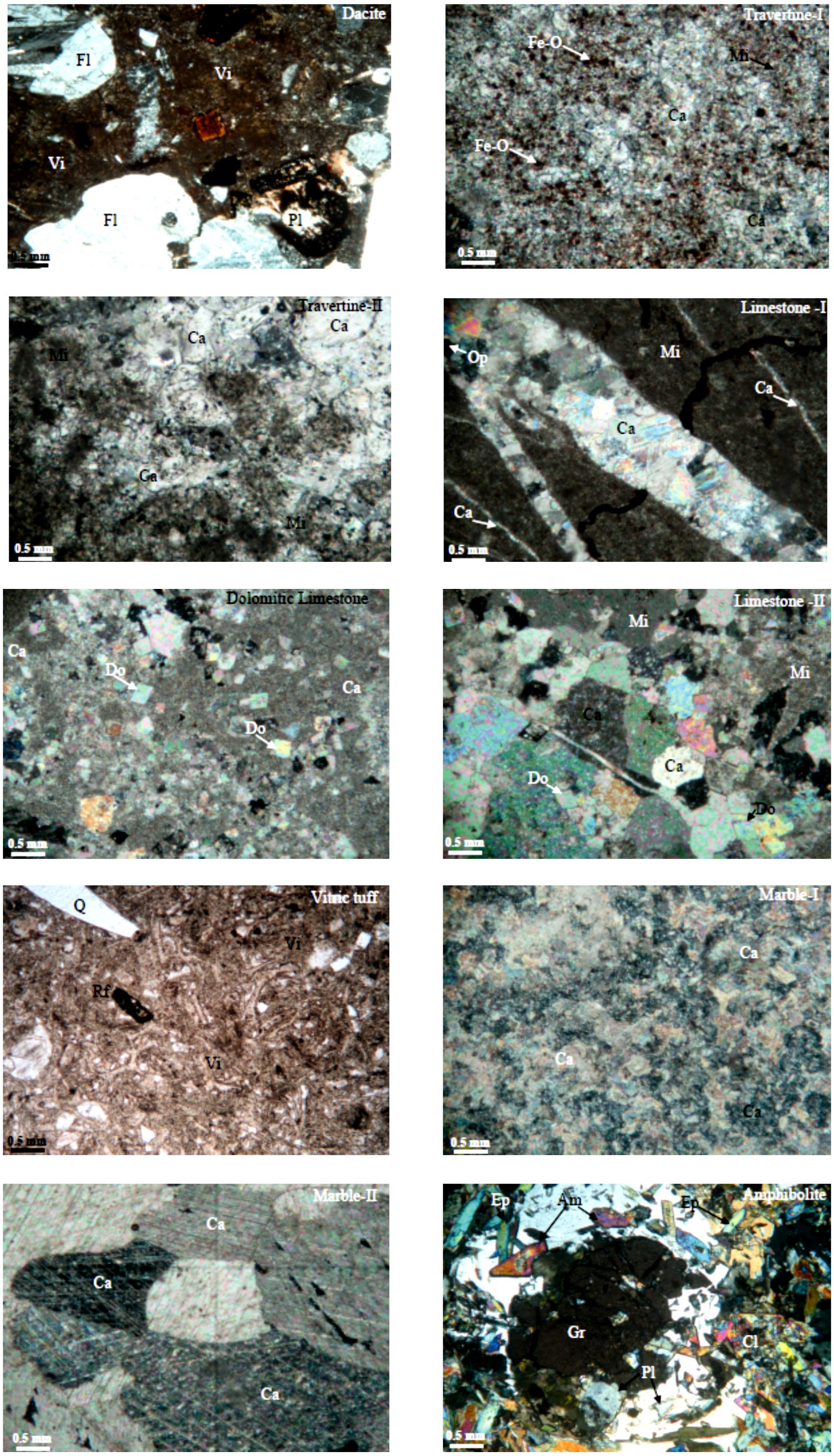

Figure 2: The typical petrographic images of the samples (Q: Quartz, Rf: Rock fragment, Vi: Vitreous groundmass, Or: Orthoclase, Pl: Plagioclase, Bi: Biotite, Fl: Feldspar, Mi: Micrite, Ca: Calcites, Op: Opaque minerals, Do: Dolomite, Fe-O: Iron oxides cement, Am: Amphibole, Ep: Epidotic, Gr: Garnet, Cl: Chlorite, Mu: Muscovite)

Physical properties

The physical properties of the samples, including dry density (ρ_d), saturated density (ρ_{sat}), effective porosity, total porosity, water absorption (W_a) and specific gravity (G_S), were determined according to ASTM-C 830 (ASTM, 2000). Dry density, saturated density, effective porosity, and water absorption were determined using the saturation and buoyancy methods. This method is suitable for the tested samples of this study, because they have no friable and swelling potentials. Total porosity was measured by crushing the rock into a fine powder and measuring its volume by fluid displacement in a pycnometer. The total volume of pores was calculated as the difference between the volume of the sample and that of the crushed particles. Five samples from each stone type in the form of cylindrical were used and then the mean values were obtained. The results of these determinations are given in Table 3. Although pores of Travertine-I are partly filled with iron oxides cement, it has a lower density than the other samples (except Vitric tuff). This can be due to its higher porosity level in comparison with the other samples.

