

Abstract

Efflorescences identified as acetate salts were found on over 150 ceramic objects in a museum in Belgium. The objects were treated by salt concentration reduction. An investigation was undertaken into the exact causes of the damage. Analyses were conducted of efflorescences, wash solutions, air pollutants and display-case materials. The relative humidity and temperature inside and outside the cases were measured over one year. The importance of the location and nature of the salts and implications for the active and preventive conservation of ceramics, or other calcareous museum objects, susceptible to similar damage are discussed.

Keywords

acetate, salt, ceramics, damage, preventive conservation

A collection of ceramics damaged by acetate salts: conservation and investigation into the causes

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Introduction

In 2000, X-ray diffraction (XRD) analysis of efflorescence samples found on ceramic tiles in the collection of the Broelmuseum in Kortrijk, Belgium, revealed the presence of thecotrichite ($\text{Ca}_3(\text{CH}_3\text{COO})_3\text{Cl}(\text{NO}_3)_2 \cdot 7\text{H}_2\text{O}$). The complex acetate salt is believed to have formed on the surface of the tiles after reaction with acetic acid vapours in the display cases (Halsberghe 2001, 2003). In 2001 more dramatic efflorescence growth was noted (Figures 1 and 2) on some objects. A thorough survey revealed damage and efflorescence on 160 ceramic objects of the collection; lead and tin glazed tiles of various origins and dates (Figure 3). XRD analyses, again, revealed mainly thecotrichite.

Damage caused by acid vapours to museum collections containing ceramics, limestone, metals, geological specimens or shells, is a well documented problem (Halsberghe et al. 2005), but damage on such a scale had never been noted in a ceramics collection. The causes of the problem were investigated to determine the appropriate measures for conservation and prevention. This included:

- observation of efflorescences and damage before sampling. The descriptions will aid recognition of similar problems at an early stage
- conservation of the damaged objects
- analyses of efflorescences and wash solutions to identify the ions and determine their origin



Figure 1. Abundant whisker growth (thecotrichite) on Nishapur plate

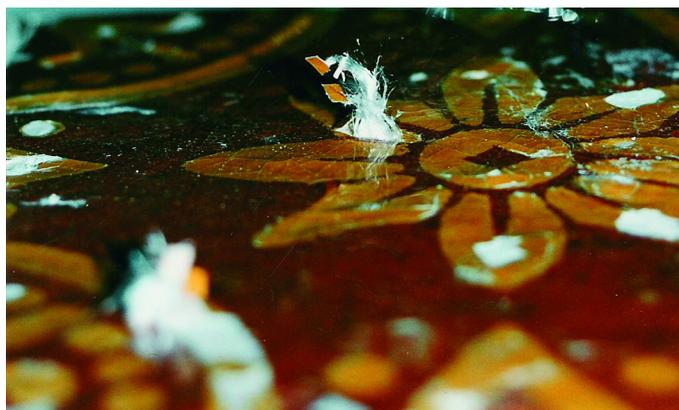


Figure 2. Long bundles of thecotrichite expelling glaze particles on Flemish tile (inv. 5545)



Figure 3. Display cases A–D with Flemish tiles



Figure 4. Thick layers of fluffy efflorescences growing on Dutch tin-glazed tile (inv. 3922). Left of the tile there is a heap of debris, fallen off another tile



Figure 5. Back of tin-glazed tile (inv. 5202) with salt pustules

- determination of the concentration of acetic acid vapours inside the display cases to determine any relationship between pollutant concentration and observed damage
- study of materials used in the cases to locate the source of acid vapours
- monitoring of relative humidity and temperature inside the cases to provide information about conditions in which damage occurs
- formulation of advice for conservation and prevention.

Observation

Not all efflorescences are as obvious as the ones pictured in Figures 1 and 2, so examination under magnification with a binocular microscope is recommended (Halsberghe 2003). The following external signs indicate the presence of damaging salts: flaking of glaze or particles, pitting or powdering of the surface, debris on the shelves under the objects (Figure 4) and fresh lacunae (which are usually distinct from old ones because of the colour). Salt efflorescences come in different shapes (crystal habit) and sizes, which are less an indication of the nature of the salt than of the environmental conditions in which the salts crystallized (Arnold and Zehnder 1991). On the objects studied here, salt crusts, salt pustules (Figure 5 and 6), thick layers of fluffy efflorescences (Figure 4) (composed of many tiny needles or whiskers), and larger bundles of whiskers (Figure 2), up to several centimetres long, were observed.

