OPTIMIZING THE RIGIDITY OF GELLAN AND AGAR GELS FOR CLEANING SENSITIVE ACRYLIC EMULSION PAINTED SURFACES

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Abstract

This paper is based on the explorative study aimed at furthering the understanding of cleaning of acrylic emulsion painted surfaces and evaluating the effectiveness of gel cleaning with rigid gels without affecting the original paint layer. In the present study, the gellan and agar gelling materials were exploited for cleaning acrylic emulsion painted surfaces by optimizing their rheological properties using two application methodologies. The gellan and agar gels were used on soiled and unsoiled acrylic painted surfaces and micro-photographed at 50x magnification using Dinolite digital microscope under normal, raking and ultraviolet fluorescence light. The experimental results have demonstrated better contact, no loss of water and fair cleaning results for gellan gel as compared to agar. The effectiveness of the cleaning operation was also observed using ATR-FTIR and SEM-EDX in the present study. The results can be extended for cleaning of other sensitive painted surfaces.

Keywords: Acrylic paint; Cadmium; Cleaning; Gellan; Agar; Swelling

Introduction

Surface cleaning of painted surfaces is usually a straightforward process but the cleaning of acrylic emulsion paintings is very complicated and challenging due to the high susceptibility of soiling of acrylic paints and sensitivity of the film to the aqueous and solvent mode of cleaning. Acrylic paint films always remain soft and tacky due to low glass transition temperature, low molecular weight and low minimum film-forming temperature [1]. The problem of soiling of acrylic paints and their permanent imbibing in the paint matrix have been an area of utmost concern [2, 3]. The swelling capacity of acrylic paints is about 100 times more than oil paint films. Acrylic paints also show high sensitivity to a range of solvents whose swelling capacities are dependent on their position in the polarity scale. The swelling of acrylic paint is minimum on both the higher and lower ends of the polarity scale with maximum swelling by chlorinated solvents and aromatics [4]. Therefore, investigations started on providing the solution to prevent the surface from soiling in the first place, so that the need for cleaning does not arise. Investigations on finding suitable varnishes for the clean surface of the acrylic paint have been one such step in precluding the necessity to clean the painting. Several studies have been conducted to understand cleaning efficiency and efficacy using infrared spectroscopies, SEM-EDX, ESEM, AFM etc. [5-7]. Several analytical examinations were employed to study the after cleaning effects on the paint film. X-ray photoelectron spectroscopy (XPS) revealed that the aqueous cleaning methods via swabbing with de-ionized water in combination with ecosurf and/or tri-ammonium citrate have shown an increase in the electrical charge on the surface thereby indicating the leaching out of the surfactants from the bulk paint.
film [8]. Near-edge X-ray absorption fine structure (NEXAFS) also provided information with XPS on the extraction of surfactants, residues left on the surface due to the employed cleaning systems, the difference in the pigment concentration on the paint surface etc. [9].

In addition to the solvent sensitivity, the selectivity of organic solvents has also been an issue while cleaning the acrylic painted surfaces. In this circumstance, gels may provide an effective solution of cleaning to this kind of sensitive painted surfaces. Many gelling materials have been tested and found effective in controlling the cleaning mechanism by restricting the movement of liquid in the gel matrix. However, viscous gels such as gels with cellulose ethers, polyacrylic acid gels, etc. have a tendency of leaving the residue on the surface on which they have been applied. There has been clearance concern always associated with these physical gels [10]. Now, there is a growing body of research exploring the rigidity factor of gels to adapt for cleaning of surfaces sensitive to water, minimizing the risk of gel residues. Application of rigid gels was introduced by Richard Wolbers in the field of conservation which has been subsequently researched by others. Two gelling materials viz. agar and gellan have been tested so far for cleaning on many types of surfaces, their properties have been studied, and their adaptability in different solvents at different pH levels have been tested.

