

# Should we Clean Plastics like we Clean Paintings?

John Morrison and Petronella Nel

Cleaning plastics poses a significant issue in cultural collections. Highly susceptible to attack from mechanical, organic and ionic cleaning agents, it can seem impossible to find products that adequately clean plastic materials without causing damage in the process. This paper aims to address these issues. It explores how techniques, knowledge and decision-making processes used in painting conservation can be adapted and used to deliver sophisticated, inexpensive, and accessible strategies for cleaning plastics.

Using plasticised polyvinyl chloride as a case study, this paper demonstrates how principles of pH, ion concentration, polarity, chelation, gel-formulations and colloidal interface can be used to arrive at optimal methods for cleaning plastics. Combinations of Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR), optical microscopy, colourimetry and accelerated ageing were used to determine the effectiveness of and damage levels of the cleaning processes being evaluated. Ultimately it was found that neat solvents, detergents and acidic or basic aqueous solutions should not be used unaltered but should rather be tailored specifically to the needs of the polymeric material being treated.

## 1. Problems in Cleaning Plastics

As relatively new and unfamiliar materials in collections of cultural materials, the conservation literature on cleaning plastics is marked by unique deterioration problems and few documented or widely accepted options for treatment. More so than other major classes of materials, plastics have chemical characteristics that make them prone to soiling. Being electrical insulators, plastics have strong static surface forces, making them susceptible to attracting particulates (Shashoua 2008: 207). Certain polymers, such as polyesters are particularly vulnerable because they contain ester linkages, where oxygen atoms with free electrons accelerate the material's uptake of moisture and statically charged surface contaminants (Shashoua 2008: 102). Some plastics also contain plasticisers and other liquid additives, known to form sticky surface deposits that attract and bind dust particles (Shashoua 2008: 186-187, Shashoua 2003: 76). From a cross institutional survey, Barabant found soiling is widely reported in plastics held in collections of cultural materials (Barabant 2012). Soiling was also prevalent in a 1993 survey of objects at the Victoria and Albert Museum (Williams 2002).

These issues can be difficult to remedy, as plastics are also vulnerable to damage from conventional conservation cleaning agents. With mechanical cleaning the principal problem is scratching (Kavda *et al.* 2016). Certain kinds of

mechanical action can result in visible scratching from granular material, or changes in colour and gloss due to micro-scratching (Moncreiff and Weaver 1992). Shashoua (2008: 153-4), Grattan and Williams (1999) and Coxon (1993) stress the risks associated with solvent cleaning, as almost all plastics are organic polymers, which can be swelled or solubilised by organic solvents (Shashoua 2008: 129).

Aqueous solutions are generally less harmful to water-insoluble polymeric materials (Shashoua 2008: 210-212; Grattan and Williams 1999). However, polyesters can be damaged by ionic solutions, as their ester linkages are prone to hydrolysis under acidic and alkaline conditions (Timár-Belászy and Eastop 1998: 61; Shashoua 2008: 164). Similarly, the amide functional groups of polyamides are also prone to breakage under acidic conditions (Coxon 1993: 405; Williams *et al.* 1998: 4).

As literature on the cleaning of plastics is developing, it is difficult to find clear answers to these soiling removal problems. However, when surveying literature on the topic of cleaning in conservation more broadly, many of the cleaning problems encountered with plastics also appear with the cleaning of paintings. With decades of innovative research and practice in its wake, it seemed timely to investigate how the materials and techniques used in contemporary approaches to the cleaning of paintings might advance methods for the cleaning of plastics. This

paper explores this proposition by reviewing aqueous approaches to the cleaning of paintings and reporting the findings of cleaning trials. Samples of plasticized polyvinyl chloride (PVCp) were artificially soiled and cleaned with (i) conventional cleaning agents and (ii) aqueous systems like those used in paintings conservation. Results were evaluated using several analytical methods.

## 2. Aqueous Methods for Cleaning Paintings

Paint-films are usually bound by organic polymers, natural or synthetic, and thus exhibit similar cleaning problems to plastics. Like synthetic polymers, materials used as paint media or as coating materials become more polar as they degrade, and thus increasingly prone to attracting particulate matter over time (Wolbers 2000: 158; Wolbers and Stavroudis 2012). Like plastics with high additive content, paintings can also be affected by surface 'bloom' caused by soap-like materials precipitating from the paint film or coating media (Wolbers 2000: 25). The problem with cleaning paintings is especially challenging when soiling materials bear only fractional differences in polarity to the original paint layer beneath. Common examples of such soiling materials include degraded coatings and cross-linked deposits of organic material.

In response to these challenges, aqueous systems, including colloids and high-viscosity polymeric solutions developed to clean painted surfaces are beginning to find broader application in paper and objects conservation (Cushman 2017: 324). Aqueous cleaning mixtures in paintings conservation are often tailored solutions, mixed from a range of chemical reagents to target and isolate soiling materials, while minimising damage to the constituent materials of the substrate being treated (Stavroudis *et al.* 2005; Wolbers 2000; Wolbers and Stavroudis 2012).

Tailoring an aqueous system begins with a characterisation phase, where the substrate is examined to flag its solubility and permeability issues (Wolbers 2000: 158-160). If feasible, further characterisation of the soiling material can inform the removal strategy (Wolbers 2000: 4, 158; Horie 2010: 47, 58). After this, the pH of the solution is optimised for safe and effective cleaning. A higher pH will deprotonate and solubilise acidic materials, or

form insoluble metal hydroxide precipitates, while a lower pH can swell oily materials (Wolbers and Stavroudis 2012: 503). Conductivity of the solution should be isotonic: no greater than 10-20 times that of the material being treated. If the conductivity is too low, the material will swell, if too high, ions from the cleaning solution will be driven into the substrate (Wolbers and Stavroudis 2012: 502-503). Solution pH can be adjusted to a safe pH 6-8 window with the use of buffers and the addition of basic or acidic materials. Conductivity is controlled to a safe isotonic range by diluting the stock solution with deionised water followed by a readjustment of pH to the safe pH 6-8 range.

