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THE USE OF AGAR AS A SOLVENT GEL IN OBJECTS CONSERVATION

CINDY LEE SCOTT

ABSTRACT

Agar, or agarose, is a rigid polysaccharide gel that has found use in conservation cleaning treatments of three-dimensional porous objects in recent years, most notably by Italian conservation scientists Marilena Anzani and Paulo Cremonesi. Used strictly as an aqueous gel, it has shown great promise as a poulticing material on porous plaster substrates for the removal of surface particulate matter and water-soluble soiling.

Agar is readily soluble in hot water, stable in both alkaline and acidic conditions, and (prior to adding other materials) is a safe, non-toxic, and eco-friendly material. The dispersion rate of agar can be tailored to the treatment by adjusting the concentration of the solution. In addition, agar acts as a molecular sponge; the gel, when used with solvents, is both a poulticing material as well as a solvent gel, solubilizing the impurities, drawing them away from the surface, and holding those materials within its gel matrix. Post-treatment analysis of cleaned surfaces and used gels using Fourier transform infrared spectroscopy and ultraviolet-induced visible fluorescence photography indicate that the gels show great promise with respect to clearance.

This paper builds upon the work of Anzani et al. (2010) by using agar as a support material for multiple solvents as well as other aqueous cleaning solutions. Specifically, its uses for the cleaning of and adhesive reversal on three-dimensional objects are explored.

1. INTRODUCTION

At a conference on cleaning held in Valencia in June 2010, Italian chemist Paolo Cremonesi presented a paper on the use of rigid agar gels for the conservation and cleaning of outdoor plaster busts. The research he presented in Valencia, and that he published in later months with his colleagues in Milan, showed that agar gels have great promise as cleaning materials for three-dimensional objects (Cremonesi 2013). In their studies, they used the gel primarily with deionized water. Given their degree of success, the author wondered if other cleaning agents, such as solvents, could be added to the gel to increase its versatility, and how the addition of such agents would affect the stability and working properties of the gel.

The studies published by Anzani et al. (2010) and Cremonesi (2013) explore the use of agar as a sol, or colloidal solution, which allows for its application on three-dimensional materials (the rigid nature of the gel had previously restricted its use to two-dimensional materials). Since these studies were originally published, the usage of agar gels in conservation has gained ground in North America, as exemplified by an upcoming workshop sponsored by the Smithsonian Institute in June 2012.

2. AGAR

Agar is a rigid gel derived from the cell walls of a species of red algae of the *Gelidium* or *Gracilaria* families (Davidson and Jaine 2006). Agar gels can be classified as reversible sol-gels or reversible hydrocolloid gels. A sol-gel starts from a colloidal solution (the sol) that acts as the precursor for an integrated network of polymers (the gel) (Sol-gel 2012). Herein, the warm, semi-solid form of agar will be referred to as the sol, while the cooled, more rigid form will be referred to as the gel.

According to the Lonza Bench Guide on the physical properties of agar, the gelation mechanism involves a “shift from a random coil in solution to a double helix in the initial stages

of gelation, and then to bundles of double helices in the final stage" (Lonza n.d.). Each stage of gelation is reversible and is reached by the addition or subtraction of heat.

At the molecular level, agar consists of two polysaccharides: agarose, which forms approximately 70% of the mixture and has the greatest gelling tendency, and agaropectin (fig. 1). The polymeric backbone structure of both of these molecules consists of alternating galactopyrose molecules which form agarobiose units (Fisheries and Aquaculture Department of the Food and Agriculture Organization of the United Nations 1990). The agarobiose units form long chains with an average molecular mass of 120,000 daltons, or roughly 400 agarobiose units (Lonza n.d.).

Agar powder can be purchased in a number of purities, ranging from highly purified agarose used in biological studies to food grade agar. Both this author's research and that conducted by Anzani et al. (2010) and Cremonesi (2013) showed little to no detectable differences between the FTIR spectra of food grade samples and those that were of analytical grade. Experiments conducted at UCLA were done using an analytical grade of agar powder ground to 80 mesh; those conducted at the Museums of New Mexico used a food grade agar purchased from Moor Agar Inc., also ground to 80 mesh. Some initial experiments at the Museums of New Mexico were also conducted using flake agar purchased from the local Whole Foods, and ground in a coffee grinder.

