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Removing Ingrained Soiling from Medieval Lime-based Wall Paintings Using Nanorestore Gel Peggy 6 in Combination with Aqueous Cleaning Liquids

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ABSTRACT

This paper describes a cleaning method for removing ingrained carbonaceous dirt from lime-based wall paintings by utilizing hydrogels in combination with aqueous cleaning liquids. Nanorestore Gel[®] Peggy 6 has a number of advantages over traditional cleaning methods, and it is capable of holding large amounts of liquid, but it limits liquid penetration into the substrate. Cleaning action occurs only at the interface, without affecting the surrounding area or leaving residues. Furthermore, its viscosity makes it an ideal tool for treating irregular, vaulted surfaces. Laboratory experiments conducted on limewashed model tiles were decisive in the design of *in situ* experiments on fifteenth-century wall paintings. The cleaning efficiency of each method was assessed and quantified using colorimetry, 2-D FTIR mapping and image analysis using Cultural Heritage ImageJ. SEM-EDX was used for seeking residues. Results of both the model and *in situ* cleaning tests show a significant reduction of ingrained dirt when comparing Nanorestore Gel[®] Peggy 6 to traditional methods. Best visual results were obtained when cleaning with hydrogel loaded with 5% triammonium citrate was following by swabbing with a wet sponge. However, using water alone with the gel yielded almost as good a result *in situ*, making cleaning possible without any risk of residues.

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Ingrained dirt; Nanorestore Gel[®] Peggy 6; wall paintings; murals; microemulsions; chelating agents; anionic surfactants; Akapad dry sponge

Introduction

Removing dirt is a common treatment for wall paintings, often posing serious challenges. Ingrained soiling, as opposed to superficial deposits, is particularly difficult. This paper presents the results of testing a new method, where hydrogels are used in combination with aqueous cleaning liquids to remove ingrained dirt from porous lime-based wall paintings. Results of experiments carried out on models and on medieval paintings present a solution when traditional methods are insufficient or damaging.

The significance of this study

There is a multitude of medieval lime-based wall paintings in northern Europe. In Scandinavia alone, there are several hundred decorated churches where paintings have been exposed in artificially heated interiors for decades. Coal-burning stoves, used until the middle of the twentieth century (Brimblecombe 2003, Legnér 2012), were a major source of soot, as were candles, which continue to be used today (Pagels et al. 2009). The wall paintings gracing vaults were particularly vulnerable, and their soiling has both aesthetic and physical consequences. Many of the paintings were executed on a layer of limewash using a technique that visually resembles watercolor paintings, where

the white background plays a prominent role in the design. The paintings were generally executed *a secco*, and lime was a common binding medium. A typical feature, significant for cleaning, is the uneven surface, created by the bristles of a limewash brush. Due to the prominence of the white background, dirt is very visible and disturbs readability. Furthermore, numerous examples have been found in which soiling has provided sustenance for microorganisms (Caneva, Nugari, and Salvadori 1991; Sterflinger and Pinar 2013).

Challenges

This study focused on the main constituent responsible for the visual alteration of paintings, i.e. partially combusted carbonaceous particles formed by stoves and candles. These fine particles (< 1µm) have high specific surface areas, and due to their absorptive properties, contain large amounts of organic matter (Saiz-Jimenez 2003; Pagels et al. 2009). Particulate deposits can become so thick that the paintings are obscured, and in the past these were often washed with water and sponges, facilitating penetration of dirt into the substrate. This explains why conservators currently experience this type of soiling as ingrained and thus difficult to remove (Grau-Bové and Strlič 2013; Martin de Fonjaudran 2014).

The most commonly used dry-cleaning methods for removal of superficial dirt (Heiling 2012; Emmanuel 2016) do not remove ingrained dirt. Wet cleaning with poultices also has disadvantages: application is difficult, contact times are long, and there are risks of leaving residues and tidemarks. However, most problematic is the introduction of large amounts of water, which often initiates damaging processes. Some liquids commonly applied with poultices, e.g. tri-ammonium citrate (TAC), acknowledged for its efficiency for the removal of inorganic and organic matter (Phenix and Burnstock 1992), may pose problems when absorbed in calcareous materials (Gervais et al. 2010).

