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Evaluation of effectiveness of silicoorganic treatments using hydric properties

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Abstract

In this work the effectiveness of silicoorganic treatments for the consolidation, with or without waterproofing, of silicified stones (one conglomerate and four sandstones) from Zamora is analyzed, provided that these treatments are applied in the same way. Changes in the following properties, have been monitored: total and free porosity, real and apparent density, absorption coefficient, imbibition coefficient and capillary absorption coefficient. These intrinsic stone properties and the treatments employed have a significant effect on changes in these variables and lead to differences in the transport of fluid in the stone. This is the first time that the Canonical Biplot has been applied to this type of data to determine the control of the effectiveness of silicoorganic treatments applied to rocks. It was observed that the double action treatment RC80, which includes both waterproofing and consolidating, is more

Keywords: Silicified stones, silicoorganic treatments, porosity, hydric properties, Canonical Biplot.

Introduction

The effectiveness of consolidation treatments with or without waterproofing can be measured by analyzing, prior to and after their application, different hydric properties of stone materials such as total and free porosity, real and apparent density, absorption coefficient, imbibition coefficient and capillary absorption coefficient. These hydric properties indicate the extent to which fluids can circulate throughout the pore network and are intrinsic to each material. As a result, they influence the introduction of conservation products into stone.

The porosity of stone material, either macro or micro, determines which of the hydric properties are important. Previous studies regarding this (Iñigo et al., 2001), using Anova (Analysis of Variance, a classical statistical method for analyzing possible comparisons in one variable), show that free porosity and the capillary absorption coefficient are the properties that vary the most for silicified granites from Avila (granites that have undergone paleoalterations and which have given rise to silicified materials). Later on, Iñigo et al. (2006) show, using these silicified granites from Avila, that the imbibition coefficient could also be used for determining the effectiveness of conservation products. In order to evaluate the effectiveness of consolidation with or without waterproofing treatments, and to verify their suitability, the hydric properties and color changes on the surface of different types of materials (bricks, stone, etc.) before and after applying several treatments were analyzed. These types of analyses have also been carried out by several other authors (Hammecker et al., 1992; Valdeon et al., 1992; Vicente, 1996; Villegas-Sánchez and Espinosa-Gaitán, 2001; García-Garmilla et al., 2002; Berardi et al., 2002; Espinosa-Gaitan et al., 2002; Quaresima et al., 2002; Alcalde-Moreno et al., 2003; Buj and Gisbert, 2006; Luque et al., 2008; Torrisi, 2008; Alejandre and Villegas, 2009; García et al., 2010; Rivas et al., 2011; Galvana et al., 2014; García-Talegón et al., 2015, 2016a; Arezki et al., 2016; Costa et al., 2017). In addition, Martín et al. (2002) designed an instrument that allows a wide range of temperatures to be maintained and controlled automatically using a computer. This instrument is used to automatically acquire data without interrupting the process (water absorption by capillarity and by total immersion, sorption and desorption of water and water vapor permeability) and is essential because some of the previously mentioned properties are temperature dependent. This instrument allows these properties to be measured at actual temperatures; that is, temperatures to which the stones of monuments are exposed.

Porosity, besides being considered a hydric property, can be calculated using other methods such as the adsorption of nitrogen (it determines the microporosity of the material) and mercury intrusion. These methods can be used to determine the effectiveness of conservation treatments by calculating the porosity before and after their application. Our research group has carried out work in this field (Iñigo et al., 2007) and determined that RC70 was the most efficient product over RC80 for decreasing porosity. This is because the surface of the samples treated with RC80 does not allow real porosity to be determined. For this reason, it is necessary to determine values for more than one property using several techniques, allowing the most effective treatment to be identified.

Total porosity of a material is the sum of "trapped" porosity (pores that are not connected to the external surface and fluids are unable to circulate freely), and "free" porosity (pores are connected to the external surface and easily accessed by fluids). Total porosity thus controls fluid transport through the stone at ambient temperature and pressure (Meng, 1992; Brandes and Stadlbauer, 1992).

The penetration of liquids or other fluids inside the pore network of a solid material can be evaluated using the capillary absorption coefficient (CAC) (Meng, 1992; Cordova and Gomez, 1997).

In the present work we report on the effect of consolidation treatments, with or without waterproofing, on hydric properties (total and free porosity, real and apparent density, absorption coefficient, imbibition coefficient and capillary absorption coefficient) when non-weathered stones are submitted to different silicoorganic conservation treatments. The use of the Canonical Biplot allowed us to establish possible variations among different types of stone materials depending on the treatment applied.

