

Salts in Ceramic Bodies III: An Experimental Study on Desalination

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Abstract

This study evaluates the efficiency of various immersion methods in the removal of soluble salts previously introduced into ceramic tiles. Two types of ceramic bodies were tested to evaluate the effect of porosity on the desalination procedure: earthenware and stoneware tiles prepared ad-hoc for this study. The desalination methods compare long-term immersion, repeated washings by total immersion and intermittent washings that included a partial drying period between successive washings. While the last two methods were more effective in removing more soluble salts, such as NaCl, long-term immersion appears to be more efficient for less soluble salts such as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, or even Na_2SO_4 in the case of lower porosity ceramics.

Keywords: desalination, ceramics, salts, immersion, desalination efficiency

Salze in poröser Keramik III: Ein experimenteller Beitrag zum Entsalzen

Zusammenfassung

In dieser Arbeit wird die Wirksamkeit einiger unterschiedlicher Immersionsmethoden für die Entfernung von früher eingebrachten Salzen aus porösen keramischen Ziegeln untersucht. Zwei Arten keramischer Werkstoffe wurden geprägt, um den Einfluss der Porosität auf das Entsalzen untersuchen zu können: Steinzeug- und Steingutziegel, die speziell für dieses Projekt hergestellt wurden. Für das Entsalzen wurden die folgenden Varianten verglichen: langzeitiges Eintauchen, wieder-

holtes Waschen durch vollständiges Eintauchen und wiederholtes Eintauchen mit dazwischenliegenden Perioden zum teilweisen Austrocknen. Die zwei letztgenannten Varianten waren leistungsfähiger, wenn es sich um den Austrag gut löslicher Salze wie CaCl handelt. Das langzeitige Eintauchen ist dagegen wirksamer für das Entfernen weniger löslicher Salze wie $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ oder sogar Na_2SO_4 im Falle der keramischen Proben mit geringer Porosität.

Stichwörter: Entsalzen, Keramik, Salze, Eintauchen, Wirksamkeit des Entsalzens



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1 Introduction

One of the most difficult problems in the conservation of salt-laden porous building materials or archaeological ceramics is the removal of the salt, which has long been recognized as one of the primary sources for their deterioration [1,2,3]. Many studies have been carried out to evaluate different methods and procedures to extract these damaging compounds from porous materials [2,3,4,5]. The procedures, based on the solubility of the salts in water, range from simple immersion to forcing the water through the interior of masonry while a poultice, applied to the surface, serves to collect the emerging salts. Several examples can be cited illustrating these techniques. In the simplest case, large marble slabs were desalinated utilizing a prolonged immersion, i.e., three months, in deionized water [6]. For the specific case of the removal of gypsum crusts from the surface of buildings, repeated fine mistings of the wall were used, allowing the wall to dry between the washings. This repetitive procedure proved to be far more effective than a single washing [7].

The above examples, from architectural structures, can be complemented with those applied to smaller objects. In the case of archaeological ceramics, total immersion is one of the standard approaches [8]. Poultices have also been recommended for especially friable ceramics [4] and in some cases a combination of both techniques have been applied to desalinate Dutch tiles [3]. A critical review of the various desalination methods can be found elsewhere [9].

The aim of the present study was to evaluate the effectiveness of total immersion procedures by comparing results from prolonged immersion with those of successive washings with and without partial drying in between. For this purpose the study used ad-hoc ceramic tiles impregnated with individual salts and their mixtures, which had been used for a previous study [10,11].

2 Experimental

2.1 Ceramic tiles

Two sets of ceramic tiles were used, a more porous earthenware and less porous stoneware. Table 1 summarizes the most relevant information of these tiles. Further information on the manufacturing and characteristics of these tiles can be found elsewhere [10,12].

The tiles were impregnated with individual salts, sodium chloride, NaCl, sodium sulfate, Na₂SO₄, and calcium sulfate, CaSO₄, or mixtures of NaCl with each of the other two. Further details of the impregnation methodology are described in the first paper of this study [10]. The tiles were used to determine their moisture uptake with increasing or minor fluctuations of relative humidity [11].