Once the pH and conductivity of the aqueous solution are established within safe parameters, further elements can be added to modify the properties of the cleaning solution. Some of these agents include:

- *Chelators* to target low solubility product metal salts.
- *Enzymes* to target carbohydrates and animal proteins.
- *Oxidation/Reduction* agents to target tarnishing on pigments, metal hydroxide precipitates, and certain forms of biodeterioration.

Once the aqueous phase has been tailored to the cleaning problem, colloidal materials, including surfactants, gels and other emulsifiers can be added to the cleaning system to increase the detergency, emulsify low polarity solvents, or modify viscosity.

Emulsions are created when two usually immiscible solvents are brought into a continuous dispersion of minute droplets of one liquid (dispersed phase) into another (continuous phase) by a gelling agent and/or surfactant. When emulsified, the solubility parameters of each solvent are not affected, so the cleaning agent can operate in two completely different solubility regions simultaneously. For this reason, emulsions are employed in paintings conservation for complex soils with both water soluble and insoluble components (Wolbers and Stavroudis 2012: 519; Stavroudis 2012). Oil in water emulsions occur when a low polarity solvent is dispersed into a high polarity solvent; a water in oil emulsion is the reverse. A 'microemulsion' is

achieved when the dispersed droplets are only nanometers in length (Baglioni *et al.* 2014). Another advantage of emulsions is that the continuous phase acts as both a suspension aid and a barrier, which limits the spread of solvents and solubilised material through the substrate being treated (Baglioni *et al.* 2014). This holds the treatment agent at the surface, preventing soiling materials from being spread deeper into the pores of the paint film while cleaning.

Gels are rheological materials that modify the viscosity and evaporation time of cleaning solutions. They restrict diffusion of the solution at the surface of the material being treated and enable longer contact times (Wolbers and Stavroudis 2012: 510). A gel is comprised of at least two distinct phases: (i) a mobile liquid phase, and the (ii) 'scaffold'-like structure of the gelling agent (Angelova *et al.* 2017: 231). In some gels, the polymer scaffolding is flexible, stretching under shear force and contracting when the force ceases. This creates forces that draw liquid in and out of the gel during cleaning (Stavroudis 2017: 215; Wolbers and Stavroudis 2012: 511). Like emulsions, these forces within the gels help to ensure soiling materials are not redeposited and limit diffusion of emulsified solvents through the paint film and support. Other gels, such as agarose and gellan gum, behave more like a 'molecular sponge' (Scott 2012: 72), slowly drawing contaminants into the gel through capillary action (Scott 2012: 72-73). These gels are especially useful when the active ingredients in the cleaning agent can damage the paint film or supporting material, and their diffusion needs to be tightly controlled.

### Case Study: Methods for the Cleaning of plasticised polyvinyl chloride

Some of the benefits of these paint cleaning systems, such as a means for controlling the release of solvents, and ways to modify the properties of water to selectively target metal salts or polymers, should have obvious benefits for the cleaning of plastics. These include less leaching of additives, less solvent damage to polymers, and more tools to target specific surface contaminants like metal salts and biopolymers. Plasticised polyvinyl chloride (PVCp) was selected to be a good case study

polymer to test the effectiveness of these methods, for it is often affected by soiling (Barabant 2012), contains a high proportion of easily extracted additives, and is well-known for being problematic when removing water-insoluble soils (Balcar *et al.* 2012).

To test holistically and evaluate these paintings conservation derived methods for the cleaning of PVCp, the experiment design ensured the cleaning trial:

- Tested soiling scenarios likely to occur in cultural collections.
- Evaluated cleaning agents for effectiveness using quantitative and qualitative methods.
- Considered the cleaning agents for their sustainability and practicality.

To this end, samples of commercially available PVCp were artificially soiled to simulate two scenarios often encountered in the museum environment: (i) aged PVCp, with a sticky leached-plasticiser film on the surface with trapped particulates, and (ii) an oily stain on fresh material. Samples were cleaned using conventional cleaning agents used in other previous cleaning trials of PVCp and tailored aqueous solutions like those used in paintings conservation. Results were evaluated using Optical Microscopy, Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (FTIR-ATR), weighing and technical examination. Results were scored and compared using an evaluation matrix, which combines the factors mentioned above.

### 3.1 Background: Cleaning of Poly Vinyl Chloride

PVC is a slightly crystalline thermoplastic polymer (Shashoua 2008: 251-252). Commercial blends are usually mixed with additives, including plasticisers, stabilisers, fillers, pigments, and release agents. Plasticisers are the most frequently encountered additive and may constitute anywhere between 5 to 50% of the material. Phthalate plasticisers commonly found in PVC (Shashoua 2008: 184-187; Huys and van Oosten 2005: 336), are viscous, oily liquids attached to the polymer through weak Van der Waals bonds. Phthalates are added to increase flexibility and remain mobile between the polymer chains after manufacture (Shashoua 2008: 159, 184). Generally, PVC blends containing plasticisers are

referred to as plasticised polyvinyl chloride, PVCp or flexible PVC.

PVCp is a problematic material in museum collections because of its degradation mechanism. The primary mode by which the PVC polymer degrades is dehydrochlorination (Shashoua 2008: 184-188; Shashoua 2003; Osawa 1992). Here, the polymer loses chlorine atoms, which gradually converts the polymer chains into highly light-sensitive polyene sequences (Shashoua 2008: 170). Meanwhile, the plasticiser migrates from the bulk of the material to the surface to form a sticky film, which traps and binds particulates in an unsightly water-insoluble grime. Plasticiser loss also causes shrivelling and embrittlement of the bulk polymer. Over time, the leached plasticiser evaporates, or hydrolyses to form crystals of phthalic anhydride and phthalic acid. Plasticisers also inhibit the growth of polyene sequences, and so their loss further accelerates degradation of the polymer (Shashoua 2008: 186-187; Shashoua 2003: 76).