Sampling

Identification of the salts, or its components, may reveal the source of the efflorescence. Appropriate measures may only be determined when such information is known. Simple tests and analytical methods for the identification of acid induced salts are the subject of another paper in this volume (Halsberghe et al. 2005).

The objects with the efflorescence should be photographed. Abundant whisker growth is best sampled with a brush or spatula onto a clean sheet of paper, then transferred to a glass or polypropylene vial that is appropriately labelled. Most acetate salts are stable during storage if kept cool and dry. In many cases, harder salt pustules have been observed on the surface of the objects. These may be sampled with a scalpel. In the case of tiny quantities of efflorescence, it is advised to collect samples using a binocular microscope.

Conservation

The origin of the acetate ions in the salt was thought to be acetic acid vapours from materials of the showcases. Replacing acidic materials with inert materials like glass or Perspex was the first recommendation. But, as the identity and concentration of the salts remaining in the objects was unknown, the risk of further damage by crystallization remained. Strict control of the relative humidity (RH) in the cases was not feasible. It was not known what level or range of RH for the salt mixtures would be safe, and individual RH control of the display cases was not an option at that time. Salt concentration reduction ('desalination') was therefore the approach chosen. This was done systematically to obtain analytical results.

Several precautionary measures were needed before washing (Halsberghe 2001). Objects and any decoration should be tested for resistance to water. Water sensitive objects require consolidation before any treatment with water. Consolidation was not necessary here, but loose glaze flakes were fixated before washing. Paraloid[®] B72 (7 per cent in acetone-ethanol (85–15 per cent v/v)) was applied to a piece of Japanese paper placed over the loose flakes. This held the flakes in place and allowed the salts to be dissolved. After washing and drying the objects, these zones required fixation again before removing the paper.

The salt concentration was reduced by washing the objects in static baths of demineralized water. After removal of efflorescences the objects were weighed and placed individually in a bath of 2 litre of water. This same quantity of water

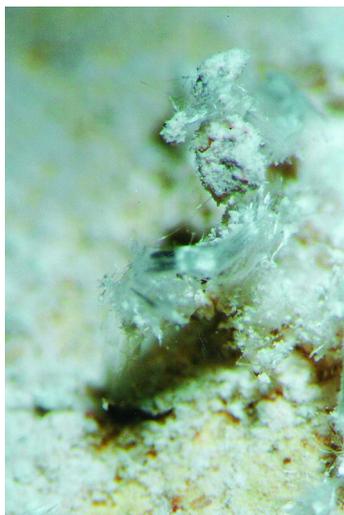


Figure 6. Detail of Figure 4. Under magnification bundles of thecotrichite can be observed. These enclose particles of ceramic

was used for subsequent baths. The progress of the process was monitored by measuring the conductivity of the water with a multi-parameter instrument (pH/Cond 340i by WTW) with a TetraCon 325 conductivity cell. The water was changed at 24 and 48 hours after which time the measured conductivity had decreased significantly. The washing process was halted when the conductivity remained below 100 $\mu\text{S}/\text{cm}$. The objects were dried slowly to constant weight. A 100 ml sample of water was taken from each bath, and refrigerated in small HPDE bottles.

Damaged objects in the storage area

During examination of the remaining tiles in the storage area (the old kitchen cabinets of the historic house), several seriously degraded ceramics were found, some beyond repair.

Of the tin glazed Dutch tiles (inv. 3922), belonging to the same tile panel with biblical scenes, the decorative tiles from the edges of the tile panel and the triangular corner tiles are kept in these kitchen cabinets. Eight triangular tiles, wrapped in pieces of ordinary copy paper, were undamaged. The unprotected tiles showed substantial loss of material by disintegration of the body and spalling of ceramic particles up to several square centimetres wide and several millimetres thick (Figure 7). Several other lead glazed Flemish tiles, many from groups displayed in cases A–D (Table 1) showed severe loss of glaze particles and pitting of the unglazed parts (Figure 8). Efflorescences could not be seen with the eye, but several samples were taken with the aid of a binocular microscope.



