WELL, THAT DIDN’T WORK, NOW WHAT? STAIN REDUCTION ON A 10TH CENTURY IRANIAN CERAMIC

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This article presents the treatment of an Iranian 10th century dish from the Freer Gallery of Art in Washington, DC. The dish bore disfiguring stains along most of the joins and, as a result of its appearance, had not been on view since 1982. An investigation of the stains and subsequent treatment was undertaken to reduce their visual impact. Analyses were conducted in order to identify the nature and source of the staining. Although a specific material was not identified, analyses provided clear indications that the stains were caused by earlier treatments. Various methods of stain reduction were investigated. Of the methods deemed safe for the object, none was completely effective at removing the staining, but one reduced it somewhat. Thus, the ceramic was treated in order to obtain a consistent appearance. After the sherds were joined back together and the losses and cracks were filled, several approaches to loss compensation were considered, from different levels of integration of the stained areas to covering the most disfigured ones. A dialogue with the curator and several conservators led to the decision to paint over the stained areas to allow for the display of the object and for the public and scholars to appreciate it.

KEYWORDS: Ceramic, Stain, Stain reduction, Stain removal, Chelator, Bleach, Inpainting, Loss compensation, Sodium citrate, Carbamide peroxide, Agarose, Gel, Poultice

1. INTRODUCTION

The Smithsonian’s Freer Gallery of Art and the Arthur M. Sackler Gallery (Freer|Sackler) in Washington, DC together form the national museum of Asian arts. The collection consists of the arts of both East and West Asia, including Islamic art. An Iranian 10th century dish in the Freer collection was badly stained along its joins, leaving a distracting and disfiguring appearance that limited its ability to be exhibited (fig. 1).

Indeed, the curator for Islamic Art had been asking for several years whether the staining could be removed or reduced, but the stain reduction treatment was expected to be complex and time-consuming. For this reason, treatment had not yet been undertaken. However, conservation of the dish was made possible by a two-year fellowship that was funded by the Hagop Kevorkian Fund and dedicated to the conservation of Islamic ceramics. The dish was one of the highlights of this fellowship.

2. DESCRIPTION

The object is a shallow, wide-rimmed dish of about 43 cm (17 in.) in diameter. The reddish-buff and porous body is covered completely with a white slip. A transparent, lead-based glaze, characterized qualitatively using portable XRF analysis, is applied on the whole ceramic except for the foot. The only decoration is a line of Kufic inscription that runs horizontally across the dish, from rim to rim, and two letters, at the top and bottom. The inscription is executed in brownish-black slip, except for three letters in red slip.

3. CONDITION

The object was broken into 40 sherds and displayed only a few small losses. Glaze losses were present along many of the breaks and on the rim, as well as some chips of glaze and body. Losses, chips, and glaze
losses represent approximately 5% of missing original surface. The dish was joined, filled, and inpainted, but the appearance of the repairs was not satisfactory. The staining that prompted the current treatment of the object is light- to medium-dark-brown and located along most of the break lines. The brown stain lines do not start right at the edge of the sherds but rather one to a few millimeters in from the edge. On several sherds, there is a lighter-brown line of staining a couple of millimeters further in from the edge, and even a third line in a few cases (fig. 2).

Other areas of staining in addition to the edges are located across some, but not all, of the sherds. In general, this staining is more yellow and more diffuse than the staining on the edges. Finally, a few areas show dark-gray staining.

Light iridescence of the glaze was observed on several of the sherds and a few show a more matte and rough surface that indicates some chemical degradation of the glaze, which could be due to an acidic burial environment or to the composition of the glaze. The surface of the brown inscriptions has a matte and whitish appearance, also likely due to chemical degradation. The degradation noted appeared to be stable.

The glaze has craquelure overall and many very small pin holes, both most likely due to the firing process of the ceramic. Overall the surface of the ceramic is relatively clean of grime or dust. On the underside of
the unglazed foot, however, a reddish-brown accretion is present that appears to be iron based and possibly burial related (fig. 3).

