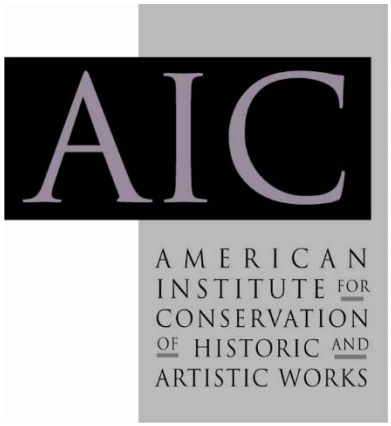

Article: The surface revealed: Cleaning of two painted plaster sculpture
Author(s): Richard C. Wolbers and Margaret A. Little
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THE SURFACE REVEALED: CLEANING OF TWO PAINTED PLASTER SCULPTURES

Richard C. Wolbers and Margaret A. Little

Abstract

In approaching surface cleaning of an art object, treatment typically starts with analysis of the object to understand the materials used to create it, and the nature of the soiling on the surface. This information aids in designing the cleaning system used in the treatment. In situations where the nature of the soiling and/or the geography of the object's surface are complex, it can also be important to find a way to monitor and measure the efficacy and level of cleaning of the surface.

An approach to designing a cleaning system and measuring the level of cleaning will be discussed in the context of the cleaning of two painted plaster busts in the collection of the Winterthur Library Archives. The busts, one of Pierre Samuel du Pont de Nemours, the other of his first wife Nicole Charlotte Marie Louise Le Dée de Rencourt, are copies of plaster busts attributed to Louis Simon Boizot which date to 1775-6. These objects were chosen for inclusion in a Winterthur Museum exhibition, and required treatment prior to exhibit, specifically cleaning, minor paint consolidation and loss compensation. The painted surfaces of the busts were heavily soiled, and removing these soils efficiently and evenly presented an interesting challenge, given the range of surface materials (including a possible original painted surface), overpaint and exposed plaster surfaces.

Introduction

In May 2004, the exhibition "The Winterthur Library Revealed: Five Centuries of Design and Inspiration" opened at Winterthur Museum. At the entrance to the exhibit, two painted plaster busts from the Winterthur Library Archives were installed. The life-size busts depict Pierre Samuel du Pont de Nemours (du Pont de Nemours), patriarch of the du Pont family in the United States, and his first wife Nicole Charlotte Marie Louise le Dée de Rencourt (Madame du Pont). These plaster busts, made in the 19th century, are copies of busts made in France in 1775-6, possibly by Louis Simon Boizot (Hughes, 2003). The maker of the Winterthur Archives plaster busts is unknown, as are the circumstances and precise date of manufacture. However, it is know that a number of copies of the original plaster busts were made for du Pont family members (Anonymous, 1992).

When the busts were first examined prior to treatment, they were found to be in overall poor condition (Fig. 1). The most serious condition issue was the heavy accumulation of dirt on the surface. Some of the dirt was loosely held to the surface and could be mechanically removed with a soft brush and vacuum. However, a significant amount of the dirt was ingrained in the painted plaster surface and could not be mechanically removed. Overall, the paint was well adhered to the plaster substrate, though there were areas where paint was lost, abraded or poorly

adhered to the substrate.

Figure 1: Busts of Madame du Pont (OB 373b) on left and du Pont de Nemours (OB 373a) on right, before treatment. Winterthur Library Archives.

The plaster itself appeared to be in stable physical condition. X-radiographs (taken with Staveley Instruments CPX 160 X-Ray System at 100kV, 2.5 mA for 2 minutes) revealed that the busts were hollow cast, probably in a two piece mold, and that there was no internal metal armature. The x-radiographs also revealed that there were no cracks which would undermine the physical stability of the busts. There were losses of plaster, both large and small, and this afforded an opportunity to examine the plaster on break surfaces. In both objects the plaster appeared "hard" and did not crumble if touched. Qualitatively, though, the plaster of Madame du Pont's bust seemed somewhat "softer" than that of du Pont de Nemours.

Both busts had been repaired at least once in the past. Repairs were evident to the unaided eye because the color of the inpainting and/or fill material no longer blended with the original surface. When the objects were viewed in both long and short wave ultra violet light (Fig. 2) the fills and inpainting were more clearly visible, as was a discontinuous yellow fluorescence present on the paint surfaces, indicating the presence of a varnish coating. On unpainted plaster surfaces, the yellow fluorescence was completely absent.

Figure 2: OB 373a (on right) and OB 373b (on left) in long wave ultra-violet light, before treatment. Winterthur Library Archives.

After initial examination of the busts, their condition was discussed with Heather Clewell, Winterthur Library Archivist and curator for the objects, and goals for the treatment were established. Because the busts would be prominently displayed in the exhibit, it was felt that the conservation treatment should return them to visual and physical state as close to "original" as possible. That meant that dirt/grime needed to be cleaned from the painted surfaces in a way that maintained visual harmony both within a single bust and between the two busts; where necessary paint would be consolidated to the substrate; losses of plaster would be compensated; and finally areas of restoration would be inpainted to visually integrate with the cleaned painted surfaces.

Cleaning the painted plaster surfaces would clearly be the most challenging aspect of the treatment. First, there was the large and complex three-dimensional surface to consider. The cleaning system – the material used and its method of application – would have to be one which could be used both on flat surfaces and also hard to reach three-dimensional folds and crevices found on the objects.

Second was the need to maintain visual harmony both within a single object and between the two busts. This was challenging because of the uneven distribution of soiling on the surfaces of the

two objects, and further complicated by the fact that the treatment would need to be accomplished quickly to meet an installation deadline. Realistically the treatment would be carried out by more than one conservator and reaching the aesthetic goal established by the curator would be made more difficult by the number of hands involved in the treatment.

Third, the ultraviolet light examination and analysis of paint cross sections indicated the presence of a varnish coating on the painted surface. Cross sectional analysis of paint samples taken from the busts suggested the presence of significant amounts of Pb and Ca in both paint and substrate; these metals could conceivably contribute to the tenacious binding of soiling materials on the paint surface.