Dried agar, either as a powder or raw flakes, is insoluble in cold water, though it dissolves easily in boiling water. The minimum temperature at which the polymeric chains cross-link to cause gelation is 85ºC. An agar sol needs to be cooled to below 40°C to form the gel. Once cooled, it is easily reheated to its sol state, a process that can be repeated multiple times without change to the working properties of the gel (though a loss of water through evaporation should be compensated for with each reheating) (Chaplin 2009; Anzani et al. 2010, 42).

Agar gels exhibit high gel strength even at concentrations less than 6%. They are stable up to 65°C and are not enzymatically degraded by most bacterial species. They are stable in both highly alkaline and acidic conditions, and, prior to the addition of other cleaning agents, they are completely non-toxic and natural. It is an ideal gel matrix for biomedical applications because it is biologically inert with controlled ionic properties (Fisheries and Aquaculture Department of the Food and Agriculture Organization of the United Nations 1990).

The porosity of an agar gel is directly related to the concentration of agarose within the dispersion phase. Thus by altering the concentration, it is possible to manipulate viscosity, absorption, and dispersion as needed by a given treatment or experiment (Agar 2012). The

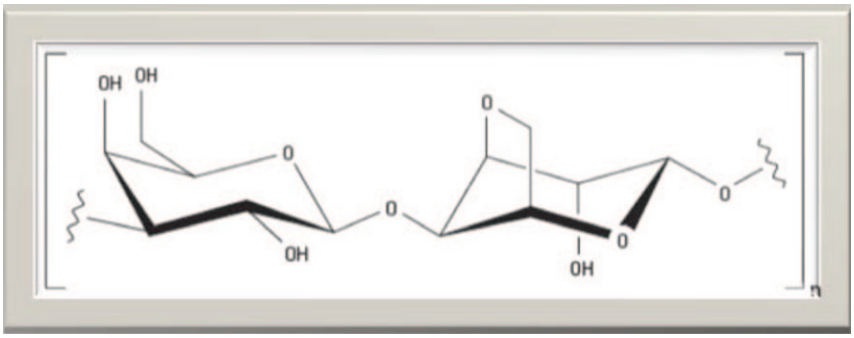

Fig. 1. Agarobiose structural unit (Courtesy of http://bio.lonza.com/uploads/tx_mwaxmarketingmaterial/Lonza_BenchGuides_SourceBook_Appendix_B_)

average pore size for a prepared gel between 2-7% (w/v) is typically between 100 and 300 nm (Lonza n.d.). It is this porosity that allows agar gel to act as a molecular sponge, the process being absorption by means of gradients of concentration, as well as osmosis (Anzani et al. 2010). The degree of absorption possible for a given solute is influenced by both the concentration of the agar gel and by the particle size of the solute. For example, in two instances of removing gouache paint from a plaster surface, a large particle sized blue pigment and a finer particle sized orange pigment, it was found that a 2% (w/v) solution of agar gel was required to clean the blue gouache, while a 3% (w/v) solution worked well for the orange gouache. While the binding medium and substrate were the same, the particle size of the respective pigments differed; as such, a modified treatment approach was necessary.

Diffusion studies were carried out by Anzani et al. (2010) on agar gels prepared using only water. Their experiments found that after a period of twenty minutes, a 4% (w/v) agar gel will diffuse up to 2mm into a given porous substrate, in this case, gypsum, while a 2% (w/v) gel will diffuse to a depth of 4mm under the same conditions. In comparison, the researchers also applied a cotton poultice soaked in water and found that the water had diffused to a depth of 5-6mm after only 3 minutes (fig. 2). This study found a concentration of around 3-4% to be ideal, but for highly sensitive materials, a higher concentration is recommended - sometimes up to 8%, which can be easily mixed. On highly textured or porous surfaces, it is not advisable to use concentrations lower than 2%, as the dried residues can be exceedingly difficult to remove; they form a very thin film that can be removed mechanically, but with difficulty.

2.1 PREPARATION OF AGAR GELS

Preparing an agar gel is relatively simple. Measured amounts of agar powder and cold water are mixed. The mixture is then heated to a temperature above 85°C. Heating can be done either in a microwave or on a hot plate, ensuring the solution is well mixed before, during, and after the heating process. After heating, a measured amount of solvent, chelating agent, oxidizer, or other cleaning agent is added to the agar sol and stirred in to achieve homogeneity. The agar can be allowed to cool and applied as a gel cut to the desired shape and size, or applied with a brush or spatula while warm as a sol .