4. PREVIOUS TREATMENTS

A review of the conservation records and the removal of restoration materials were helpful in tracing a history of successive campaigns of repair, at least four. During a first campaign, sometime after excavation of the object, the sherds were joined with an unknown adhesive that later started to discolor with age.

Fig. 2. Detail before treatment, dark lines of staining, secondary lighter-brown lines, and third lines are indicated by arrows (from the bottom up). (Courtesy of the Freer Gallery of Art and Arthur M. Sackler Gallery, photograph by Claire Cuyaubère)
It appears that a second intervention attempted to remove this first adhesive; quite probably, the use of a solvent caused the adhesive to migrate into the porous sherds. Further attempts at cleaning adhesive residues seem to have pushed them further in, up to a few millimeters away from the sherds’ edges. The sherds were then joined using a second adhesive.

The residues of the first adhesive progressively darkened and cross-linked within the ceramic body. At some point, it seems that about a dozen additional breaks to the dish occurred; a third repair campaign consisted of joining these new breaks with a third adhesive. These joins can easily be distinguished from older ones, as they do not have associated staining. The losses and cracks were filled and painted, with the paint extending over the stained areas on the entire dish in order to mask them.

These first three campaigns of restoration must have occurred before 1954, when the object was acquired by the Freer Gallery of Art. When the dish was examined in 1964, it was noticed that some of the fills and overpaint were flaking. It was found to be extensively broken, repaired, and overpainted along all joins and fills, as visible on a before-treatment photograph from 1964 (fig. 4).

All of the overpaint was removed at that time, which revealed the staining; during this treatment, the fills were resurfaced. The inpainting approach on these fills was to mimic the dark staining rather than cover it. The Department of Conservation and Scientific Research holds records dating back to the beginning of the Freer Tech Lab in 1951—quite fortunately, there is a good treatment record for this object. The
report indicates that concealing the stained areas with overpaint was considered in 1964, but this was not done owing to a lack of time. At that time, the staining was thought to have been caused by some material that penetrated into the sherds during burial. Our most recent analyses, however, seem to indicate that the staining was more likely due to old repairs. The dish was exhibited a few times during the 1970s and early 1980s but had not gone on display since 1982 because of its disfiguring stains.

5. ANALYSES

Analyses were carried out to attempt identification of the nature and cause of the staining and accretion on the foot. Portable XRF spectroscopy was used to identify the reddish-brown accretion on the reverse of the dish. Iron, calcium, and strontium were found. After discussion with the curator, it was agreed that a historical reason for the presence of this sediment was unlikely and that it probably came from burial. It was decided to reduce the accretion as much as possible to avoid potential staining of the ceramic during subsequent stain reduction treatments.
Examination under longwave UV radiation showed no fluorescence of the stains under the glaze. Several additional analytical methods were used to identify the staining along the breaks, which were carried out during the conservation treatment once the edges of the sherds became accessible. A section of glaze loss is present directly above an area of stained slip on a small fragment of the dish, making the staining more accessible for analysis. This area was examined using SEM and qualitatively analyzed with energy dispersive x-ray spectroscopy (EDS). The image of the slip in SEM was not easily readable, which may indicate the presence of an organic stain. Although qualitative in nature, EDS showed that the stained areas contained very small amounts of iron, significantly less than would have been expected if the main cause of discoloration was an iron-based material that migrated into the sherds during burial, further supporting the possibility that an organic material may have been causing the stain. A sample was taken for analysis using pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS), microscopic reflectance infrared spectroscopy (MRIR), and microscopic transmission infrared spectroscopy (MTIR). Although the initial suspicion was that the adhesive potentially responsible for the staining was shellac, none of these analyses were able to confirm this. The results indicated the presence of organic material in the stained area but were inconclusive in terms of identifying a specific adhesive. This is probably explained by the presence of several different adhesives on the edges of the sherds from various conservation campaigns that subsequently penetrated into the porous body and slip.

