CONSERVATION TREATMENT OF ENCRUSTED CERAMICS

by

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□ ABSTRACT

An encrustation obscuring the surface of sherds from the Trants site inhibits analysis of the decoration and coloration of the Saladoid ceramics. A treatment using a weak concentration of hydrochloric acid (HCl) dissolves the calcareous encrustation and renders the design elements visible for the analyst. No flaking, discoloration of slip or paint, or other detrimental effects have been observed on the surfaces of treated ceramics. This paper discusses safety measures required when using HCl, the procedures used to determine if the encrustation is soluble in HCl, and the results of an analytical report. Constraints of the treatment must be considered beforehand because HCl adversely affects some pottery constituents (shell temper) and may similarly affect others (residues), thereby potentially invalidating analyses performed subsequently.

Resumen

La incrustación que oculta la superficie de los tiestos del sitio Trants impide el análisis de la decoración y coloración de las cerámicas Saladoides. El tratamiento con una concentración débil de ácido hidroclórico (HCl) disuelve la incrustación calcárea y hace visibles para el analista los elementos del diseño. No se ha observado descascaramiento, decoloración del engobe o la pintura, u otros efectos perjudiciales sobre la superficie de las cerámicas tratadas. Este artículo discute las medidas de seguridad requeridas cuando se usa HCl, los procedimientos usados para determinar si la incrustación es soluble en el HCl, y los resultados del informe analítico. Se deben considerar de antemano los constreñimientos del tratamiento, porque el HCl afecta adversamente algunos constituyentes de la cerámica (desgrasante de conchas) y puede afectar de manera similar a otros (residuos), invalidando potentcialmente de ésta manera los análisis llevados a cabo posteriormente.

□ INTRODUCTION

This paper discusses a treatment to remove insoluble encrustations adhering to ceramic fragments from the Trants site (MS-G1), Montserrat. A compact and tightly-adhering layer of insolubles was present on the exterior and interior surfaces of many of the Trants ceramics. The encrustation covered fully the sherd's surfaces (including broken edges) in extreme cases. The encrustation was insoluble in water, and it still adhered to the sherds even though they had been field cleaned of dirt by washing in water and using soft brushes and hand action. No detergents had been applied. Field efforts to remove the encrustation by mechanical means (scraping with a metal implement) were found to be unsatisfactory because they resulted in scoring of the sherd's surface.

Gardner, the Conservator for Carnegie Museum of Natural History, examined the encrusted sherds after Watters, the excavator, apprised her of the problem. The encrustation obscured the designs on incised pottery and the designs on and colors of painted ceramics; it even masked surfaces of unpainted pottery (Figures 1-3). Designs and painting were concealed totally in the most extreme cases, where the encrustation was thick and dense (Figure 4). It is important to understand that the pottery from Trants generally is hard and well fired. It contained no pieces of unbaked clay and no sherds with fugitive paints (dissolvable in water). Poorly fired, soft pottery (such as griddle sherds) would require a different kind of treatment.

Gardner's prior experience in treating encrusted materials from archaeological sites allowed her to identify provisionally the layer of insolubles as being calcareous in nature. Gardner decided to test hydrochloric acid (HCl) for its applicability to the encrusted sherds from Trants because calcareous encrustations react to that acid. She had used the HCl on ceramics from the Near East during her training at the Smithsonian Institution.

Conservators and some archaeologists know that insoluble encrustations of calcium carbonates or calcium sulphates, which form on the surfaces of ceramics buried in soils derived from limestone or containing lime, can be removed with acids (Hodges 1987). Our testing confirmed that hydrochloric acid was very effective on the Trants ceramics, so we did not test other agents to determine their effectiveness in removing the encrustation. However, we do recommend such testing since other agents may also perform well. Those used most commonly are hydrochloric acid, nitric acid, and the organic acids — oxalic acid, citric acid and acetic acid (Buys and Oakley 1993; Sease 1987). Should the treatment with HCl not have worked or been inadequate for the problem posed by the Trants sherds, other procedures would have been tried using these other acids. The Conservation and Restoration of Ceramics by Buys and Oakley (1993), provides a very good series of tests to be conducted before using any acid treatment on pottery.

The treatment was performed initially by Watters in 1992 on sherds from the 1990 excavations at Trants. Brown processed many more sherds obtained during the 1995 Trants project, and recent reports about the Trants ceramics (Reed 1999; Reed and Petersen 1999) are based on those treated sherds.