Table 1: Average size, weight, volume and open porosity (both 24-hour immersion in cold water and 5-hour in boiling water ASTM C67-97) for the earthenware (E) and stoneware tiles (S) used in the study

	E	S
Dimensions	5.7 x 5.6 x 1.4 cm	5.3 x 5.4 x 1.4 cm
Weight	71.61 g	76.90 g
Volume	44.68 cm ³	40.07 cm ³
Open Porosity (24 hours)	17.7%	5.1%
Open Porosity (5 hours)	19.1%	7.8%

2.2 Determination of salt concentration

Both conductance measurements and conductimetric titrations were used for the quantitative determination of the amount of salts removed from tiles by the different procedures used. Conductance measurements were carried out on standard solutions at ambient lab conditions where the temperature ranged from 17° to 22°C. Calibration curves were obtained with standard solutions of NaCl, Na₂SO₄, and CaSO₄, correlating conductance to molarity ranging between 0.001M and 0.01M. For solutions containing only one salt, this technique provided results as accurate as those obtained through titration.

For solutions of mixed salts, conductometric titrations were necessary to estimate the concentration of the individual salts. The titrants were 0.1M and 0.0141 M silver nitrate for chlorides and 0.005 M and 0.05M barium chloride for sulfates. The titrations of these ions were carried out on separate aliquots of the test solution. Although silver sulfate forms a relatively insoluble salt, it was found that the ratio of Cl⁻/SO₄⁼ concentration in the titrated solutions was such that this interference was negligible [12].

From test titrations with standard solutions of the mixed salts it was found that the actual titration of the sulfates could be circumvented. The procedure used was as follows:

- the total conductance of the solution was measured;
- the chloride ion was titrated;
- knowing the concentration of sodium chloride in the solution, the contributing conductance of this salt was estimated using the standard concentration-conductance plot;

- this conductance was subtracted from the total conductance of the original solution;
- the remaining conductance corresponds to the contribution of the sodium sulfate and the concentration of this salt was then obtained from the standard concentration-conductance plot.

Periodically, for solutions that had higher concentrations of sulfates, i.e., those containing Na_2SO_4 , this ion was titrated directly to confirm the calculated values.

2.3 Desalination Methods

2.3.1 Prolonged Immersion

This method was only used on tiles containing individual salts. The tiles were simply soaked in either 150-ml or 300-ml of deionized water. The choice of 150-ml was based on its being the minimum amount of water necessary to cover a single tile placed in a 250ml beaker. The tiles were slowly immersed into the beaker containing the pre-measured amount of water. They sat on their edge resting against the side of the beaker thus allowing water to surround them completely. When 300-ml of deionized water were used, the tiles were stood up on glass beads to allow exposure on all sides to the water. To be able to compare results between the two solutions, pairs of tiles were chosen with the closest amount of salts.

Once the tile was immersed in the water, the beaker was covered and conductance measurements were taken every hour for the first eight hours and daily afterwards. The tiles were left until the difference between two successive conductance measurements taken at a 24-hour interval was negligible (<5% change). This occurred after ten days, for earthenware, and eight days, for stoneware tiles, for both volumes of wash water.

Stoneware tiles containing CaSO_4 gave erratic results. This can be attributed to the combination of the negligible amount of this salt and the low-porosity of these tiles.

It should be noted that for the earthenware tiles containing NaCl , given the high concentration of salt in the tile, it was necessary to change the solution after the first 2.5 hours (for the 150-ml experiment) and after 3 days (for the 300-ml experiment), to be able to continue to monitor the conductance of the solution.

2.3.2 Repeated Washings

In this experiment, also carried out on tiles only containing individual salts, the immersion solution, either 150 or 300-ml, was changed after short intervals. The intervals chosen were 1-hour for tiles containing NaCl , and 2-hours for tiles containing Na_2SO_4 or CaSO_4 . A total of five washings were made.