Proposed solution

Nanorestore Gel® Peggy 6 (PG6) provides a tool for solving challenging cleaning tasks on historic surfaces (Eriksson et al. 2017). The hydrophilic poly(vinyl alcohol)-based structural network renders the gel capable of holding large amounts of aqueous liquid, while the highly retentive properties limit the liquid's penetration so that cleaning occurs only at the interface, without affecting the surrounding area or leaving residues (Mastrangelo et al. 2017). High flexibility makes PG6 ideal for treating irregular surfaces. Weak intermolecular attraction forces between the gel and the substrate ensure good adhesion as well as easy removal (no intervention layer is necessary). Such properties make these hydrogels excellent media for the application of well-known aqueous cleaning agents, such as chelators, surfactants and microemulsions (Wolbers 2000; Baglioni et al. 2014; Chelazzi, Giorgi, and Baglioni 2018).

Experimental

Model tile preparation

Laboratory experiments were conducted on carbonated limewashed lime-based plaster applied to clay tiles, which imitated the substrate of medieval wall paintings.¹ An artificial dirt, comprising carbon black and paraffin oil, was prepared following published recipes, and applied by brush (Balcar et al. 2012). Repeated applications of 10% ethanol following soiling enhanced penetration of the artificial soiling. This allowed for preparation of model material where soil removal was expected to be particularly problematic, thus posing the greatest possible challenge in the cleaning experiment.

Cleaning with PG6 loaded with aqueous liquids

The following liquids were tested:

- Deionized water (DI)
- 2% and 5% TAC, pH adjusted to 7.5

- Nanostructured cleaning liquid, Nanorestore Cleaning Polar Coating S (PCS), containing an anionic surfactant, sodium dodecyl sulfate (SDS)
- Oil-in-water microemulsion, Nanorestore Cleaning Apolar Coating (ApC), containing SDS

Application time was 90 s, repeated on ten randomly selected tiles, which allowed for statistically viable results considering experimental anomalies. The working protocol followed common guidelines for these products (Baglioni, Chelazzi, and Giorgi 2015).² Subsequent rinsing (90 s) was performed with water-loaded PG6.

Color measurements before and after cleaning assessed efficiency. CIELAB color coordinates, $L^*a^*b^*$ with the specular component included, were measured with a portable Minolta CM-2600d version 1.08 spectrophotometer using $d/8^\circ$ measuring geometry. The color of each test area was measured nine times. Color differences were calculated using the 1976 CIELAB formula, $\Delta E_{ab}^* = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}}$.

Colorimetry could not detect the transparent oily component of the artificial dirt. Instead, micro-reflectance Fourier transform infrared (FTIR) spectroscopy 2-D imaging was used to detect oil after cleaning. Intensity maps, with a spatial resolution of 5.5 μm , were obtained with a Cary 620–670 FTIR microscope equipped with an FPA 128 \times 128 detector. Spectra were recorded directly on samples in reflectance mode, with open aperture and a spectral resolution of 4 cm^{-1} , collecting 128 scans for each spectrum, in the 4000–900 cm^{-1} range.

Detection of possible residues from cleaning liquids

Experiments were performed on unsoiled surfaces, focusing on TAC and ApC residues. Firstly, both liquids were applied by pipette on the surface and left to dry. To seek residues, samples were analyzed with a Hitachi S-3400N VP scanning electron microscope using energy-dispersive X-ray analysis (SEM-EDX), equipped with two Bruker X-Flash 6130 EDX detectors. Nitrogen (for TAC), or sodium and sulfur (for ApC) were used as references, as these are not present in limewash. Secondly, liquids were applied using PG6 following the procedure for aqueous liquids.