Literature surrounding the cleaning of PVCp is marked by studies with differing results and evaluation methods (see Balcar *et al.* 2012; Hackett 2014; Muñoz 2014; Sale 1988; Huys and van Oosten 2005). Nevertheless, all studies report concerns about the impact of cleaning agents on plasticisers. Various studies have shown that solvents and detergents can draw plasticiser from the polymer during cleaning (Muñoz *et al.* 2014; Sale 1988). Yet there are certain cleaning problems that demand solvents and detergents, such as oily hydrocarbon based stains (Balcar *et al.* 2012), leached plasticiser films (Muñoz *et al.* 2014) and removal of adhesive residues (van Oosten 2005). Perhaps the biggest cleaning challenge for PVCp, then, is finding ways to remove soiling materials – water-insoluble materials in particular – without displacing plasticiser and other additives from the bulk polymer.

#### 4. Experimental

The aim of the experiment was to find a cleaning method that would remove tough, partially water-insoluble soils that could be realistically encountered in the museum environment. The 'ideal' cleaning agent was defined in terms of its:

- (i) *Effectiveness* in removing soiling materials with minimal damage to the bulk material.
- (ii) *Sustainability* for humans and the environment.
- (iii) *Practicality* in cost, working time, ease of preparation, and workability.

*Effectiveness* was measured by comparing how closely the cleaning agent was able to return the appearance and physical properties of the sample to its original state prior to soiling. *Sustainability* was given by the ecological, health and physical hazards associated with the material, as well as its production related issues. *Practicality* was given by cost, speed of use, difficulty in preparation and workability.

The four key stages of the experiment are described below:

1. Firstly, samples were cut from raw material and characterised using analytical methods and technical examination techniques.
2. They were then cleaned, using a standard method.
3. After cleaning, the samples were run through the same gamut of tests and examinations, and the changes in physical and chemical properties noted.
4. Finally, the experiment entered an 'Evaluation phase', where changes observed between the first and second round of analysis and examination were tabulated and scored to generate a series of numerical vectors denoting the effectiveness of each cleaning agent. Practicality and sustainability (discussed in more detail below) were also scored and results integrated into the final evaluation matrix.

#### 5. Sample Preparation and Characterisation

##### 5.1. Sample space and size

Firstly, appropriate sample numbers were determined. The experimental design involved two soiling scenarios (and an unsoiled control), and eleven different cleaning agents (including no cleaning) for each soiling scenario. This made 33 variables each requiring triplicates, producing 99 samples in

total. Samples were prepared from a commercially available 0.5 mm thick transparent PVCp (Zone Hardware® *Clear PVC Door Curtains*). It bore ATR-FTIR absorption peaks consistent with PVC polymer (3000–2840, 1332, 1252, 876 and 615  $\text{cm}^{-1}$ ) and a phthalate plasticiser (1720, 1464, 1380, 1258, 1123, 1073, 958 and 743  $\text{cm}^{-1}$ ) (Shashoua 2008: 137). Each sample was cut to the microscope slide dimensions of (25.4 x 76.1 mm).

### 5.2. Analysis

Prior to soiling, each sample was subjected to a series of analytical tests. It was important these tests could characterise the sample material and would detect any changes after soiling and cleaning.

Changes in visual appearance due to soiling/cleaning were determined by measuring sample colour, colour change ( $\Delta E_{00}$ ) and gloss using a BYK® Spectro-Sphere Gloss colour spectrophotometer (observer 10°, illuminant D65 Tungsten, colour space CIE2000, average of 10 readings), with data acquisition and processing performed with the OnColour® software (Fields *et al.* 2004).

Owing to its usefulness in identifying and characterising both organic materials, and certain functional groups in inorganic materials, impurities after soiling/cleaning were identified by collecting IR spectra using a Bruker Alpha-P FTIR spectrometer equipped with a diamond ATR window. All spectra were recorded in the spectral range of 4000–375  $\text{cm}^{-1}$  with 32 co-added scans at a spectral resolution of 4  $\text{cm}^{-1}$ . Prior to each sample, the diamond ATR crystal was cleaned with isopropanol, a cleanness test function performed, and a new background measurement collected. Data acquisition and processing was performed with OPUS software (version 7.5, Bruker).

Optical microscopy (BX-51, Olympus) with a digital camera (DP70) with transmitted or reflected light at various magnifications was used to examine and detect changes in surface deposits and topography of the sample. Data acquisition was performed using the DP Controller® and DP Manager® Software.

Sample mass was measured on an analytical balance (GR-200, A&D Instruments). This last method was especially important, as a decrease in mass is an indicator of plasticiser loss (Sale 1988).

Variances in results between triplicate samples are expressed in standard deviations, given by error bars in graphs, and  $\pm$  in text. Qualitative visual observations were also recorded to aid interpretation of data.

### 5.3. Soiling

Once the baseline data and characterisation of every sample was complete, samples were soiled, using one of the two soiling scenarios devised to simulate real cleaning problems occurring in museum environments.

The first scenario was 'aged PVCp', with a surface plasticizer film containing trapped dust and particulates. To simulate this, samples were aged for 4 weeks (672hrs) using a UV accelerated weathering testing chamber (QUV/se, Q-lab) with UVA 340nm fluorescent lamps at an irradiance of 1.2  $\text{W}/\text{m}^2$  at 60°C according to ISO 4892-1 and 3, initiating formation of a visible film of plasticiser on the surface. After this, baseline data for the samples had changed, so they were analysed and characterised again. Samples were then gently pressed face down into the dirt mixture and the excess brushed away. The dirt mixture was derived from a standard recipe, containing a mixture of key classes of organic and inorganic materials (see Table 1), emulating key

Mixture used for this study

Material and Manufacturer	Portion w/w
Carbon/Lamp Black pigment JNH Art & Conservation Materials Suppliers, Australia.	25%
Wheat Starch Archival Survival, USA	12.5%
Gelatine Powder Laboratory Chemicals, AUS	12.5%
Red Iron Oxide Pigment Walker Ceramics, AUS	12.5%
Kaolin AJAX Chemicals, AUS	12.5%
Bolonga Chalk Langridge Artist Colours, AUS	12.5%
Silica Gel Ajax Finechem, AUS	12.5%

Table 1 Dry dirt mixture

constituents found in common dirt (Ormsby 2010). Samples were left for 5 hours, before conducting the cleaning tests, to allow the dirt mixture to settle and congeal.