6. TREATMENT

The treatment of the object started with surface cleaning, removal of paint and fill materials, and disassembly (fig. 5). This was carried out using a combined mechanical and solvent approach, with acetone and deionized water (depending on the nature of the various paints, fill materials and adhesives present) with cotton wool swabs, cotton wool poultices, and a scalpel.

The diversity of materials found in use on the object for inpainting, fills, and adhesives (particularly the two adhesives that must have been used during the second and third campaigns of restoration) confirmed the history of successive restorations. The removal of old fill material also revealed that the glaze was poorly adhered in areas, which is partly the result of manufacture, but also suggested the presence of soluble salts, although none were visible. Microchemical tests confirmed the presence of chlorides in the body of the dish. Before the central sherds could be taken apart, the iron-rich accretion inside the foot ring had to be reduced, as there was a concern that it could bleed into the unglazed body and create additional staining. It was mechanically reduced with the tip of a bamboo skewer, brushed off with a stiff brush, and cleaned with acetone on a cotton wool swab. While this did not remove the staining on the porous body and slip, it reduced the bulk of the accretion, which seemed sufficient to avoid bleeding.

6.1 Stain Reduction

Stain reduction tests started with common solvents applied on cotton wool poultices. Deionized water, ethanol, acetone, isopropanol, ethyl acetate, ShellSol 340 HT and xylene were tested and found to have no effect on the staining. Varying the type of poultice material was tested using a range of poultice materials, such as α-cellulose powder, diatomaceous earth, ion-exchanging resin Amberlite XAD, and K-dry tissues. The test poultices were left in place between 5 and 15 minutes and then removed while slightly damp, with no discernible difference in appearance. The poultices also were left in place until completely dry but, again, the stains did not react.
We then consulted with Bruno Pouliot and Lauren Fair from the Winterthur/University of Delaware Program in Art Conservation before subsequent steps were taken to reduce the staining. In collaboration with Richard Wolbers, they have developed a method of stain reduction on ceramics that has proven very effective on other examples of tenacious staining (Pouliot, Fair, and Wolbers 2013). This method is based on the sequence of the action of a chelator, followed by a bleach, and then a rinse. For the application of these active agents, poultice materials are selected for their capillarity properties and the control of liquid that they provide.

On their advice, several chelating agents were tested, starting with those that have broad ranges of effectiveness and then moving on to more target-specific chelators. The abundance of small sherds provided many opportunities to test various parameters and combinations of chelators and bleaches. Sodium citrate, EDTA disodic salt (ethylenediaminetetraacetic acid disodium salt dihydrate), Tiron (4,5-dihydroxy-1,3-benzene-disulfonic acid disodium salt monohydrate), HBED (N,N´-di(2-hydroxybenzyl) ethylenediamine-N,N´-diacetic acid monohydrochloride hydrate), and acetylacetone were assessed as possible chelators. Since Tiron and HBED both target iron during chelation, these were included in the tests along with the more general chelators in case the small amount of iron detected in the stained area of slip was indeed a factor in the discoloration. As suspected based on the analysis of the
stain, Tiron was not effective at reducing the darkening. Since HBED also focuses on iron, it was difficult to get into solution, and is relatively expensive, it was ruled out as a possible treatment. Acetylacetone, which is a solvent and a mild chelator at the same time, did not appear to reduce the staining at all. Both sodium citrate and EDTA showed some effectiveness; however, sodium citrate was selected because it is less aggressive.¹

Among oxidizing bleaches, hydrogen peroxide is probably the most commonly used in stain reduction. A stabilized version of hydrogen peroxide, carbamide peroxide, is made by adding to it an equal molar amount of urea. Carbamide peroxide acts more slowly than hydrogen peroxide because it takes longer to break down into hydrogen peroxide and carbamic acid, and then into hydrogen and water. This confers a more progressive and uniform action to the bleach, making it more efficient at reducing organic stains (Norquest 2008). Therefore, carbamide peroxide was chosen for the second step of the stain reduction sequence.