Strength characterization

The strength characterization of the samples, including uniaxial compressive strength and Brazilian tensile strength, were determined according to ISRM (1981). Five samples from each stone type in the form of cylindrical were used, and then the mean values were obtained. The results of these determinations are given in Table 3. According to the classification of rocks based on the uniaxial compressive strength by Broch & Franklin (1972), the Travertine I and II are classified as rocks with high strength (15-50 MPa), and the other samples are considered rocks with very high strength (50-160 MPa).

Table 3: The physical properties and strength characteristics of the samples under study

Rock type	Dry density (ρ_d) (g/cm^3)	Saturated density (ρ_{sat}) (g/cm^3)	Effective porosity (P_{Ef}) (%)	Total porosity (P_T) (%)	Water absorption(W_a) (%)	Specific gravity (G_S)	Uniaxial compressive strength (UCS) (MPa)	Brazilian tensile strength (BTS) (MPa)
Rhyolite	2.45	2.49	4.15	6.49	1.69	2.62	130.2	18.3
Ignimbrite	2.63	2.64	1.48	1.87	0.56	2.68	94.4	12.9
Granite-I	2.63	2.65	1.26	1.87	0.48	2.68	98.7	12.1
Granite-II	2.58	2.58	0.91	1.90	0.35	2.63	80.5	14.5
Dacite	2.59	2.59	0.96	1.53	0.37	2.62	117	18.4
Travertine-I	2.38	2.41	3.36	11.52	1.41	2.69	33.1	5.2
Travertine-II	2.41	2.44	2.93	10.07	1.22	2.68	44.4	4.1
Limestone-I	2.70	2.71	0.37	1.46	0.14	2.74	95.6	7.8
Dolomitic Limestone	2.68	2.69	0.77	1.11	0.29	2.71	83.0	10.5
Limestone-II	2.68	2.69	0.46	1.47	0.17	2.72	93.3	12.9
Vitric tuff	2.18	2.30	12.21	16.15	5.61	2.60	101.5	11.2
Marble-I	2.75	2.76	0.22	0.72	0.08	2.77	96.0	11.3
Marble-II	2.69	2.70	0.44	1.10	0.16	2.72	64.7	5.8
Amphibolite	3.06	3.07	0.35	0.97	0.12	3.09	147.0	19.2

Salt crystallization

Salt crystallization, which a rock undergoes at and near the earth's surface, is one of the most powerful decay mechanisms. When water reaches the pore network of a rock, it may carry various salts in the solution. When this occurs, highly concentrated salt solutions may yield large volumes of precipitates (Goudie & Viles, 1997). Several mechanisms have been proposed to explain the decay caused by soluble salts to porous materials. These mechanisms include generation of crystallization pressure, hydration pressure, thermal expansion, osmotic pressure, and chemical weathering (Rodriguez-Navarro & Doehne, 1999). Sodium sulfate salt is extensively found in both damaged cement structures and building stones (Rodriguez- Navarro *et al.,* 2000). It is highly damaging and causes completely different decay effects. Occurrence of sodium sulfate salt in nature is quite common and distributed worldwide (Goudie & Cooke, 1984). This salt is commonly used in the accelerated decay testing of stones because its crystallization is highly damaging (Rodriguez-Navarro & Doehne, 1999). In fact, this salt typically is ranked as the most effective salt in salt crystallization experiments (Goudie, 1993).

Salt crystallization test

There are many standard tests based on the total immersion of samples in a salt solution: the American standard test ASTM (C-88, C-128), the Germany standard test DIN (52111), and the Spanish standard test UNE-EN (12370). The small difference between them is their temperature, relative humidity, sample size, salt concentration, and number of cycles (Jefferson, 1993; Goudie, 1999).

In this study, the Spanish standard test UNE-EN (12370) was used. The quantification of sample durability against salt crystallization was measured by the Dry Weight Loss (DWL) of the samples after the test. In this test, five samples were used, and a 14% w/w Na_2SO_4 solution was used. First, clean and dry samples were introduced into a container and completely covered with the solution at 20°C for a period of 4 h (immersion stage). Second, the samples were removed from the solution, and settled into the drying oven at 60°C for a period of 16 h (drying stage). Finally, the samples were subjected to room conditions 20°C for a period 4 h (cooling stage). The duration of this cycle is 24 h and the test procedure was repeated for 20 cycles (see Fig. 3).