In situ cleaning tests

Final cleaning tests were carried out on fifteenth-century wall paintings in Skamstrup Church, Denmark, where dirt is particularly difficult to remove. Tests conducted on vaults were repeated three times, concentrating on white backgrounds. Based on results with aqueous liquids, the selection of cleaning liquids was narrowed, and the effect of longer

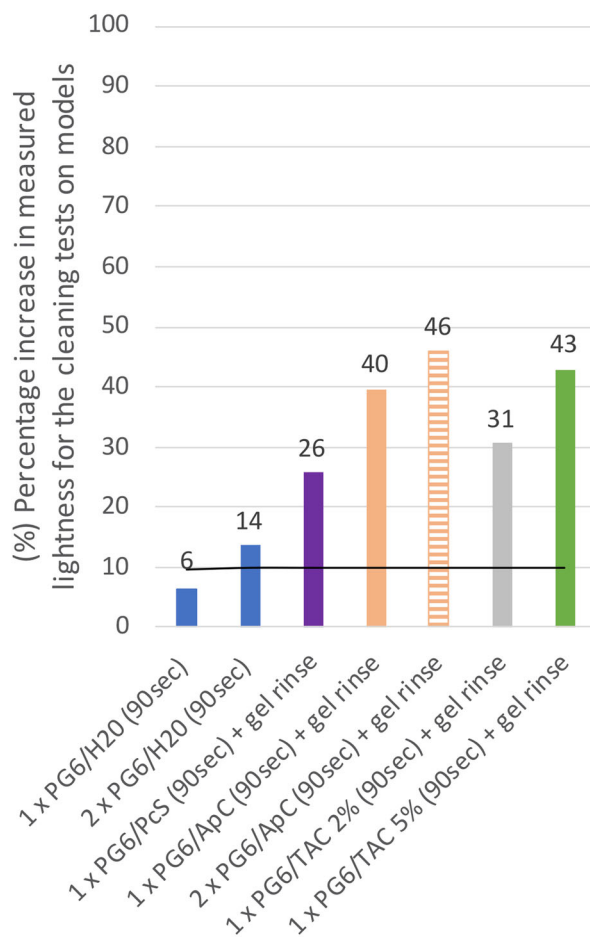


Figure 1. Color measurements as % increase in lightness for model tiles after cleaning with PG6 loaded with aqueous liquids.

contact times was explored. Furthermore, an alternative rinsing procedure involving mechanical action with an absorbent synthetic sponge, Saugwunder (SW), was tested. In addition, the commonly used yellow Akapad soft no. 404101 sponge and a water-moistened SW sponge were assessed for performance in relation to gel cleaning. The use of both sponges followed established protocols based on long-term practical use, consistent with manufacturers' guidelines.

Colorimetry was used to assess cleaning efficiency. Due to more challenging working conditions compared to laboratory experiments, the number of

measurements was reduced to five. To supplement colorimetry, software-based image analysis using ImageJ and a collection of plug-ins was organized in a semi-automated workflow, Cultural Heritage ImageJ and a collection of plug-ins was organized in a semi-automated workflow (Martin de Fonjaudran 2014), which allowed for evaluation of efficiency, based on three parameters:

- Mean RGB values of grayscale images (0–255) were measured and the % increase in 'white level' after cleaning was calculated