Given the combustible nature of most solvents habitually used by conservators, the solvent should be added after heating. In the case of alkaline materials, a colour change has been noted when the agar is prepared at a high pH that is not noted when the alkaline material is added

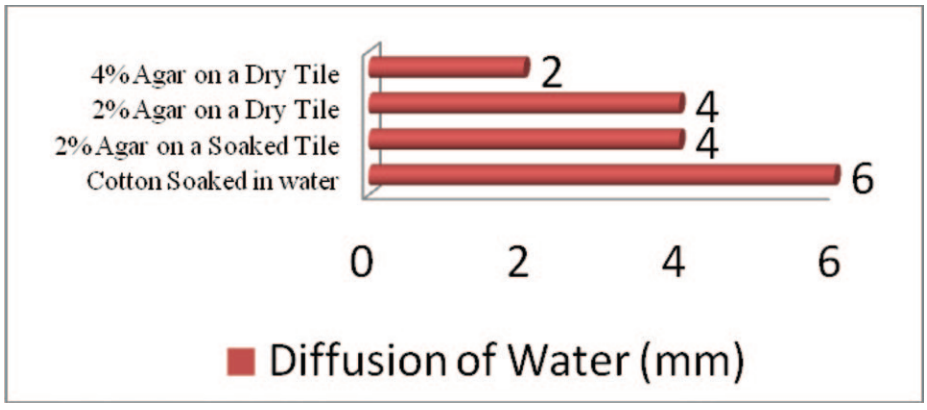

Fig. 2. Diffusion of water in agar and cotton on a gypsum substrate
(Courtesy of Anzani et al. 2010)

to the warm sol after heating (fig. 3). The nature of this colour change is not fully understood at this point, but can cause staining on some substrates and as such, should be avoided.

Removal of the gel after treatment is a simple matter: one need only lift an edge of the gel and peel it off. The softness and elasticity of the gel makes removal complete. Any small residues, particularly around edges, will tend to detach from the substrate and spontaneously flake off after drying. Those that do not can be moistened with warm water and brushed off easily. The entire application process can be seen in the figure below (fig. 4), where a mixture of agar, ethanol, and water were used for the removal of gouache over-painting on the plaster fills of a Gallina-Largo ceramic.

Generally speaking, the gel is removed before it has dried out completely, but should the gel dry out, current evidence has not indicated increased risk of damage to the object. Anzani et al. (2010) have even recommended allowing the gel to dry out completely if there is a need to remove deeply penetrating stains or salts.

The drying time of agar can be influenced by the thickness of its application. Even at thicknesses of less than 2mm at an ambient RH of 50%, the gel will stay moist for more than eight hours without being covered. Covering the gel can extend the drying period over several days, though it should be noted that the risk of mould growth is increased with extended dwell time and multiple applications should be considered instead.

3. EXPERIMENTAL

Experimentation and research into agar gels occurred in two phases: the first was conducted as a component of the author's thesis research at UCLA; the second was conducted during her internship with the Museums of New Mexico.

During the first phase, experiments were conducted on ceramic test tiles created in the lab. In this instance, agar was explored strictly as a support material for other chemicals that could remove shellac from previously restored ceramics. The ceramic test tiles were made of terracotta coated with a kaolinite-type slip and fired in a furnace at 900 °C; these tiles were either coated or mended with a 20% (w/v) mixture of seed lac and ethanol.

As a part of the first phase of study, the agar sol-gels mixed with ethanol, acetone, and 5M sodium hydroxide (NaOH), as well as various combinations of these three materials were tested. Evaluation of the efficacy of the agar gels in this context, including clearance, was

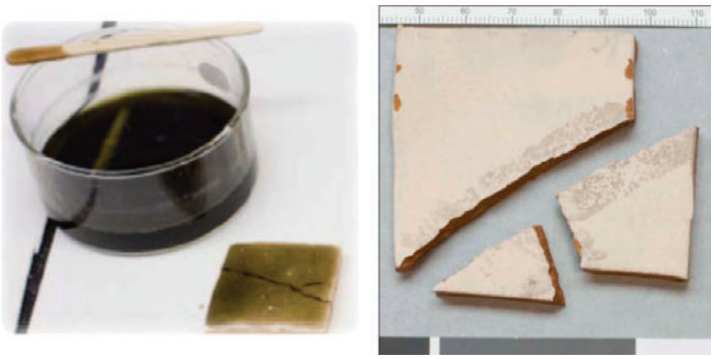

Fig. 3. Discolouration of agar gel in the presence of heat and NaOH and resulting staining (Courtesy of Cindy Lee Scott)