Several poultice materials provided different levels of flow control, absorption, and capillarity. K-dry tissue, α-cellulose powder, cotton wool, Laponite RD gel, Amberlite XAD, and agarose gel blocks were tested. Poultices were tested on the break edges and on the glazed surface directly above the staining. The latter method of application was eventually chosen, as the stains were located in the slip layer a couple of millimeters away from the break edges, requiring deeper penetration of the solution to reach the stain than if applied from the surface of the glaze. K-dry tissue did not introduce nearly enough liquid to penetrate the craquelure of the glaze and reach the slip layer and would not provide enough control if more liquid was added to overcome this limitation. α-Cellulose powder and Amberlite XAD did not offer very good adherence to the ceramic’s surface and edges. Laponite RD would have been difficult to clean off the porous edges and did not provide enough contact with an interleaving layer. Agarose rigid gel blocks offered good control of the amount of liquid delivered and were easy to use, resulting in their selection as the poultice material. They were chosen as the method of application since the liquid is held closer to the surface with less penetration to the body of the ceramic than other methods. Capillarity and flow can be further controlled by variation of the concentration and thickness of the agarose gel blocks (Norquest 2008; Pouliot, Fair, and Wolbers 2013).

To prepare these blocks, agarose powder is heated on low heat with deionized water until a viscous liquid forms. The liquid is then poured into Petri dishes up to approximately 1 cm thick. Agarose cools down into a rigid gel that can be cut into blocks of the desired dimension and shape. The blocks of agarose are then soaked in the chelator or bleach solution for a couple of hours before being used. Exchanges with the liquid take place in the pores of the gel blocks; they can later deliver the solution in which they had been soaked. They could be applied specifically to the stained areas and, therefore, achieve a localized application (fig. 6).

After testing 1%, 2%, and 4% (w/w) agarose blocks, 2% (w/w) agarose was chosen because it delivered enough liquid with satisfactory flow control. Literature on stain reduction recommends a 2% (w/w) concentration for sodium citrate. For both the chelator and the bleach, the pH was adjusted to 8 with sodium hydroxide, which was a compromise between enhanced efficiency and safety of the ceramic.

Sequences of 2% (w/w) sodium citrate applications, followed by 3%, 5%, or 10% (w/w) carbamide peroxide as necessary, and rinsing in baths of deionized water were tested on some of the small sherds, with the rinse also serving as desalination treatment. The baths were changed once a day, in general for about three days, after monitoring the conductivity. The goal was to lower the conductivity to a stable
level, not to entirely desalinate the sherds, which was feared could lead to leaching of certain elements from the body or glaze.

None of the sequences tested seemed to break down or move the staining, however, even when repeated one or two more times. The method described by Pouliot, Fair, and Wolbers (2013) recommends trying repeated applications at lower concentrations of bleach first but that tenacious stains may require the use of more concentrated solutions of bleach. In several case studies presented in their article, the technique is used with up to 20% (w/w) carbamide peroxide in order to reduce some staining. Initial tests of the procedure with 3% to 10% (w/w) carbamide peroxide on fragments from the dish proved to be ineffective. The concentration of carbamide peroxide was brought to 15% and then 20% (w/w), which was the only one that had an effect on this staining. The glaze and body of the ceramic were monitored under magnification during the tests and care was taken with the application of the agarose blocks to limit penetration of the solutions.

During testing, the duration of application of the agarose blocks increased progressively from 15 to about 40 minutes for the chelator applications, the same range for the bleach applications, and up to an hour for the darkest stains. Plastic wrap was placed loosely over the sherds to slow evaporation.
The most effective sequence for reducing the appearance of the staining was found to be 2% (w/w) sodium citrate and 20% (w/w) carbamide peroxide, both at pH 8 and applied through 2% (w/w) agarose gel blocks, followed by soaking in deionized water for rinse and desalination. Each 40-minute application was renewed twice, then the sherd was left to dry before the next step of the stain reduction sequence. Although this sequence was not able to remove the staining completely and the stain reduction was not homogenous among the sherds, it lightened the overall appearance significantly (fig. 7). After consultation with the curator regarding the appearance, it was decided to proceed with the overall treatment of the dish using this protocol.