2.3.3 Intermittent Washings

This method was applied to tiles containing individual salts as well as their mixtures. To improve salt removal efficiency, the tiles were allowed to dry partially between washings. The cycle included two steps: immersion of the tiles in 150-ml of deionized water; and, partial air-drying the tiles between soakings. The same immersion-time was used as in the previous case: tiles with NaCl or its mixtures, were left in water for one hour, tiles with individual sulfate salts, for two hours. After each cycle, all tiles regardless of the salts they contained, were left to air-dry for four hours. This time frame was chosen because it falls between the times it takes both tiles to reach the critical moisture content.

3 Results and Discussion

3.1 Prolonged Immersion

For desalination by total immersion, recommended procedures include prior climatization of the ceramic body in a 100% RH [13] or gradual immersion of the object into the water to eliminate air trapped in the pores [2,14]. In the present experiment, the small size of the tiles and the similarity in porosity obtained for the

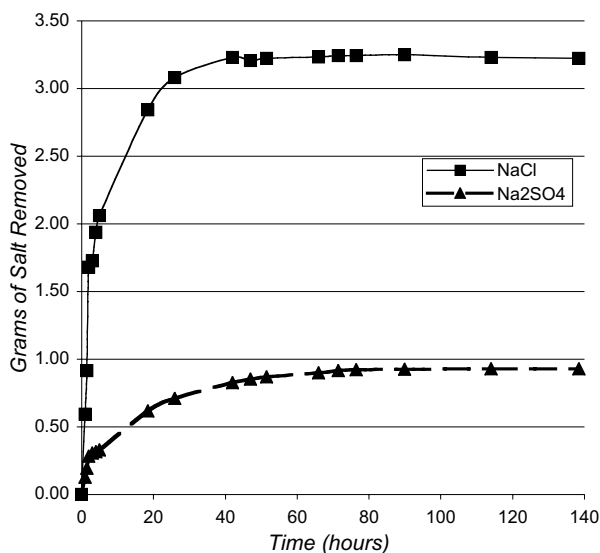


Figure 1: Desalination curves (grams of removed salt vs. time) for prolonged immersion in 150-ml of water of earthenware tiles containing NaCl or Na₂SO₄. Note that for the NaCl-containing tile, the water was changed once, at 2.5 hours.

tiles when measured by total immersion in cold and boiling water [10] suggested that this extra step was not required since water at room temperature infiltrated most open pores.

The desalination curves obtained are similar for all tiles, and as illustration the curves for earthenware tiles containing either NaCl or Na₂SO₄ when immersed in 150-ml of water are shown in Figure 1.

The percentages of salts removed by prolonged immersion are reported in Table 2. For the case of earthenware tiles containing NaCl, the values may be higher because of the fact that the solution was changed once during the experiment as mentioned above. The percentages of salts removed are computed based on the sum of the amounts removed in both solutions. The table also mentions whether the tile had been used for a previous experiment, such as RH cycling or measurement of the sorption isotherm, as described in a previous paper [11]. Since during these experiments, some tiles effloresced and lost some salt, the average values presented in this table and in subsequent ones, slightly underestimates the amount of salts extracted.

The amount of salt extracted is directly proportional to the solubility of the salt and the porosity of the ceramic body. Another factor that appears to be significant, particularly for the low porosity stoneware tiles, is the previous history of the ceramic object. Tiles that had been cycled at a higher RH released more salts. Although the number of tiles used in the experiment is not enough to confirm this hypothesis, the suggested trend is plausible. Tiles cycled between 44-55% RH for six months showed some deterioration which indicates that the salts were mobilized towards the surface.

Table 2: Percentage of salts removed from salt-laden tiles after prolonged immersion in 150-ml or 300-ml of deionized water. Immersion times were of 10 days for earthenware tiles and 8 days for stoneware tiles. The previous history of the tiles, such as RH cycling or measurement of the sorption isotherm, is also indicated.