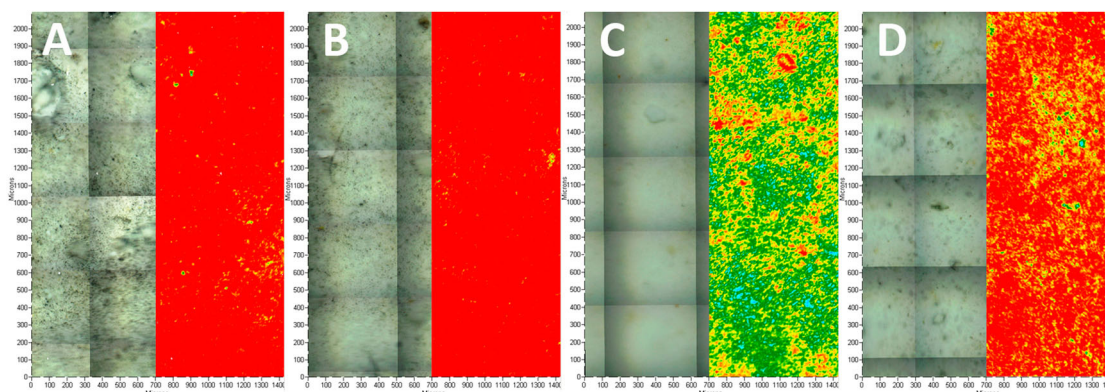


Figure 2. Macrophotographs (left) and 2-D FTIR intensity maps (right). (A) Artificially soiled surface. (B) PG6 with DI water. (C) PG6 with ApC. (D) PG6 with 5% TAC. Level of oil residue: red – high; yellow – medium; green – low.

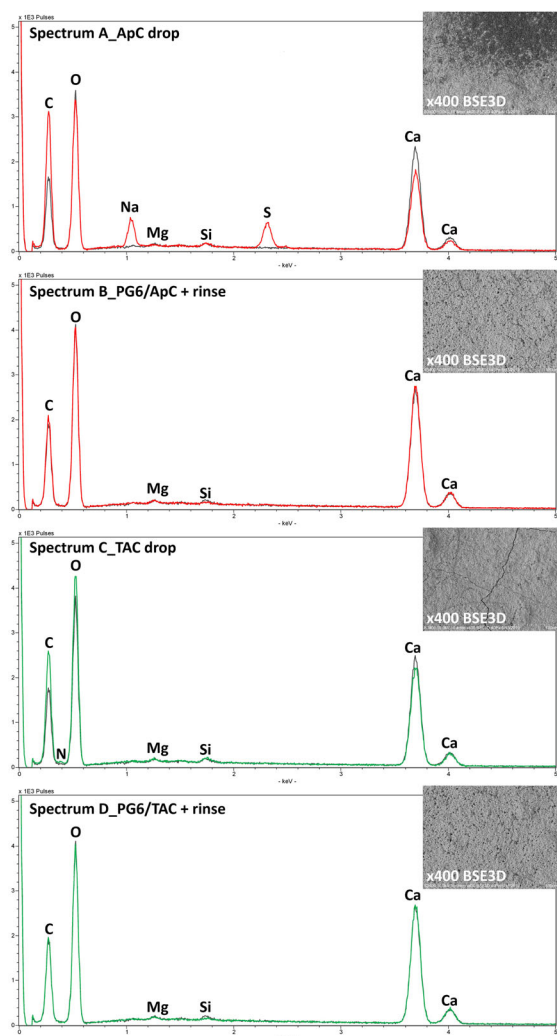


Figure 3. SEM-EDX of ApC and TAC residues on CaCO_3 . (A) ApC applied by pipette. (B) ApC with PG6 followed by rinsing. (C) TAC applied by pipette. (D) TAC with PG6 followed by rinsing. Spectrum of CaCO_3 is included for reference (gray). Backscattered electron images are seen in each upper right corner.

- The % of surfaces presenting minimal changes after cleaning (areas presenting a difference in RGB value below 15).
- The % of surfaces with visible particulate matter removed after cleaning (areas presenting a difference in RGB value above 25).

Macro photographs of a limited selection of identical tests were captured before and after cleaning using a Nikon D810 equipped with a Tamron 90 mm f/2.8 macro lens fixed on a tripod with micro-positioning equipment. Image capturing sequences, calibration chart for correction processes, and plug-ins used for image correction and analysis are described elsewhere (Fox et al. 2018).