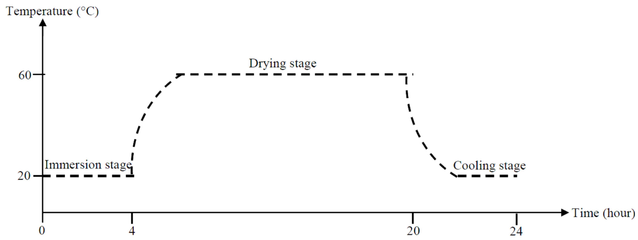

Figure 3: The generalized temperature curve for the salt crystallization cycle

At the end of the 20 cycles, the tested samples were cleaned with distill water to eliminate salt. Then samples were dried until they reached a constant weight. The DWL was calculated in the end of this stage. For example, the change in visual appearance of Travertine-I before and after salt crystallization test is illustrated in Fig. 4. The results are given in Table 4, and graphically illustrated in Fig. 5.

Table 4: Dry Weight Loss (DWL) of samples by salt crystallization

Rock type	DWL (%)	Rock type	DWL (%)
Rhyolite	6.1	Limestone-I	3.1
Ignimbrite	8.8	Dolomitic Limestone	6.0
Granite-I	5.5	Limestone-II	2.9
Granite-II	4.3	Vitric tuff	20.1
Dacite	7.9	Marble-I	1.3
Travertine-I	11.2	Marble-II	5.8
Travertine-II	15.6	Amphibolite	1.9

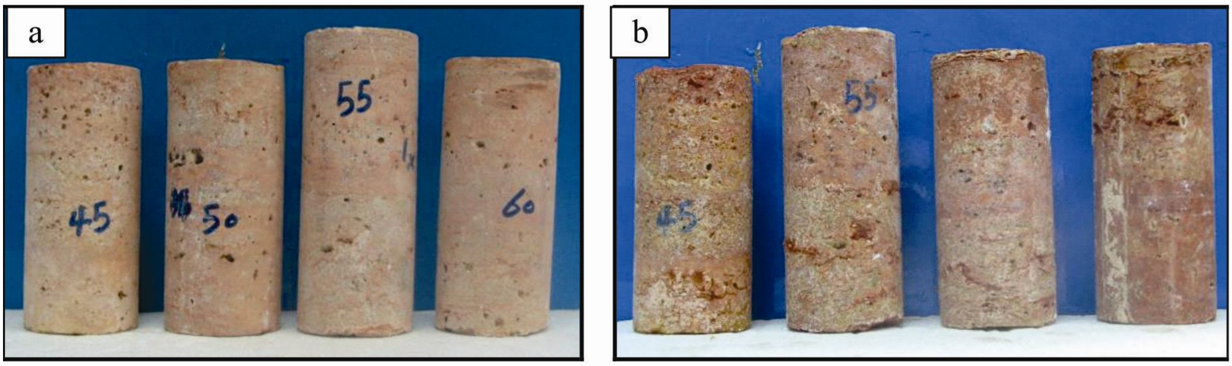

Figure 4: The change in visual appearance of Travertine-I a) before and b) after salt crystallization test

The durability of samples against salt crystallization mechanism can be connected not only to their physical properties, but also to their strength characteristic (as it is the material resistance to salt crystallization pressure which creates tensile stress over the pore surface). As can be seen in Fig. 5, Vitric tuff, Travertine-I and II have the most weight loss after the salt crystallization process. This can be the result of having a higher porosity level in comparison with the other samples (Table 3). Moreover, Marble-I and Amphibolite have minimum weight loss compared with the other samples. This shows that porosity can be the clue for estimating durability.

Fig. 5 also shows that there is no relationship between weight loss and rock type. In fact, rocks of the same origin can exhibit significantly different durability against salt crystallization. As a result, the rock type alone (at least for the samples used here) does not provide enough information regarding samples durability against salt crystallization.