Tile / Salt	% Salt Removed 150-ml	Previous experiment	% Salt Removed 300- ml	Previous experiment
E / NaCl	96.2%	43-55% RH	96.0%	43-55% RH
E / Na ₂ SO ₄	85.6%	43-55% RH	88.3%	0-11% RH
E / CaSO ₄	70.8%	43-55% RH	50.0%	0-11% RH
S / NaCl	86.9%	43-55% RH	78.6%	0-11% RH
S / Na ₂ SO ₄	76.0%	43-55% RH	59.1%	0-11% RH

It can also be observed that the relative amount of salts extracted was not significantly influenced by the volume of wash-water used. Although it has been suggested that larger amounts of water are needed to effectively desalinate objects the present experiments do not confirm this. The suggested water to object volume ratio is in the order of 4 [2] or 10-ml water per gram of object [14]. The experiments show that a volume ratio of 3 (and weight ratio ~ 2 ml/g), was as effective as a volume ratio of 6 (and weight ratio ~4 ml/g) and that both weight ratios were significantly below recommendation. These results are influenced by the small size of the tiles, their porosity and by the previous history of the ceramic body.

3.2 Repeated Washings

The wash-times chosen, 1 hour for NaCl and 2 hours for the sulfate salts, were based on the first inflexion points in the desalination curves obtained by the data above and shown in Figure 1. The percentage of salts removed during each of the five successive washings is reported in Table 3. For comparison, the estimated per-

Table 3: Percentage of salts removed during each of the five successive washings with 150-ml or 300-ml of water as well as the total amount. For comparison the amount extracted in prolonged immersion for the equivalent amount of time is reported. Washing times were 1-hour for NaCl- and 2-hours for Na₂SO₄-containing tiles. The previous history of the tiles is also indicated. NT means that the tile had not been used for any experiment.

Tile/Salt	Prev.	Salts Removed (%) 150-ml					Total	Prol.	Prev.
	Exper.	1.Wash	2.Wash	3.Wash	4.Wash	5.Wash	Imm.	Exper.	
E/NaCl	Sorpt.	49.7%	4.5%	3.2%	2.6%	2.4%	62.4%	~54%	43-55%
E/Na ₂ SO ₄	0-11%	26.3%	5.6%	4.4%	4.4%	4.4%	45.1%	~40%	43-55%
E/CaSO ₄	N.T.	8.5%	2.3%	1.5%	1.5%	1.5%	15.4%	~25%	43-55%
S/NaCl	Sorpt.	30.9%	0.90%	0.90%	0.90%	0.90%	34.5%	~24%	43-55%
S/Na ₂ SO ₄	43-55%	16.7%	1.7%	1.7%	1.7%	1.7%	20.0%	~25%	43-55%
Salts Removed (%) 300-ml									
E/NaCl	0-11%	47.8%	4.2%	4.2%	2.8%	2.5%	61.5%	~49%	43-55%
E/Na ₂ SO ₄	45-55%	33.1%	6.0%	4.8%	4.0%	4.0%	51.9%	~35%	0-11%
E/CaSO ₄	N.T.	8.5%	3.1%	3.1%	3.1%	3.1%	20.9%	~26%	0-11%
S/NaCl	Sorpt.	27.5%	1.8%	1.3%	1.2%	1.1%	32.9%	~23%	43-55%
S/Na ₂ SO ₄	0-11%	14.5%	1.6%	1.6%	1.6%	1.6%	19.4%	~23%	0-11%

centage of salt removed during the total immersion procedure for a length of time equal to the sum of the five successive washings is included. The table also lists the experiments for which these tiles had been used and described elsewhere [11].

Repetitive washings are generally more effective at removing salts, particularly NaCl. For Na₂SO₄ in earthenware tiles the differences are smaller, ranging from ~5% when the tile used for prolonged washing (150-ml) had been cycled at a higher RH than that for repeated washing, to ~15% for the inverse case (300-ml), showing the importance of the previous history of the tiles in question. When Na₂SO₄ is present in stoneware tiles it would appear that prolonged immersion is always more effective at removing it. This is also the case for CaSO₄ in either type of tile. This suggests that there is a distinct correlation between the solubility of the salt to be removed and the porosity of the tile. This topic is addressed in the following paper [15].