Results

Aqueous liquids

Figure 1 shows the results of laboratory tests. The average ΔE^* between clean and soiled surfaces was 22.9. In all conducted measurements the lightness L^* accounted for more than 99% of the change, hence the chromatic values, a^* and b^* , were insignificantly altered in this study. The calculated percentages for cleaning efficiency were determined by comparing color measurements of test areas after cleaning to clean model surfaces. The black line corresponds to $\Delta E_{ab}^* = 2.3$. Values below this are scarcely perceptible (Mokrzycki and Tatol 2011). Best results were obtained when loading PG6 with 5% TAC (green), followed by ApC (orange), 2% TAC (gray) and PCS (purple). Poorer results were obtained with water-loaded PG6 (blue). However, none of the tested systems removed all

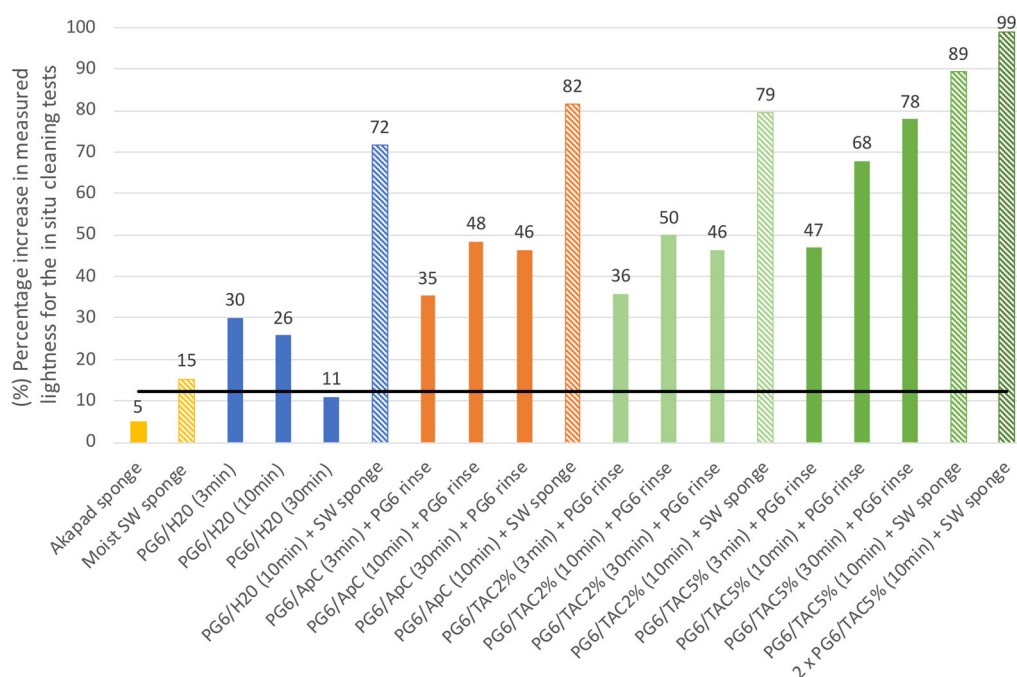


Figure 4. Percentage increase in measured lightness for the *in situ* cleaning tests.