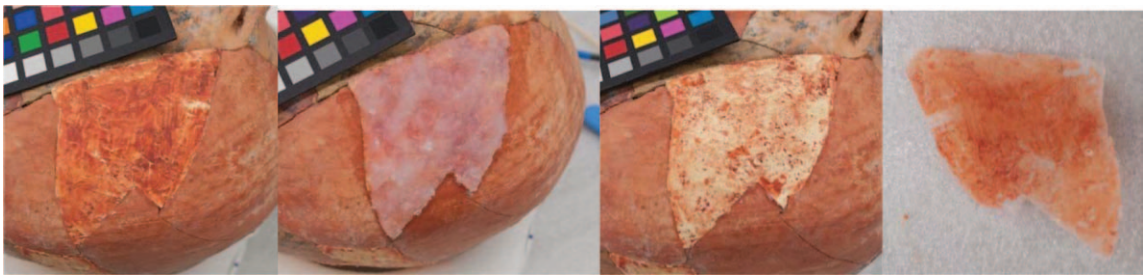

Fig. 4: (left to right) Before treatment; application of agar; after treatment; agar after removal (Courtesy of Cindy Lee Scott)

conducted using visual analysis with binocular microscopy, UV-visible fluorescence imaging, and FTIR spectroscopy.

Preliminary results showed excellent working properties when the agar sol was mixed with either ethanol or NaOH, as well as good clearance after treatment. To increase clearance and ease of removal for lower concentration gels with increased porosity, Japanese tissue paper was used as an intermediary layer; this additional layer did not seem to impede the absorptive action of the gel in any way.

During the second phase, several classes of cleaning agents were tested for their use in agar gels. Care was taken to note the working properties of each mixture, colour changes that could potentially lead to staining, and the degree of miscibility. Because of limited access to analytical instrumentation, each of these observations was qualitative in nature, and will require further research. The classes of cleaning agents explored in preparation for this paper include solvents, surfactants, chelating agents, oxidizers, and acids.

With respect to working properties, the best results were achieved with ethanol, followed by Stoddard solvent, then acetone, and finally xylenes, which, if added in too high a concentration, could cause the gel to completely dissociate. None of the solvents caused colour changes that were concerning (Table 1).

With respect to other cleaning agents, excellent mixing results were achieved for the chelating agent, oxidizer, acid and base tested (Table 2). Additional testing should be done with other examples of these classes of materials in the future. Surfactants could not be mixed at all, causing complete dissociation of the gel, for reasons that will be discussed in greater detail below.

Table 1. Results of solvent/mixtures. All solvents were added to an 8% w/v agar gel after heating during sol-phase.

Solvent	Miscibility	Colour Changes?	Working Properties
Ethanol	Mixes readily	None	Excellent
Acetone	Destabilizes the colloid mixture	Becomes slightly opaque	Difficult/Good
	-Effect is mitigated by the addition of EtOH		
Xylenes	Destabilizes the colloid mixture	Becomes opaque	Difficult
Stoddard solvent	Mixes well	Becomes slightly opaque	Medium/Good

Table 2. Results of mixing tests using other cleaning agents

Cleaning Agent	Miscibility	Colour Changes?	Working Properties
Triton XL-80N (1% w/v in H_2O) (Surfactant)	Gel dissociation	None	Gel dissociation
Orvus Paste (1% w/v in H_2O) (Surfactant)	Gel dissociation	None	Gel dissociation
Ammonium citrate (3% w/v in H_2O) (Chelating Agent)	Excellent	None	Excellent
Hydrogen peroxide (3% w/v in H_2O) (Oxidizer)	Excellent	None	Excellent
Phosphoric acid (10% v/v)	Excellent	None	Excellent
Sodium hydroxide (5M)	Excellent	Yellows slightly (temporary)	Excellent

In addition to the above tests, plaster tiles were cast in the labs at the Museum of New Mexico in order to further test the gel using these different cleaning agents in a number of cleaning challenges. The plaster tiles were coated with the following materials:

i. Alizarin dye
ii. Garnet shellac (20% (w/v) in ethanol)
iii. Phthalo green watercolour (in gum Arabic)
iv. Turquoise blue gouache
v. Iridescent bronze acrylic emulsion paint
vi. PVA wood glue
vii. Soil

After each of the tiles was 'mis-treated' with their respective cleaning challenges, they were artificially aged with cycles of heat and humidity. This was accomplished by placing the tiles in an oven at 70°C with a beaker of water to increase humidity to roughly 60-70% for 24 hours, and then removed to the ambient atmosphere of the lab for another 24 hours; this cycle was repeated for a 14 day period.