6.2 Reassembly and Fills
The next step of the treatment was consolidation of the vulnerable areas of glaze along the edges of losses with 5% (w/w) Paraloid B-72 in acetone. The same solution was used to seal the porous body on sherd edges and glaze losses.

Joining the sherds back together was a challenge owing to the number of sherds—40 in total—and the condition of the edges, which had been eroded by burial and several campaigns of repair. Some joins barely had any point of contact, particularly around the bottom of the dish. Thirty-five percent (w/w) Paraloid B-72 in acetone, bulked with calcium carbonate and hydrophobic fumed silica, served as both adhesive and fill material in these areas (fig. 8).
Other losses were filled with Paraloid B-72 bulked with glass microballoons (0.34–0.4 mm in diameter), and the surface of all losses, glaze losses, and cracks were filled with Modostuc spackling compound (fig. 9).

6.3 Inpainting
When inpainting the fills began (with Golden acrylic emulsion paints and Rhoplex WS-24), questions arose regarding the approach to the integration of the stains. Several options were considered, such as inpainting the joins the color of adjacent stained areas, which made the stains appear darker and more noticeable. Another option consisted of painting the joins the color of the unstained glaze, which gave the dish an appearance more in keeping with an approach commonly used in the conservation of archaeological material.

Although an archaeological approach is acceptable in many cases, the current aesthetic for the Islamic galleries at the Freer|Sackler is that of art objects rather than archaeological artifacts and the curator
prefers losses to be more integrated. The staining had been preventing the dish from being exhibited for over 30 years. Even though now reduced, the stains would still bother the curator and deter her from displaying the object.

As a result, a major treatment had been undertaken with a less than satisfactory outcome since it did not sufficiently reduce the appearance of the staining to allow for its display—the primary reason for treating the object. The treatment included a stabilization aspect, but neither the soluble salts issue nor the instability of the glaze were apparent before the removal of the old repairs and were not the impetus for taking on such a complex and long treatment.

Although all conservators who saw the dish at that point, both from the Freer|Sackler and elsewhere, found that the appearance of the dish had improved overall, the goal of the stain reduction treatment was not achieved. A few suggested covering the stained areas with paint to make them less distracting, which had already been mentioned as an option in the treatment report of 1964. This was not an intervention that had been considered during the current treatment because it meant applying paint over original
surfaces. As the various experiments into ways of inpainting the losses and cracks proceeded, however, it became clear that as long as inpainting was limited to filled surfaces, it would not be possible to achieve an appearance that would meet the aesthetic requirements of the curator.

The “overpainting” option was carried out in a small area (fig. 10) to provide an idea of the visual result as well as of the amount of paint needed to mask the stains.

The tested area was chosen because it was one of the most distracting ones, with the stains creating an apparent network. The stained areas to be overpainted were first sealed with 5% (w/w) Paraloid B-72 in acetone. The overpainting was done with consecutive layers of Golden acrylic emulsion paints, applied only where necessary, with a fine brush (fig. 11).

Once the tested area was painted, each of us was dissatisfied with the result. We were uncomfortable with the principle of painting over an original surface and found the painted area more distracting than the staining, probably in part because, after working on this ceramic for so long, it was difficult to have an

Fig. 10. During treatment, obverse with losses and joins inpainted the color of unstained glaze. On the lower right corner, dark stains are partially overpainted as a test. (Courtesy of the Freer Gallery of Art and Arthur M. Sackler Gallery, photograph by Claire Cuyaubère)
objective eye. It was decided to show the object to colleagues from various specialties in the Department of Conservation and Scientific Research, and to conservators visiting the labs. Surprisingly, the feedback received was almost unanimously positive, with most viewing the possibility of exhibiting the dish as the primary benefit since the painting could be done in a way that would not permanently affect the dish.