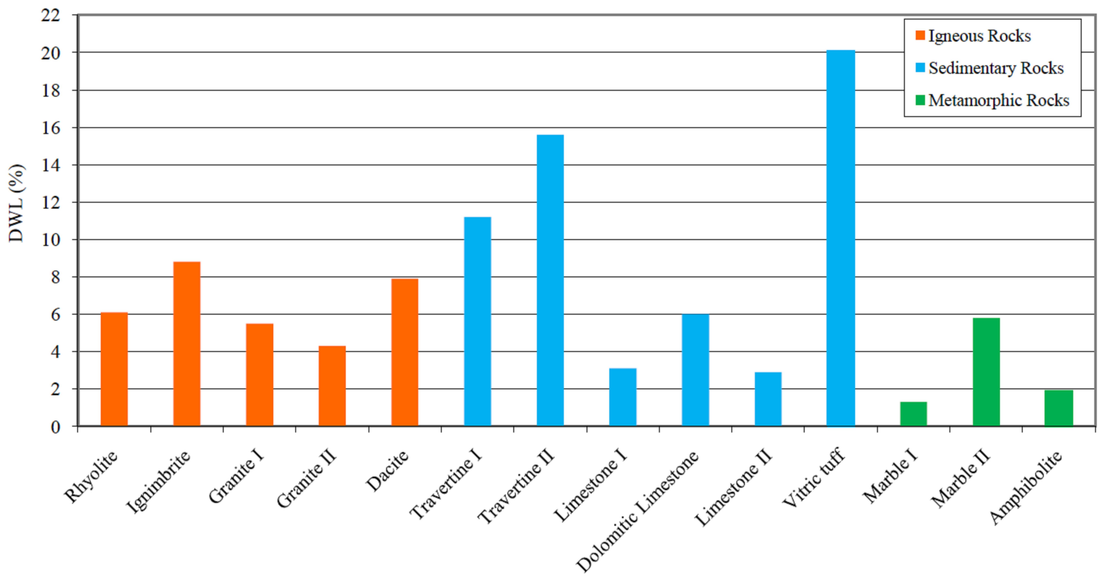

Figure 5: Dry Weight Loss (DWL) of samples by salt crystallization

Statistical analysis of the tests results

Among the most common accepted methods of investigating empirical relationships between rock properties, such as durability, physical properties, and strength characteristics, are simple and multiple regression analyses. In this study, we have used both simple and multiple regression analyses for estimating the samples durability.

Simple regression analyses

In order to investigate the relationship between DWL and physical properties and strength, linear and non-linear simple regressions were undertaken with 95% confidence level, and the determination coefficient (R^2) was calculated to find relationships. Authors attempted to develop the best correlation between different variables to achieve the most reliable empirical equation. Figs. 6–8, show the relationship between DWL and physical properties (effective porosity, total porosity, and water absorption) for tested samples. There are both a logarithmic and a linear relationship between DWL and the effective and total porosity, respectively. A moderate correlation (R^2 = 0.83/0.87) was found between DWL and effective/total porosity. Similarly, a logarithmic relationship was observed between DWL and water absorption with a determination coefficient of 0.84.

One of the most important physical properties that characterizes the stone durability against salt crystallization is porosity, particularly effective porosity. Effective porosity is a key factor in controlling the uptake and transport of weathering agents such as salt solutions within porous building materials. Therefore, building materials' durability against salt crystallization can be estimated from effective porosity, i.e.: the ratio of the volume of connected porous to the total volume of the sample. The effective porosity is closely related to total porosity and water absorption. Consequently, stone durability is related to effective porosity, total porosity, and water absorption. For example, Rhyolite samples have lower DWL (higher durability) than Vitric tuff samples. This can be due to lower physical properties (effective porosity, total porosity, and water absorption) of Rhyolite samples.

Figs. 6–8 demonstrate that although the trend of data shows an increase in DWL_ with the increase in effective porosity, total porosity, and water absorption_ due to inappropriate distribution of data, there is a poor meaningful relationship between them. In fact, this shows that, in addition to physical properties, other samples properties such as strength affect the durability against salt crystallization.