With these issues in mind, criteria were established for the cleaning system to be used in the treatment of the plaster busts:

- The system/method needed to be effective in removing dirt without damaging the varnish coating, paint or plaster substrate.

- It was critical that whatever cleaning system/method was chosen would allow the conservator to control the level of cleaning so that visual harmony could be maintained.

- An objective means of monitoring the level of cleaning would be crucial given the emphasis on the aesthetic appearance of the busts after treatment. Without a means of evaluating the level of cleaning, it could be difficult to maintain visual harmony given the unevenness of soiling.

Literature Review

Literature relating to the conservation of plaster objects, both painted and unpainted, was reviewed to gain an understanding of methods used to clean plaster objects in the past (Andre 1977; Beale et al. 1977; Canadian Conservation Institution n.d.; MacKay 1997; Maudueche 1992; Mel'inkova and Lebel 1978; Richie 1933; Stable et al. 2002). In terms of cleaning plaster and painted plaster surfaces, there did not appear to be a single method of treatment recommended. The choice of cleaning system was dictated by whether or not a painted or other decorative layer was applied to the substrate, the nature and condition of the paint and/or the plaster substrate, and the nature of the soiling to be removed. However, most articles indicated that while water could be effective in cleaning a plaster surface, it should be used sparingly as it could damage the plaster.

Analysis and Materials Characterization

To determine the appropriate cleaning method for the busts, instrumental analysis of samples of plaster substrate and painted decoration were performed in the Scientific Research and Analytical Laboratory at Winterthur Museum (Mass and Carlson, 2004). Analysis of paint cross sections from both objects were also carried out in the Paintings Conservation Laboratory at

Winterthur Museum.

Samples taken from the two busts were divided into two portions. One portion was mounted in Bioplast resin (Ward's Scientific). The embedded samples were initially sectioned on a belt-type sander, and prepared using progressively finer bonded abrasive clothes (Micro-Mesh Inc.) to a grit of 12,000, lubricated with mineral spirits. The samples were cover-slipped prior to examination, again using mineral spirits. The other portion was used for analysis of the paint binding media and elemental composition of the substrate by infrared spectroscopy.

Images of the cross sectioned samples were recorded on Kodacolor 200 print film using a Nikon Labophot microscope equipped for epi-illumination in UV and normal light. The magnification for each sample was 125x. For the UV light shots, the illumination conditions were a 360-430 nm excitation, 430 nm suppression filter, from an HBO 100 W mercury source. For the normal light image, a tungsten-halogen lamp (15W, Osram) was used without any additional color correction, but with cross polarization.

Cross sections of paint samples were photographed in normal light and UV light. They were then photographed in UV light after the following stains were applied: 4% Triphenyl Tetrazolium chloride (TTC) in methanol; 0.2% Rhodamine B (RHOB) in ethanol; and 0.2% Alexa Fluor 488 in a 0.05M borate buffer at a pH of 9.0.

The stained samples were then re-polished slightly (8000 grit adhesive) to a uniform cross sectional view using a fresh Micro-Mesh abrasive cloth, then sputter-coated with carbon and examined at various magnifications using a scanning electron microscope or SEM Topcon ABT 60 electron microscope with an Evex x-ray microanalysis system. A 20kV beam voltage was used in conjunction with a 22 mm working distance. Backscatter images were obtained for these samples, as well as Energy Dispersive Spectra (EDS) for additional elemental and distribution information.

Synopsis of Analytical Data

The results of qualitative energy dispersive x-ray microanalysis (carried out using ArtTAX µ-XRF spectrometer, molybdenum tube, 50 kV, 600µamp, 100 sec), Fourier transform infrared (FTIR) microspectroscopy (carried out using Thermo-Nicolet Magna 560 FTIR spectrometer, Nic-Plan microscope, 120 scans, 4 cm^{-1} resolution, range 4000-650 cm^{-1}) and scanning electron microscopy and energy dispersive x-ray microanalysis (SEM-EDS) indicate that both objects were made primarily of gypsum ($CaSO_4 . 2H_2O$) with small amounts of plaster of Paris ($CaSO_4 . ½ H_2O$), indicating an incomplete conversion of plaster to gypsum upon mixing with water (Mass and Carlson, 2004).

SEM-EDS was used to examine the paint on the surfaces of both objects. Lead was identified as the primary pigment in the paint. This analysis also identified silicon in the soil on the surface of the sample (Fig. 3).