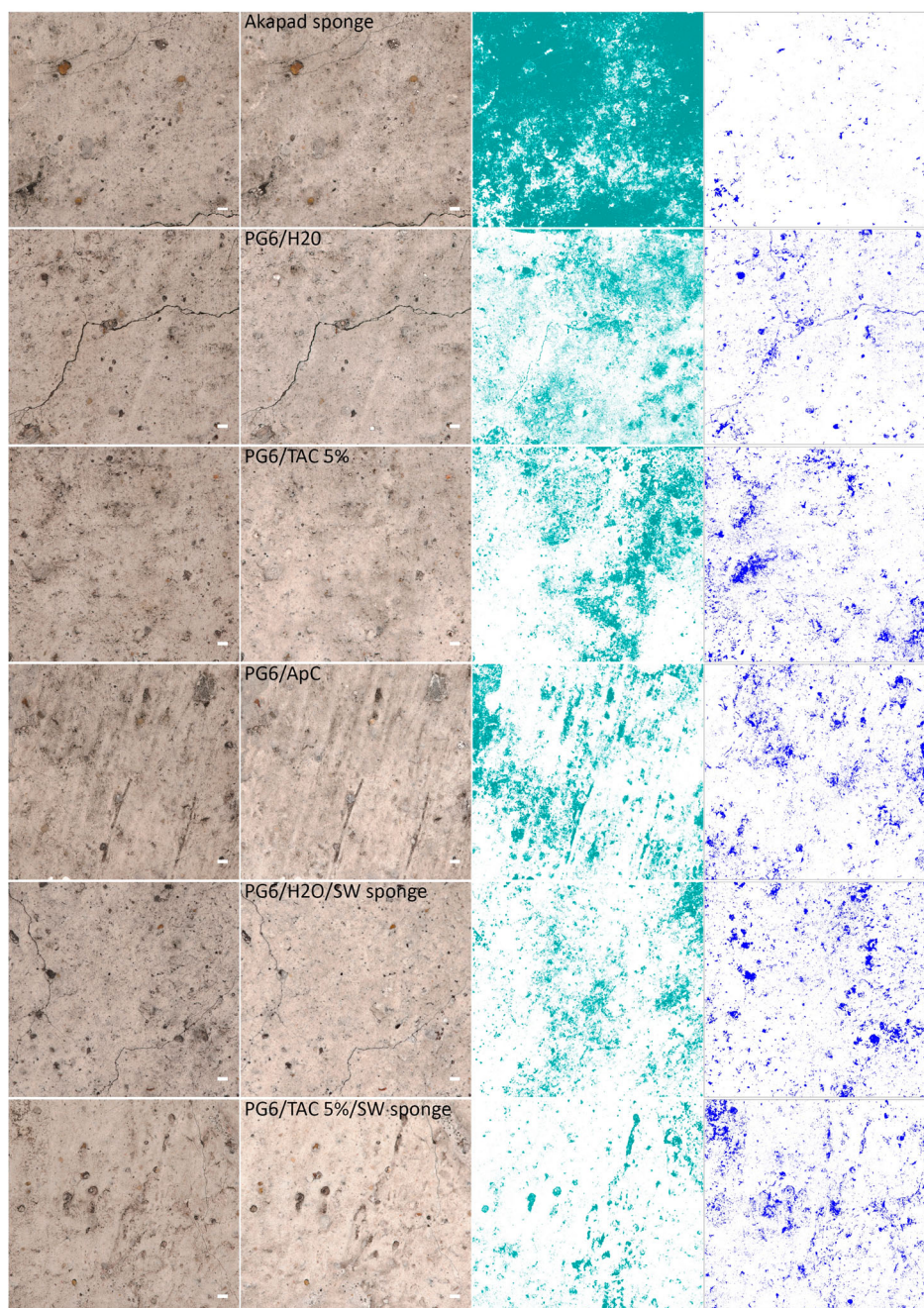


Figure 5. Software-based image analysis results of *in situ* cleaning tests. Columns from left to right: 1. Macrophotographs of trial areas before cleaning; 2. Macrophotographs of trial areas after cleaning; 3. Image analysis of trial areas with minimal changes after cleaning (turquoise); 4. Image analysis of trial areas showing where particulate matter was removed during cleaning (blue). White scalebar on bottom right of images = 1 mm.

soiling. It is noteworthy that cleaning improved when treatments were repeated (pale columns).

2-D FTIR chromatic scale maps (Figure 2) show changes in intensity in the CH stretching bands for paraffin oil, at $2800\text{--}3029\text{ cm}^{-1}$. They are distinguishable from the calcium carbonate substrate (CaCO_3), which has absorptions around 2972 and 2868 cm^{-1} . A strong presence of the oil is represented in the maps by red pixels (A). Blue/ green pixels represent the absence of CH stretching absorptions, with yellow as a medium range. Figure 2 shows that ApC (C) was significantly better at reducing the organic component of the artificial soiling. A smaller reduction was

achieved with 5% TAC (D), whereas DI water (B) had no effect, as expected.

