

CLAY POULTICES IN SALT EXTRACTION FROM ORNAMENTAL STONES: A STATISTICAL APPROACH

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Abstract—The extraction of salts by layered (bentonite) and fibrous (sepiolite) clay poultices from stone materials currently used in Heritage buildings was studied. Analyses were performed on stones affected by salts but not submitted to humidity, and also on stones affected by salts and simultaneously submitted to continuous or intermittent humidity during the salt-extraction process. From the experimental results and the statistical analysis, the application of two successive poultices is sufficient for an efficient extraction of Cl^- and NO_3^- in the absence of humidity. However, under humid conditions, the efficiency of the method is reduced considerably. For SO_4^{2-} , the use of hydrotalcites (anionic clays) is recommended for more effective extraction.

Key Words—Bentonite, Conservation, Historical Heritage Buildings, MANOVA-Biplot, Ornamental Stones, Poultices, Salts, Sepiolite, Weathering of Stone.

INTRODUCTION

Haloclasty, the breaking of stone materials because of the stresses generated by rain and the growth of salts inside their pore network, is one of the most common processes involved in the decay of stone (Vicente, 1994, 1996; García-Talegón *et al.*, 1999). Problems related to the presence of salts in the pore network of stone and other materials were addressed in detail by Evans (1970) and Arnold (1996).

Conservation of heritage buildings often requires the removal of salts from stone. ‘Desalting’ of stones by application of absorbent poultices is a widely used technique, although it is chiefly empirical in nature and no in-depth studies have been reported on the processes involved in this procedure. In addition to the conditions involved in the use of poultices (*e.g.* contact time, temperature), the efficiency of the procedure also depends on the nature of the poultices and the type of ions to be removed. No general agreement exists about the beneficial effects of salt removal or the suitability of the use of poultices. Bromblet and Verges-Belmin (1966), Ling and Smith (1966), Simon *et al.* (1966) and Weber *et al.* (1996) addressed the problems arising when the stone is contaminated by mixtures of ions, and they detected an increase in sulfate content and subsequent surface degradation in ornamental stones, following salt extraction by poultices.

In this study, we report a protocol developed to determine the behavior of clays used for removing salt under simulated conditions where poultices are commonly used. The aim was to reproduce in the laboratory the two situations usually found in practice in weathered stones for which conservation desalting is

recommended: (1) salt-laden stone in humidity-free conditions (*e.g.* from archaeological excavations, where the walls are largely unaffected by humidity); and (2) stone continuously or intermittently affected by humidity (*e.g.* walls currently affected by humidity).

MATERIALS AND METHODS

Blocks $5 \times 5 \times 5$ cm in size of a deeply-weathered natural granite (with a total porosity of 28.40%) were studied. The mineralogical and petrophysical characteristics of this granite were published by García-Talegón *et al.* (1994) and Iñigo *et al.* (1994). The blocks were immersed for 4 d in 0.5 M solutions of NaCl, NaNO_3 and Na_2SO_4 , respectively, to a height of 1 cm above the base of the blocks in order to produce total impregnation via capillary uprising. After the blocks were removed from the bath and dried at room temperature, a salt extraction process was performed using poultices of bentonite or sepiolite. Two extraction protocols were used: D (dry) and W (wet) conditions, as described below.

The sepiolite, S, and bentonite, B, used are commercial clays provided by the TOLSA Company (Madrid, Spain). Their specific surface areas are $240 \text{ m}^2/\text{g}$ (S) and $120 \text{ m}^2/\text{g}$ (B). Their X-ray diffraction (XRD) patterns and differential thermal analysis (DTA) curves are shown in Figures 1 and 2, respectively. The XRD patterns were recorded in a Philips PW 1030 instrument, using $\text{CuK}\alpha$ radiation ($\lambda = 1.5405 \text{ \AA}$) and a Ni filter. The DTA curves were recorded in air, in a Perkin-Elmer DTA 1700 apparatus, with a vertical furnace, chromel-alumel thermocouples, and at a heating rate of $10^\circ\text{C}/\text{min}$; the instrument was coupled to a Perkin-Elmer 3600 Data Station.

[†] Deceased.

