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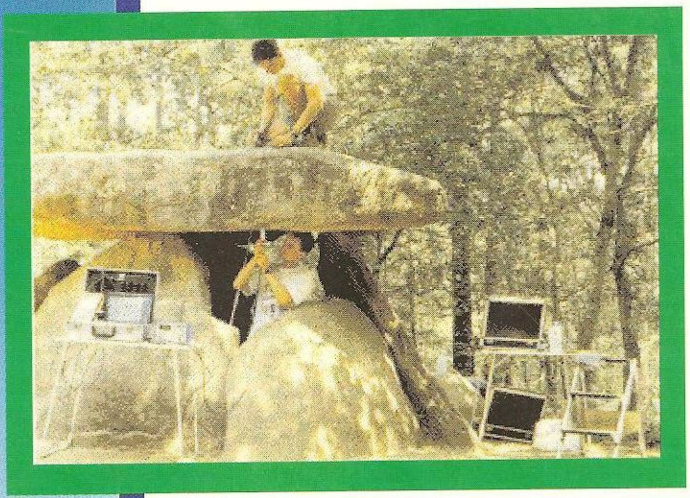

Degradation and conservation of granitic rocks in monuments

Protection and conservation of
European cultural heritage

Research report No 5

INFLUENCE OF CONSOLIDATION AND HYDROFUGATION TREATMENTS ON THE PHYSICAL PROPERTIES OF AVILA NATURAL AND ARTIFICIALLY AGED GRANITE; A STATISTICAL APPROACH.

Iñigo, A. C.*, García-Talegón, J.*, Trujillano, R.*, Vicente-Tavera, S.**.

*Instituto de Recursos Naturales y Agrobiología, IRNA-CSIC, Salamanca, Spain.
**Dpto. Estadística y Matematica Aplicadas, Universidad de Salamanca, Spain.

ABSTRACT

The variation in the petrophysical properties of five varieties of granitic materials when artificially aged and/or submited to different treatments of consolidation and hydrofugation, has been studied.

The statistical study informs about the influence of the characteristics of the rocks and treatments on the properties studied. Those varying most with the treatment were: % imbibition, coefficient of absorption, capillarity and free porosity.

Combined treatment with consolidant plus hydrofugant was much more effective than when these were used alone.

INTRODUCTION

Prior knowledge of the behaviour of stone materials in a given environment is crucial for predicting future problems arising in evolution on the stone since the behaviour of a particular material depends on its composition (chemical, mineralogical, petrophysical, etc.) and on the environmental characteristics of the site in which it is employed.

The aim of the present work was to gain insight into the behaviour of granitic (*sensu lato*) rocks in a continental environment with a low degree of atmospheric pollution. To do so, an experiment was designed with five types of granites with highly differentiated characteristics. These were subjected to climatic conditions reproducing the strong contrasts of continental climate and assaying consolidation and/or hydrofugation treatments in order to determine the degree and type of alteration undergone by the different granitic facies in each case and the effect of the treatments on a series of petrophysical characteristics that are fundamental to the behaviour of these materials.

MATERIALS AND METHODS

Five varieties of natural granite, ranging from sound natural facies, with a low degree of porosity and high resistance to decay, to weathered facies with high porosity and containing strongly reactive components (swelling clay, smectite) in their composition were used: fine grain grey granite (M1), coarse grain grey granite (M2), ochre granite (M3), red granite (M4) and white granite (M5).

The products assayed were: RC70, ethyl silicate (consolidant); RC80, ethyl silicate with methyl resin (consolidant) and a polysiloxane resin (hydrofugant); H224, an alkylpolysiloxane oligomer (hydrofugant). All products were supplied by Rhône-Poulenc.

A summary of the most important petrophysical characteristics affecting the behaviour of the samples is given in the table 1.

A more detailed reference can be found in [Iñigo et al., 1994].