Materials and Methods

One white siliceous conglomerate (Z1) and four siliceous sandstones facies (TG: white sandstone with large-sized grains, TB: white sandstone, TO: ochreous sandstone and TR: red sandstone) with different porosity values and containing strongly reactive components (swelling clays, and opal that dissolves in basic solutions, iron oxyhydroxides, which can come to the surface of the stone through dissolution and dragging due to freeze/thawing processes, changing the characteristics of the materials such as the surface color, etc.) were used in this study. Their mineralogical origin, chemical and petrophysical properties have been described elsewhere (Añorbe-Urmeneta, 1997). All materials are widely used in construction and are currently being used in the conservation intervention of the Cathedral and other buildings in Zamora (Spain).

The untreated stones and their corresponding treated counterparts are known to be affected by the specific climate of the area, which is Mediterranean, tending towards being continental, and the different microclimates inside buildings (Iñigo et al., 2000; Iñigo and Vicente-Tavera, 2002). In this climate there are daily abrupt changes in temperature which can lead to fissures. In addition, processes of freezing/thawing giving rise to fissures, scales, etc. and processes of haloclasty due to the effects of atmospheric and underground and/or sub-surface water pollution, give rise to efflorescence and crusts.

The following commercial conservation products were tested: RC70 (ethyl silicate) as the consolidant and RC80 (a mixture of ethyl silicate, a methyl resin, and polysiloxane) as the consolidant plus water repellent. All products contain a catalyst, which favors polymerization, and were provided by Rhodia Siliconas Spain, Ltd.

The treatments were carried out as follows: both consolidation treatments (RC70 and RC80) followed the recommendations by Iñigo et al. (2001, 2006), which promotes the penetration of products inside stone samples (5 cubic samples measuring 5x5x5 cm in each group). The treatment was carried out in four consecutive steps, one immediately after the other: 1) immersion of the stone samples in white spirit (also used as a solvent for the conservation products) for 30 min; 2) immersion in a white spirit solution + conservation product (5% w/w) for 8 hours; 3) immersion in a white spirit solution + conservation product (40% w/w) for 24 hours and 4) immersion in a white spirit solution + conservation product (75% w/w) for 48 hours. Curing extended for one month at room temperature and ambient laboratory conditions.

The following hydric properties were analyzed: total porosity (NF, 1973a) and free porosity (NF, 1973a, 1973b), absorption coefficient (NF, 1973a, 1973b), real and apparent density (NF, 1973a), imbibition coefficient (NORMAL, 1981) and capillary absorption coefficient (NORMAL, 1986).

The Canonical Biplot was performed to analyze variation between the five different untreated stone varieties and their corresponding treated samples.

The Canonical Biplot is complementary to the MANOVA analysis (Multivariate Analysis of Variance), but includes all of the characteristics of the Biplot method (Gabriel, 1971, 1972, 1995), and can be used to discriminate the set of groups of previously established populations. This technique was later developed and completed (Galindo, 1986; Amaro et al., 2004; Vicente-Villardón, 2018), and applied to the field of Cultural Heritage conservation (Iñigo et al., 2014, 2017, 2019; García-Talegón et al., 2016b).

The results are summarized on several factorial planes, where the variables are represented as vectors that start out from a hypothetical origin and the means of the different groups as stars surrounded by confidence circles in the same reference system. If two variables are represented with a very small angle, then the variables are highly correlated, and if they are opposite their correlation is inverse. Additionally, if the angle is close to perpendicularity, their correlation is minimum. When projecting all of the star markers perpendicularly onto the directions of any of the variables, the order of the projections in the direction of those variables is equivalent to the value of the population means for that variable. If two confidence circles are projected perpendicularly on one of the variables and the intervals of both projections do not overlap, this is tantamount to saying that there are differences between both means (Student's t-test); the amplitudes of the circles will depend on the significance, α, determined (MSD, Bonferroni corrections, etc.). These interpretations are subject to a series of measurements of the quality of representation for the different planes (inertia absorption of the planes, the goodness of the projections of the measurements on the variables for the dimensions selected, etc.).