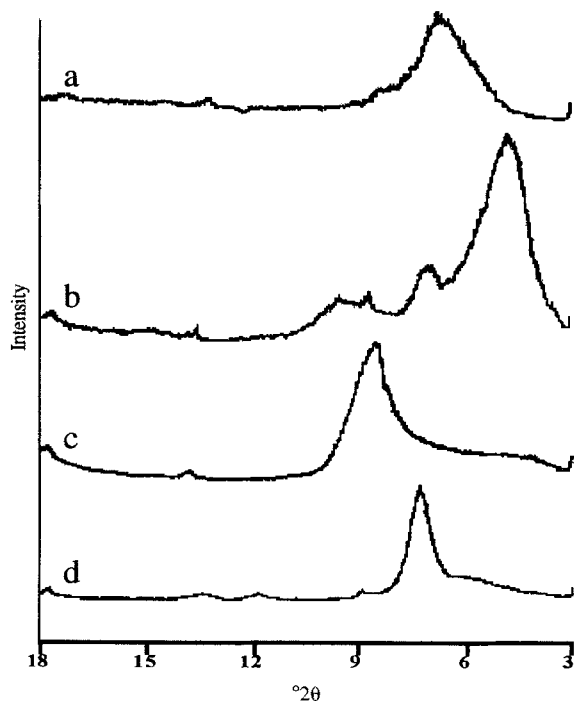


Figure 1. XRD patterns of sepiolite and bentonite used in poultice preparation: (a) untreated bentonite; (b) oriented aggregate of bentonite with glycerol; (c) bentonite heated at 500°C; and (d) untreated sepiolite.

D Protocol

Poultices were prepared by wetting the clays (B or S) with distilled water, and placing the poultice on the top surface (poultice 4 mm thick) of three salt-impregnated blocks. The block was placed on a dry surface and the sides were covered with paraffin to avoid evaporation through the sides (*i.e.* to ensure it occurred only through the poultice). The blocks were covered with a plastic film to prevent drying of the poultice (when dried, poultices peel off and lose contact with the stone block). The plastic film was removed after 3 d and the blocks were allowed to dry at room temperature. The poultice peeled off spontaneously and was analyzed. The same blocks were treated twice more, following the same method, to assess the efficiency of salt extraction.

W Protocol

This protocol was identical to protocol D, but the block was placed on a humid surface for 4 d. Then, the block was moved to a dry surface for 3 d. The blocks were subjected to two additional cycles of wetting and drying.

After each poultice was removed from the blocks, it was dried to constant weight. Then, ion extraction from the poultices was performed following the NORMAL (1983) procedure. Analysis of the different salt

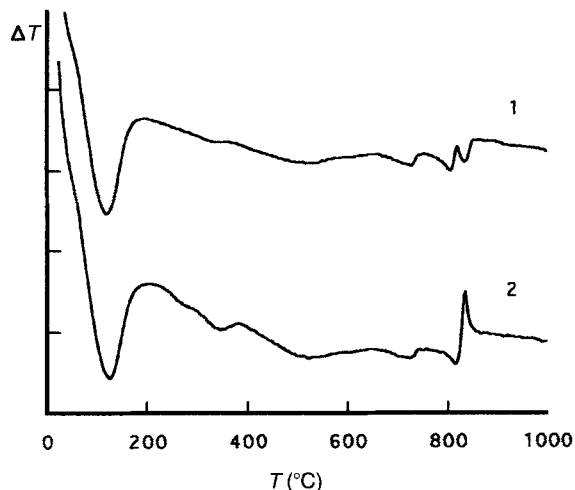


Figure 2. DTA traces of bentonite (curve 1) and sepiolite (curve 2) used in poultice preparation.

species extracted was accomplished using a Metrohm ionic chromatography system, equipped with a CI 709 pump, a CI 732 conductivity detector, a CI 733 separation chamber, and a Metrohm MSM (CI 753) suppressor module, with a CI 752 peristaltic pump.

After extraction with the bentonite poultices, the granite blocks were split into three horizontal portions with thicknesses of 1.5 cm (X), 1.5 cm (Y) and 2.0 cm (Z) from top to bottom, as shown in Figure 3. After grinding and homogenization, the soluble salts in each portion were analyzed following the method of NORMAL (1983). Analysis was also performed on reference blocks impregnated with salt solutions, but not submitted to extraction.