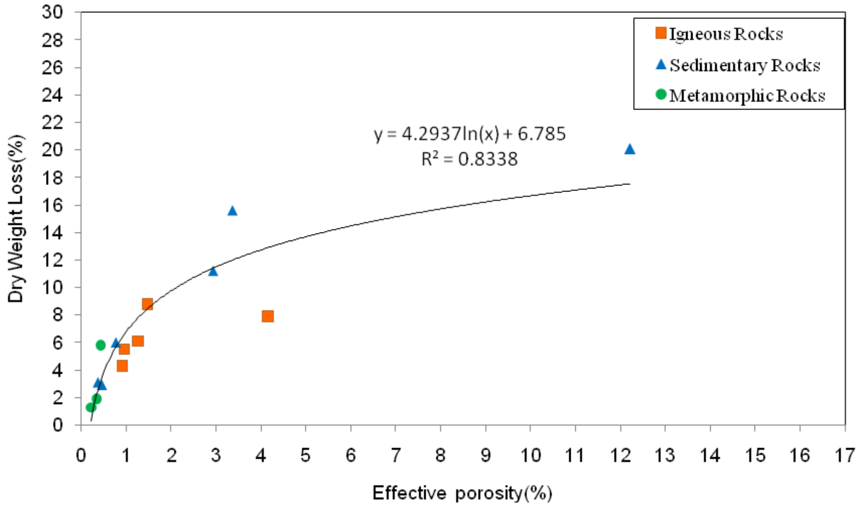

Figure 6: The relationship between Dry Weight Loss (DWL) and effective porosity

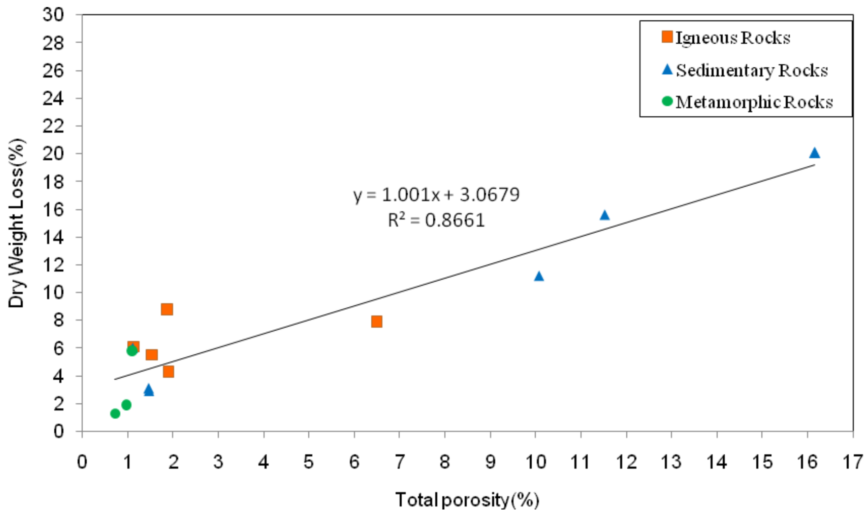

Figure 7: The relationship between Dry Weight Loss (DWL) and total porosity

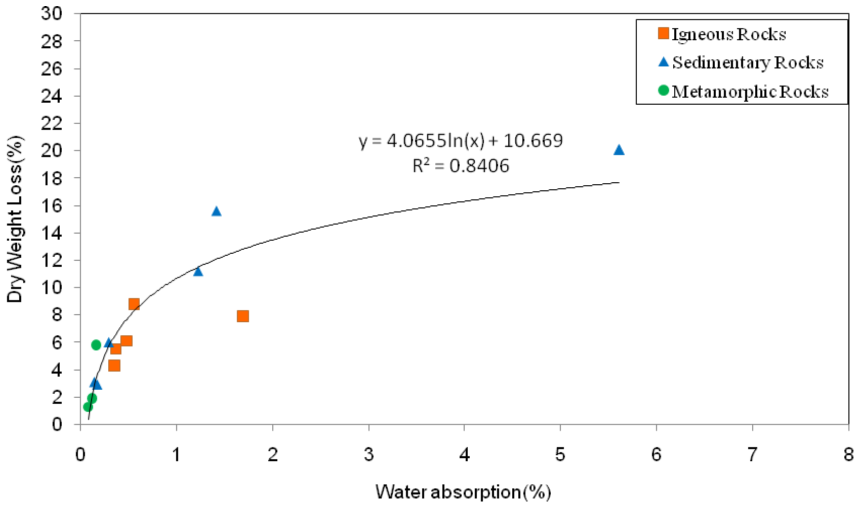

Figure 8: The relationship between Dry Weight Loss (DWL) and water absorption

Figs. 9 and 10 show the relationship between DWL and uniaxial compressive and Brazilian tensile strength. It can be seen that there are strong relationships between those with determination coefficients of 0.20 and 0.14, respectively. These determination coefficients are much lower than determination coefficients between DWL and physical properties (0.83, 0.87 and 0.84). From the analysis between DWL and uniaxial compressive and Brazilian tensile strength, it is concluded that no meaningful relationship between them can be drawn. In fact, stone durability against salt crystallization is more affected by physical properties rather than strength. For example, Vitric tuff has a very high strength level (UCS equal to 101.5 MPa); however, because of its high porosity, it shows low durability against salt crystallization. Table 5 summarizes the results of simple regression analysis.

Table 5: Summarized the results of simple regression analyses

Regression equations	Determination coefficient (R^2)
$DWL = 4.2937\ln(P_{Ef}) + 6.785$	0.83
$DWL = 1.001P_T + 3.0679$	0.87
$DWL = 4.0655\ln(W_a) + 10.669$	0.84
$DWL = 263.46UCS^{-0.867}$	0.20
$DWL = 23.255BTS^{-0.605}$	0.14

Dry Weight Loss, DWL; Effective Porosity, P_{Ef}; Total Porosity, P_T; Water absorption, W_a; Uniaxial compressive strength, UCS; Brazilian tensile strength, BTS