Agar also known as agar-agar is a polysaccharidal complex and is derived from a species of red algae of the Gelidium family [11]. It is composed of agarose, a linear polysaccharide with strong gelling properties, and agarpectin, a non-gelling sulfated polysaccharide. Agarose consists of approximately 70% of the mixture and is responsible for the gelation. It is also available in the market in pure form. It forms more transparent gels than the cloudy agar gels. Agar can control the release of water very efficiently because the agarpectin in agar reduces the pore size of the polymer thus retarding the expulsion rate of water from the gel thus making a good humidifier for many conservation requirements [12]. Agarose has a higher rate of syneresis than agar due to the absence of agarpectin, a sulphated polysaccharide and is also very expensive to agar. Therefore, agar has been found more in usage than agarose in the conservation laboratory. It is also considered as reversible sol-gels. When the agar is warm and liquid in the solution, it is considered as sol, as the solution cools, the gel is formed which is like a rigid cake or cube [11]. In the gelation mechanism of agar, a shift from a random coil in solution to a double helix occur in the initial stages of gelation and to bundle of double helices in final stages. The gelation mechanism is reversible and this reversibility is induced by the application of heat [13]. The concentration of agar can be adjusted from fluid gel to rigid gel as per the desired application and sensitivity of painted surface to water [14]. The rigidity of agar is useful for water sensitive surfaces as it does not impregnate the paint layers due to its rigid structure and retains the water in its gelling matrix while allowing the water to come in contact with the painted surfaces [15]. It also magnifies the details of the surface on which it is applied thus creating a ‘lens effect’ which can be useful for conservators to see the magnified textures and details which is not visible with an unaided eye [16]. The efficacy of the gel can be improved by altering the concentration of agar and solvent, the temperature of the poultice of application, the length of application and the number of applications on the surface [11]. One of the limitations of the rigid gels is that there is difficulty in establishing proper contact with the uneven and not so flat surfaces as they are rigid. To overcome this limitation, a variant of agar named Pyhtagel has been proposed which is more flexible and transparent than agar and suitable for the uneven surfaces [12]. Ultraviolet radiation of 365 nm, however, showed some faint fluorescence at some area particularly along the edges of treated areas which was also confirmed by P. Cremonisi [12].

Gellan is a high molecular weight polysaccharide *Sphingomonas elodea* (formerly called as *Pseudomonas elodea*). Molecules of gellan gel are in the disordered coil (single chain) upon heating in aqueous solutions. The molecules transform into an ordered double helical conformation upon subsequent cooling, followed by associations between the helices through weak interactions, such as hydrogen bonds and van der Waals forces. Thus ordered helix of double strands are formed at low temperature and at high temperatures, polysaccharides of single strands develop which reduces the viscosity [17]. Gellan gels are stable at a wide range of pH which can gelify the deacidifying agent and reducing agent for deacidification and
reductive bleaching processes [18]. The acyl content guides the properties of gellan gum gel. For the conservation purposes, a deacylated form of gellan gum is used which forms hard and brittle gels in front of cations [18]. Besides being more transparent than agar, the rigid gel obtained from Gellan is more effective in terms of water retention, at low concentrations of 1-2% [19]. Recently a comparative study was done on the cleaning of paper between the immersion treatment and by application of rigid gellan gum to find which of these two methods remove the sizing material less. It was found that application of gellan gum removed gelatine much lesser than by immersion treatment and found its efficacy in controlling volume and water release timing to paper during aqueous treatment [20]. FTIR analysis showed that it does not leave residues on the paper. It was also helpful in removing acidity from the paper as HPLC measurements showed the pH of paper increased after the treatment. This indicated that the cleaning with rigid gel was effective and did not induce any morphological changes to the paper [21].

The principal objective of this research was to assess the suitability of the hydrogels by exploiting their rigidity for the cleaning of acrylic emulsion paints and their after-effects on the paint surface. For this purpose, two rigid gelling materials, both coming from the food industry were used in five different concentrations. Two cleaning methodologies for cleaning, one by direct application of gels on the paint surface, leaving for few minutes for gel to take action and the second method to harness the rigidity of the gels as wet erasers were used. The idea of using these gelling materials as wet erasers is that since acrylic painted surfaces are very soft and tacky, application of any dry cleaning materials such as erasers or sponges entails the risk of polishing the surface and increase in gloss. These erasers which are basically polyvinyl chloride erasers also contain plasticizers which are left on the paint surface as permanent residue [22]. The assessment of the efficiency of gelling materials were in terms of controlled release of water, swelling on direct placement on the paint surface ease of removal of the gels, clearance issues and changes in the surface topography. In this study, visual analysis, digital microscopy was used to examine the surface before and after cleaning. The SEM-EDX study was carried out for elements present in the control samples and any migration from the bulk paint film to paint surface and FT-IR analysis for estimating the acrylic binder, finding any swelling and residue left on the paint surface.