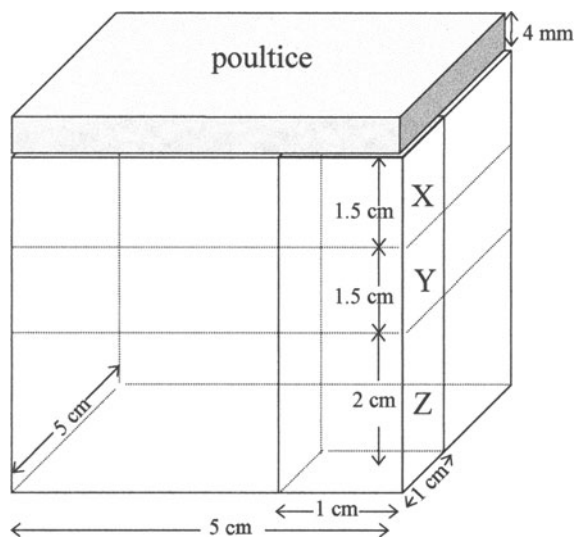


Figure 3. Schematic representation of the blocks used in the study.

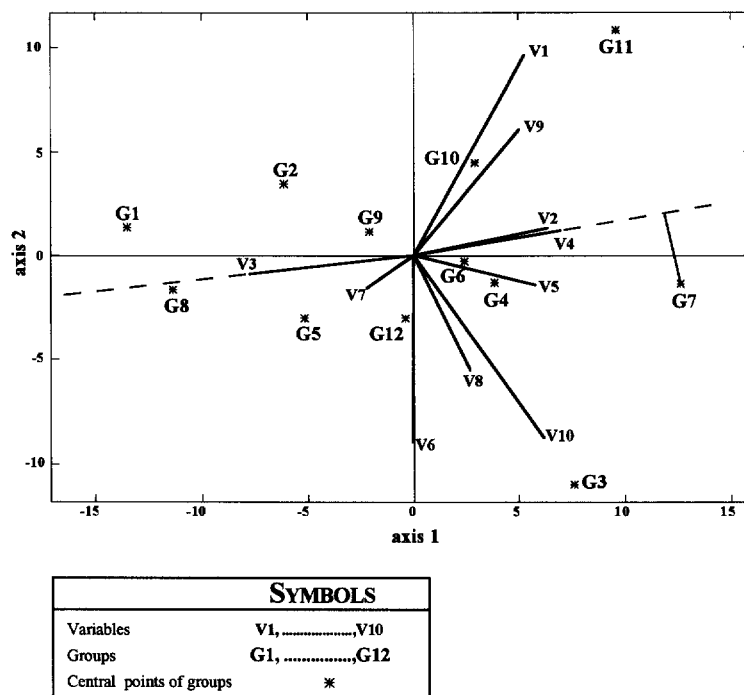


Figure 4. Joint plot on the first plane of a canonical biplot.

Statistical method (MANOVA-biplot)

Statistical analysis of the results by classical statistical methods (ANOVA) for individual variables, treated independently, showed no differences between the properties of the different slabs, treatments, nature of clay, *etc.* However, the physicochemical results showed beyond doubt that differences did exist. For this reason, biplot methods were used to assess the relationships existing between the different variables studied.

One of the best statistical methods among descriptive multivariate analyses is the biplot method described by Gabriel (1971), Galindo (1986) and Gower and Hand (1996). Biplot methods are techniques for the simultaneous representation, in reduced dimensions, of a matrix of dimensions $n \times p$, where n generally represents n rows (elements) and p refers to the corresponding variables measured on those elements. The current trend is to consider biplot methods as graphic representations or tools for diagnosis of more formal models where the structure of the model is considered *a priori* or *a posteriori*, respectively, in the data matrix. As a representation method, Gabriel (1972, 1975), Vicente-Villardón (1992) and Gower and Hand (1996) proposed the biplot method as an alternative tool to multivariate analysis of variance (MANOVA), canonical analysis or discriminant analysis.