We decided to overpaint the stains in a larger area and to present it to the curator, who confirmed that despite the stains being lighter as a result of the treatment, she would not exhibit the object for the reopening of the Freer|Sackler Galleries if the stains were still visible. On the other hand, she was particularly happy with the appearance of the overpainted areas. The visual result was enough to make her change her mind and bring the ceramic back into the design of the Islamic Galleries for the reopening.

At that point, the pros and cons of this solution were assessed in order for us to make a final decision on the integration of the losses and stains.

The first issue was covering original surface; but not only can the overpainting be easily removed, the stained areas would also be sealed before being painted over. Additionally, the areas were overpainted during the third restoration; thus, previously unpainted areas of the glaze would not be painted in the current treatment. Furthermore, no section of the inscription would be overpainted.
The other main concern was whether the visual result was satisfactory. However, the goal of overpainting the stains was to allow the public to appreciate the object without being distracted by the network of stains, not for the inpainting to necessarily be invisible. The overpaint should be discernible at a close distance. Even given these concerns, the curator, as well as a number of conservators, still favored the overpainted areas instead of a stained dish.

As a result of these discussions, we decided to cover up the stained areas on the front of the dish (fig. 12). As few layers of paint as possible were applied in order to limit the issue of variation in topography of the inpainted and glazed surfaces. Dilute Rhoplex WS-24 was applied over the paint to approximate (but not quite match) the glossiness of the glaze. The difference in topography and glossiness between the overpaint and the glaze make the joins visible under certain lighting conditions (particularly in raking light) but discreet for a nontrained eye in gallery lighting.

The back of the dish was inpainted in a much more visible way to ensure that potential handlers would be aware of the level of breakage of the dish (fig. 13).

Fig. 12. Obverse after treatment (Courtesy of the Freer Gallery of Art and Arthur M. Sackler Gallery, photograph by Claire Cuyaubère)
Fig. 13. Reverse after treatment (Courtesy of the Freer Gallery of Art and Arthur M. Sackler Gallery, photograph by Claire Cuyaubère)

Fig. 14. Obverse before (left) and after (right) treatment (Courtesy of the Freer Gallery of Art and Arthur M. Sackler Gallery, photograph by Claire Cuyaubère)
7. CONCLUSION

In conclusion, the complex nature of this treatment and the complicated decision process it necessitated provided an opportunity to experiment with various methods of stain reduction. Although the stain reduction treatment was not as successful as had been hoped, the method did reduce the appearance of the stains significantly (figs. 14, 15). The issue of inpainting versus overpainting was a chance to reflect on the importance of accessibility of an object and whether a less than ideal but reversible outcome is preferable to another 30 years in storage.

We hope that, in the future, a more effective stain reduction method will be found for this ceramic. In the meantime, it can be exhibited and appreciated by the public and scholars.

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NOTE

1. Citric acid (used in the form of sodium citrate) is a good chelator of organic materials held in ceramic-like substrate (Pouliot, Fair, and Wolbers 2013) and EDTA is a stronger chelator than citric acid (Pouliot, Fair, and Wolbers 2014), meaning that it is efficient at chelating additional elements, which seemed unnecessary for an equal apparent effectiveness on the stains discussed here.
REFERENCES


SOURCES OF MATERIALS

Rhoplex WS-24, Glass Microballoons (0.34–0.4 mm in diameter)
Conservation Resources International, LLC
5532 Port Royal Rd.
Springfield, VA 22151
800-634-6932
www.conservationresources.com

Lonza SeaKem LE Agarose
Fisher Scientific
800-766-7000
www.fishersci.com

Golden Heavy Body Artist Acrylics
Dick Blick Art Materials
P.O. Box 1267
Galesburg, IL 61402-1267
800-828-4548
www.dickblick.com

Modostuc Spackling Compound
Peregrine Brushes and Tools
267-888-6657
www.brushesandtool.com

Paraloid B-72
Talas
330 Morgan Ave.
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