The second scenario simulated fresh PVCp with a greasy, oily stain composed of a sebum-like synthetic fatty acid mix of mineral oil and 20% (w/w) solution of palmitic acid in isopropanol (Balcar *et al.* 2012: 237). A 2:2:1 dirt, mineral oil and palmitic acid mixture produced a soiling mixture with excellent staining properties and was used to soil samples for the cleaning trials. 0.5 ml of the soiling mixture was applied to each unaged PVCp sample and was left to absorb for ten days before cleaning.

## 6. Cleaning Trial

Five cleaning agents that performed well in previous studies were used in the trials (see Table 2):

A further six cleaning solutions were 'tailored' to the cleaning task using paintings methods and materials. To this end, the cleaning agents were tailored in such a way that they would preferentially remove soiling and degradation products without affecting the PVCp polymer or its additives.

The first solution to be trialed was a chelating solution comprising deionised water, citric acid, disodium EDTA (ethylenediaminetetraacetic acid), and sodium hydroxide, adjusted and diluted to suit the surface of the sample material. The conductivity of the PVCp sample material was found to be between 17 and 100  $\mu\text{S}/\text{cm}$ , and the pH was just under 6.0. Thus it was decided an acceptable pH and conductivity for the cleaning solution was 6.0 and 1000  $\mu\text{S}/\text{cm}$  respectively. At this pH, EDTA can complex with and remove metal salts in the soiling mixture, whilst posing little risk of hydrolysing ester linkages in the phthalate plasticiser. This solution is sometimes referred to as the 'University of

Delaware Solution E', though in this trial it is referred to as the EDTA Solution, buffered to pH 6 with citric acid.

A series of gelled solutions were then created by mixing the EDTA solution with range of colloidal materials, to improve detergency and solvation of water insoluble components (see Table 3). The first gel formulation, titled the 'HPMC' gel, comprised the EDTA solution, mixed with a mild, biodegradable non-ionic detergent, Ecosurf EH-9™, to aid in detergency of organic materials. It was then gelled with 2% (w/v) hydroxypropylmethylcellulose (4000 cps). The next two emulsion-gel formulations were based on polysaccharide materials. A 4% (w/v) Agarose gel was prepared in the EDTA solution and was then soaked in and loaded with ethanol for two hours. A 2% (w/v) Xanthan gum gel was

Cleaning Solution Name Recipe	
<i>EDTA Solution</i>	~.16g DiSodium EDTA ~.16g Citric Acid in 100ml of water Buffered to 5.9pH with NaOH Conductivity: 1000 $\mu\text{S}/\text{cm}$ -1
<i>Emulsion No. 1</i>	96ml 0.5M SDS Solution 2ml Pentanol 2ml n-Heptane
<i>Emulsion No. 2</i>	50ml Deionised Water 50ml White Spirit 6 drops Synperonic A7
<i>HPMC Gel</i>	100ml EDTA Solution 1g Ecosurf EH9 2g Hydroxypropylmethylcellulose
<i>Xanthan Gel</i>	100ml EDTA Solution 2g Xanthan Gum 10g White Spirit
<i>Agarose Gel</i>	100ml EDTA Solution 4g Agarose Soaked in Ethanol for two hours

**Table 3** Tailored aqueous solutions introduced in this study

Cleaning Agent	Recommended in:
<i>Iso-Propanol 100%</i>	POPART (2012) Sale (1988)
<i>n-Heptane 100%</i>	Muñoz <i>et al.</i> (2014)
<i>Ethanol &amp; Water 1:1 v/v</i>	Muñoz <i>et al.</i> (2014) Sale (1988)
<i>Dehypon LS45 1.0% in H<sub>2</sub>O</i>	Huys & Van Oosten (2005) POPART (2012)
<i>Orvus WA Paste 1.5%</i>	POPART (2012) Sale (1988)

**Table 2** Conventional cleaning agents used in other studies

prepared with the EDTA solution and loaded with 10% (v/v) white spirit/water. The Agarose gel was only evaluated on the first soiling scenario (aged PVCp), because it would not conform to the surface of the oily soil samples, or the unsoiled control samples. For these two sample sets, the Xanthan gel was used instead.

The final two complex systems to be created and tested were non-thickened emulsions, based on mixtures of materials that have performed well in previous studies. The first emulsion is an oil-in-water micro-emulsion, comprising a water continuous phase, and an n-heptane dispersed phase. The surfactant and co-surfactants are sodium dodecyl sulphate (SDS – the active ingredient in Orvus® WA Paste, an anionic detergent) and pentanol respectively. The solution was prepared based on a recipe and instructions in Kartsev *et al.* (2000). The second solution is a cheaper and more readily available emulsion. Here, water and white spirit are mixed 1:1 (v/v) to form a two-phase solution, to which a few drops of Synperonic® A7 is added to form the emulsion. This emulsion is sometimes referred to as the 'V&A Mix'. The idea behind testing these emulsions is that they would simultaneously act on both the water soluble and insoluble phases of the soiling mixture.

### 6.1 Cleaning Method

A standardised method of application was used for all the non-gelled cleaning agents. Samples were cleaned with cotton swabs using a 'rolling-and-lifting' action (Ruhemann 1968: 203). By reducing shearing force, this method allows observation of the effects of the cleaning agent independent of mechanical force. The HPMC and Xanthan gels were brushed onto the sample through a low-density Japanese tissue paper, covered with glad wrap plastic film and left for five minutes. The solubilised residue was then cleared with the EDTA solution, as it is standard to rinse the bulk of non-volatiles in cleaning solutions with adjusted water (Stavroudis 2017: 217). For the Agarose preparation, the gel was left in contact with the sample for sixty minutes (Scott 2012). A strip of Mylar® was placed on top of the gel to prevent undue solvent evaporation. Neat solvents and solvent mixtures were simply left to evaporate under ambient conditions. Aqueous systems were given a final rinse (using the same swabbing action) with deionised water to

remove any residual EDTA and citric acid residues. This step differs from paintings conservation, where final rinsing is usually done with mineral spirits, ammonium acetate or volatile methyl siloxane (Wolbers 2000: 157-165; Moskalik-Detalle *et al.* 2017; Stavroudis 2017).