THE TREATMENT

We had an effective treatment, the proper laboratory with appropriate materials at hand, and therefore proceeded with the project. Small insignificant sherds initially were tested to determine the effectiveness of the treatment and to assure that the pottery did not have a calcareous temper (e.g., shell or limestone temper). Calcareous temper is drastically weakened by hydrochloric acid, and could cause the pottery to disintegrate.

Most workers (Sease 1987) state that the acid bath should be dilute, not exceeding 5 percent. Too high of a concentration will over-clean the pottery and produce a raw, porous surface, or so soften the surface that subsequent cleaning could leave brush marks. Acid cleaning is a harsh treatment on even strong pottery and should be used only when necessary. The diluted hydrochloric acid we used is designated 2N or 2 normal. A conservation chemist recommended the 2N grade to Gardner many years ago, and other chemists have affirmed this is probably the most used grade of hydrochloric acid in conservation labs. We had tested the 1N (one normal) grade earlier in the project, to ascertain whether this more dilute solution would react with the encrustation on the Trants pottery. It did not. When we tested the 2N HCl, we were rewarded immediately with a bubbling reaction and the dissolving away of the accretion.

The procedures we followed in this treatment are presented in a flow chart (Figure 5) on which are shown the stages where decisions (posed as questions followed by "yes" or "no") must be made in the process. There are several parts of the flow chart that warrant discussion. The sherd should be immersed in water to thoroughly wet its paste all the way through, before being placed in hydrochloric acid. A saturated sherd will retard the entry of HCl (and dilute what does enter) into the interstices of the paste, where it is not needed. The sherd is encrusted on its surface, not within its paste. One conservator (Sease 1987: 96) recommends that the sherds be placed in water for an hour before the acid treatment, and also recommends tapping or shaking the container to release air bubbles trapped in the paste.

Bubbling and frothing will emanate from the encrustation as soon as the sherd is immersed in HCl. Bubbling persists until an encrustation is dissolved away or the reaction stops because the acid is depleted (has lost its strength) in which case it must be replenished. Special attention should be given to a stream of small bubbles rising from the sherd because that can indicate reaction by calcareous temper. However, a stream of bubbles also may be simply the displacement of trapped air in the matrix of a poorly saturated sherd. As long as ten minutes in HCl may be necessary to dissolve the most heavily encrusted sherds, but relatively few Trants sherds required this length of time. It is preferable to immerse the sherd several times for short periods than leaving it immersed in acid for an extended period. A medicine dropper with dilute HCl may be used on localized spots of encrustation rather than placing the entire sherd in the acid bath.

After treatment, it is important to remove all traces of HCl by dilution in water. If the acid is not removed totally, it can cause dissolution of the sherd over time. Immediately upon being removed from the HCl, a sherd should be rinsed in the first water bath for about two minutes followed by a second water bath rinse of five to ten minutes, to flush acid concentrated on the sherd's surface. Thereafter, a sherd is put into the third bath, the soak bath, in which the water is changed completely at least four times at intervals of about 12 hours. With two rinse baths and four soak baths, each sherd undergoes six separate changes of water, and no harm is done by even more rinses or soaks. The containers we used for rinses and baths held generous amounts of standing water. Some conservators advocate leaving treated ceramics in slowly running water for a period of hours. As a general rule, you cannot rinse too much, and you want to ensure that all acid residue is entirely flushed from the interstices within the body of the sherd.

After completing the final stage of the soak bath, the sherd is placed on a plastic tray lined with absorbent paper towels to wick away the moisture. Provenience data for unlabeled pottery must be carefully monitored throughout the treatment; we found that plastic bags with these data sheets could be readily clipped to the various containers used in the process (Figure 6). The sherd is allowed to air dry gradually; it should not be exposed to direct sunlight.

Additional information about the procedures outlined on the flow chart is provided in Endnotes to this paper.

□ SET UP AND SAFETY

The tables in the research area were useful for initially sorting the sherds, holding the containers for the extended soak baths, and drying the treated pottery. The HCl treatment was performed in a separate area on counters near sinks, where a water supply was readily available (Figure 7). The immersion of sherds in HCl was conducted exclusively in glass beakers and glass dishes, although sherds waiting to be processed were held on plastic trays (Figure 8). A full face shield protected the preparator's face and a forceps were used to place sherds in the acid (Figure 9).

Safety precautions when using hydrochloric acid MUST be observed at all times. It should be used in a laboratory where proper equipment and supervision are available, and where there is an adequate supply of water. Hydrochloric acid should not be used in a field situation.