Sample	Nt %	N48 %	S48	DA (g/cm^3)	DR (g/cm^3)	A (g/cm^2x $^+h^{1/2}$)	K_1 (Kg/m^2 S) x10^{-7}
Fine Grey Granite	1.12 ± 0.11	1.02 ± 0.08	0.91 ± 0.05	2.60 ± 0.02	2.63 ± 0.01	n.m.	2.3 ± 0.5
Coarse Grey Granite	0.66 ± 0.10	0.62 ± 0.06	0.95 ± 0.04	2.66 ± 0.01	2.68 ± 0.01	n.m.	2.4 ± 0.5
Ochre Granite	28.40 ± 1.75	21.02 ± 1.22	0.74 ± 0.05	1.77 ± 0.03	2.47 ± 0.02	0.08 ± 0.01	47.6 ± 7.3
Red Granite	20.13 ± 1.43	13.49 ± 0.90	0.67 ± 0.05	1.98 ± 0.04	2.48 ± 0.02	0.05 ± 0.01	25.6 ± 4.6
White Granite	21.04 ± 0.97	12.69 ± 0.72	0.60 ± 0.05	1.87 ± 0.02	2.37 ± 0.02	0.04 ± 0.01	35.6 ± 6.7

Nt total porosity to water
S48 Hischwald coefficient (N48/Nt)
DR Real density
K_1 Water vapour permeability
N48 porosity at 48 hours
DA Apparent density
A Weight of water absorption by surface unit and time $^{1/2}$
n.m. not measurable under the experimental conditions

Table 1: *Petrophysical characteristics of the Avila granites.*

The procedure followed in the experiments was: Proofs (5 x 5 x 5cm) of different types of stone were subjected to 25 cycles of cold/heat and freezing/thawing, from -20°C to 110°C, in a simulation chamber following the recommendations of Tiano and Pecchioni (1990) with a view, first to determining the degree of weathering achieved and, second, preparing artificially aged material for the experiments involving consolidant and/or hydrofugant treatments.

The assays with the different treatments were performed on proofs of natural stone and on previously aged samples.

The intensity of the weathering process and the efficiency of the treatments (hydrofugation and/or consolidation) was evaluated by monitoring a series of properties of stone from quarries, stone after aging and treated stone. These were: weight loss during aging, weight gain during treatments, % of imbibition, free porosity, total porosity, capillarity, apparent density, real density and the absorption coefficient.

The consolidation treatments were conducted following the method of impregnation according to the NORMAL 20/85-specified protocol, slightly modified, in order to facilitate penetration of the product into the stone. Treatment was performed in three steps: 1st, 8 hours with a solution of the commercial product in solvent (white spirit) at 5%; 2nd, 24 hours with a 40% solution and 3rd, 40 hours with a 75% solution.

Hydrofugation treatment was applied to the surface of the stone with a brush in two successive steps at 24 h intervals [Esbert et al., 1991].

In all experiments, the number of proofs was calculated previously according to a theoretical design in order to obtain statistically significant results.

RESULTS

The results were analyzed according to a one-way (ANOVA) factorial design for aging and a three-way design with their interactions for the treatment.

1) Assays on artificial ageing

The aging intensity reached was determined by weight loss in the proofs assayed. For statistical analysis, only one variation factor was considered (type of stone).

Figure 1 shows the different types of behaviour of the M1 and M2 samples -sound facies- whose weight losses are minimum as compared with those shown by the M3, M4 and M5 naturally weathered in the quarry. This difference in behaviour was striking in nearly all the properties examined later, such that although the analyses were done for 5 types of stone, subsequent contrasts were made with the M3, M4 and M5 samples.