Figure 7. Tin-glazed tiles in the storage cabinets (inv. 3922) with very visible destruction caused by halite crystallization



Figure 8. Flemish tiles in storage area. Lead glaze damaged by halite crystallization

Analyses

Difficulties associated with the analyses of acid induced efflorescences, and the comparison of results obtained with various analytical methods, are discussed elsewhere in this volume (Halsberghe et al. 2005). The purpose of the analysis of the efflorescence and wash solution samples was to find out more about the conditions of formation of complex acetate salts like thecotrichite. The exact composition and concentration of the salt species and ions of the efflorescences, as well as the salt solutions (and subfluorescences) inside the objects was of particular interest. The results of analyses by XRD, Fourier transform infrared spectroscopy (FTIR), ion chromatography (IC) and atomic emission spectrometry (AES) are in Tables 2–4.

The predominant salt species in the efflorescences of the objects on display is thecotrichite. Halite is also found, mainly on the Bible tiles in display case F. In one case, a tiny efflorescence sample was identified as nitratine. This display case is exposed to direct sunlight in the evening. The calcite and quartz identified with XRD are particles of the ceramic body mixed in with the efflorescences. The efflorescences in the storage area are all halite. This is consistent with the results

Table 1. Description of damaged objects

Display case	Number	Object	Origin	Date (century)	Measurements	Efflorescence and damage
Nishapur case	1	Round dish, slip decoration and lead glaze, inv. 5363.	Persia, Nishapur	12	Round, Ø 20.5 cm	Long whiskers on half of the upper surface, a few bundles at the back. Numerous glaze and terracotta particles missing, glaze is crizzled.
A	17	Lead glazed tiles, inv. 5594, 5534 and 5597.	Flanders	18	Square, sides between 135 and 149 mm	Lifting of glaze particles, lacunae. Salt pustules and small whiskers.
B	13	Lead glazed tiles, inv. 5595 and 5555.	Flanders	18	Square, sides between 143 and 150 mm	Efflorescences in lacunae at the front and sides, plenty of glaze particles on bottom of the shelf.
B	10	Lead glazed tiles, inv. 5559 and 5545.	Flanders	19	Square, sides between 135 and 142 mm	Lifting of glaze particles, visible and spectacularly long whisker bundles (inv. 5545).
C	22	Lead glazed tiles, inv. 5576, 5571 and 5550.	Flanders	18	Square, sides between 126 and 130 mm	Fresh lacunae, lifted glaze particles, visible efflorescences.
D	2	Lead glazed floor tile fragments, inv. 1006.	Tournai	14		Pitting of the front surface and back. Tiny efflorescences visible with binocular microscope.
D	2	Lead glazed tiles, inv. 1622 and 1598.	Flanders	15	Square, sides between 50 and 65 mm	Efflorescences and fresh lacunae.
D	1	Lead glazed tile, inv. 1281.	Flanders	16	Square, side 150 mm	Fresh lacunae.
D	1	Lead glazed hearth tile, inv. 1255.	Germany	17	Rectangular, sides 90 mm × 130 mm	Efflorescences at the unglazed back side, including tiny particles of terracotta.
D	1	Lead glazed tile fragment, inv. 3556.	Flanders	17	Square, sides 100 mm × 100 mm	Efflorescences and fresh lacunae.
D	11	Lead glazed tiles, inv. 5540, 5713, 3451, 3449, 3446, 1610, 3631 and 3958.	Flanders	18	Square, sides between 110 and 145 mm	Salt pustules, tiny whiskers, fresh lacunae with efflorescences.
D	1	Lead glazed tile, inv. 7106.	Flanders	19	Square, side 139 mm	Efflorescences and fresh lacunae.
E	5	Tin glazed tiles, inv. 1229, 1192, 5143, 5492 and 5142.	Holland	17	Square, sides between 127 and 130 mm	Efflorescences at the unglazed sides, including tiny particles of terracotta.
E	1	Tin glazed tile, inv. 5378.	N. France	18	Square, side 125 mm	Abundant efflorescence growth including tiny particles of terracotta.
E	3	Tin glazed tiles, inv. 5202, 5377 and 5376.	N. France	19	Square, sides between 123 and 130 mm	Efflorescences at the unglazed back sides, including tiny particles of terracotta.
F	72	Tin glazed tiles with biblical scenes, inv. 3922.	Holland	18	Square, sides 130 mm	The unglazed sides of most tiles is covered with a thick layer of fluffy efflorescences, many particles of the terracotta body and some of the glaze have spalled, some up to several square centimetres! Debris is found on the shelves.