Detection of residual deposits from liquids

SEM-EDX was used to detect any residues on a hydrophilic substrate. Major peaks for calcium, carbon and oxygen were visible in all samples, due to the elemental composition of the mortar (CaCO_3). Silicon and magnesium are considered trace elements in limewash.

SEM-EDX detected marker peaks of sodium and sulfur, indicating the presence of the anionic surfactant

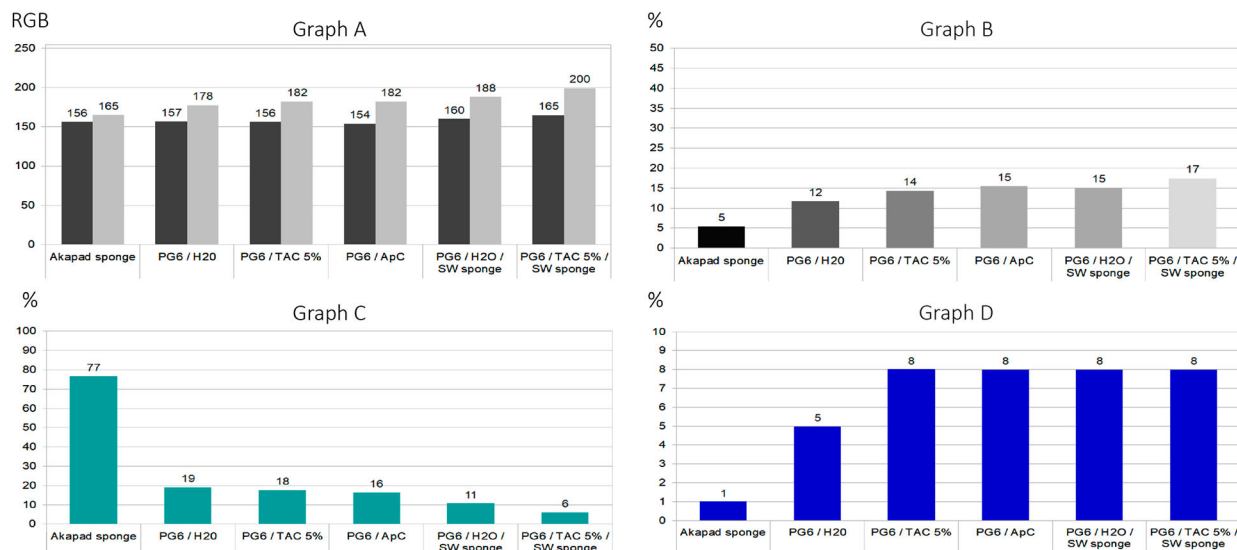


Figure 6. Quantitative data extracted from the software-based image analysis shown in Figure 5. (A) Mean RGB values of grayscale images (0–255) before and after cleaning. (B) Percent increase in lightness after cleaning. (C) Percent of surface with minimal changes after cleaning (turquoise in Figure 5). (D) Percent of surface where particulate matter was removed during cleaning (blue in Figure 5).

SDS ($\text{NaC}_{12}\text{H}_{25}\text{SO}_4$), as seen in Figure 3, spectrum A, where ApC was deposited by pipette but not rinsed.

The 3-D backscattered electron image (BSE) showed surfactant residue, recorded as dark gray. When ApC was applied loaded into PG6 followed by rinsing, residual deposits were below the detection limit, and the EDX spectrum was identical to that of a clean CaCO_3 surface (B).