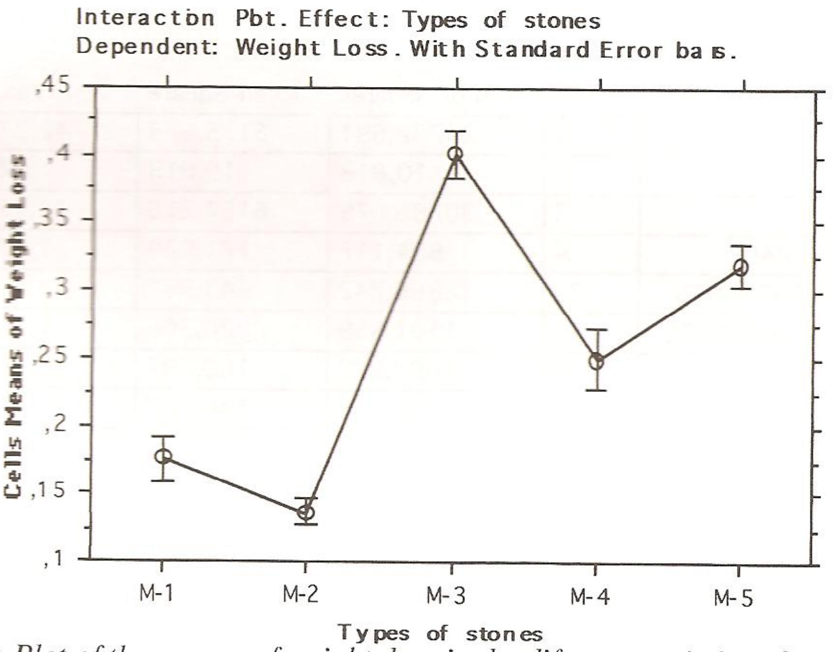

Fig. 1: *Plot of the means of weight loss in the diferent varieties of granites.*

Table 2 compares the variations in weight of the different granite facies used during artificial ageing of the stone ($p<0.01$).

Type III Sums of Squares

Source	df	Sum of Squares	Mean Square	F-Value	P-Value
Types of stones	4	,77671	,19418	42,08350	,0001
Residual	80	,36913	,00461		

Dependent: weight loss

Table 2: *One way ANOVA (types of stones)* .

2) Treatment with consolidants and/or hydrofugants.

In the proofs from quarries, aged and subjected to different treatments, the above-described properties were studied to determine the efficiency of the treatments assayed

2-1 Weight gain: On analyzing the variations in this parameter no significance was found in the effect of the hydrofugants, which elicited a very small weight gain. However, the consolidant plus hydrofugant combination had an appreciable effect. On comparing the effects of the consolidants with those of consolidant plus hydrofugant, the differences were found to be significant ($p<0.01$).

2-2 Coefficient of absorption, % (FN, 1973, B10-503): Since the values of this variable do not correspond to a direct petrophysical parameter but rather reflect the relationship between two variables (free porosity/total porosity), variability is difficult to monitor and interpret.

Table 3 shows a dual interaction between the treatments and types a stone. Once analyzed, an effect of H224 was found ($p<0.01$). The overall effect of consolidated and untreated samples *versus* samples treated with consolidants plus hydrofugant gave significant variations ($p<0.01$).

Type III Sums of Squares

Source	df	Sum of Squares	Mean Square	F-Value	P-Value
STONES	4	24702,691	6175,673	42,276	,0001
DEGRAD.	1	10,813	10,813	,074	,7864
TREATMENTS	5	30786,079	6157,216	42,150	,0001
STONES * DEGRAD.	4	684,117	171,029	1,171	,3319
STONES * TREATMENTS	20	12866,742	643,337	4,404	,0001
DEGRAD. * TREATMENTS	5	1101,539	220,308	1,508	,1995
STONES*DEGRAD.*TREAT.	10	1007,972	100,797	,690	,7299
Residual	65	9495,178	146,080		

Dependent: C. ABS.

Table 3: *Three way ANOVA (factorial design).*

2-3 Real density, $g \cdot cm^{-3}$ (FN, 1973, B10-503): The overall analysis afforded significance in the three types of interaction (sample, aging and treatment). In analysis of these interactions separately, significance in the effect of H224 in the natural samples was observed. The aging process induced significant changes in this variable ($p<0.05$).

2-4 Imbibition, % (NORMAL, 7/1981): In the analysis of the variations occurring in this variable the following were found: an effect of treatment ($p<0.01$), producing a triple interaction. Separately within this triple interaction, treatments with RC80, RC80 + H224 and RC70 + H224 were seen to have a pronounced effect on the percentage of imbibition (Fig. 2). When the effects of RC80 were compared with those of RC70, differences were found ($p<0.01$). The plot (Fig. 2) reveals the more effective behaviour of RC80 with respect to RC70. The effect of H224 was also significant ($p<0.01$).