Table 2. Results of ion chromatography analysis of some samples

Object	Inv.	IC of the wash solutions: total salt extracted in mg						IC of efflorescences: mg/g sample								
		Cl ⁻	NO ₃ ⁻	SO ₄ ²⁻	Na ⁺	K ⁺	Mg ⁺	Ca ²⁺	CH ₃ COO ⁻	Cl ⁻	NO ₃ ⁻	SO ₄ ⁻	Na ⁺	K ⁺	Mg ⁺	Ca ²⁺
1. Objects in display cases																
Nishapur vase, with lead glaze (LG)	5363	988	8195	806	646	619	279	2322	216	49	270	0	2	2	1	161
LG Tile Kf1	5694	632	4825	133	326	157	115	1470	232	50	273	0	1	1	0	167
Tile B1 with tin glaze (TG)	3922	1278	2320	62	433	262	80	1150	140	59	111	11	19	—	—	101
TG tile B2	3922	1460	2171	58	470	198	58	1225	114	51	82	0	25	—	—	74
TG tile B3	3922	1485	2657	53	456	285	61	1243	90	54	68	0	18	—	—	65
TG tile B4	3922	1590	2914	70	524	364	82	1324	163	51	126	6	12	—	—	112
TG tile B5	3922	1686	2822	109	548	372	79	1229	162	75	101	0	36	—	—	90
2. For comparison: objects kept in the storage area																
LG tile	5542	—	—	—	—	—	—	—	0	584	0	—	—	—	—	—
TG tile	9145	—	—	—	—	—	—	—	0	650	0	—	—	—	—	—
3. For comparison: Object from other museum with efflorescences identified as calcilicite																
Cyprus vase	NS 80	798	<1.7	61	82	53	42	478	229	173	0	0	15	1	1	143

— Not analysed. The Nishapur plate and the Cyprus vase had plaster fillings that were not removed before washing, hence the sulphates.

of the IC of the efflorescences, where the concentrations of the identified ions reflect the composition of the cotrichite, and halite for the Bible tiles B1–B5. Analyses of the wash solutions proved interesting. No acetates were found, but high concentrations of nitrates and chlorides were found in all objects. Calcium was the prevalent cation, with some sodium and potassium also present. These

Table 3. Other results of analyses

Sample	Display case	Object inv.	X-ray diffraction	FTIR	Polarizing light microscopy
1. Objects in display cases					
Nishapur plate	Nishapur case	5363	thecotrichite	thecotrichite	thecotrichite
LG tile	B	5595	—	—	nitratine (NaNO ₃)
LG tile	D	5713	—	—	thecotrichite
LG tile	D	1610	thecotrichite, calcite	thecotrichite	thecotrichite
TG tile	E	5378	—	—	thecotrichite
TG tile	E	5202	thecotrichite	thecotrichite	thecotrichite
TG tiles (various)	F	3922	thecotrichite, halite, calcite, quartz	thecotrichite	thecotrichite
2. For comparison: objects kept in the storage area					
LG tile	5542	—	—	halite	
TG tile	9145	halite	—	halite	
LG tile	5589	halite	—	halite	
3. For comparison: object from other museum with efflorescences identified as calclacite					
Cyprus vase	NS 80	calclacite, quartz	calclacite	calclacite	

Table 4. Analysis results (IC and AES) on wash solutions of tiles of one tile panel (inv. 3922), conserved in three different environments