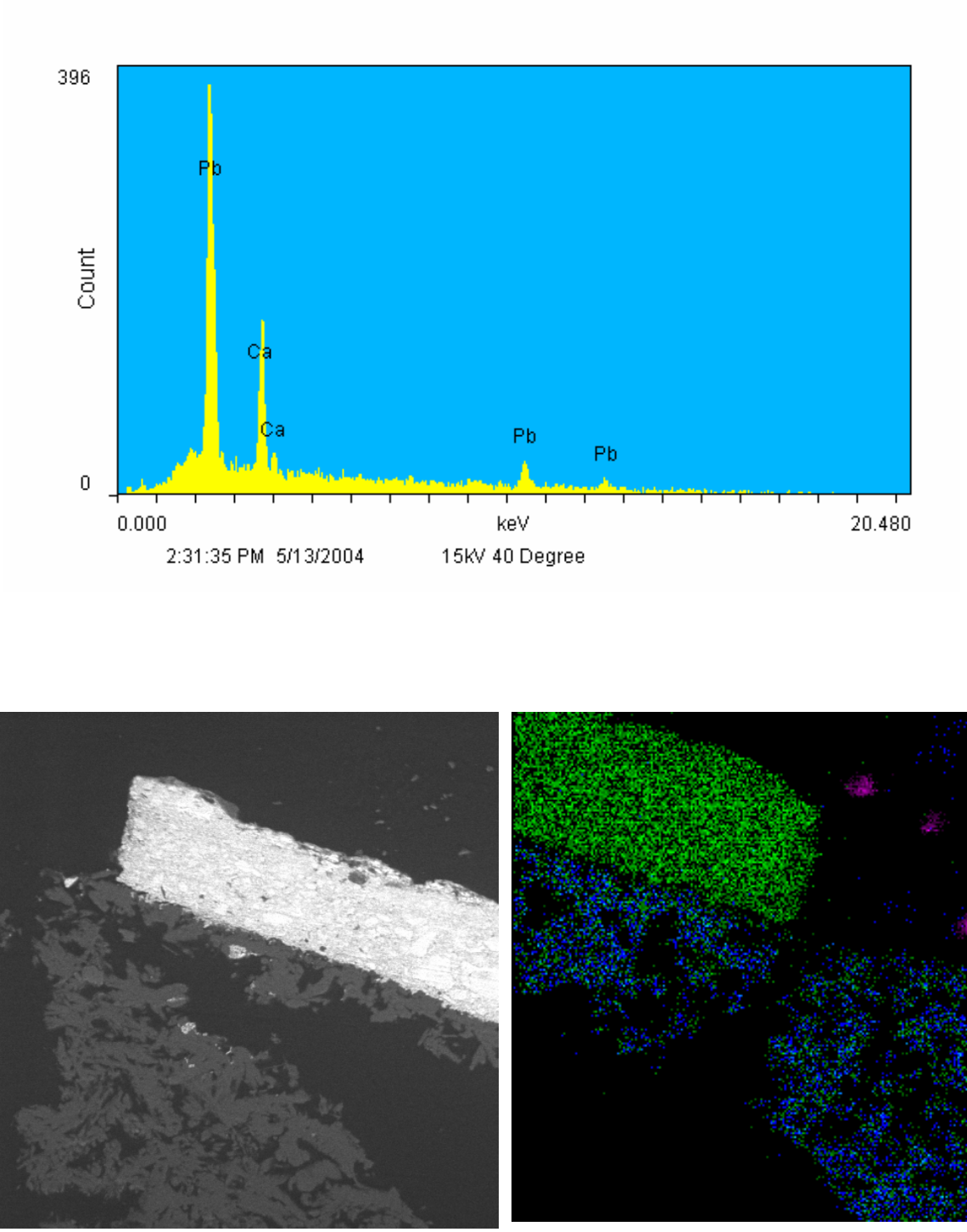
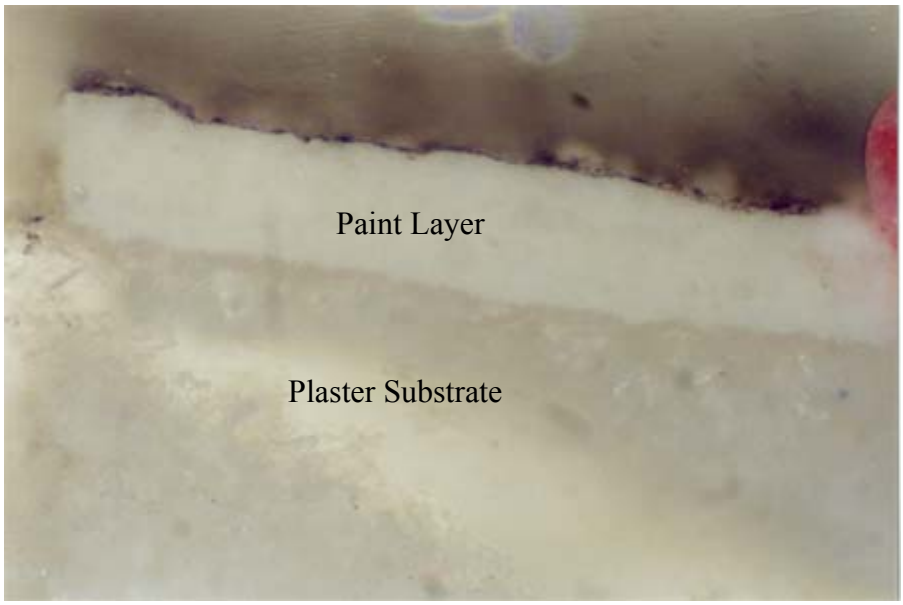
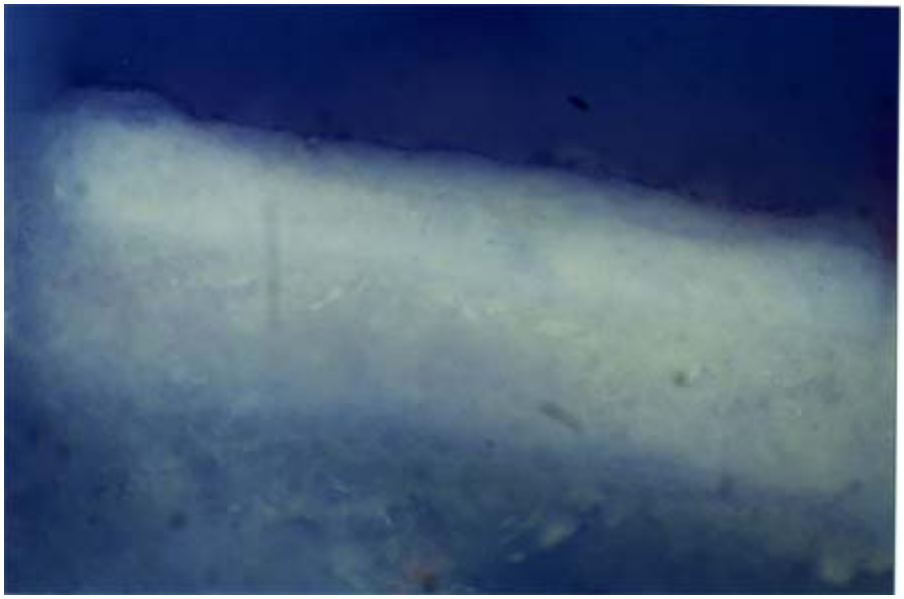