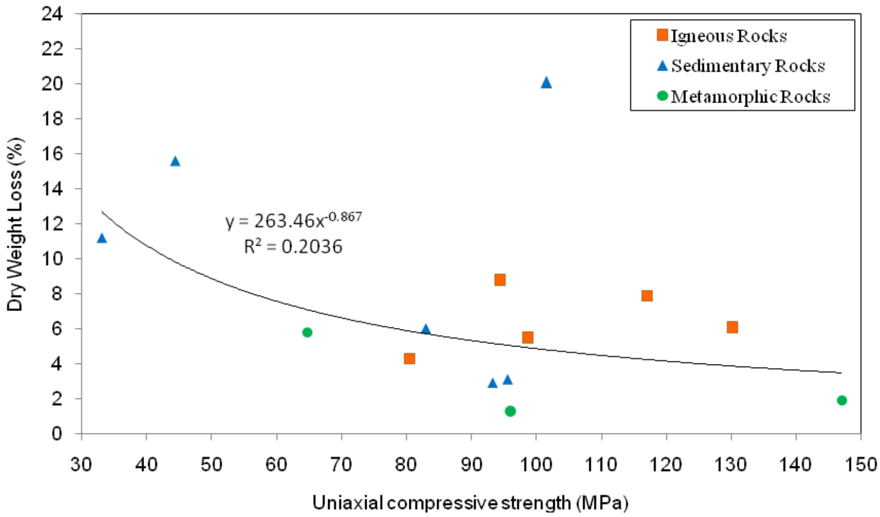

Figure 9: The relationship between Dry Weight Loss (DWL) and uniaxial compressive strength

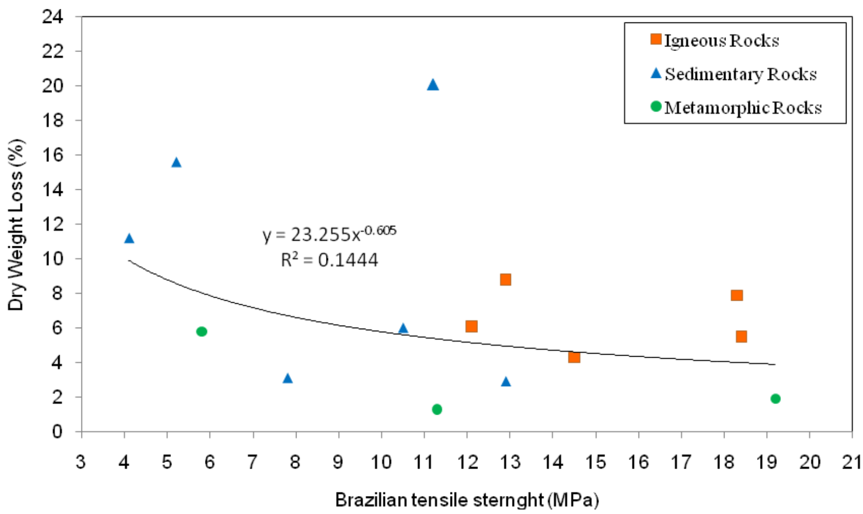

Figure 10: The relationship between Dry Weight Loss (DWL) and Brazilian tensile strength

Multiple regression analyses

Multiple regression analyses were used for estimating stone durability against salt crystallization. In these analyses, DWL, after salt crystallization, was considered to be the dependent variable, which was dependent on physical properties and strength. The best-fit curves were determined using the least square method, and the fitting quality was investigated by multiple determination coefficients (R^2) and the plots of measured DWL values versus estimated samples. The equation of the best-fit line, 95% confidence level and the determination coefficient (R^2), were determined for each equation. The general equation for estimating the DWL after salt crystallization is shown as follows:

$$DWL=\alpha_0+ \alpha_1 P+ \alpha_2 S \quad (1)$$

Where *DWL* is the Dry Weight Loss estimated value for samples after salt crystallization, *P* is physical properties (effective porosity, total porosity, and water absorption), *S* is the strength of samples (uniaxial compressive strength and Brazilian tensile strength), α_0 is a constant, α_1 and α_2 are the regression coefficients of *P* and *S*, respectively. The method of least- squares, which minimizes the sum of squared deviations between the fitted and measured data, estimates the coefficients, Using the data for physical properties and strength, the authors have illustrated DWL in Tables 3 and 4. The best correlations between the variables, generally achieved using a best-fit surface curve, are given in Table 6.

Table 6: Proposed models for estimating DWL after salt crystallization from Eq (1)

Regression equations	Standard error of estimate (S)	Tabulated F-ratio	F-ratio	Determination coefficient (R^2)
$DWL=10.122+1.398\ P_{Ef}-0.065UCS$	2.42	3.98	26.097	0.826
$DWL=4.690+0.930\ P_T-0.015\ UCS$	2.62	3.98	21.493	0.796
$DWL=10.426+3.022\ W_a-0.065\ UCS$	2.47	3.98	24.865	0.819
$DWL=8.190+1.368\ P_{Ef}-0.335\ BTS$	2.70	3.98	19.824	0.783
$DWL=4.238+0.935\ P_T-0.081\ BTS$	2.63	3.98	21.270	0.795
$DWL=8.413+2.950\ W_a-0.333\ BTS$	2.77	3.98	18.609	0.772