Surfaces treated with TAC ($\text{C}_6\text{H}_{17}\text{N}_3\text{O}_7$) presented similar results, though detection of TAC-characteristic elements was more challenging. A small peak for nitrogen combined with raised values for both carbon and oxygen (compared to the CaCO_3 reference) confirmed the presence of TAC when applied by pipette and not rinsed (C). When the chelator was applied following the PG6 protocol, the amount of citrate after rinsing was below the detection limit for EDX (D).

In situ cleaning tests

Figure 4 shows the results of *in situ* cleaning tests in Skamstrup Church. The calculated percentages for cleaning efficiency were determined by combining visual observations with color analysis. The average ΔE^* between E_h^* and soiled surfaces was 19. Again, the lightness L^* accounted for 99% of all color changes. The subjectively assessed value $L^* > 85$ was determined to be detrimental, denoting an over-cleaned surface. The black line denotes the perception limit.

Akapad sponge (yellow) and water-moistened SW sponge (hatched yellow) showed hardly perceptible improvement. The results for water-loaded PG6 (blue) were slightly better. Loading PG6 with ApC improves the efficiency of cleaning (orange). The best cleaning

results were obtained when gels were loaded with 5% TAC (green). It is noteworthy that cleaning is greatly improved when swabbing with SW sponge (hatched blue, red and green).

Software-based image analyses supplemented color spectrometry. Figure 5 presents the efficacy of each method from the poorest (top) to the best (bottom). Akapad performed significantly worse than other methods with the lowest % increase in ‘white level’ (Figure 6, graphs A and B), lowest removal of visible particulate matter (D) and corresponding highest % of surface with changes after cleaning (C). The gel rinsing procedure regardless of cleaning liquid (H_2O , ApC, TAC) gave medium improvement (2nd to 4th rows). Again, cleaning is greatly improved when swabbing with the SW sponge (5th and 6th row).

Discussion and conclusion

PG6 provides a viable solution for complex cleaning situations on wall paintings. Both model and *in situ* tests showed a similar trend using different cleaning liquids and demonstrated that traditional methods performed poorly on ingrained soiling. However, as important as it was to use model tiles in preliminary tests, they provided more challenging conditions than those found *in situ*. The difficulty in cleaning model tiles could be explained by a higher oil content in the artificial dirt. Thus, the models presented a pessimistic scenario to challenge the application protocols of the cleaning solutions.

Hydrogel cleaning followed by swabbing (SW sponge) gave significantly better results. Adhesion of recalcitrant dirt was weakened using PG6. This enabled removal during subsequent swabbing,

possibly due to its swelling and detachment under gel contact. This led to the most positive outcome of this study, showing the efficiency of water alone as a cleaning agent, when in combination with gel.

However, the best results were achieved with TAC-loaded PG6. Research on dirt removal confirms its efficiency on this type of soiling (Phenix and Burnstock 1992; Fardi et al. 2018). In this study, the relatively short application times enabled by the gels were a very important aspect of the working protocol, as it has been shown that the dissolution mechanisms of calcium involve a series of complex processes, which are more detrimental with longer contact times (Gervais et al. 2010).

This study focused on the cleaning of white backgrounds not only because dirt is visually more disturbing in these areas, but also because limewash provided homogeneous conditions allowing assessment and quantification of efficiency, which would be complicated on colors. To make this cleaning method viable for wall painting conservation, future research should focus on removing dirt from paint layers. Initial steps have already been taken using water-loaded hydrogels on poorly-bound pigments, where superficial dirt was removed (Segel et al. 2020). A successful use of gels in combination with liquids for ingrained dirt removal on painted areas will provide a much-needed solution to problems currently challenging wall paintings conservators.

Notes

1. Midtgaard 2020.
2. Nanorestore Cleaning Technical Sheet (5 March 2019). Retrieved from http://www.csgi.unifi.it/products/downloads/cleaning_ts_eng.pdf. Nanorestore Gel Peggy Technical Sheet (5 March 2019). Retrieved from http://www.csgi.unifi.it/products/downloads/gelpg_ts_eng.pdf.

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