Using the most efficacious cleaning agent for each(as determined through solubility testing), the agar gel was applied in its sol form and left in place for 15 minutes, after which time it was removed. Generally, only one application was evaluated, though in the case of the blue gouache, a second application at a lower concentration was tested. Each test was evaluated for the following:

i. Efficacy and efficiency of cleaning
ii. Visible damage or morphological changes to the substrate
iii. Working properties of the gel
iv. Ease of removal/clearance

3.1 RESULTS

Solvent mixtures with agar gels were inadequate in the cleaning of either the plaster tile coated with alizarin, or the plaster tile coated with shellac, though some reduction is visible on the alizarin tile. On the shellac tile, clearance was achieved only through the addition of 5M NaOH, which did cause some slight pitting to the plaster. All treatment efforts for removing the watercolour from the tile were unsuccessful, likely due to the use of a phthalo green dye-based

Table 3. Results of cleaning tests

Tile	Solvent	Efficacy	Damage	Working Properties	Clearance
Alizarin Dye	Ethanol	Moderate	None	Excellent	Total
Garnet Shellac	1:1 EtOH and Acetone	Minimal	Lac Dye stains	Good	Total
	NaOH and EtOH	Moderate	Lac Dye stains	Good	Good
Water Colour	Water/EtOH	Minimal	None	Excellent	Total
Gouache	Water	Needs lower concentration	Tidelines	Excellent	Total
Acrylic Emulsion	Acetone	No change	None	Excellent	Total
PVA Adhesive	Water	Excellent	None	Excellent	Total

watercolour. As previously discussed, there was good success removing the gouache using a 2% (w/v) agar in water alone. Acetone on a cotton swab proved to be quite efficacious for the removal of the acrylic emulsion, whereas an acetone/agar mixture proved to be completely ineffective. Treatment of the PVA with agar was highly successful. The extended contact time with the adhesive has proven a very effective means for its removal. A high degree of success was achieved using agar on the soiled tile. A single treatment showed significant cleaning without damage to the surface. The results are summarized in table 3.

It should go without saying that in any conservation treatment using an agar gel, the efficacy of treatment depends upon the suitability of the chosen cleaning agent. Efficacy can be enhanced by increasing or decreasing the respective concentrations of agar and solvent, or by changing the temperature, length, and number of applications.

Some other noteworthy results are as follows:

a. The addition of ethanol to the gel increases the efficacy of treatment through improved wettability.
b. When trying to mix the gel with acetone, the addition of ethanol can increase the miscibility of the solution.
c. Chemically complexed materials, such as the alizarin, cannot be removed by solvation alone; additional steps must be taken to reverse the chemical complex. This can be accomplished by lowering the pH of the gel to create an acidic environment.
d. Agents that disrupt hydrogen bond formation, such as chaotropic agents and some classes of surfactants, can significantly decrease melting and gelling temperatures, and can even inhibit the formation of the gel entirely. When adding such agents to the gel after gelation has occurred, a disassociation of the gel has been observed.
e. For the cleaning of heavily soiled or saturated substrates, the gel should be reapplied intermittently to avoid saturation of the gel and diffusion of the solubilised material back into the substrate. The more heavily soiled or contaminated the surface, the more often the gel should be changed.

3.2 CLEARANCE

Clearance methodologies for agar gels are as simple as they are effective; "the surface freed from the gel has suffered no [physical] manipulation" (Anzani et al. 2010, 48). This rigid gel is not adhered to the object in any way; it is simply held in place by gravity, and depending on the texture and shape of the surface to which it is applied, physically. Such a methodology has many advantages.

This ease of clearance when using agar stands in contrast to more traditional cleaning methodologies, in which clearance of post-cleaning gel residues can involve a high degree of physical interaction with the surface. Further, the more complex mixtures of surfactants, solvents, and other chemical gelling agents, such as polyacrylic and PVOH gels, can leave a greater variety of residues, particularly on porous or uneven surfaces; there is evidence that such residues can interact with the surface as they age.