Following a method used by Orsmy *et al.* (2010), samples were cleaned until they reached their 'clean point': a point at which the sample is optically clear, or a point at which there is no visible change in the sample from swabbing. This method was chosen because it is understood not all cleaning agents work at the same rate. Gentle cleaning agents provide safe cleaning but require more working time; aggressive cleaning agents work quickly, though with less control.

After cleaning, samples were left to rest for one month before being physically/chemically analysed. This was intended to allow the surface of the material to recover and normalise after the initial impact of cleaning, so results would reflect the permanent effects of cleaning, not temporary displacement of the surface chemistry of samples immediately after cleaning. During this time, samples were stored on lipped, polypropylene trays and covered with a sheet of archival cardboard lined with Mylar® film, preventing deposition of dust and exposure to light. The lipped design of the trays created a small gap between the tray and cardboard covering, facilitating ventilation and off-gassing of the samples after cleaning.

### 7. Evaluation

To evaluate the overall performance of the cleaning agents, a detailed evaluation matrix was used to generate three vectors describing the effectiveness, sustainability, and practicality of the cleaning agents for each soiling scenario. Effectiveness was determined quantitatively by a sum of five vectors, each scored out of 100. The total score for effectiveness is thus given by a number from 0 to 500, where zero is the best result. Practicality and sustainability were each scored out of 100. Thus, the overall total is a score out of 700. Metrics for each of these scores was based on undesirable attributes rather than desirable attributes, and so a lower score indicates a better result.

The five categories of the effectiveness score were:

- I. *Visual quality of cleaning*, given by the colour change before soiling and after cleaning in  $\Delta E_{00}$ , multiplied by a scaling factor such that the worst performing cleaning agent scored 100. A lower score indicates more visual fidelity to the sample's original appearance.
- II. *Effectiveness in removing oil components of soiling mixture*. This was given by how effectively the cleaning agent removed the unwanted oily components of the soiling mixture. In the aged soiling scenario, this meant reducing the level of plasticiser at the sample surface to the sample level it was at before ageing. This was calculated by measuring the change in plasticiser index (PI) before ageing and after cleaning (as per standard method Deutsches Institute für Normung (DIN) 53-405-1981 *Testing plasticisers: determination of migration of plasticisers*. Available from <https://www.din.de/de>). Plasticiser index is an integer in per cent used to map shifts in plasticiser concentration on the polymer surface. For PVCp, it is calculated by comparing the change in relative intensity of infrared spectra between the carbonyl (C=O) stretch peak at  $1722\text{ cm}^{-1}$  and the  $\text{CH}_2$  in-plane deformation peak at  $1426\text{ cm}^{-1}$  at the start-phase and end-phase of the experiment (Shashoua 2003; Muñoz *et al.* 2014). In the oily soil scenario, remaining mineral oil surface residue is detected by mapping shifts in ratios between methyl ( $-\text{CH}_3$ ) and alkane (C-H) stretches. Mineral oil contains a higher ratio of alkane groups to methyl groups than the plasticizer in the PVCp sample material, so an elevated alkane peak at  $2852\text{ cm}^{-1}$  against the methyl peak at  $2958\text{ cm}^{-1}$  in an IR spectrum indicates the presence of mineral oil. These ratios were multiplied by a scaling factor such that the worst performing cleaning agent scored 100. A lower score indicates better removal of oily materials in the soiling mixture.
- III. *Effectiveness of dirt removal of soiling mixture*. This was given again by the ATR-FTIR data. Each of the residual peaks and features of

the dirt components and/or degradation products and palmitic acid present in the sample's IR spectrum after cleaning (see Table 4 for breakdown of how each component was identified on IR spectra) was given a score from 0–3, where 3 denotes a 'strong' peak, and 1 a 'trace'. The sum of the values of the peaks was added, then multiplied by a scaling factor. For sample set 2, phthalate degradation products such as carboxylic acids and alcohols were also evaluated. Again, the final score ranged from 0–100, where 100 indicates the highest amount of contaminant features in the IR spectra.

- IV. *Scratching and surface damage*. Each sample group's extent of scratching was assessed using microscopy and scored a number between 0–2, where 0=no scratching, 1=oderate, 2=severe. Any residue, bloom or other surface damage added an additional point to this score. This score was then multiplied by 33.33, to generate a score out of 100.
- V. *Sample mass change*. Using the analytical balance data from the unsoiled control samples, the loss in sample weight in percent was scaled such that the sample with the highest depreciation in mass scored 100.

Practicality was given by four scores out of 25, which included:

- I. *The price of the cleaning agent* in AUD, scaled to a figure between 0–25.
- II. *The speed of cleaning*: the average number of swab rolls required to clean the samples, again scaled to a figure between 0–25.
- III. *The difficulty in preparation*, given by the number of ingredients required plus the number of hours taken to prepare the mixture, scaled to a figure between 0–25.
- IV. *Workability*: the sum of aspects that made the cleaning agent difficult to work with, including difficulty to control, unpleasant and/or hazardous fumes and vapours, poor dirt suspension and difficult clearance. The sum of these factors was scaled to a figure between 0–25.