Let M be a matrix of n individuals by p variables and assume that the n individuals are divided into mu-

tually exclusive k classes (groups), each with n_i individuals ($i = 1, 2, \dots, k$) ($n = n_1 + n_2 + \dots + n_k$). The aim is to obtain a simultaneous plot of the rows (groups) and columns (variables) of the data matrix in such a way that the groups are separated with maximum discriminatory power. This type of biplot is referred to as a MANOVA biplot by Gabriel (1995) and a biplot of canonical variables by Vicente-Villardón (1992).

The results of this analysis are usually plotted as dispersion diagrams, (Figure 4), with markers (asterisks) for the groups (G) and vectors for the different variables (V). If the projection of a marker point of a group (*e.g.* $G7$) is close to a column marker (the end of the vector, *e.g.* $V4$), it is concluded that that group takes high values in that variable. If two column markers are close, both variables are correlated. These conclusions are not, however, straightforward, as they may depend on other factors, such as the goodness-of-fit of the variables on the plane, the absorption of inertia of the plane, *etc.*

RESULTS

Experimental results

Table 1 and Figure 5 show, respectively, the concentration of anions extracted (averaged values for three blocks tested) with bentonite poultices (B) under both extraction conditions (D and W) studied. The ratio between the amount of Cl^- extracted with protocol D (Table 1, Figure 5) for the first poultice and for the

Table 1. Amount of anions (meq/cm²) extracted by three successive poultices of bentonite (average values and standard deviations of three assays) under D and W conditions.

	1st	2nd	3rd	1st/2nd	1/st/3rd
D Conditions					
Cl ⁻	0.041 (0.014)	0.014 (0.005)	0.009 (0.006)	2.880	4.680
NO ₃ ⁻	0.034 (0.014)	0.010 (0.004)	0.005 (0.002)	3.400	6.740
SO ₄ ²⁻	0.053 (0.019)	0.013 (0.006)	0.007 (0.003)	4.200	7.500
W Conditions					
Cl ⁻	0.143 (0.011)	0.110 (0.019)	0.078 (0.004)	1.290	1.820
NO ₃ ⁻	0.133 (0.017)	0.092 (0.015)	0.069 (0.012)	1.450	1.940
SO ₄ ²⁻	0.236 (0.047)	0.176 (0.023)	0.140 (0.011)	1.340	1.680

second poultice is 2.880, and between the first and the third is 4.680. For NO₃⁻, the ratios between the amounts extracted by the first and the second poultices is 3.400, and between the first and the third, 6.740. For SO₄²⁻, the values are 4.200 and 7.500, respectively. The amounts extracted by the third poultice using the D protocol are <0.01 meq/cm². For extractions following protocol W (Table 1, Figure 5), the relationship between the amounts extracted with the first and second and the first and third poultices are as follows: Cl⁻, 1.290 and 1.820; NO₃⁻, 1.450 and 1.940; and SO₄²⁻, 1.340 and 1.680.

The sepiolite poultices under D conditions (Table 2, Figure 6) extracted appreciably lower amounts of ions than those of bentonite. The relationships between the amounts extracted with the first and second poultices and the first and third are Cl⁻, 1.691 and 4.070; NO₃⁻, 1.794 and 6.160; and SO₄²⁻, 1.926 and 5.450, respectively. Under W conditions, (Table 2, Figure 6), the relationships between the first and second, and first and third extractions are Cl⁻, 1.486 and 2.684; NO₃⁻, 0.913 and 1.205; and SO₄²⁻, 2.291 and 3.275, respectively.

To determine the depth of extraction with the poultices, the concentrations of anions (Table 3, Figure 7) remaining in parts X, Y and Z of the blocks subjected to the extraction process (D,W) with three successive poultices of bentonite were determined. The ion contents remaining in the different parts of the blocks after extraction under D conditions are very low in X, slightly higher in Y, and much higher in Z, which has the same ion contents as the reference blocks. Under W conditions, the opposite results are observed: the greatest ion contents are found in X and they decrease dramatically in Y, and increase slightly in Z.

The mass balance of ions between the salt-treated reference block and the poultice-extracted blocks (*i.e.* the sum of ions extracted and determined in the poultice and those remaining in sections X, Y and Z) was determined using a column of granite (Figure 3) subjected to 1 cm² of poultice on the top face. The density of the granite used was 1.77 g/cm³ (Iñigo *et al.*, 1994) and thus the masses of parts X, Y and Z were 2.6, 2.65 and 3.50 g, respectively. The sum of average val-

ues extracted per cm² of three successive poultices of bentonite, the total ion content remaining in parts X, Y and Z of the column after extraction, and the total ion contents of parts X, Y and Z of the reference column are given in Table 4 for methods D and W, respectively.