Safety precautions to be observed when performing this treatment or whenever using hydrochloric acid include:

(1). Before initiating any treatment, become thoroughly familiar with the information provided in the Materials Safety Data Sheet (MSDS) for hydrochloric acid (MSDS is the standard fact sheet available within the United States).

(2). Use small containers of acid when pouring into glassware. They are less cumbersome than a large and heavy bottle and thus decrease the chance of an accident. Remember that accidents require immediate emergency attention, lots of water, and can entail liability and insurance issues.

(3). Always wear protective clothing, a chemical splash goggle or face shield, and thick rubber gloves (Commoner 1984:153-158 lists acceptable kinds of gloves to use).

(4). We recommend using hydrochloric acid already diluted to the appropriate grade (2N). If you find you must dilute a stronger grade, always add the acid to the water, NEVER add water to the concentrated acid. Adding water to acid produces large amounts of heat and can cause sputtering and spitting of the acid. Add acid to water slowly and stir continuously to dissipate the heat that will be generated.

(5). Be sure to have bicarbonate of soda (clearly labeled) readily available as the antidote and a source of water for flushing acid spills. Avoid getting acid on clothing or your skin as serious burns can result. If this occurs, immediately flush the area with copious amounts of water and then rinse the area with a dilute solution of bicarbonate of soda. Acid should be used only in close proximity to a water source.

(6). Use hydrochloric or any other acid in a well-ventilated area. Do not inhale the fumes, which can cause serious damage to the eyes, nose, throat, and lungs (McCann 1979:188, 196, 225).

(7). Dispose of used acid in a safe place after diluting it thoroughly with water.

OTHER ANALYSES

It may prove necessary to measure the removal of soluble salts from the treated ceramics. During the HCl treatment, the insoluble carbonates are converted into a soluble chloride that can be easily washed away, such that the process then becomes one of desalination or salts removal (The Conservation Unit of the Museums & Galleries Commission 1992: 94-95). The Anthropology Conservation Lab uses a YSI Model 33 conductivity meter that measures in micro mhos (other meters measure parts per million). The decrease in conductivity resulting from successive washes and soaks is a measure of the degree of removal of soluble salts from treated objects. Other options include use of EM Quant Test Strips by the Merck Company or a silver nitrate test. It is important to establish a baseline reading from the tap water you are using. Paterakis (1987) has a comprehensive discussion of the removal of soluble salts from ceramics and the various ways to monitor their removal.

We submitted two samples of the encrustation to Dr. Richard Newman, research scientist and geologist at the Museum of Fine Arts in Boston. The samples provided virtually identical results (Newman 1999). In both samples, calcite (calcium carbonate) and quartz were detected using FTIR microspectrometry and X-ray diffraction. Analysis by X-ray fluorescence in an electron beam microprobe detected calcium as the major element; smaller amounts of silicon, aluminum, phosphorous, magnesium and iron were also found. The microprobe analyses imply that in addition to the major compounds of calcite and quartz, other compounds are also present in the encrustation, possibly including a clay, iron oxide(s), and a phosphate (possibly calcium phosphate).

Newman (1999) also analyzed red and white paint samples from three Trants sherds, two which had been treated with HCl. No analytical difference was detected between treated and untreated sherds. FTIR microspectrometry consistently detected silica and quartz in the three white samples but only silica in the three red samples. The microprobe analyses indicate that the whites were probably made from a calcareous clay containing some quartz. The reds are colored by iron oxide (hematite).

One unexpected result of these analyses is Newman's (1999) deduction that the red and white samples are fired glazes, not paints. Caribbean archaeologists almost invariably refer to such materials as paint, and therefore we present Newman's reasons for characterizing them as glazes. He states:

"The 'silica' detected by FTIR analysis in all of the samples, whites as well as reds, has a spectrum typical of glass. This, coupled with the lack of any more than a trace of any clay mineral(s) in the samples, implies that the red and white colors are fired glazes, not paints. The small solid pieces that were analyzed in the microprobe had rather porous structures, which suggests that they were not fired at extremely high temperatures" (Newman, 1999:1).