Material	Features
<i>Carbon Black</i>	Significant baseline shift due to strong absorbance of carbon (Tomasini, Siracusano & Maier 2012).
<i>Gelatine</i>	Distinctive amide bands at 1626 cm <sup>-1</sup> , 1530 cm <sup>-1</sup> & 1447 cm <sup>-1</sup> , and OH stretching at 3290 cm <sup>-1</sup> (Stuart 2007; Derrick Stulik & Landry 1999).
<i>Starch</i>	Peaks at 3289 cm <sup>-1</sup> , 1641 cm <sup>-1</sup> , 994 cm <sup>-1</sup> , 525 cm <sup>-1</sup> , 572 cm <sup>-1</sup> , 436 cm <sup>-1</sup> , 406 cm <sup>-1</sup> (Stuart 2007; Derrick Stulik & Landry 1999).
<i>Iron Oxide</i>	Peaks at two distinctive peaks at 3687 cm <sup>-1</sup> & 3615 cm <sup>-1</sup> , twin peaks at 1030 cm <sup>-1</sup> & 1007 cm <sup>-1</sup> & 911 cm <sup>-1</sup> . Two large bands at 520 cm <sup>-1</sup> , 434 cm <sup>-1</sup> (Stuart 2007; Derrick Stulik & Landry 1999).
<i>Kaolin</i>	Peaks at 3685 cm <sup>-1</sup> & 3615 cm <sup>-1</sup> . Asymmetric twin peaks at 1001 cm <sup>-1</sup> & 911 cm <sup>-1</sup> . 3 large bands at 527 cm <sup>-1</sup> , 459 cm <sup>-1</sup> & 408 cm <sup>-1</sup> (Stuart 2007).
<i>Bologna Chalk</i>	Peaks at 3498cm <sup>-1</sup> , 3396 cm <sup>-1</sup> , 1619 cm <sup>-1</sup> , 1105 cm <sup>-1</sup> , 667 cm <sup>-1</sup> , 597 cm <sup>-1</sup> , 422 cm <sup>-1</sup> (Stuart 2007; Derrick Stulik & Landry 1999).
<i>Silica Gel</i>	Peaks at: 1057 cm <sup>-1</sup> , 797 cm <sup>-1</sup> & 447 cm <sup>-1</sup> (Stuart 2007).
<i>Mineral Oil</i>	Mineral oil has a higher C-H stretch peak at 2852 cm <sup>-1</sup> , and a shorter C-H peak at 2958 cm <sup>-1</sup> , whereas in PVCp, the 2958 cm <sup>-1</sup> is significantly higher than the 2852 cm <sup>-1</sup> peak.
<i>Palmitic Acid</i>	Shows significant C-H stretches below 2900 cm <sup>-1</sup> , and much lower C-H stretches over 3000 cm <sup>-1</sup> . The carbonyl peak in palmitic acid sits at 1698 cm <sup>-1</sup> , while the PVCp carbonyl peak is at 1722 cm <sup>-1</sup> .
<i>Carboxylic Acid degradation products</i>	Broad, asymmetric bands to the lower wavenumber region of plasticisers carbonyl peak (Pimental Real 2008, p. 749; Muñoz <i>et al.</i> 2014, p. 240), O-H stretching centres around 3400 cm <sup>-1</sup> (Derrick Stulik & Landry 1999, p. 93).
<i>Alcohol degradation products</i>	Broad O-H bands centring at 3289 cm <sup>-1</sup> (Muñoz <i>et al.</i> 2014, p. 240)

**Table 4** FTIR identification of soiling materials

To compare the sustainability of each product, the 'Chemistry Scoring Index', introduced by Verslycke *et al.* (2014) was used to generate indices denoting the hazardousness of the cleaning agents to human health, and the environment, as well as their physical hazards and reactivity. The scoring matrix makes use of the Global Harmonised System (GHS) and Oil Spill Prevention, Administration and Response (OSPAR) Harmonised Offshore Chemical Notification Format (HOCNF) (<https://www.ospar.org/work-areas/oic/chemicals>) to generate a weighted score for the hazardousness of a chemical, or chemicals in a mixture. The GHS and HOCNF provide universal and accessible criteria to identify and measure the extent of hazards that specific chemicals pose to people and the environment. HOCNF classifications specifically relate to threats posed to marine life. Usually, these classifications can be found in the Safety Data Sheet. The matrix was altered to incorporate the sustainability of the cleaning agents in a broader, more holistic sense. Modifications incorporated the renewability of the chemical components, and the social and ecological consequences of their production as components of

the matrix. Sustainability was given by the cleaning agent's CSI index scaled to a figure out of 100.

## 8. Results and discussion

The results for overall performance are presented in **Chart 1**. The consensus from these results is that tailored aqueous systems provide more efficient removal of soiling material than off-the-shelf materials, provided that the gelling agent is compatible with the material, and that the solvent volume is kept to a minimum. Of these tailored solutions, it was found that <sup>®</sup>HPMC Gel is the most versatile, and best performing cleaning agent for PVCp for both soiling scenarios, closely followed by the SDS-Based Emulsion No. 1. Aqueous solutions contain lower volumes of active ingredients, significantly improving their sustainability profiles (Wolbers 2017). Hence, the EDTA Solution, the HPMC Gel, Orvus<sup>®</sup> WA Paste score well in terms of sustainability. Practicality issues associated with neat solvents include volatility and difficulty managing vapours. Tailored aqueous solutions were adversely scored by their difficulty in preparation. **Table 5** summaries the key pros and cons of each mixture.



Chart 1 Total score.

Cleaning Solution Name	Pros	Cons
<b>Isopropanol</b>	Evaporates quickly, residue free, little preparation required.	Poor dirt pickup.
<b>Heptane</b>	Evaporates quickly, residue free.	Poor dirt pickup, damages PVCp material, very flammable and dangerous to store, derived from petroleum.
<b>Ethanol &amp; Water</b>	Easy to prepare, gentle yet effective cleaning for aged sample material.	Ineffective on oily soils.
<b>Dehypon LS45</b>	Simple to prepare.	Poor ecological impact, difficult to source, adverse ecological impacts, slow, ineffectual cleaning.
<b>Orvus WA Paste</b>	Simple to prepare, low ecological impact, good cleaning results across both trials, very cost effective.	Very slow to clean.
<b>Emulsion No. 1</b>	Fast and effective cleaning results in both soiling scenarios.	Some components hazardous to health and the environment, difficult to prepare.
<b>Emulsion No. 2</b>	Effective cleaning simpler to prepare than most emulsions, easy to source ingredients.	Poor environmental fate, derived from petroleum, caused significant loss of plasticiser.
<b>EDTA Solution</b>	Controlled and effective cleaning for aged PVCp.	Less effective for oily soils, very slow to clean, some difficulty in preparation
<b>HPMC Gel</b>	Excellent cleaning in both soiling scenarios, eco-friendly and non-hazardous.	Difficult to prepare, requires thorough clearance.
<b>Xanthan Gel</b>	Very fast and visually effective cleaning.	Emulsified solvent leaches plasticiser, residue difficult to clear after cleaning.
<b>Agarose Gel</b>	N/A	Wholly unsuitable for cleaning PVCp.