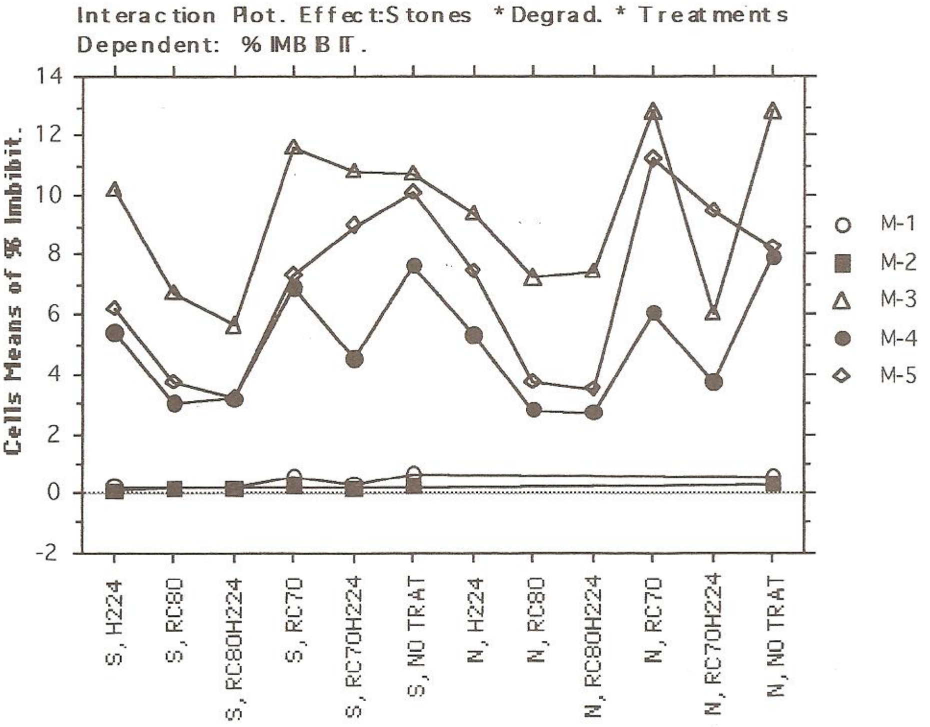

Fig. 2: *Interaction plot for % of Inbibition.*

2-5 Capillarity, g · cm^{-2} · S$^{-1/2}$ (NORMAL, 11/1985): Analysis of the variations in this variable reveals a clear effect of the treatment applied (Fig. 3). The effect of H224 was significant as compared with the untreated samples ($p<0.01$). On comparing the aged samples, significant differences were found between the proofs tested with RC70 and RC80 and those treated with these compounds plus H224 ($p<0.05$).