Figure 3. Du Pont de Nemours (OB 373a) sample: x-ray spectrum (top), electron backscatter image (bottom left) and elemental map (bottom right; Ca , blue; Pb, green; Si, red)

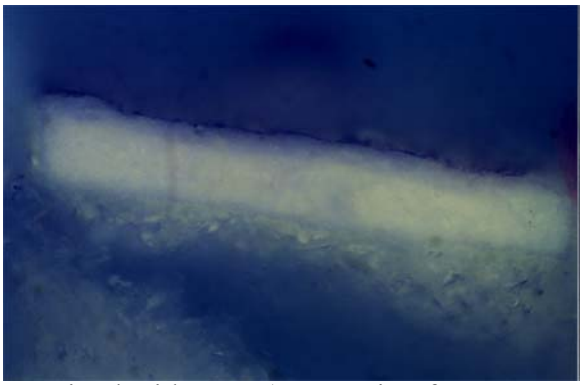
Figure 4 shows the paint cross section taken from the bust of du Pont de Nemours (OB 373a), and Figure 5 shows the paint cross section from the bust of Madame du Pont (OB 373b).



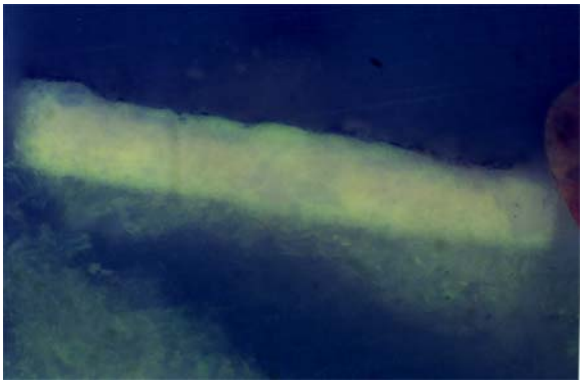
a. Visible Light



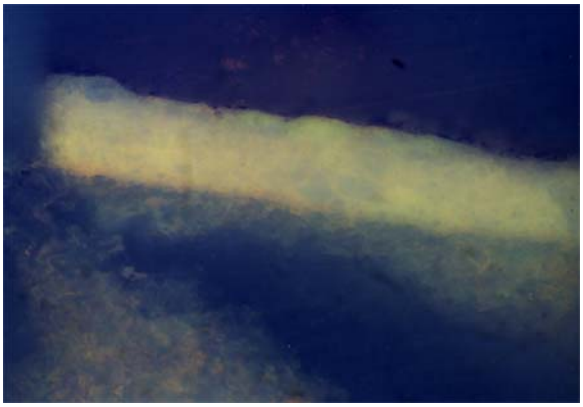
b. Ultraviolet Light



c. stained with TTC (no reaction for carbohydrates)

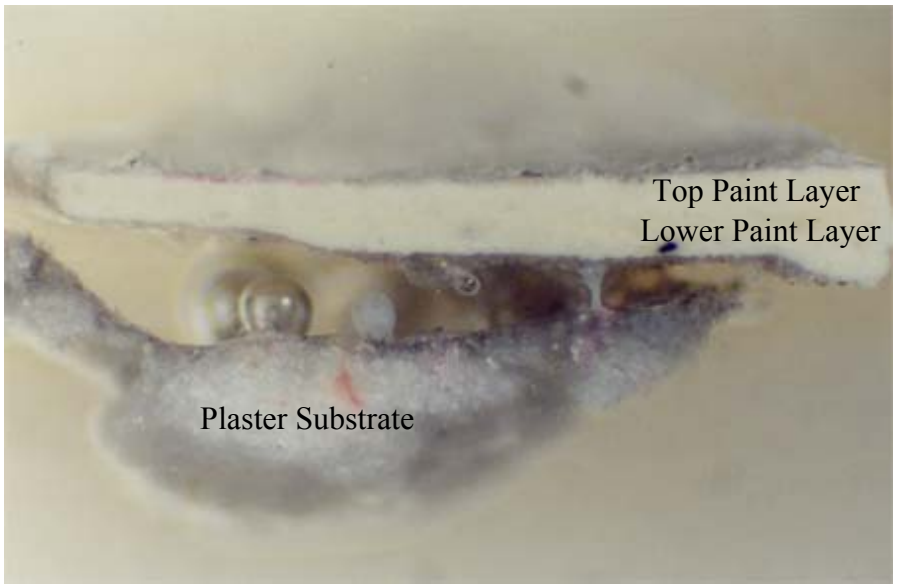


d. stained with Alexa Fluor 488 (note positive reaction for protein as indicated by green fluorescence in paint layer)

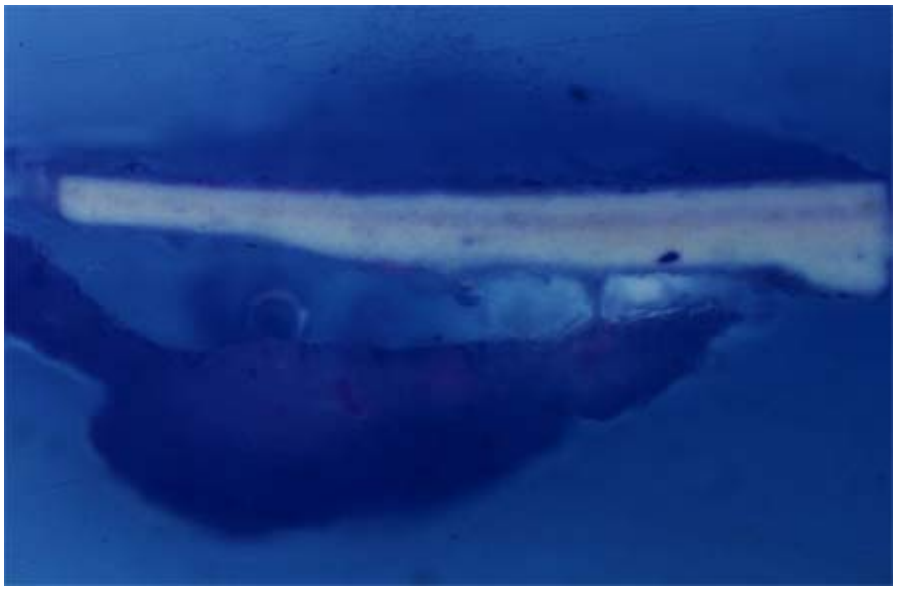


e. stained with RHOB (note positive reaction for oil as indicated by red-orange fluorescence in paint layer)