The Canonical Biplot is a comprehensive technique that analyzes all variables together, minimizing Type I risk, while the classic individual ANOVAS are used for each variable. Likewise, the Biplot representation allows us to easily visualize all the possible differences between the different groupings for all of the variables analyzed

In our case, there are 8 variables (the previously mentioned hydric properties) and the nomenclature used was YZ, where Y indicates the type of rock (Z1, TG, TB, TO, and TR) and Z the type of treatment (untreated, RC70 and RC80). Thus, Z1RC80 corresponds to rock Z1 treated with RC80.

Results and Discussion

The variation in weight of the treated samples with respect to the untreated samples provides an approximation of the amount of product introduced through the pore network of the stones. The weight gain observed in the varieties treated with RC70 varied between 0.48 - 1.10% and between 0.90 - 1.11% in the samples treated with RC80, when using the original weight of the corresponding untreated rocks as a reference.

Table 1 shows the means and standard error of the hydric properties for each treatment and stone variety.

After applying the multivariate Canonical Biplot technique, we obtained a Wilk's lambda of 34.0991 ($p<0.001$), which can be interpreted as though there were differences among the means of the joint measurements of the populations (groups) for all variables. Regarding the absorption of inertia, the first three axes absorbed 92.913% of the total inertia.

Although porosity was not low (≈11%), no changes were detected in any of the hydric properties studied using the different silicoorganic treatments (Table 1 and Figs 1 and 2) on the white silicified conglomerates Z1, because when analyzing the graphs the projections of the confidence circles of the groupings (Z1-Z1RC70 and Z1-Z1RC80) overlap. This indicated that these types of properties were not suitable for determining the effectiveness of the various treatments applied. However, in the silicified sandstones of Zamora, which have greater porosity, the following variations were observed:

Total Porosity (TP)

In Fig 1, differences were detected only between the treated and untreated samples of varieties TR and TO.

Free Porosity (FP), Absorption Coefficient (AC), Real Density (RD)

In Fig 1, differences were observed between the treated and untreated stones and between the samples treated with RC70 and RC80. The values obtained for the samples treated with RC80 were lower than the values obtained for those treated with RC70. This indicated that the consolidant RC80 decreases free porosity (FP), the absorption coefficient (AC) and real density (RD) more than RC70, as it substantially reduces the circulation of fluids through the pore network. This may have occurred due to the fact that RC80 contains polysiloxane derivatives that are hydrophobic in nature.

Apparent Density (AD)

In Fig 1, differences were observed between the treated and untreated samples of TR, and between the RC70 and RC80 treatments. In Fig 2, differences were only detected in varieties TB and TO among the samples treated with RC80 and the untreated samples.

Imbibition Coefficient (IC)

In Fig 1, differences were observed between the treated and the untreated samples of all stone varieties studied. However, no differences were detected among the samples treated with RC70 and RC80, except for TR.

Capillary Absorption Coefficient (CAC)

This variable behaved the same as the variables of Free Porosity (FP), the Absorption coefficient (AC) and real density (RD); although no differences were detected for TB treated with RC70 and RC80 as shown in Fig 1. Again this could be due to the hydrophobic nature of the polysiloxane derivatives.

In view of the Canonical Biplot results, the most determinant hydric properties capable of distinguishing between high and low efficiency of the different treatments were the capillary absorption coefficient, the absorption coefficient, real density and free porosity. These properties presented greater variations with respect to the different stone substrates and the different conservation treatments applied. In addition, the imbibition coefficient was also able to distinguish between the effectiveness of the treatments, although to a lesser extent because there were fewer variations. As was observed, these hydric properties are directly related to the circulation of fluids inside the pore network and are intrinsic properties of the stones both before and after treatment.

In summary, we can affirm that the most effective treatment for these types of stone varieties, under the conditions applied, is the consolidant plus water repellent, RC80, due to its double action (consolidant + water repellant), as shown in Fig 1. This treatment decreases the hydric properties related to the transport of fluids, such as porosity, absorption coefficient and coefficient of capillary absorption, more than treatment RC70, and at the same time increases real density. However, RC70 can be used as a preconsolidant due to its density and the fact that its viscosity is lower than that of RC80.

In this work, additional hydric properties have been identified, capable of confirming the effectiveness of the treatments used in this study on the various types of stone, that had not been previously identified in other studies using different types of stones (silicified granites) (Iñigo et al., 2001, 2006). It can be said that the response of a stone material to a particular treatment depends on its intrinsic characteristics. Therefore, it is advisable to study different types of treatments on each stone substrate.