Materials and Methods

Preparation of painted canvas samples
The medium grained, pre-primed (titanium dioxide), acid-free cotton duck canvas was used for preparing the painted samples. Cadmium yellow medium paint from Camel paint brand was used for the paint application on the canvas. A mechanism was devised for paint application with local materials such as glass plate, scale and some clips on canvas samples in order to obtain even thickness throughout the film on all samples (Fig.1). The paint was applied in a drag-down technique to a dry thickness of 100±20µm. A multi-pigmented mock acrylic painting was also prepared on titanium dioxide pre-primed stretched canvas. Each sample was kept in drying state for six months in museum condition. To simulate the type of museum dirt on the stretched painted canvas, a layer of synthetic soiling mixture prepared as per the international protocol mentioned elsewhere was applied on the paint layer [7, 8, 23].

Preparation of hydrogels from gellan and agar
Low acyl variety of gellan gum (KELCOGEL®CG-LA, material number 20006521) was obtained from CP KELCO (Burzin and Leons), Mumbai, India. Calcium acetate of Fisher Scientific and agar powder of Rankem brand was procured from a local supplier. All materials used as received without any further modification. The process of preparing gels with gellan and agar was almost the same. Selected amounts of agar and gellan gels were measured, dispersed and mixed in cold de-mineralized water in borosil glass beakers using glass rods. The mixtures were stirred for a while and then they were placed on the induction cook-top for a couple of minutes at 600W. Agar solution was heated until the time all agar granules that were stuck on the inner side of the glass beaker melted and dropped into the solution. In case of
gellan gel preparation, calcium acetate in a ratio of 0.40 g/L was added to the dispersed gellan gum powder in de-mineralized water. Calcium acetate was added as sequestrant to bind the soluble calcium in gellan solution as calcium ion in the solution would inhibit the total hydration of LA gellan gum. Upon heating, gellan powders dissolved completely in two minutes of heating. All the gel solutions were well mixed before, during and after the heating process. After heating, hot solutions of both gellan and agar were poured into the melinex trays and allowed to cool for a couple of hours. Several small melinex trays were prepared to provide the high flexibility to the trays so that entire gel sheet can be taken out of the trays. After the solutions cooled, within two hours all solutions formed gels. Agar formed a thick cake like pale yellow gels whereas gellan formed water containing colourless transparent sheets (Fig. 2). Thus both powders formed rigid gels and rigidity increased with the increasing concentration and after a certain percentage, the powder could not be completely dissolved in de-mineralized water even upon heating for a longer period. Though some amount of solvents, chelating agents or other cleaning agents can be added to these gels when they are in sol form or after the formation of a gel, in this study, also to test the cleaning efficiency of both gelling materials, no other cleaning agents were added. Both gellan and agar gels were prepared in five concentrations of 1, 2, 3, 4 and 5% and were applied on unsoiled acrylic painted samples and on soiled acrylic painting. Five samples were studied for each concentration of both gels. A total of fifty samples were prepared for this study.

![Fig. 1](image1.jpg)

**Fig. 1.** The paint application on the pre-primed canvas with locally available materials

![Fig. 2](image2.jpg)

**Fig. 2.** Preparation of gellan gum gel and placement of gels on the painted samples

**Gel application process**

The gel cleaning tests were conducted on acrylic painted mock-up canvas samples. Rigid gels were cut into desired pieces and placed with steel spatula on the painted samples and allowed to stay for 30 minutes to observe and compare the extent of problems these gel cleaning treatments can induce on the paint layer. Then these gels of different concentrations were also applied on the synthetically soiled acrylic painted canvas (on the stretcher). The application of
gellan gel is also very simple. Just the sheet of gellan gum or the piece of gellan gel has to be placed on the painted surface. Then a piece of melinex was placed on the gellan gel and a glass piece was placed on the gel to provide some pressure and even contact of the gel to the painted surface. No clearance step was introduced to any of the samples after cleaning. In order to exploit the rigidity characteristics of these gels, different methods of application were tested. First, gels as cubes were placed on the samples and the gel cubes stayed on the paint surface due to gravitation. In another method, these gels were used as water containing wet erasers and the cleaning action involved rubbing of painted surface with slight pressure ensuring the minimum to no friction generated due to the rubbing action.