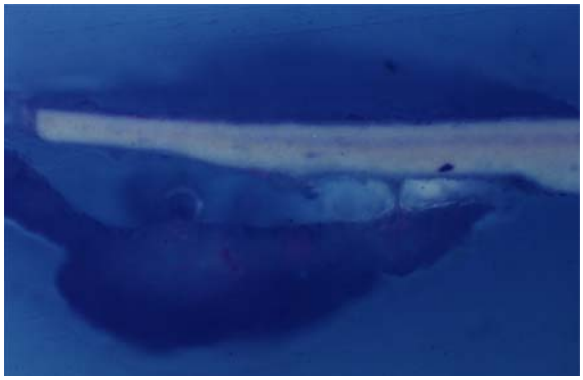
Figure 4 a-e. du Pont de Nemours (OB 373a), cross-sectional view, 125x magnification



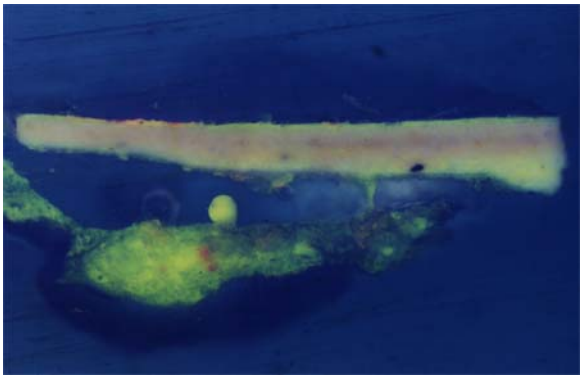
a. Visible Light



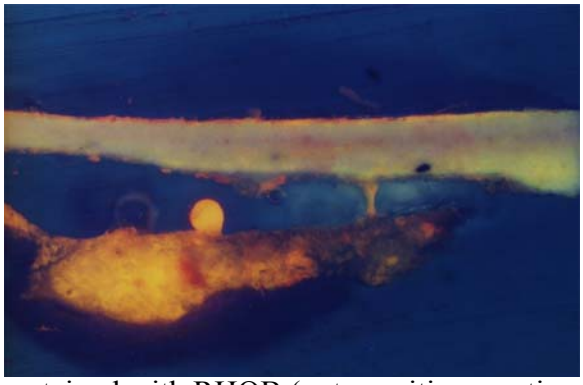
b. Ultraviolet Light



c. stained with TTC (no reaction for carbohydrates)



d. stained with Alexa Fluor 488 (note positive reaction for protein as indicated by green fluorescence in paint layer)



e. stained with RHOB (note positive reaction for oil as indicated by red-orange fluorescence in paint layer)

Figure 5 a-e. Madame du Pont (OB 373b), cross-sectional view , 125x magnification

Portions of the samples held in reserve from each portrait bust were analyzed by infrared spectroscopy for additional characterization of paint binding media. A Thermo-Nicolet IR100 bench top spectrometer was used to characterize the samples. All spectra were the result of identical instrumental run parameters (gain: 4; no. of scans 32; 4000cm^{-1} to 400cm^{-1} spectral range, and converted to absorbance spectra). Baseline corrections, smoothing, and library searches were performed using Thermo-Nicolet Encompass proprietary software. The samples were usually divided by scalpel; pressed with a piston, and run "neat" on a Thermo-Nicolet Thunderdome ATR cell. Spectral results for the gypsum substrate and paint from the bust of du Pont de Nemours are shown in Figure 6.