The sum of ions remaining in parts X, Y and Z and the amount extracted per 1 cm² of poultice (Table 4), is slightly lower than the values found for the reference columns under D conditions, but slightly higher under W conditions. This small error is probably related to the fact that the concentrations in parts X, Y and Z were not uniform (a decrease in salt concentration upwards is expected), and thinner slices were not possible with the saw available.

To generalize the results of the present work, a statistical study using a MANOVA-biplot in three formats was used.

Ions extracted by the clay poultices. The MANOVA-biplot analysis performed gave a global result (Wilks' lambda) of $p < 0.05$. Differences occur between the average amounts of salts adsorbed by the poultices in the four groups compared. The absorption of inertia of the first three factorial axes is 100%, that of the first plane being 88.39%. Differences were detected in the first three axes: $p < 0.01$, $p < 0.01$ and $p < 0.05$, respectively.

Figure 8 shows the MANOVA-biplot representation on the first factorial plane. The first axis discriminates the BW from the remaining samples, due to its higher salt content. The second axis discriminates the extractions performed under D conditions from those performed under W conditions. All the variables are highly correlated. On performing individual ANOVA analysis on each initial variable, differences at $p < 0.05$ were only found in Cl⁻ for the third poultice, and in SO₄²⁻ for the second and third poultices (shown in bold type in Figure 8).

Ions remaining in parts X, Y and Z of the block after extraction by poultices under D conditions. Global analysis of the amounts of salts remaining under D conditions (Wilks' lambda) gave $p < 0.01$. The absorption of inertia of the first factorial plane is 94.27%.

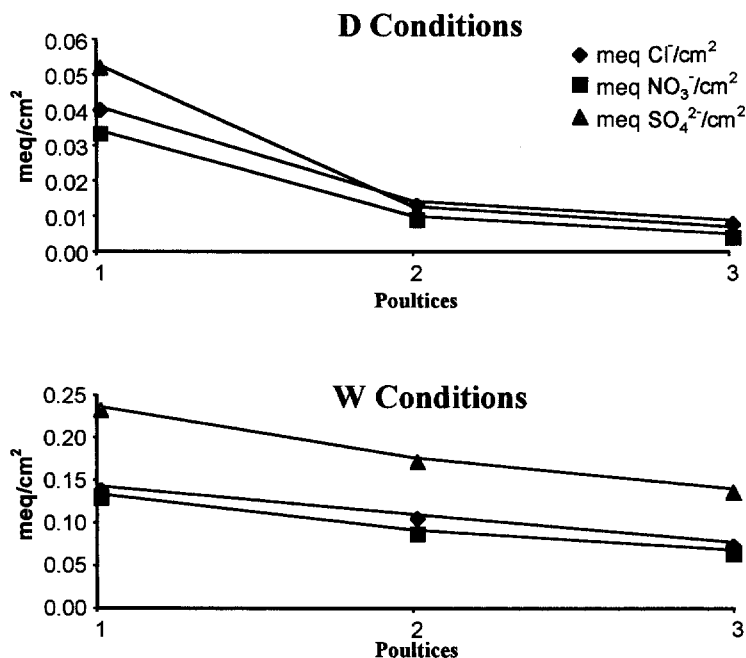


Figure 5. Amount of anions extracted (meq/cm²) by application of three successive poultices of bentonite (average values of three blocks) under D and W conditions.

The first two axes show a *p* value of <0.01. The individual ANOVA analysis of the four variables show *p* > 0.05.

Figure 9 shows the representation of the first factorial plane, where only the average (central points) of the groups are represented. The first axis discriminates the salt contents in parts Y and Z of the blocks after extraction with bentonite poultices (BY and BZ, respectively) from the salts remaining in all three parts (X, Y and Z) after extraction with sepiolite. The second axis discriminates BZ from BY. This discrimination is related to the salt contents in part Z and it is much higher than in part Y.