Tile number	Total mass of ions extracted per tile (mg)						Average concentration of ions per group (mg)						Average mass of extracted salt per tile		
	Cl ⁻	NO ₃ ³⁻	SO ₄ ²⁻	Na ⁺	K ⁺	Mg ⁺	Ca ²⁺	Cl ⁻	NO ₃ ³⁻	SO ₄ ²⁻	Na ⁺	K ⁺		Mg ⁺	Ca ²⁺
Group 1: tiles on display in display case F															
B1	1278	2320	62	433	262	80	1150	1500	2577	70	486	296	72	1234	6.2
B2	1460	2171	58	470	198	58	1225								
B3	1485	2657	53	456	285	61	1243								
B4	1590	2914	70	524	364	82	1324								
B5	1686	2822	109	548	372	79	1229								
Group 2: tiles conserved in the storage cabinets, uncovered															
V1	1467	2460	71	630			987	1495	2806	89	716	n.a.	n.a.	1044	6.2
V2	1600	3144	69	724			1147								
V3	1402	2831	118	825			869								
V4	1326	2242	67	537			1044								
V5	1682	3354	122	863			1176								
Group 3: triangular tiles conserved in the storage cabinets, wrapped in copy paper															
H1 and 2	1091	2076	104	590			804	1094	2051	167	607			808	4.7
H3 and 4	1097	2027	230	624			812								

ions originate from the previous history or use, possibly during burial or attachment to non-isolated walls, where the salts accumulated as absorbed groundwater evaporated. Some calcium comes from the calcite in the object dissolved by the acid. The analyses also showed that three washings were more than sufficient to remove most of the salts. The presence of sulphates in the wash solutions is mainly due to the presence of plaster, as filling on the Nishapur plate and as mortar on the tiles.

The only salt species found on the objects in the storage area is halite. Even if these objects contain the same salt solutions as the ones in the display cases in the museum and small concentrations of acetic acid were measured in that environment (see below), no traces of acetates or nitrates were found in the efflorescences.

Gaseous air pollutants inside the showcases

The acetic acid vapour responsible for the acetate ions present in salts within the objects was suspected to come from the storage environment of the objects because there were many objects with simultaneous damage, the objects originated from several different places and times, and organic acids had already been detected in environments where other objects damaged by thecotrichite were found (Grzywacz and Tennent 1994).

In 2003 the concentrations of different gaseous pollutants were measured in several display cases and halls of the museum, in collaboration with Velichka Kontozova of the University of Antwerp, Belgium. Other researchers repeated

some measurements several months later. Discussions of these comparative results are published elsewhere (van Bommel et al. 2005).

Outside the display cases the measured concentrations of acetic acid vapour were negligible. However, concentrations inside the cases were elevated, implying that the source of the acid was within the case. The highest concentrations, 358–467 ppbv, were in cases with the Flemish tiles (A–D). The lower concentrations (139–249 ppbv) were in cases with more porous objects, the Nishapur and the Bible tiles. These objects also have a higher concentration of calcium carbonate. Calcium carbonate (up to more than 20 per cent) is added to clay used for tin glazed ceramics to improve glaze adhesion. Simultaneously, measurements of concentrations in display cases with non-porous porcelain objects were conducted. These cases are identical to the Nishapur display case. The concentrations of acetic acid were 13–33 per cent higher.

In the storage area and kitchen cabinets, the concentrations measured were low (around 60 ppbv) and identical, most probably because the cabinets do not close well and air is free to circulate.

Materials of the cases

The museum obtained new display cases in 1988. The frame of the display cases is black metal, in which glass panels (top and four sides) rest. The bottom shelf and the backing panels are made of plywood covered with a textile-reinforced plasticized PVC material, surface treated with an acrylic polymer. Plywood is a known source of acetic acid. PVC is known to release gaseous hydrochloric acid as it degrades. This was not measured, but this material may have contributed to the damage.

The environment

For a period of one year, five dataloggers (Elsac 764 Environmental Monitor) were used to monitor the environment in three display cases where damage was found, in the museum hall and in the storage area.

The glass museum cases were found to buffer changes in RH and temperature occurring in the unconditioned museum galleries, even in very hot and humid conditions such as the summer of 2003. Significant changes take place inside only if the change outside persists over a long period.