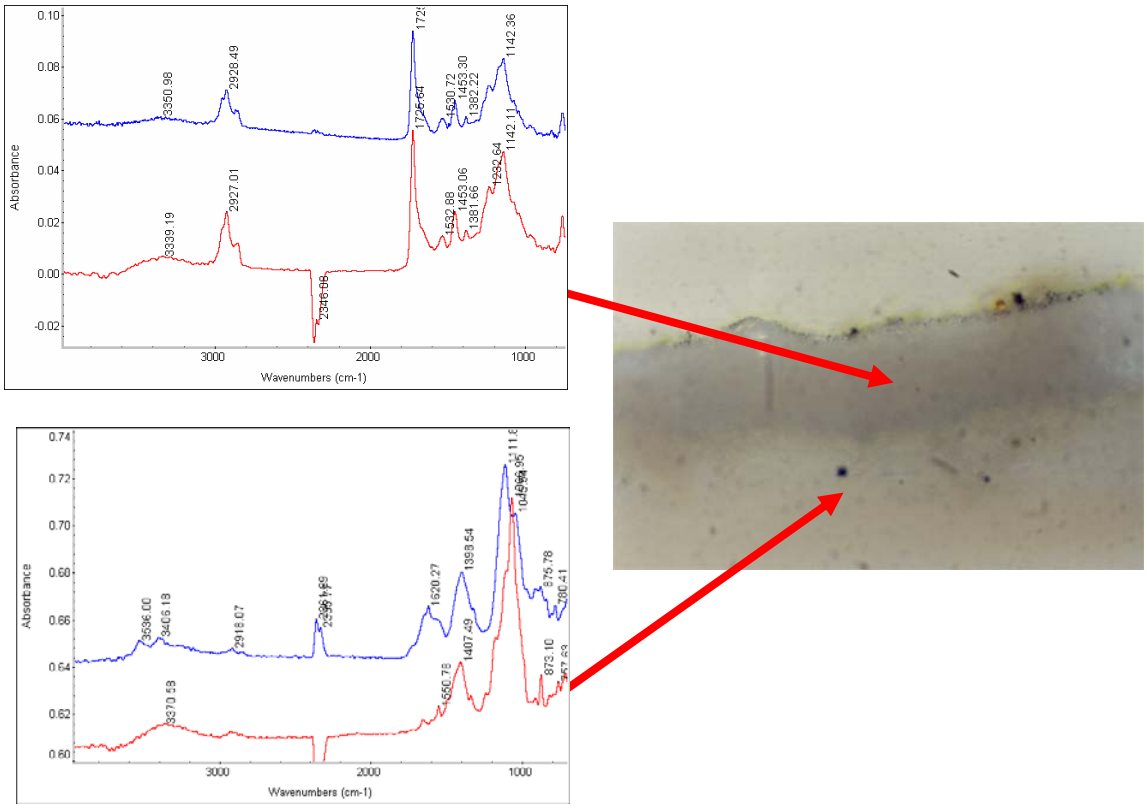


Figure 6. Top Left: FTIR Spectra of du Pont de Nemours Paint (blue) and a reference spectrum for a casein/oil paint standard (red). Bottom Left: FTIR Spectrum to the plaster substrate material of the bust (blue) and a reference spectrum for a gypsum standard (red). The cross-sectional detail (right) shows the approximate arrangement to the two materials tested.

From cross sectional views and media staining, it would appear that the plaster surface of both busts was sized with a proteinaceous material prior to painting. On both busts, the paint binder appears to be an emulsion (e.g. a blend of protein and oil binding materials); this observation seems to be confirmed by the infrared spectra obtained from the paint films as well. The paints in both cases appear to be lead containing materials (e.g. lead white as the bulk colorant). The soiling layer predominantly appears to contain both lead-based compounds and calcium-based compounds.

One additional note: during cross sectional sampling, there were some samples that at higher magnifications appeared to carry a droplet or spray like application on the surface of the paint layer, in effect fixing the accumulated oil to the paint surface in those areas where it had been applied. Figure 7 is a cross sectional image in normal light of one such sample from the bust of du Pont de Nemours.

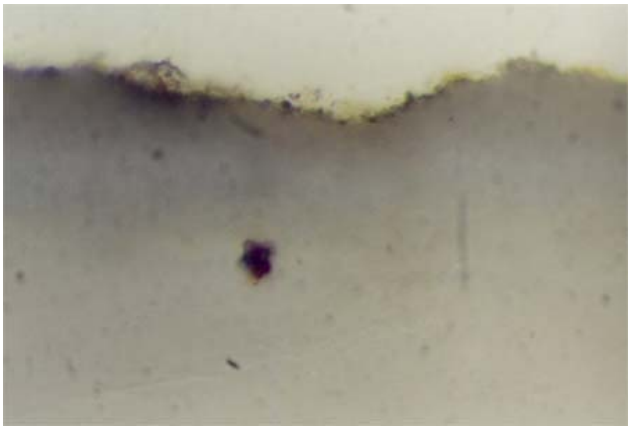


Figure 7. Cross sectional image, normal light 250x from du Pont de Nemours bust (OB 373 a).

Materials and Methods

Though the literature indicated that water should be used sparingly in the cleaning of plaster, initial cleaning tests indicated that aqueous cleaning systems were more effective than solvents in removing dirt and grime from the painted and plaster surfaces. In evaluating cleaning systems for general soil removal on the two busts, five solution properties for aqueous cleaning systems were specifically tested for their efficacy or contribution to the overall cleaning effect: pH, solution conductivity, the nature and strength of a chelator or chelators needed for the dissociation of relatively low solubility soil salts, the form and strength of surfactant needed, and the solution viscosity.

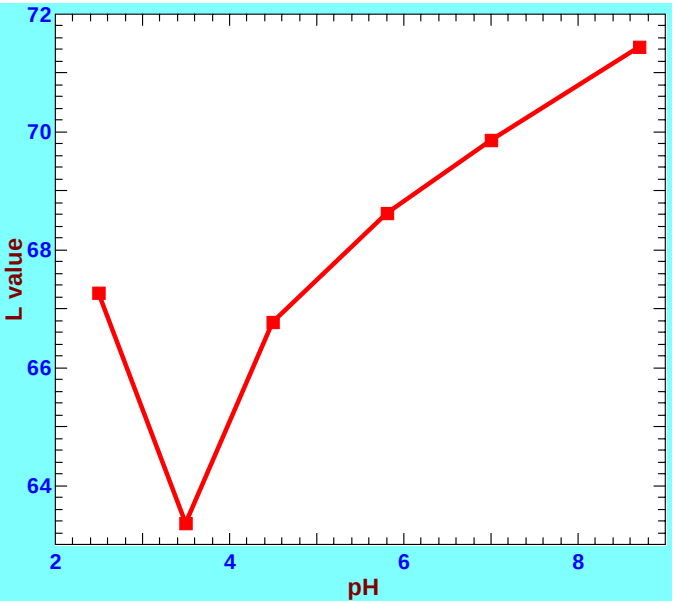
pH

A series of test solutions were created to evaluate the pH sensitivity of the paint/plaster surfaces on both busts. Standard buffer solutions were prepared at a 0.05M concentration for the following buffers, adjusted as close to the individual pK_a's for each material.

Buffer	pK_a
phosphate	(1) 2.15
citrate	(1) 3.13
citrate	(2) 4.76
succinate	5.6
BIS-TRIS	6.44
triethanolamine	7.76
borate	9.23

The test buffer solutions were applied with a cotton swab. Generally these test cleanings were done on the base and on the reverse of each bust.

The cleaning effect of the buffered test solutions was monitored used the Minolta CR100 chromameter to compare the brightness of test cleaning areas to the average brightness measurement obtained for the relatively unsoiled paint/plaster surfaces in a protected area of the busts. For the du Pont de Nemours bust the average of brightest areas measured L* 78.68 a* +0.37 b* +13.14 (where L=light,dark, a=red,green, and b=blue,yellow); for Madame Du Pont the average of brightest areas measured L* 68.84 a* +1.16 b* +13.45. The treated area was rinsed with a swab dampened with de-ionized water and allowed to dry 60 minutes prior to measurement. For the du Pont de Nemours bust the relative brightness or L value recovered on cleaning is recorded here in Graph 1 as a function of pH.