Ions remaining in parts X, Y and Z of the blocks after extraction by poultices under W conditions. In the global analysis, the amounts of salts remaining in W samples showed no differences compared to the average values of the six groups (Wilks' lambda with *p*

> 0.05). The same analysis showed a *p* value of 0.05 for the original variables.

DISCUSSION

The total amount of ions extracted with three successive poultices is considerably higher under W than under D conditions. The ratio between the average amounts extracted under W and D conditions with bentonite poultices is Cl⁻ 5.00; NO₃⁻ 5.92; and SO₄²⁻ 7.59. Under W conditions the ions migrate toward the surface by water flow; under D conditions, mobilization of ions occurs in the wet part by diffusion from the poultice, and below a certain depth, the extraction effect is zero. The ion contents in part Z of the blocks are the same as in the reference column.

The amounts extracted with the third poultice are small at <0.01 meq/cm² in the blocks treated under D conditions. Thus, only two extractions are necessary

Table 2. Amount of anions (meq/cm²) extracted by three successive poultices of sepiolite (average values and standard deviations of three assays) under D and W conditions.

	1st	2nd	3rd	1st/2nd	1st/3rd
D Conditions					
Cl ⁻	0.019 (0.003)	0.011 (0.003)	0.005 (0.002)	1.691	4.070
NO ₃ ⁻	0.016 (0.004)	0.009 (0.002)	0.003 (0.001)	1.794	6.160
SO ₄ ²⁻	0.026 (0.005)	0.014 (0.004)	0.005 (0.001)	1.926	5.450
W Conditions					
Cl ⁻	0.085 (0.018)	0.057 (0.007)	0.032 (0.001)	1.486	2.684
NO ₃ ⁻	0.042 (0.017)	0.046 (0.006)	0.035 (0.007)	0.913	1.205
SO ₄ ²⁻	0.175 (0.034)	0.077 (0.014)	0.054 (0.003)	2.291	3.275

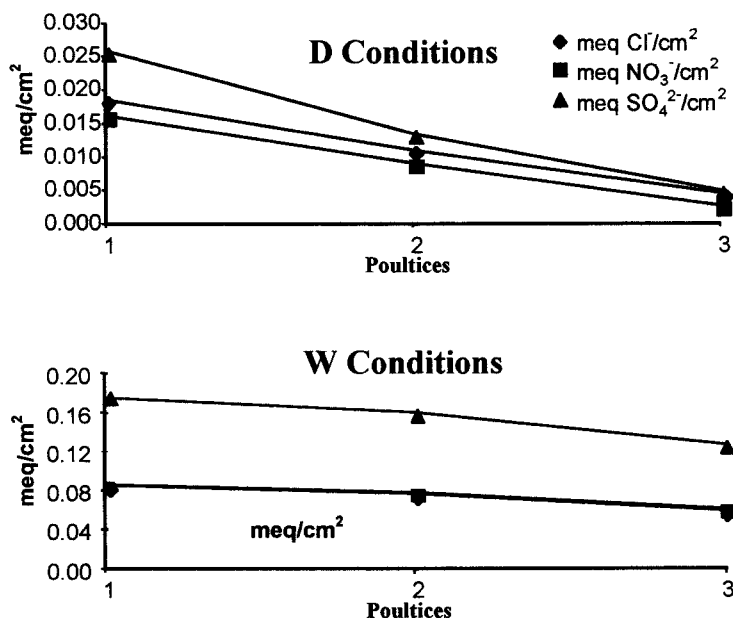


Figure 6. Amount of anions extracted (meq/cm²) by application of three successive poultices of sepiolite (average values of three blocks) under D and W conditions.

under this condition for efficient desalting of the surface. In extractions performed under W conditions, the third poultice still extracts appreciable amounts of ions, especially sulfate ions, which may reach 0.14 meq/cm². Under D conditions, the salt contents remaining in the blocks after the application of the three poultices are smaller in part X than in Y, and smaller in part Y than in Z. In part Z, the salt content is the same before and after extraction, suggesting that at this depth (>3 cm) the poultices have no effect.

When extraction is performed under W conditions, the greatest concentrations of salts remain in part X and the smallest in part Z. The constant flow of water during the process carries the salts toward the surface and the ion contents are appreciably higher in part A after the extraction process than before, especially sulfate (Figure 7). This result probably occurs because of the low mobility of this doubly valent anion, which is difficult to remove by simple physical adsorption.