Dry Weight Loss, DWL; Effective Porosity, P_{Ef}; Total Porosity, P_T; Water absorption, W_a; Uniaxial compressive strength, UCS; Brazilian tensile strength, BTS

The degree of fitness to a curve can be measured by the value of the determination coefficient (R^2), which measures the proportion of variation in the dependent variable can be explained by the variation of independent variable, and the standard error of the estimate, S, which is an important measure for shows how close the actual data points fall to the estimated values on the regression curve, are given in Table 6. Values of R^2 show that the proposed models fit the data well and are capable of estimating the DWL of samples. To test the global usefulness of the proposed models, analysis of variance for the regression was performed. For this purpose, the F statistics test was performed to test the global usefulness of the model. This test is widely used in regression and analysis of variance. The null hypothesis for this test is $H_0 : \alpha_1 = \alpha_2 = 0$, against the alternative hypothesis H_1: at least one of α_1, α_2 is not equal to zero. F statistics test follows an F-distribution with the numerator degree of freedom 2 and with the denominator degrees of freedom 11, is 3.98. In this test, a 95% level of confidence was chosen. The calculated F-ratio of the models is given in Table 5. Since all the computed F-ratios of the models in all cases are greater than F-ratios tabulated, the null hypothesis is rejected. It is concluded that there is a clear relationship between DWL and physical properties and strength; therefore, the models are appropriate for estimating the DWL caused by salt crystallization.

The statistical models proposed in this study were evaluated by comparing their results with each other. The estimated values of the DWLs were then plotted against the measured values for all samples using 1:1 slope line (Figs. 11 and 12). A point lying on the line shows an exact estimation. The figures indicate that the data points fall close to the 1:1 slope line and are scattered uniformly around it; suggesting that models with the assumed coefficients are appropriate to estimate the DWL using physical properties and strength.

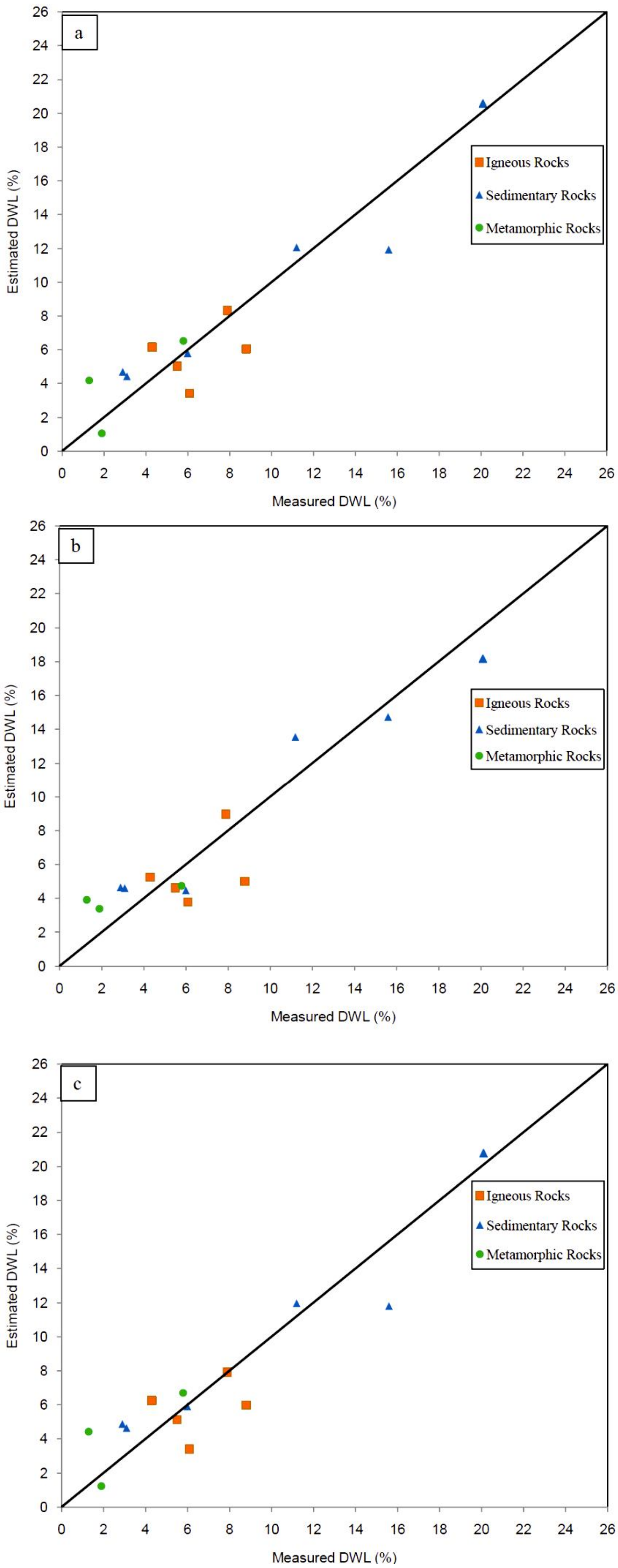

Figure 11: Measured DWL versus Estimated DWL from Eq (1) using **(a)** P_{Ef} and UCS **(b)** P_T and UCS **(c)** Wa and UCS