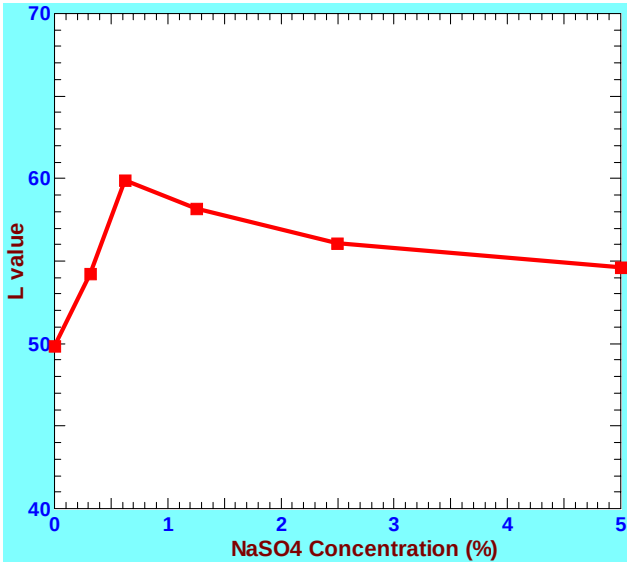
Graph 1. The relationship between L value and pH of cleaning solution.

No single test solution completely recovered the relative brightness of the average unsoiled surface, L 78.68 in the case of the du Pont de Nemours bust. The test cleaning results did suggest that soil removal was at a minimum at, or close to, pH of 3.5 (presumably close to the iso-electric point of the proteinaceous binder in the paint), and that raising the pH as high as 8 (with triethanolamine) was a more effective strategy than lowering the pH below the apparent iso-electric point where the recovered brightness was about 92% of the unsoiled average surface.

Conductivity

As an additional solution parameter, the overall conductivity of any potentially useful aqueous solution that contained ionizable materials was considered in our evaluation of aqueous methods for cleaning. A set of standard solutions were prepared with varying amounts of $NaSO_4$, to evaluate the general effect of increasing ionic strength on the paint/plaster surfaces of the busts. Again, the effect of the sulfate test solutions was monitored used the Minolta CR100 chromameter to compare the test cleaning results to the average brightness measurement obtained for the relatively unsoiled paint/plaster surface in a protected area of the bust (Madame Dupont: average of brightest areas measured L* 68.84 a* +1.16 b* +13.45; du Pont de Nemours: average of brightest areas L* 78.68 a* +0.37 b* +13.14). The test buffer solutions were applied with a cotton swab; the area treated rinsed with a swab dampened with de-ionized water, and the area thus treated was allowed to dry 60 minutes prior to measurement. Generally these test cleanings were done on the base and on the reverse of each bust.

On the du Pont de Nemours bust (see Graph 2), as the sulfate concentration was increased from zero to 1%, the conductivity rose from 0 to about 10^4 microSeimens (µS), with a concomitant rise in apparent cleaning effect. At this conductivity (10^4 µS), the recovered brightness was only about 76% of the average unsoiled surface. In addition, the brightness of the test-cleaned surfaces actually leveled off slightly at higher concentrations of sulphate. The data suggested that higher conductivities added no additional cleaning effect beyond about 10^4 µS.



Graph 2. The relationship between L value and $NaSO_4$ concentration

Chelators

The initial cross sectional and analytical data suggested that both high levels of lead (from the paint) and calcium (from the plaster support) were likely metallic cations to be present on the paint/plaster surfaces, and therefore most likely to be present as part of insoluble complexes or salts accumulated there. Several chelating materials were tested for their efficacy in cleaning the soiled paint surfaces:

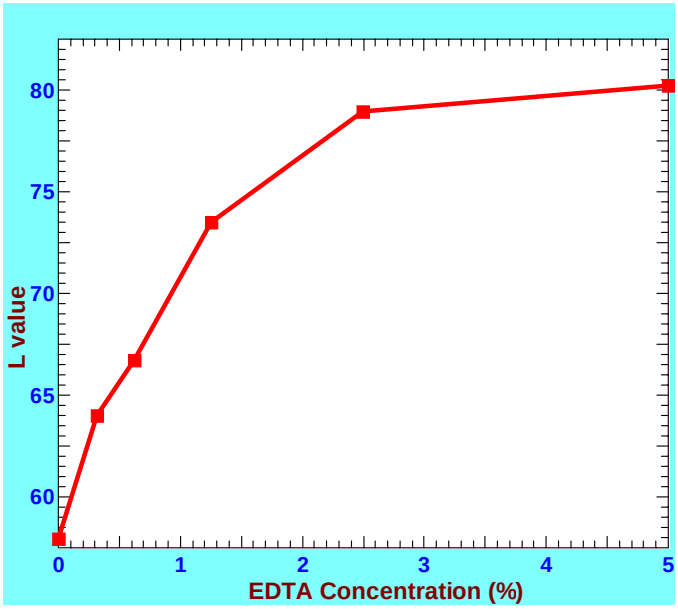
- citrate
- ethylenediamineteraacetic acid (EDTA)
- ethyleneglycoltetraacetic acid (EGTA)
- N-(hydroxyethyl)-ethylenediaminetriacetic acid HEDTA (reduced EDTA)
- nitrilotriacetic acid (NTA)

These chelators generally exhibit increasing formation constants in this order (formation constants from Dean 1985):

Chelator	Formation Constant Ca^{+2}	Formation Constant Pb^{+2}
Citrate	4.66	8.32
NTA	6.41	11.39
HEDTA	8.14	15.5
EDTA	10.96	18.04
EGTA	11.0	14.71

While the specific lead and calcium salts present on the soiled paint/plaster surfaces were not characterized, both exhibited concentrations of each metal ion accumulated on their surface. In the case of the du Pont de Nemours bust, the SEM-EDS data suggested a relatively higher proportion of lead to calcium at the paint surface. All of the chelators were made into test solutions at a constant molar concentration (.05M) and at the same pH (8.0 adjusted with triethanolamine) for comparison purposes. Only the EGTA and EDTA containing solutions appeared to have any cleaning effect on the accumulated soils (presumably because of the higher affinity for Pb^{+2}). The concentration vs. cleaning effect curve for EDTA on the du Pont de Nemours bust surface is shown in Graph 3.