Table 3. Amount of anions (meq/g stone) remaining in parts X, Y and Z of a 1cm²-base column of granite after successive application of three bentonite poultices under D and W conditions.

	X	Y	Z	Reference
D Conditions				
Cl ⁻	0.027	0.029	0.054	0.054
NO ₃ ⁻	0.024	0.025	0.054	0.054
SO ₄ ²⁻	0.054	0.085	0.119	0.119
W Conditions				
Cl ⁻	0.046	0.015	0.013	0.054
NO ₃ ⁻	0.049	0.013	0.011	0.054
SO ₄ ²⁻	0.132	0.022	0.041	0.119

Thus, an extractant that would trap this ion by ion exchange is more suitable. The use of hydrotalcites (anionic clays) under the conditions reported by Trujillano *et al.* (1998) is recommended.

Application of two consecutive bentonite poultices under slightly humid, relatively dry conditions (protocol D) is sufficient to reduce the salt content in granite to acceptable values. This is supported by comparison of the amounts of Cl⁻ and NO₃⁻ extracted by the first and third poultices.

When extraction is performed under very humid conditions (protocol W), the amounts extracted with

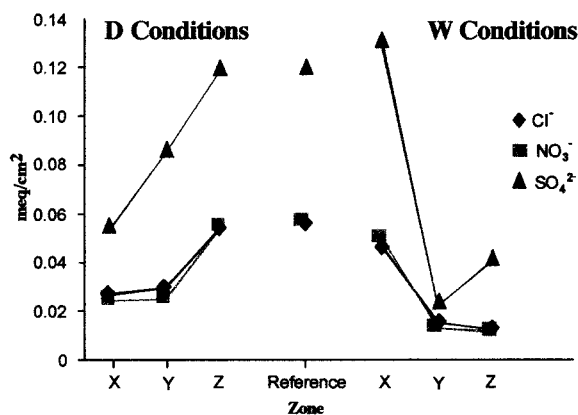


Figure 7. Amount of anions (meq/cm²) remaining in parts X, Y and Z of the blocks after application of three successive poultices of bentonite (average values for three blocks) under D and W conditions.

Table 4. Total amount of ions remaining in parts X, Y and Z of a 1 cm²-base column after extraction with poultices. Ions extracted by three successive poultices, sum of X + Y + Z + extracted and total ions in parts X, Y and Z of the reference column.

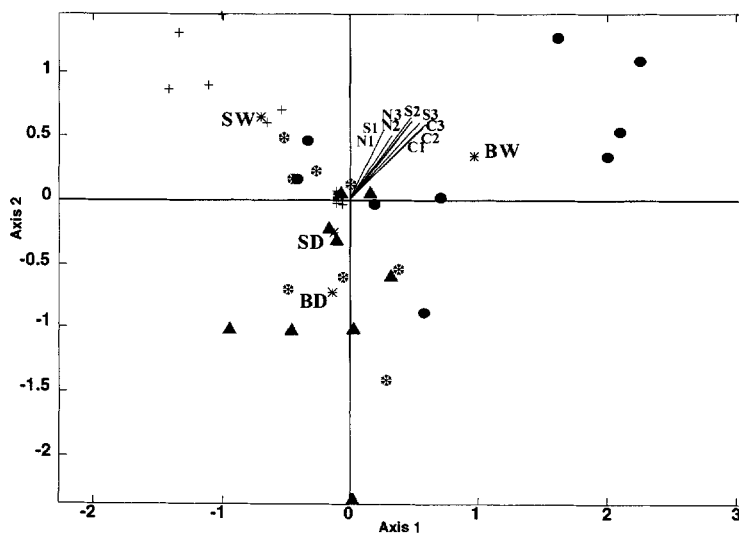
	X	Y	Z	Extr.	Extr. + X + Y + Z	Reference
D Conditions						
Cl ⁻	0.072	0.078	0.195	0.061	0.411	0.480
NO ₃ ⁻	0.065	0.066	0.192	0.050	0.371	0.491
SO ₄ ²⁻	0.143	0.022	0.423	0.073	0.864	1.050
W Conditions						
Cl ⁻	0.122	0.041	0.045	0.331	0.538	0.480
NO ₃ ⁻	0.131	0.034	0.040	0.294	0.499	0.491
SO ₄ ²⁻	0.394	0.059	0.143	0.553	1.104	1.050

three successive poultices are roughly equivalent. In the case of walls subjected to high humidity, the migration of salts to the surface is enhanced because solutions continuously allow salts to move to the surface. Thus, desalting by poultices is not appropriate in this case.