Table 5 Summary of the pros and cons of each cleaning solution

### 8.1 Effectiveness.

The scoring results for effectiveness are shown in [Chart 2](#). Cleaning agents most effective at reducing the soiling mixture, and providing the best visual cleaning result appear to be those that combine:

- (i) Strong chemical affinity for the soiling material.
- (ii) Good soiling suspension and detergency properties.

It is clear to see why a strong affinity for the soiling material produced a better cleaning result. It is axiomatic in conservation that like dissolves like, and thus it makes sense that ionic cleaning agents strongly reduce peaks associated with the inorganic dirt mixture in the IR spectra, and detergent solutions reduce the oily portions (see [Table 4](#) for wavenumbers) very effectively. As an exception to this rule, solvents alone do not appear to be very effective at removing the IR peaks associated with the oily materials in both soiling mixtures. It is surmised that for cleaning PVCp, it is not enough to simply solubilise soiling materials. Because PVCp is very sticky and is liable to draw contaminants through the plasticizer and absorb oily materials ([Balcar et al. 2012](#)) the best cleaning systems appear to be those with a strong detergency mechanism

to prevent the soiling mixture from being redeposited into the material's substrate.

An interesting facet of the visual cleaning quality data is the inverse correlation between the redness of the sample material after cleaning and the presence of EDTA in the cleaning solution. Samples not cleaned with EDTA were generally redder than those cleaned with EDTA after cleaning. Given the only red component in the soiling mixture is red iron oxide, results indicate EDTA's effectiveness as a metal sequestering agent, consistent with its general use in conservation as a treatment for iron staining ([Timár-Belazsy and Eastop 1998: 222](#)).

In findings consistent with previous studies ([Sale 1988; Muñoz et al. 2014](#)), it was observed that neat solvents and solutions with a high solvent volume cause significant sample weight loss, indicating leaching of plasticizer during cleaning. Solvents in emulsified systems had mixed success in this regard. The Xanthan gel solution showed a weight loss of  $0.3\% \pm 0.02\%$  after cleaning, a comparatively significant reduction. Conversely, the Synperonic® A7-based Emulsion No. 2 caused less weight loss, and the SDS-based microemulsion only showed weight loss within the standard deviation of the control samples. Perhaps then, only micro-emulsions offer the necessary amount of control to deliver low polarity solvents as cleaning agents for PVCp.



Chart 2 Effectiveness score.

In terms of mechanical damage, it is noted that cleaning duration and mechanical damage are not related. Instead, it was noticed that the non-ionic cleaning solutions – Isopropanol, n-Heptane and Dehypon LS45 – saw greater amounts of scratching, most likely due to an inability to solubilise and remove rough, metal and ionic based soiling materials. Though no gel residues were detected on the IR spectra for any of the gelled solutions, it was possible to observe residues on the samples cleaned with Xanthan gum gels via microscopy. Also a small deposit of gel-like material was identified on one of the samples cleaned with HPMC gel. It is unclear whether these deposits will impact the material in the future.

### 8.2 Sustainability.

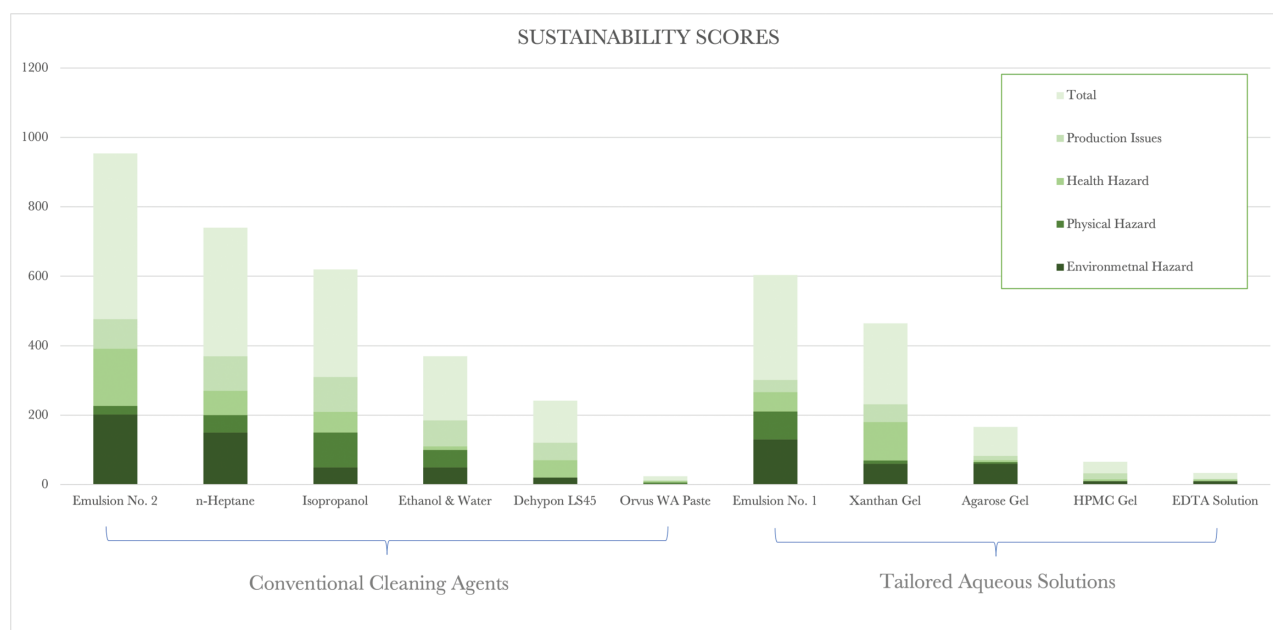
The sustainability results are summarised in **Chart 3**. The CSI sustainability matrix data show that to create more sustainable cleaning agents, limiting the quantity of active ingredients in solutions is just as important as choosing ingredients that have fewer undesirable effects in their pure form. For this reason, aqueous solutions generally post lower/more ideal CSI scores than organic solvent-based recipes, because they typically contained lower proportions of active ingredients, even though the active ingredients are not necessarily without health or environmental hazards themselves. For example, while EDTA is a relatively non-problematic material

when used in the small portions required in conservation cleaning formulations, it poses serious ecological problems when released into the environment in industrial quantities ([Jessop et al. 2015: 2669-2670](#)).