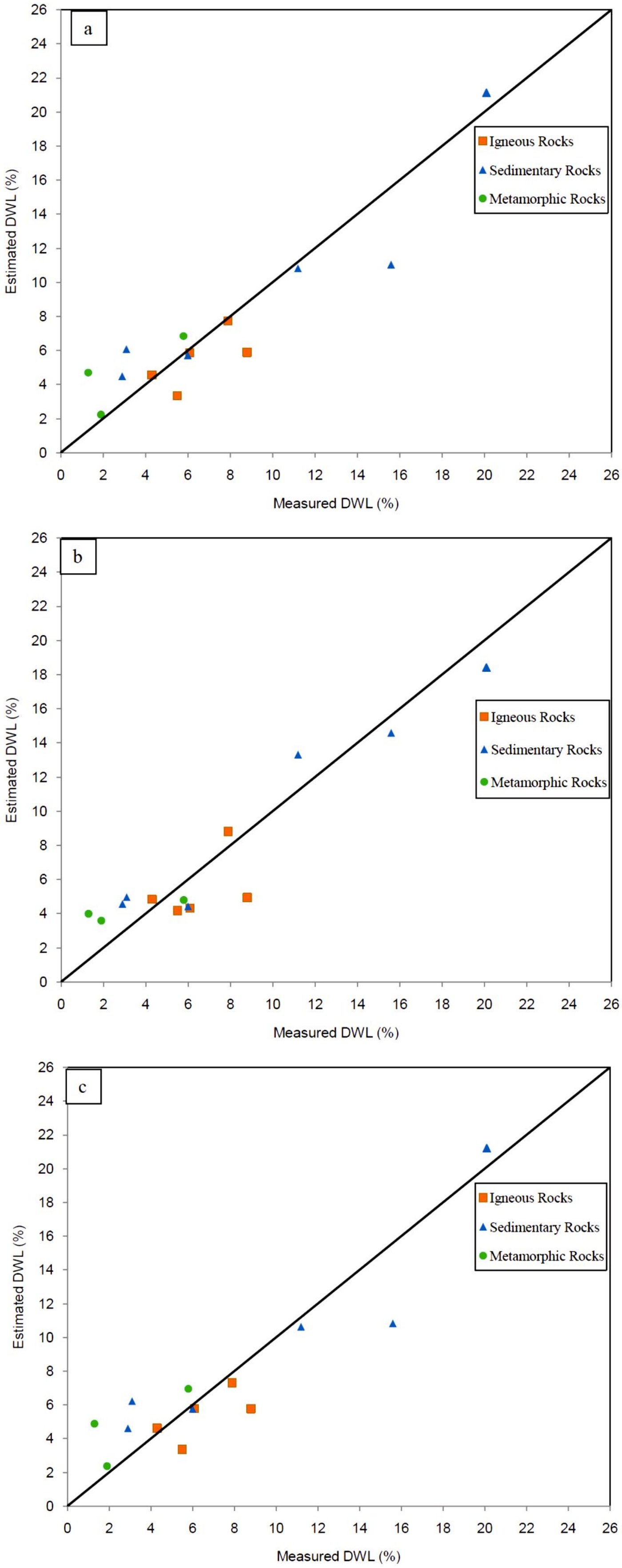

Figure 12: Measured DWL versus Estimated DWL from Eq (1) using **(a)** P_{Ef} and BTS **(b)** P_T and BTS **(c)** Wa and BTS

Conclusions

In this study, laboratory testing was carried out on 14 samples of building stones to investigate the possible relationships between stone durability and salt crystallization using both the physical properties and strength. Our study shows rock type, physical properties, and strength alone (at least for the samples used here) cannot provide enough information regarding durability of salt crystallization, although there is a moderate correlation between stone durability and physical properties. However, there is a high correlation between stones durability and physical properties and strength when both of them are considered. These results demonstrate the importance of both physical properties and strength in the stone's durability against salt crystallization. The statistical models for estimating the stone's durability against salt crystallization using both the physical properties and strength were proposed. These models were developed by multiple regression analysis and then statistically checked. The application of the models is that the physical properties and strength measurement of the stone can be used for estimating its durability. Consequently, these models help the researchers to save time. In addition, the models provide significant advantages for a rapid stone durability assessment.

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