**Instrumentation**

ATR-FTIR spectra were obtained by using Nicolet i550 FT-IR (Thermo Scientific) using a Spectra-Tech ATR objective with diamond crystal. All the samples were scanned using 15 scans at a resolution of 4 cm\(^{-1}\). The wave number region studied was between 4000 cm\(^{-1}\) and 400 cm\(^{-1}\) and the resulting characteristic peaks of infrared transmission were recorded. No corrections were made to any spectra. Dino-lite 7915MZT Dino-Lite edge was used to capture the images before and after cleaning at 5MP. A separate UV torch was attached to the microscope for capturing the UV photography. They were micro-photographed at 50x magnification and images were taken in normal, raking and ultraviolet fluorescence light. Carl Zeiss EVO 50 SEM was used to capture the images of control and after cleaning at 100×, 250×, 500× and 1000× magnification. The samples for SEM imaging were coated with gold and for EDX analysis the samples were carbon sputtered. Images were taken in high vacuum mode. The accelerating voltage was set at 20kV at a working distance of 8mm at a resolution of 2250nm. EDX analysis was conducted with Bruker and the data were processed with Roentag software.

**Results and Discussions**

Gellan gels and agar gels both were found very effective in controlling the release of water thereby moistening the painted surface sufficient for the soil to stick to the gel. The removal of these gels was also very easy due to no adhesion between these gels and the paint surface. However, there was a problem of syneresis found with agar gels, as it released some water on the painted surfaces which were deposited on the surface. There was no case of syneresis noticed with gellan gels. The handling property of gellan gel was also far better than the rigid agar gels. The gels formed with gellan at all concentrations from 1 to 4% had very good flexibility. Gels formed with gellan were like a water-containing sheet whereas with agar were like a rigid cake. The agar cake broke into fragments/crumbs while cleaning leaving some crumbs to stay on the painted surface. The release of gellan gel was also much better than agar and the entire sheet could be released with great ease. It was apparent that the contact time played a significant role in controlling the effect of aqueous based treatment and that the increase in the contact time may have led to swelling. Therefore, the contact time should be carefully assessed and continuously monitored. In case of gellan gel, the swelling was noticed on all samples at all concentrations. The prominent swelling was observed on all samples of gellan at 4%. However, at gellan at 3%, the swelling observed was relatively lesser than samples of other concentrations. However, the swelling was not noticed when the samples were left to dry for 24 hours. In case of agar gel, the swelling was seen in almost all samples at the gel concentration of agar at 1, 2 and 3%. Swelling was also observed in gel concentrations of agar at 4 and 5%. Agar gels had a peculiar problem to release water from the gel matrix onto the paint surfaces. This phenomenon was observed on the samples of agar at 5% concentration. Both gels in all selected concentrations were easy to remove from the painted canvas samples and it was hoped that they don’t leave any residue on the treated surface as the gels applied were rigid. In case of gellan gel no residue was noticeable with the unaided eye as well as with dinolite digital microscope. The images were compared in both normal light and ultraviolet
radiations at 50×. Also, no cotton fibre were found on the treated surface adhered to the surface. Some cotton fibres which were loosely present did not signal the presence of any residue. However, gellan gel at 5% concentration did leave a few residues on the surface. This was possible due to the gellan gel at 5% concentration did not form a clear transparent one sheet, instead, it formed a lumpy sheet and the residue was due to some loosely held particles of the gellan gum to the lumpy mass. In case of agar gels though the gel cubes were removed with ease but the presence of cotton fibres was seen on all samples of agar at 1 and 2%. Presence of cotton was also noticed but at a lesser amount. Very thin whitish superficial layer and vertical streaks were noticed in one sample of agar at 1%, three samples at 2%, one sample at 3%, two samples at 4 and 5%. (Fig. 3). This effect was very prominent at 4 and 5% after the cleaning which probably emerged from the water absorption from the gel which was not visible through normal and raking light microscopy and became visible only under ultraviolet magnification. However, this phenomenon was not observed in any of the samples in any concentration of gellan.