Comparing the amounts extracted by sepiolite and those by bentonite under the same conditions shows that bentonite poultices extract about twice as much of the salt content.

Comparison of the initial ion contents in the blocks with those remaining in zones 1.5, 1.5 and 2 cm below the surface after the extractions, indicates that under low-humidity conditions the nitrate and chloride contents from the surface to a depth of 3 cm decrease significantly. By contrast, under high-humidity conditions, the contents at the surface are similar to the initial Cl⁻ and NO₃⁻ contents and 15% greater than the original SO₄²⁻ content. Figure 9 shows that the amount of sulfate ions remaining 1.5–3 cm below the surface is significantly smaller than that present 3–5 cm below the surface.

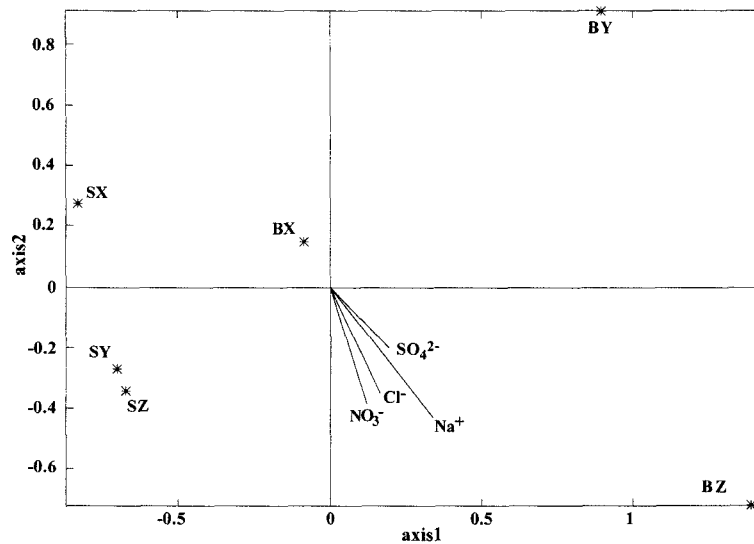
Under high-humidity conditions, no differences (*p* > 0.05) were observed between the average amounts of ions remaining in zones at three different depths below the surface. This confirms that desalting with poultices under humid conditions is not advisable.



VARIABLES	SYMBOLS
Ions of Cl ⁻ extracted with the first, second, and third poultice	C1, C2, and C3
Ions of NO ₃ ⁻ extracted with the first, second, and third poultice	N1, N2, and N3
Ions of SO ₄ ²⁻ extracted with the first, second, and third poultice	S1, S2, and S3

GROUPS	SYMBOLS		
	Label	Central points	Elements
Sepiolite under wet conditions	SW	*	+
Sepiolite under dry conditions	SD	*	⊛
Bentonite under wet conditions	BW	*	●
Bentonite under dry conditions	BD	*	▲

Figure 8. MANOVA-biplot representation on the first factorial plane (ions extracted by the clay poultices).



VARIABLES		SYMBOLS	
Ions of Cl^- remaining in part X, Y, and Z of the block after extraction		Cl^-	
Ions of NO_3^- remaining in part X, Y, and Z of the block after extraction		NO_3^-	
Ions of SO_4^{2-} remaining in part X, Y, and Z of the block after extraction		SO_4^{2-}	
Ions of Na^+ remaining in part X, Y, and Z of the block after extraction		Na^+	

GROUPS	SYMBOLS	
	Label	Central points
Sepiolite remaining in part X	SX	*
Sepiolite remaining in part Y	SY	*
Sepiolite remaining in part Z	SZ	*
Bentonite remaining in part X	BX	*
Bentonite remaining in part Y	BY	*
Bentonite remaining in part Z	BZ	*

Figure 9. MANOVA-biplot representation on the first factorial plane (ions remaining in parts X, Y and Z of the block after extraction by poultices under D conditions).

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