The tests indicated 1) only chelating materials with the highest affinity for (i.e. formation constant with) lead and calcium in the series tested would suffice for the efficient cleaning of the paint/plaster surface, and 2) that concentrations as high as 2-2.5% of EDTA could be tolerated on the surface to be cleaned without going beyond the level of cleaning (identified as L* 78.68) deemed appropriate. Both EDTA and EGTA at a pH of 8 are fully ionized, and contribute significantly to the overall conductivity of the solutions they are in (a 1% solution of EDTA raised to pH 8 with triethanolamine yielded a solution conductivity of about 10^4 μS). At concentrations of about 2 – 2.5%, maximum cleaning effect is reached with both chelators.



Graph 3. The relationship between L value and EDTA oncentration.

Surfactant

Tests were conducted using various surfactant solutions in water at about five times their critical micelle concentrations (cmc). These represent surfactant solutions of widely different strengths (i.e., solubilization effects) as defined by their hydrophile-lipophile balance (HLB) numbers.

Surfactant	CMC	HLB
Triton XL-80N	0.1 mM	12.5
lauryl sulfate	8 mM	40
Pluronic L-40 (BASF)	0.2 mM	60

The presence or absence of any of these surfactants either alone, or in concert with the buffers, chelates, and salt material ($NaSO_4$) tested seemed to be particularly effective or useful in cleaning the soiled paint/plaster surfaces of the busts.

Viscosity

Tests with standard methylcellulose preparations in water (15, 400, 4000 centipoise (cps), from Fisher Scientific) suggested that the handling properties of our optimal cleaning solution (i.e. ease of application, stirring, wiping away, and rinsing of cleaned surfaces) felt best at about 4000 cps. Increasing the viscosity of aqueous preparations was a distinct advantage when working on

vertical surfaces. Raising the viscosity seemed also to slow the diffusion of aqueous materials into the paint surfaces to which they were applied.

Results

The aqueous set of conditions or materials that seemed most likely to clean (but not over-clean) and provide the best handling properties on the painted surfaces combined a slightly alkaline pH, a strong chelator for both Pb and Ca and a slightly viscous quality. The preparation used over much of the surface area on the busts was an aqueous preparation of EDTA (2.5%), buffered to a pH of 8 with triethanolamine, and thickened with about 2-3% of methyl cellulose (>4000cps). The overall conductivity of the cleaning solution was about 2.5 x 10^4 µS, largely because of the ionized EDTA in this preparation, at this pH. It should be noted that no surfactant was needed specifically for cleaning, nor was one initially included in this preparation.

However, as treatment progressed, a modified cleaning system was necessary. The clear coating material posed a serious problem for working evenly over the surface of the busts with the simple aqueous cleaning system. It was necessary to even out the cleaning by using an emulsified version of the aqueous system with the aliphatic hydrocarbon solvent Shell Solv D-38. The emulsion consisted of a 45:33:22 v:v:v mixture of the EDTA solution at pH 8:Triton XL-80N:Shell Solv D-38.

Conclusion

Analysis of the materials and soiling on the busts of du Pont de Nemours and Madame Du Pont gave an understanding of the nature of the soiling and how it was held to the painted surface. From this, a two step process was established for removing the soil. First, removing soiling held to the surface with the clear coating material with the EDTA/Triton XL-80N/Shell Solv D-38 solution; second, applying with the 2.5% EDTA solution, buffered to pH of 8 with triethanolamine thickened with 2-3% methyl cellulose. The results of cleaning were dramatic and clearly met the expectations of the curator (Fig. 8).

It can also be said that the cleaning system met the conservation goals established at the start of the project:

- A cleaning system which could effectively remove dirt/grime held to a physically and chemically complex surface had to be devised. Understanding the ways in which the dirt was held to the painted surfaces helped in designing the two part cleaning system used in this treatment and cleaning was accomplished with out damage to the paint or exposed plaster surfaces.

- Given the complexity of the surfaces and soiling, the cleaning system needed to be flexible so that it could be varied as necessary. In the course of treatment, it was found that some areas need only the EDTA solution, or only the EDTA/Triton/Shel Solv solution, or repeated applications of one or both solutions. These variations could be

accommodated without harm to the paint or plaster surfaces.

- A method of monitoring the level of cleaning was needed so that the visual harmony within and between the surfaces of the two busts could be maintained. In the course of analysis, it was found that brightness measurements taken with the Minolta CR 100 Chromameter could be used to monitor the progress of cleaning. Having a means of measuring the extent of cleaning allowed for more than one conservator to work on the project without compromising the aesthetic of the objects.

The cleaning was the most time consuming portion of the treatment of the busts, but once that step of the treatment was completed, losses to the plaster were compensated using plaster of Paris or Flügger (a commercially prepared acrylic spackling compound, available from Conservation Resources). Fills were separated from the original plaster surfaces with Japanese tissue adhered to the plaster substrate using methyl cellulose. Finally, inpainting was executed using Winsor & Newton Gouache Paints or Charbonnel Restoration Paint.

Figure 8. Madame du Pont (OB 373a) on left, and du Pont de Nemours (OB 373b) on right after cleaning.

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Authors' Addresses

Richard C. Wolbers, Winterthur/University of Delaware Program in Art Conservation, University of Delaware, 303 Old College, Newark, DE 19716, (302) 888-4818, (wolbers@earthlink.net)

Margaret A. Little, Winterthur Museum, Route 52, Winterthur, DE 19735, (302) 888-4927, (mlittle@winterthur