### 8.3 Practicality

The scores for practicality of use are summarised in **Chart 4**. In terms of workability, the consensus from the cleaning trials is that the HPMC gel provides the best properties out of the cleaning systems trialed, as it was found to wet the material surface without running, and was not difficult to clear. Xanthan on the other hand, was found to not adhere well to the sample’s surface topography, yet was difficult to clear after cleaning. Though the SDS-based Emulsion No.2 was found to be easy to handle during cleaning, its foaming action and difficulty in clearance made it hard to use.

The major finding from comparing raw material prices is that solvents are significantly cheaper per gram than aqueous solution ingredients. However, because solvents are used in such large quantities, they are ultimately more expensive than aqueous cleaning agents used in small quantities. Interestingly, there appears to be no observed relationship between price and sustainability, as the two most sustainable solutions are also the least expensive, hopefully challenging the assumption that green conservation materials are more expensive than non-sustainable materials.



**Chart 3** Sustainability score.

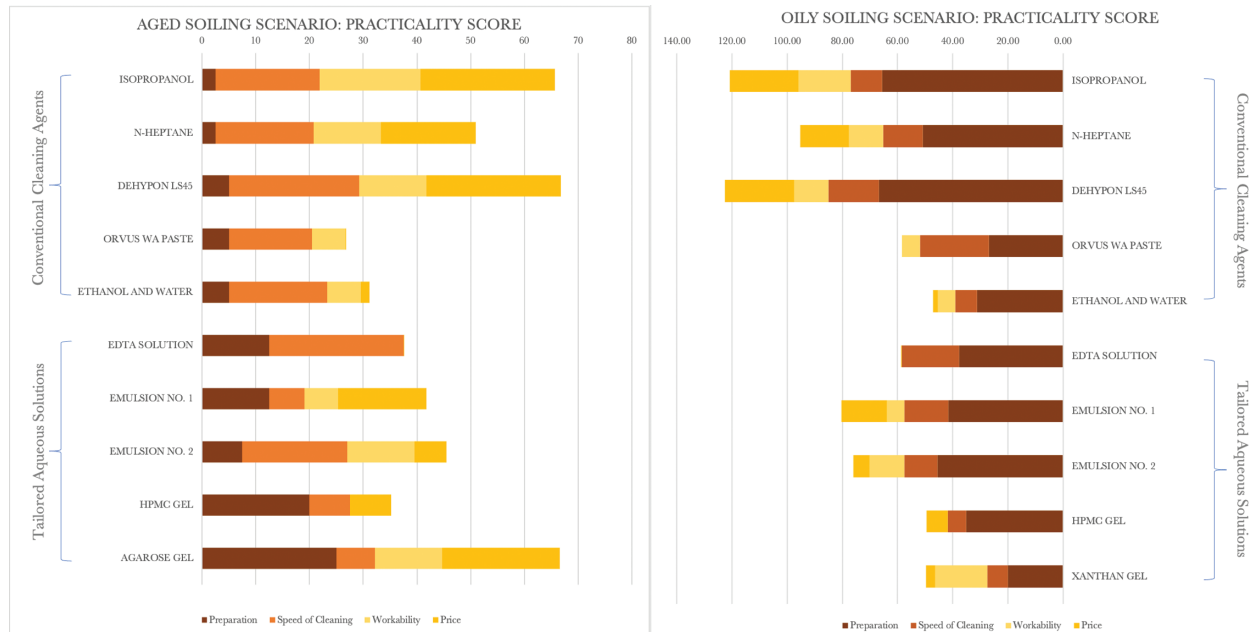


Chart 4 Practicality score.

## 9. Conclusions and Points of Departure

It is concluded from the results of the above experiment that the 'tailored' approach, the HPMC Gel and Emulsion 1 in particular, generally outperformed off the shelf formulations, such as neat solvents and simple detergent solutions in nearly every aspect evaluated. They seem to offer greener, cheaper and more effective solutions to the cleaning problems associated with PVCp. It would be timely and appropriate to further investigate and compare the long-term impacts of cleaning PVCp with both generic and paintings conservation cleaning agents/techniques. Some accelerated ageing experiments were undertaken as a part of this project. While preliminary results show promise, this field of study would benefit from larger and more comprehensive accelerated ageing studies in the future.

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## Biographies

**John Morrison** is an emerging conservator from Melbourne, Australia. Trained in objects conservation at the University of Melbourne, John is developing his skills and expertise in the fields of conservation of built heritage, with a focus on the preservation of decorative and construction materials.

[jackjohndavidmorrison@gmail.com](mailto:jackjohndavidmorrison@gmail.com)

**Dr Petronella Nel** is a Associate Professor and researcher at The Grimwade Centre at the University of Melbourne. She has a MA (Cultural Materials Conservation), PhD (Chemistry) and BSc (Honours) all obtained from the University of Melbourne in 2006, 2000 and 1990 respectively. She is leading a collaborative ARC Linkage Project 'A national framework for managing malignant plastics in Museum Collections'. She is interested in developing analytical techniques for characterizing materials to inform their preservation. [pnel@unimelb.edu.au](mailto:pnel@unimelb.edu.au)

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## Standards

- ISO 4892-1 Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance
- ISO 4892-3 Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps
- DIN 53-405-1981 — Testing plasticisers; determination of migration of plasticisers.