3.3 Intermittent Washings

Partial drying of the tiles between repeated washes has the objective of allowing the salts contained in the tile to migrate towards the surface as water evaporates. This procedure was successfully applied to in situ washing by nebulization on buildings [7]. The percentage of salt removed for tiles containing individual salts are presented in Table 4.

Table 4: Percentage of salts removed during each of the three successive washings with 150-ml water with four-hour partial drying in between. Washing times were of 1-hour for NaCl containing tiles, and 2-hours for those with Na₂SO₄. For comparison the amount extracted in prolonged immersion and repeated washings, for the equivalent amount of time is reported. The previous history of the tiles is also indicated.

Tile/Salt	Prev.	Salts Removed (%)				Repeat	Prev.	Prol.	Prev.
	Exper.	1.Wash	2.Wash	3.Wash	Total	Imm.	Exper.	Imm.	Exper.
E/NaCl	Sorpt.	46.0%	11.5%	7.6%	65.1%	57.4%	Sorpt.	~45%	43-55%
E/Na ₂ SO ₄	43-55%	50.7%	18.7%	11.7%	81.1%	36.3%	0-11%	~31%	43-55%
E/CaSO ₄	Sorpt.	25%	25%	20%	70%	15.4%	N.T.	~25%	43-55%
S/NaCl	0-11%	23.7%	5.3%	7.9%	36.9%	32.7%	Sorpt	~22%	43-55%
S/Na ₂ SO ₄	Sorpt.	12.3%	3.8%	5.1%	21.2%	20.1%	43-55%	~24%	43-55%

The results obtained show that the extraction efficiency for these salts, particularly for the more porous earthenware tile, is significantly increased when a drying period is allowed between washings. The high values obtained for both Na₂SO₄ and CaSO₄ suggest that this procedure is also more effective for the less soluble salts. The dispersion of the data is greater for these results because of the influence of the drying period, which is affected by laboratory conditions at the time the experiment was carried out. For the stoneware tiles, the increase in efficiency is not that significant and, for the less soluble sodium sulfate is still slightly below that obtained by prolonged immersion.

Table 5: Percentage of total salt removed, as well as for each individual salt, during each of three successive 1-hour washing with 150-ml water separated by a four-hour partial drying. Numbers in parenthesis give the standard deviation.

Tile/Salt	1. Wash	2. Wash	3. Wash	Total
E/NaCl+NaSulf	37.7% (±3.4)	6.9% (±0.5)	6.2% (±0.9)	50.8% (±2.6)
NaCl	37.2% (±6.5)	7.0% (±0.3)	6.1% (±0.9)	49.4% (±3.7)
Na Sulfate	39.1% (±10.8)	6.3% (±1.4)	6.6% (±0.7)	49.8% (±13.3)
E/NaCl+CaSulf	22.5% (±3.5)	9.0% (±2.1%)	8.2% (±1.6)	39.6% (±6.1)
NaCl	22.5% (±3.3)	8.9% (±2.0)	8.1% (±1.6)	39.6% (±5.9)
Ca Sulfate	17.6% (±15.6)	11.0% (±7.9)	10.1% (±4.2)	37.0% (±14.7)
S/NaCl+NaSulf	12.1% (±0.7)	3.4% (±0.4)	3.6% (±0.7)	19.1% (±1.3%)
NaCl	10.6% (±0.6)	3.4% (±0.5)	3.7% (±1.0)	18.1% (±1.9)
Na Sulfate	17.0% (±2.1)	3.3% (±0.2)	2.5% (±1.1)	22.1% (±3.7)
S/NaCl+CaSulf	20.9% (±4.4)	3.6% (±0.8)	4.5% (±2.9)	29.0% (±4.5)
NaCl	20.0% (±4.5)	3.6% (±1.5)	4.5% (±3.0)	27.4% (±4.0)