One area of concern in using a natural gelling agent is that of biodeterioration in the event that full clearance is not achieved. Agar gels have found their major use as microbiological media "as [they are] not easy for microorganisms to metabolize" (Chaplin 2009). Thus, while agar gels have traditionally been used as a growth medium in petri dishes, it is not *because* they are a food source for microorganisms, but rather, because they are not.

FTIR spectra of dry agar powder showed characteristic polysaccharide bands that were not definitively detected on the treated ceramic tiles after treatment (fig. 5); overlapping bands and the presence of common functional groups that are unrelated to treatment render a definitive identification of polysaccharides impossible. FTIR analysis of the agar gel after it was removed was not able to determine if there had been undesired leaching of minerals from the ceramic substrate, and further analysis should be undertaken to better understand this issue.

Examination under 365nm ultraviolet radiation did reveal some faintly fluorescent zones, particularly along the margins of treated areas (fig. 6) (Anzani et al. 2010; Cremonesi 2013; Scott 2012). Anzani noted a similar fluorescence when they inspected their treated gypsum tiles using ultraviolet radiation; further tests led them to believe that this halo was likely attributable more to

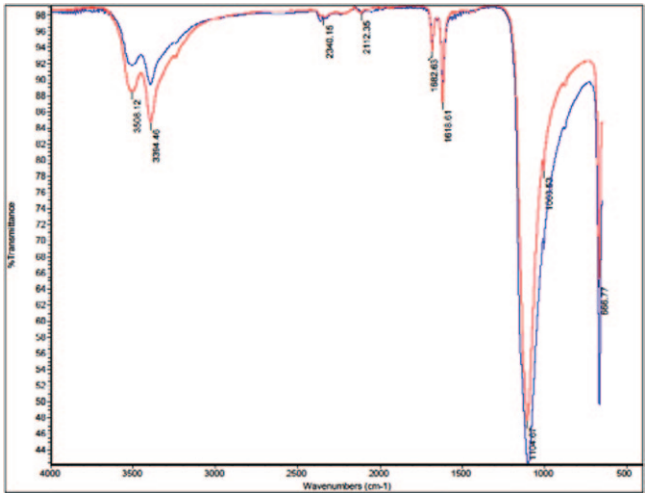

Fig. 5. FTIR spectra of an untreated control tile (blue) and a treated tile (AT) (red). This shows no noticeable or significant differences in the spectra that would indicate residues (Courtesy of Cindy Lee Scott).

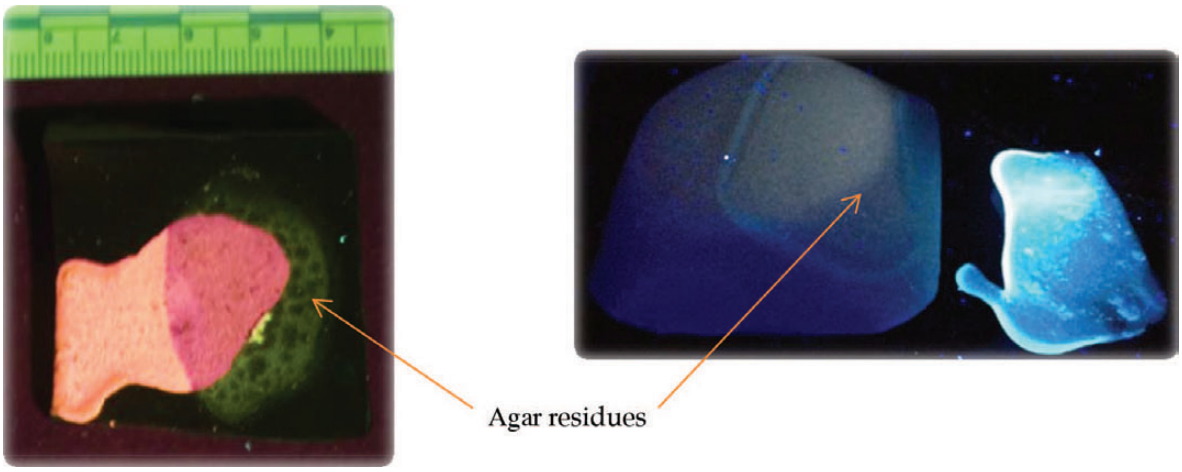

Fig. 6. UV-induced visible fluorescence photographs ((λexc = 300–400 nm) showing the distinct fluorescence of residual agar after treatment (Courtesy of Cindy Lee Scott).

the action of water on the surface than to a dry, superficial residue of the gel itself (Anzani et al. 2010). Neither possibility has yet been confirmed or refuted.