The RH inside the cases varied slowly within a range of 40–60 per cent. Temperatures were between 20 and 23 °C most of the year, with an increase to 29–30 °C in summer.

The conditions in the storage area were much more humid, varying in the range 45–75 per cent RH.

Discussion

The analyses of the wash solutions reveal that the efflorescences represent only a tiny fraction of the total amount of salt (either in solution or in subfluorescence) in the objects, even if the objects (inv. 3922, B1–B5) had a massive amount of efflorescence. If the objects are left in the same environmental conditions, this salt reserve presents a ticking time bomb. Theoretical studies of the formation of thecotrichite (Gibson et al. 2005, Linnow et al. 2005) show that a small amount of acid depositing on the objects and mixing with the salt solutions already present causes the formation of this complex salt. The monitoring has shown that this not only happens in humid RH conditions, but in 'normal' museum RH conditions, between 40 and 60 per cent RH. Removal of the sources of acetic acid is essential for the safety of ceramics containing calcium carbonate or contaminated with soluble salts. This is also true for metal objects or objects containing calcium carbonate, such as limestone, shells, or coral, as stated previously (see for instance Padfield et al. 1982; others are cited in Halsberghe et al. 2005). We do not know yet (and probably never will) what concentration of acetic acid is generally safe, for a number of reasons. The concentration of acid does not necessarily indicate the amount of acid being produced if reactive

materials or objects are present, because these act as a sink for the acid. The efflorescence growth is not instant, but, as has been observed here and in other museums, usually takes years. The concentration of acid in the air varies with the environmental conditions and over time, as new materials will release more in the beginning. Reducing the acid concentration by ventilation of the cases may not be an ideal solution if the museum environment is not adequately controlled, because large prolonged RH fluctuations may cause crystallization of the salt solutions in the objects. Alternatively, the use of absorbents has been suggested (Brokerhof 1998). This may help if absorbents are effectively placed and present an adequate surface area and if renewal of the acid absorbent materials is regular. Realistically, such a regimen is difficult to maintain, but may be an interim measure until better solutions are implemented.

The comparative study of the efflorescence and wash solutions has confirmed theory (Gibson et al. 2005, Linnow et al. 2005): the identity of the efflorescence provides a good indication of ions present in the objects. When calclacite forms as efflorescence, chloride and calcium are the major ions in the object and sodium is also likely present. Calclacite is often found on objects of Greek or Cypriot origin. Thecotrichite will form when nitrates are also present. Such is the case for tiles, and objects excavated from soils or tombs rich in nitrates.

When the environment is free of acid, sodium salts may still precipitate. In salt mixtures composed of sodium, chloride, and nitrate this may happen in the range of 30–60 per cent RH (Linnow et al. 2005). Damage due to sodium chloride was found only on the objects conserved in the humid storage area, where fluctuations between 60 and 70 per cent RH were common. Inside the showcases, with RH well below 60 per cent, halite efflorescences were found only in small quantities and only in the presence of acetate salts.

Calcium chloride and nitrate are very deliquescent salts that will not crystallize above 30 per cent RH. Being hygroscopic, they provide a liquid solution in which the acid is readily absorbed. This acid then reacts with the salt solutions and calcium carbonate of the objects and forms salts that precipitate before the acid has a chance to diffuse into the body of the object. That is why the wash waters contain very little acetate. If acid is excluded from the environment, the risk of damage by crystallization of the remaining salts is minimal if the objects are kept in moderately stable RH conditions (40–60 per cent). Sealed glass or acrylic cases that contain no source of acid and that are not exposed to direct sunlight generally provide such an environment. The salt solutions in the objects actually act as buffers in such situations. In this way, drastic treatments such as salt concentration reduction may be avoided. Prolonged soaking may cause weight losses of up to 5–10 per cent, as was noted after treatment of the Bible tiles.

Conclusion

Objects containing calcium carbonate, and in particular those contaminated with soluble salts, should be kept in environments free of acetic acid vapours, as acetic acid will dissolve part of the calcareous components and form complex acetate salts that precipitate and cause further damage even in stable conditions around 50 per cent RH. Well sealed, acid-free cases are recommended for the storage and display of such objects.

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