![Fig. 3. White chalkiness seen under ultraviolet examination at 50× in agar cleaned samples](image)

**Surface analysis**

The images taken with SEM did not show roughness or damage to the paint layer. Some particles that were noticed in the SEM images were part of the paint surface. However, in one sample cleaned with agar gel at 2% concentration showed some very fine cracks in the paint layer at 1000× magnification which might have been induced due to the swelling of the paint layer (Fig. 4). However, no cracks were seen in any other samples. The EDX data was obtained one at 100× and three different points at 500× in all samples. In the control sample, the major element detected were carbon, oxygen, and cadmium and the minor elements were sulphur, sodium and aluminum. At some points, the presence of zinc was also detected as a minor element. The presence of carbon and oxygen was from the paint binder. Cadmium originated from pigment (Fig. 5.) The presence of sulphur indicates towards the use of cadmium sulphide as a pigment. The presence of sodium and sulphur may be traced from the extender added in the paint formulation. In case of samples cleaned with gellan gum gel, in all samples from 1, 2, 3 and 4%, in the spectra taken at 500× the amount of cadmium was reduced to a very low level from 20% to 6.7% and at some point even reached to 2.15%. This trend is noticed in the EDX analysis where with the reduction in the weight percentage of cadmium, the amount of sulphur also reduced. This confirmed the use of a pigment as cadmium sulphide. The investigation also demonstrated that some cadmium pigments have been removed due to cleaning action which might have resulted due to some surfactant removal from the paint film. In some samples, traces of silicon are also present which might have originated from alumino-silicate clay present as a contaminant. In case the cleaned samples with agar gels the weight percentages of all elements were similar to the weight percentages of elements found in samples cleaned with gellan gum gels.
However, it was noticed that gellan gel samples at 5% concentration showed no depletion in weight percentage of cadmium whereas agar gel at 5% showed a loss of cadmium weight percentages (Figs. 6 and 7).
EDX result showed that no elements migrating from the paint matrix to the paint surface. This indicated that there would be the minimum to no leaching out of components of the paint film (Table 1).

### Table 1. Weight percentages of the control sample showing the presence of elements on the paint surface

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Number</th>
<th>Series</th>
<th>Wt. %</th>
<th>At. %</th>
<th>Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen</td>
<td>8</td>
<td>K-series</td>
<td>36.00</td>
<td>38.62</td>
<td>6.0</td>
</tr>
<tr>
<td>Carbon</td>
<td>6</td>
<td>K-series</td>
<td>29.93</td>
<td>32.11</td>
<td>4.3</td>
</tr>
<tr>
<td>Cadmium</td>
<td>48</td>
<td>L-series</td>
<td>19.77</td>
<td>21.21</td>
<td>0.7</td>
</tr>
<tr>
<td>Sulphur</td>
<td>16</td>
<td>K-series</td>
<td>6.27</td>
<td>6.73</td>
<td>0.3</td>
</tr>
<tr>
<td>Sodium</td>
<td>11</td>
<td>K-series</td>
<td>1.02</td>
<td>1.09</td>
<td>0.1</td>
</tr>
<tr>
<td>Aluminum</td>
<td>13</td>
<td>K-series</td>
<td>0.23</td>
<td>0.24</td>
<td>0.0</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>93.22</strong></td>
<td></td>
<td><strong>100.00</strong></td>
<td></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