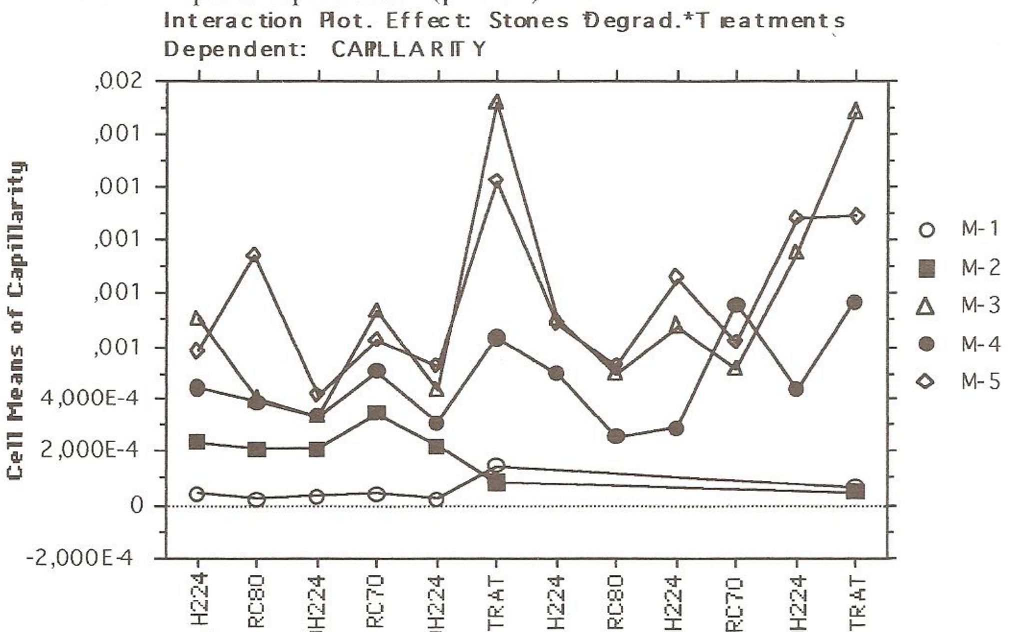

Fig. 3: *Interaction plot for Capillarity.*

2-6 Free porosity, %: In the analysis of the variations in this parameter a sample-treatment interaction was detected (Fig. 4). The effect of the samples treated with H224 compared with untreated samples was significant.Likewise, the effect produced by RC70 as compared with RC80 was highly significant ($P<0.01$).

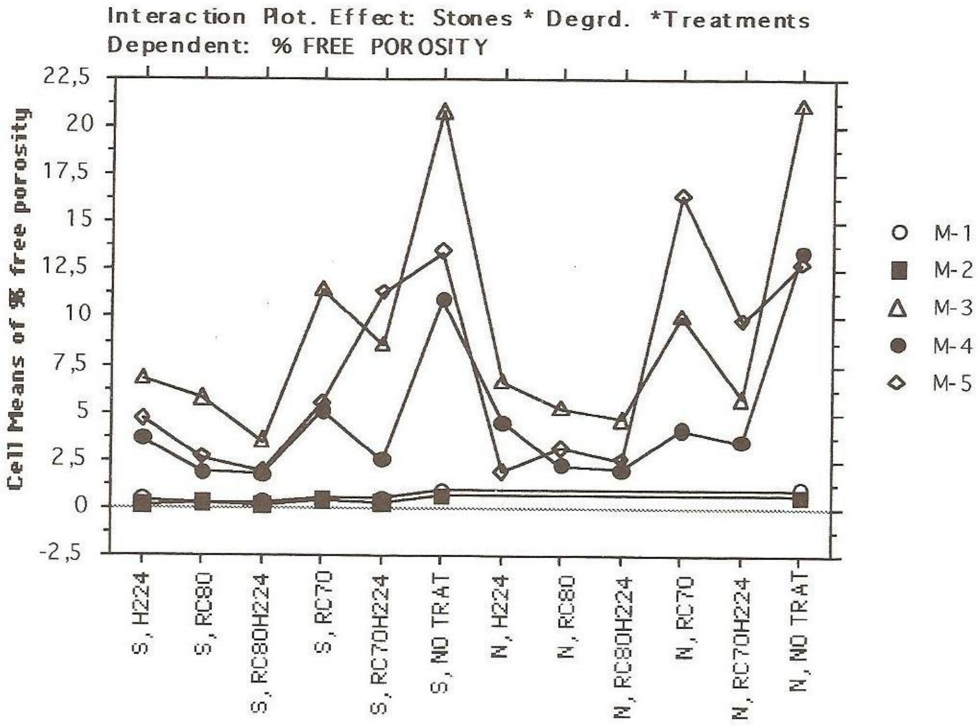

Fig. 4: *Interaction plot for % of Free Porosity.*

2-7 Total porosity, % (FN, 1973, B10-503): In this property, variations between aged and non-aged samples were found ($p<0.05$).

2-8 Apparent density, g · cm^{-3} (FN, 1973, B10-503): Little variability was found on analyzing this variable, with only small variations due to the effect of treatment.

CONCLUSIONS

From the above analyses of the different results obtained, the following conclusions can be drawn:

1) The intrinsic characteristics of the stone material (samples M1 and M2 compared with the rest) affect their behaviour crucially both as regards decay and as regards their response to treatment. It is essential to study the response of each type of material to the different treatments since it is difficult to extrapolate such information because it depends on the physico-chemical and petrophysical characteristics of each one.

2) The most variable properties of the materials with the treatment are: % imbibition, coefficient of absorption, capillarity and free porosity.

3) The combined action of consolidants plus hydrofugant is high effective with respect to the properties assayed. However, alone, both consolidant and hydrofugant have little effect on the petrophysical properties of the stone.

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