Conclusions

The results of direct observation and the statistical analysis have allowed the following conclusions to be drawn:

1) The Canonical Biplot has been successfully applied in this research field as a novel and alternative technique to classical individual ANOVA techniques.
2) In the case of white silicified conglomerates from Zamora, Z1, the hydric properties analyzed in this work cannot be used to evaluate the effectiveness of the two silicoorganic treatments tested, due to the lack of variability after their application.
3) The effectiveness of the treatments applied to the stone substrates can be determined by measuring the following properties: open porosity, absorption coefficient, real density and the capillary absorption coefficient. In addition, the imbibition coefficient can also be used to evaluate treatment efficacy, but with less reliability.
4) The variations observed for these parameters, with respect to the quality of the materials before and after treatment, show an improvement in the properties of porous siliceous sandstones from the point of view of their use as building materials.
5) The combined action of the consolidant plus water repellent, RC80, is more effective than the consolidant on its own, RC70.
6) Since the way in which stone materials respond to a given treatment depends on their intrinsic characteristics, the results were difficult to predict prior to carrying out the experiments. Therefore, it is advisable that each material is tested with each treatment under experimental conditions that are similar to those of each specific monument.

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Figure caption list

Fig 1**:** Canonical Biplot representation of the different building stones studied on the principal factorial plane 1-2

Fig 2: Canonical Biplot representation of the different building stones studied on the plane 1-3

Table 1: Average data of the hydric properties with its standard error.

SANPLE	TP[1]	FP[2]	RD[3]	AD[4]	AC[5]	IC[6]	CAC[7]
Z1	11.26	10.50	2.57	2.28	94.38	5.18	0.00103
	0.83*	0.61*	0.01*	0.02*	3.19*	0.68*	0.00005*
Z1RC70	11.91	11.46	2.59	2.28	95.19	5.03	0.00087
	0.97*	1.60*	0.03*	0.03*	5.63*	0.52*	0.00006*
Z1RC80	11.15	10.83	2.59	2.28	90.68	4.64	0.00078
	0.26*	0.56*	0.01*	0.02*	5.28*	0.22*	0.00008*
TG	16.52	15.46	2.60	2.17	93.56	7.28	0.00231
	0.37*	0.47*	0.01*	0.01*	1.50*	0.17*	0.00014*
TGRC70	15.85	12.10	2.59	2.18	76.38	7.06	0.00071
	0.23*	0.93*	0.05*	0.04*	7.01*	0.06*	0.00003*
TGRC80	4.66	1.44	2.35	2.24	30.88	3.50	0.00021
	0.53*	0.21*	0.09*	0.08*	3.23*	0.48*	0.00001*
TB	19.60	17.53	2.59	2.08	89.45	9.72	0.00230
	0.16*	0.54*	0.01*	0.01*	3.05*	1.66*	0.00006*
TBRC70	18.70	8.10	2.57	2.09	42.76	8.34	0.00079
	3.14*	2.53*	0.02*	0.10*	6.59*	2.44*	0.00015*
TBRC80	15.97	1.77	2.54	2.14	11.07	6.46	0.00025
	1.18*	0.17*	0.01*	0.03*	0.87*	0.43*	0.00006*
TO	22.00	20.01	2.58	2.01	90.96	9.38	0.00255
	0.57*	0.72*	0.04*	0.04	1.44*	0.35*	0.00015*
TORC70	20.53	14.96	2.58	2.05	72.86	9.07	0.00090
	1.15*	1.17*	0.01	0.02*	2.79*	0.44*	0.00009*
TORC80	19.10	3.26	2.54	2.05	17.08	8.04	0.00051
	0.54*	0.70*	0.01*	0.01*	3.72*	0.50*	0.00003*
TR	28.18	26.33	2.58	1.85	93.53	13.67	0.00483
	1.45*	1.03*	0.01*	0.04*	3.43*	0.90*	0.00045*
TRRC70	24.95	12.08	2.54	1.90	48.48	12.08	0.00100
	1.28*	3.02*	0.02*	0.03*	12.59*	0.78*	0.00005*
TRRC80	22.43	1.79	2.43	1.88	7.98	5.10	0.00024
	0.96*	0.08*	0.08*	0.04*	0.28*	1.22*	0.00002*

*Standard error, [1]Total Porosity (%), [2]Free Porosity (%), [3]Real Density (g/cm^3), [4]Apparent Density (g/cm^3), [5]Absorption Coefficient (%), [6]Imbibition Coefficient (%) and [7]Capillary Absorption Coefficient ($g/cm^2s^{1/2}$).