The percentage of total salts extracted, as well as of each individual salt, for tiles containing mixtures of NaCl with Na₂SO₄ or CaSO₄, is presented in Table 5. The data for CaSO₄ in the stoneware tiles is not presented because the determination of these low amounts of sulfate were at or below the detection limit of the analytical procedure. The percentages are calculated assuming that the amount of each salt introduced in the tile when in mixture is proportional to the concentration of the salts in the saturated solutions at the eutonic point. At these points, the concentrations of the individual salts when in mixture are approximately 5.6 mol/kg for NaCl and 0.6 mol/kg for Na₂SO₄, since the impregnation was carried out close to 25°C [16]; and, 6.2 mol/kg for NaCl and 0.04 mol/kg for CaSO₄ [17]. For the first mixture, the weight ratio of NaCl/Na₂SO₄ is 3.39 (327.6g NaCl / 96.6g Na₂SO₄). For the second mixture, the weight ratio of NaCl/CaSO₄ is 67.2 (362.7g NaCl / 5.4g CaSO₄).

The total amount of salt extracted is less for a mixture than if either salt were by itself. This could be attributed to ion association phenomena, i.e., ion-pairing, which occur in concentrated solutions containing different ions [18].

The extraction of NaCl from the mixtures appears to proceed in a more regular pattern, as shown by the lower standard deviations of the data, which rarely goes above ±6% and in general falls below ±1%, which was the usual reproducibility obtained for individual salts. Sulfates show a much larger dispersion of data, which increases when their amounts are at the detection level of the measuring method, as illustrated by the data for CaSO₄ in the earthenware tiles. The data for the sulfates cannot be compared to that presented in Table 4 for the individual salts, because the washing time was reduced to 1-hour for the mixtures due to the presence of the NaCl. In general, the first wash presents the highest dispersion of data, which can be attributed to the fact that the tiles have different "histories" which result in there being more or less salts concentrated at the surface. The second and third wash gave more reproducible results.

Table 6: Weight ratio of extracted amounts of NaCl/Na₂SO₄ and NaCl/CaSO₄ from tiles containing their mixtures by three successive 1-hour washing with 150-ml water separated by a four-hour partial drying. Numbers in parenthesis give the standard deviation. The theoretical ratio of the salts at their eutonic points is included as reference.

Tile/Salt	1. Wash	2. Wash	3. Wash	Total extr.	Theor. Ratio
E/NaCl+NaSulf.	3.5 (±1.5)	3.9 (±0.9)	3.1 (±0.2)	3.5 (±1.2)	~3.39
E/NaCl+CaSulf.	57.2 (±21.6)	65.3 (±36.6)	53.3 (±28.4)	68.7 (±23.8)	~67.2
S/NaCl+NaSulf.	2.1 (±0.3)	5.0 (±2.6)	6.8 (±5.0)	2.8 (±0.6)	~3.39

The ratio between the amounts of extracted NaCl to Na₂SO₄ or CaSO₄ was also calculated and the averages are presented in Table 6.

The ratios obtained show that the values, within the experimental error, are close to the theoretical value, that is, the ratio at which these two salts are extracted is proportional to their concentration in the ceramic body. In general, less sulfate is extracted, this can be attributed in part to the loss of this salt from efflorescences that occurred during the previous experiments.

4 Conclusions

The present study aimed to determine trends in the desalination of salt-laden porous ceramics and constitutes a first approach to a complex problem. It has shown that for the more soluble salts and the more porous earthenware tiles, repetitive washings are more efficient than a single prolonged immersion. Effectiveness is further increased if the ceramic body is allowed to partially dry between washings. However, the effect of partial drying and its potential damage to the object needs to be further evaluated and, if the procedure is to be used, the most effective drying time needs to be determined.

In the case of mixed salt solutions, it appears that salts are extracted in the same ratio in which they are found within the body if, as in these experiments, the distribution is presumably uniform given the small sizes of the tiles.

Finally, it has been shown that before a decision is taken regarding the best desalination procedure to use, the porosity of the ceramic body and the type of salt present is critical in determining the choice, since it would appear that for low porosity bodies and low solubility salts, e.g., gypsum, a prolonged immersion is more efficient than repeated washings.

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