Agar gels do not change the hygroscopicity of a porous substrate after treatment, and in fact, do not show any observable interaction with the treated surface, such as signs of erosion or macroscopic or microscopic etching (Anzani et al. 2010). SEM has yet to be explored for verification.

4. CONCLUSION

Agar based solvent gels show a number of important advantages and uses that can make them a good treatment option for the cleaning of certain substrates. Conversely, there are a number of disadvantages that would preclude the use of an agar-based gel.
Such advantages include:

- It is a simple compound; there is no question as to the active ingredient. The gel, on its own, is largely inert, and as such, it is only as effective as the cleaning agent it carries.
- The absorption and diffusion qualities of the gel can be easily tailored to a substrate by manipulating the concentration of agar in water.
- The porosity of the gel allows it to act like a 'molecular sponge', therefore playing the dual role of both a solvent carrier and a poulticing material. It simultaneously solubilizes the solute while drawing it away from the surface and holding it within its gel matrix, minimising any physical interaction with the substrate.
- It is easily removed without any special clearance methodologies. It also requires less training to learn how to use. There is always a reduced risk of differential clearance from one conservator to another, however.
- Agar shows wide-ranging stability when subjected to both temperature and pH fluctuations.
- Agar as a dried powder has a long shelf-life, is inexpensive, and is widely available.

Conversely:

- Agar gels must first be mixed with water. Though it can be minimised by using a higher concentration of agar, there will always be the diffusion of some water into the

substrate. If the substrate is sensitive to water, another cleaning treatment should be considered.

- Because of differences in polarity, some solvents, such as acetone, can be tricky to mix into the gel. The addition of a small amount of ethanol prior to mixing more polar solvents can mitigate this effect, but the presence of ethanol in a mixture is not always desirable. Further, the volatility of solvents such as acetone can have a rapid cooling effect on the gel, causing it to solidify before the solvent has been fully mixed. The use of a double boiler to heat the gel can help to mitigate this effect.
- Currently it is not possible to mix surfactants into the gel. The disruption of hydrogen bond formation causes the gel to dissociate and liquefy in the presence of the surfactants tested.
- There is some risk, though minimal and improbable, of biological deterioration if full clearance is not achieved.
- If using the gel as a carrier for flammable solvents, it is not possible to keep the agar solvent gel premixed as one does with Carbopol, unless it will only be used as a rigid gel and not reheated. If reheating is necessary, the solvents should be added only after heating, just prior to application.
- If one wishes to apply agar as a sol, special equipment such as a hot plate or microwave will be necessary; these are not always available or practical in a field setting.

As with any material that one uses for conservation treatment, additional research into aging, potential residues, and interaction with different substrates is required before it can and should be used on other types of materials; but the properties seen within the scope of this study have shown it to be a promising material.

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SOURCES OF MATERIALS

Agar (Powder)
Fluka Analytical
Sigma-Aldrich Corp.
St. Louis, MO USA

Agar (Powder)
Moor Agar Inc.
PO Box 1799
Rocklin, CA 95677

Agar (Flakes)
Whole Foods Markets
753 Cerrillos Rd.
Santa Fe, NM 87505

Batanas Clay
Hellenic Clay Center
S.A.55 G. Lyra
Kifissia 14564, Greece

Carpenter's Interior Wood Glue
Elmer's Products, Inc.
460 Polaris Parkway, Suite 500
Westerville, OH 43082

Grumbacher Watercolour in Pthalo Green; Winsor & Newton Designers Gouache in Turquoise
Artisans Art Supply
2601 Cerrillos Rd.
Santa Fe, NM 87505

Plaster of Paris
DAP, Inc.
2400 Boston St., Suite 200
Baltimore, MD 21224

Seed Lac, Stick Lac, Madder Lake (Alizarin)
Kremer Pigments
247 W. 29th St.
New York, NY 10001

Terracotta
Laguna Clay Company
14400 Lomitas Ave.
City of Industry, CA 91746

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