**Investigation of residues and paint swelling**

The FT-IR spectra of the control sample showed the two bands of C-H rocking at 1382cm⁻¹ and C-H bending at 753cm⁻¹ which can be attributed to the absorption vibrations of the methyl group. The strong peak of carbonyl bond stretching at 1725cm⁻¹ is of acrylate carboxyl group. There is a distinct absorption band between 1148 to 1250cm⁻¹ which is due to C-O-C stretching vibrations. The band at 989, 1062 and 844cm⁻¹ showed the characteristic absorption vibration of polymethyl methacrylate [24]. The C-H stretching frequencies were also observed strong at 2955cm⁻¹ and also well-defined peak at 2874cm⁻¹ with a shoulder at 2936cm⁻¹ (Figs. 8, 9 and 10). On the basis of the above discussion, the binding medium of the control painted sample can be estimated as of p(nBA-MMA) copolymer type. The characteristic absorption vibration band of gellan gum appears at 1637cm⁻¹ which shows the presence of glycosidic bond. However, in none of the collected FT-IR spectra from the samples cleaned with gellan gum gels showed any such peaks in this region. FT-IR spectra of gellan gum gel at 1 and 2% showed a broad band of absorption vibration of OH stretching frequencies at 3398cm⁻¹ which indicates the swelling of the paint film. At other concentrations, no such phenomenon was observed which indicates very less to no swelling of the paint films. The FT-IR signal of agar gum shows the characteristic absorption peak of OH stretching of the hydroxyl group at 3497cm⁻¹ and C=O stretching peak at 1647cm⁻¹. However, no such characteristic peaks were
observed in any of the spectra of agar cleaned samples. Though, swelling seemed more prominent in agar gels in comparison to the gellan gum gels. There was no shift in any of the peaks in any of the samples cleaned with both gellan and agar samples.

Fig. 8. FT-IR spectra of control sample showing a characteristic peak of acrylate carboxyl group at 1725 cm$^{-1}$

Fig. 9. Comparison of FT-IR spectra of samples cleaned with gellan gels

Fig. 10. Comparison of FT-IR spectra of samples cleaned with agar gels
Application of these gels as erasers

Of the various materials used for the cleaning of paintings by conservators, a number of dry cleaning materials is also used for cleaning of painted surfaces. Out of the dry cleaning agents such as dry erasers, eraser powders, sponges, different types of cloths, etc. cleaning with most of the materials are not without risk. The risks that are involved are burnishing and polishing, leaving of plasticizers in the residues. Gellan gel and agar gel both gave satisfactory results when used as wet erasers. This method was very useful to remove loosely adhered soiling and the soiling adhered considerably on the paint surface. Agar gels at all concentrations cleaned the soiled samples. The cleaned samples were smooth after the cleaning with both gels. There was no scope of burnishing or polishing of the paint surface as these gels glided on the paint surface. Since the cleaning action entailed a continuous movement of the gel, the chances of absorption of water released by gels on the paint surface were very less. Gellan gum gel basically acted more like a wipe than a dry/wet eraser, whereas agar gel acted like a wet eraser. Cleaning efficacy was found to be more or less similar. The cleaning action trapped some of the soil and dragged some of the soil. Agar gel left some crumbs and chances of these crumbs residing on the surface could not be avoided. Therefore, cleaning with agar gels require caution not to leave the crumbs of agar gel otherwise biodeterioration may induce into the paint film. However, the gellan gel was removed very easily even when they were cut into small pieces and they did not leave any crumbs or pieces. The entire piece was compact and came off very nicely. The viscoelastic property of the gellan gel rendered the flexibility to the gel as such that the gel can be placed in just one step and also can be removed in one step. There was no scope of any residue left on the gel. The gel sheets were also very transparent and the cleaning action could be observed through the gel placed on the paint surface.

Conclusion

The results presented in the paper indicated that rigidity of gels can significantly reduce the chance of leaving any residue which is impossible in any other methods of gel applications, these gels due to their rigidity and forming a strong cohesive gel did not adhere to the substrate. It was apparent that the contact time played a significant role in controlling the effect of aqueous based treatment. Digital microscopy in normal, raking and ultraviolet light allowed comparison of the cleaning efficacy and evaluate the condition of the paint surface after cleaning. SEM-EDX did not show any migration of elements from the paint matrix onto the paint surface, except the depletion of cadmium and sulphur in some cases which indicated some pigment loss due to the dry cotton swabbing after the gel removal from the paint surfaces. ATR-FTIR analysis indicated the absence of any residue of these gels, though it confirmed some degree of swelling in the paint films in some of the samples. These two cleaning systems carried through this research worked in quiet contrast to the conventional method of cleaning, in which residues do remain on the paint surface and interact with the surface. However, there were some issues of water diffusion into the substrate, specifically in agar gels. Therefore, for cleaning of any surface sensitive to water, the time of contact should be kept to a minimum. Since agar also released some amount of water in all concentrations, therefore, the risk of swelling with agar gels is more than with gellan gels. This study can be further employed on other types of paintings that are sensitive to aqueous cleaning.

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