TO TREAT OR NOT

Removal of the encrustation by hydrochloric acid certainly increases "visibility" of the sherd's designs and colors for the ceramic analyst (Figures 1-4). It also enhances the analyst's ability to study the temper and paste visible on the edges of sherds. However, the treatment we performed is time consuming and hydrochloric acid is not inexpensive. When considering whether to perform the treatment, one should evaluate the enhanced visibility factor against the expenditure of time and money in reaching a decision. One also must consider ahead of time the detrimental aspects the treatment will have on certain analyses. The dissolving of calcareous temper is one constraint to be considered. Analyses of residues, especially organic ones, adhering to sherds will be precluded or at least impeded by the HCl treatment.

Our general recommendations would be to treat only those sherds on which painting, incising, or other decorative elements are so obscured by the encrustation as to prevent or seriously hinder their analysis, and those sherds having heavily encrusted edges that preclude observation and analysis. Treating ceramics planned for display in a museum exhibit likewise is warranted. We realize that our recommendations are conservative in that we advocate the treatment of only certain categories of sherds. We also recognize, however, that the ceramic analyst, who is faced with examining the complete sherd assemblage, may disagree with our recommendations and may want all of the sherds to be treated! We first treated sherds from Trants with hydrochloric acid seven years ago. To date, none of the initially treated pottery has physically deteriorated or shows any other adverse effects from the

HCl treatment.

One objective of this paper was to provide readers with a better understanding of the procedures involved in evaluating whether archaeological materials require conservation treatment. Although our focus was ceramics, it is clear that an assessment of conservation needs can pertain equally well to other artifact categories. Yet, discussion about conservation concerns is rare among Caribbean archaeologists and publications on conservation practices for Caribbean archaeological materials are almost non-existent, with only a few exceptions (Dittert et al. 1980; Sipe et al. 1980). We regard this situation as especially troubling in view of Cummins' (1993:36-45) findings about the rapid growth of archaeological collections in Caribbean museums that, all too often, have but limited access to expertise in the conservation field.

D ENDNOTES

(1). Tools necessary: Face shield, glass dishes and beakers for acid (Pyrex® brand used in the study), forceps for handling sherds in acid, soft brushes, and at least three trays, two for rinse water baths and one for the soak bath.

(2) HCl does not affect labels composed of white gesso, ink, and acrylic resin (ACRYLOID B72).

(3) Carbonate temper can react with HCl; be wary of thin lines of streaming bubbles emanating from the sherd after the encrustation has been removed.

(4) An oily film or coating sometimes appeared (not commonly) on the surface of the first rinse bath; this may have resulted from the reaction of organic residues on certain of the sherds.

(5) About 10-25 artifacts can be processed per hour depending on the denseness of the encrustation and the sizes of the sherds.

(6) Do not mix sherds from different proveniences in rinse and soak baths; use additional trays to segregate by provenience.

(7) Change first and second rinse baths frequently as residual dirt and sand will accumulate.

(8) Work with acids only in a controlled environment such as lab; it is too dangerous to be done in the field setting.

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¬FIGURE CAPTIONS

- Figure 1. Painted sherds before (left) and after (right) treatment.
- Figure 2. Painted sherds (some incised) before and after treatment.
- Figure 3. Incised sherd (top) and undecorated sherds before and after treatment. Note the persistence of the labels.
- Figure 4. Undecorated ceramic disk before and after treatment. Painted sherds (middle and bottom) on which designs are concealed before treatment. Sherd on right in bottom row not treated in either photograph.
- Figure 5. Flow chart of the HCl treatment process.
- Figure 6. Processed sherds drying (note clipped provenience tag).
- Figure 7. Arrangement for processing sherds (acid use sector to right, water and sinks in center, rinse bath to left).
- Figure 8. Glass dishes and beakers for holding acid (left) and plastic trays (right) for sherds awaiting processing.
- Figure 9. The full face shield protection for the person performing the treatment and the use of forceps to gently lower the sherds into the hydrochloric acid.







Figure 1. Painted sherds before (left) and after (right) treatment.







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Figure 4. Undecorated ceramic disk before and after treatment. Painted sherds (middle and bottom) on which designs are concealed before treatment. Sherd on right in bottom row not treated in either photograph.

Flow Chart

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Figure 5. Flow chart of the HCl treatment process.

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Figure 6. Processed sherds drying (note clipped provenience tag).



Figure 7. Arrangement for processing sherds (acid use sector to right, water and sinks in center, rinse bath to left).



Figure 8 Glass dishes and beakers for holding acid (left) and plastic trays (right) for sherds awaiting processing.



Figure 9. The full face shield protection for the person performing the treatment and the use of forceps to gently lower the sherds into the hydrochloric acid.