GELLING IN THEORY AND PRACTICE: AN EXAMINATION OF AGAROSE GELS IN TEXTILE CONSERVATION

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ABSTRACT—Recent developments in poultice treatments have led to the introduction of agarose, a gelling agent derived from seaweed, into textile conservation and innovations in its application. Translating information on the use of agarose from published literature into actual practice has proved challenging, as predicting how these gels will work on different materials is complex.

This paper presents dissertation research carried out at the University of Glasgow to understand how the chemical and physical properties of agarose can be used to inform successful conservation treatments. Properties are assessed alongside results of tests utilizing these gels on textile substrates to better understand how the theoretical information presented in food and biological science literature informs and affects the use of these materials.

Testing examined three fibers (cotton, wool, and silk), three concentrations of agarose, and two different gel depths. The results showed that the success of a treatment is dependent on all three variables.

RESUMEN—Recientes desarrollos en los tratamientos con cataplasmas han llevado a la introducción de agarosa, un agente gelificante derivado de las algas marinas, en la conservación de textiles e innovaciones en su aplicación. La traducción de la información sobre el uso de agarosa en la literatura publicada a la práctica actual ha resultado un reto, ya que predecir cómo estos geles funcionarán en diferentes materiales es complejo.

Este artículo presenta una investigación de tesis realizada en la Universidad de Glasgow para entender cómo las propiedades químicas y físicas de la agarosa pueden ser usadas para informar los tratamientos de conservación exitosos. Las propiedades se evalúan junto con los resultados de pruebas que utilizan estos geles en sustratos textiles para comprender mejor cómo la información teórica presentada en la literatura de alimentos y ciencias biológicas informa y afecta el uso de estos materiales.

Las pruebas examinaron tres fibras (algodón, lana y seda), tres concentraciones de agarosa y dos profundidades de gel. Los resultados mostraron que el éxito de un tratamiento depende de las tres variables.

1. INTRODUCTION

Within the field of conservation, a poultice is an absorbent material that can be used to deliver water, a cleaning solution, or solvent to a substrate, usually with the aim of removing soiling or staining (Stulik et al. 2004; Poultice 2014). In textile conservation, poultice treatments are often utilized for the localized introduction of an aqueous solution or solvent when immersion cleaning is inadvisable. Most poultices work using the properties of diffusion and capillary action. When the wet poultice material is applied to a dry substrate, moisture moves into the substrate in an effort to balance the system. As the poultice begins to dry, the capillary action results in drawing moisture and any other soluble substances such as soiling from the substrate and into the poultice in order to retain equilibrium in the system (Lemiski 1998). Working with poultices requires knowledge and experience, as achieving a balance between diffusion and capillary action demands an understanding of the properties of the substrate, the poultice material, and the solvent. Poultices can be difficult to control and their use risks the formation of tidelines and migration of soiling.

The introduction of agarose, a gelling agent derived from seaweed, to the field of conservation offered a rigid gel poultice material that could theoretically offer greater control over the introduction of moisture into

a substrate and leave no residue (Warda et al. 2007). The speed at which it was adopted in a range of specialties limited the amount of research done to understand how this material would work when combined with different media. This paper examines the chemical properties of agarose and presents a study of its use with three different textile substrates undertaken at the University of Glasgow.

2. AGAR VS. AGAROSE

The difference between agar and agarose gels must be understood before a discussion of their respective uses can commence.

Agar is a hydrocolloid gelling agent extracted from seaweed (Armisen 1997). A hydrocolloid is a polysaccharide or protein most commonly used as a gelling agent for aqueous solutions (Armisen et al. 2009). Agar is the oldest known seaweed-based gel, coming into use in Japan around 1658 (Armisen 1997). It has found use in the biological sciences, mainly as a growth medium for plants and bacteria. Agar-producing seaweeds belong to the class Rhodophyceae and are termed "agarophyte seaweeds" (Armisen 1997). Agar refers to the polysaccharides produced by these seaweeds. Other seaweed gelling agents, such as alginates and carrageenan, differ according to the type of seaweed from which they originate and vary in their polysaccharides (Armisen 1997).

Agarose is the main gelling component of agar and is purified for scientific use. In conservation applications, agarose gels are unusual for their ability to be used with a range of different additives, including chelators, enzymes, water-miscible solvents, and, with some limitations, bleaches. The broad compatibility of agarose can be attributed to its structure and its neutral charge. Agar is not purified to the same extent, and thus retains other components, including charged side groups such as sulfates.

2.1. STRUCTURE AND COMPONENTS

The structure of agar was identified by Araki in 1956. In the same article, he described agar as containing two polysaccharides, agarose and agaropectin, and outlined the structure of agarose. The polysaccharides are mainly composed of galactose, a monosaccharide, while a small percentage of the structure is made of sulfate esters (Armisen et al. 2009). Agaropectin molecules are similar in form to agarose but with greater sulfate content and lower molecular mass (Armisen 1997).

Agarose is a linear polymer of agarobiose units. Agarobiose is the term used for the monomer made of β-(1,4)-(3,6)-anhydro-L-galactose and α-(1-3)–D-galactose rings (fig. 1) (Armisen 1997). Variations within these singular units can occur depending on the species of seaweed and the season in which it is harvested (Armisen 1997).

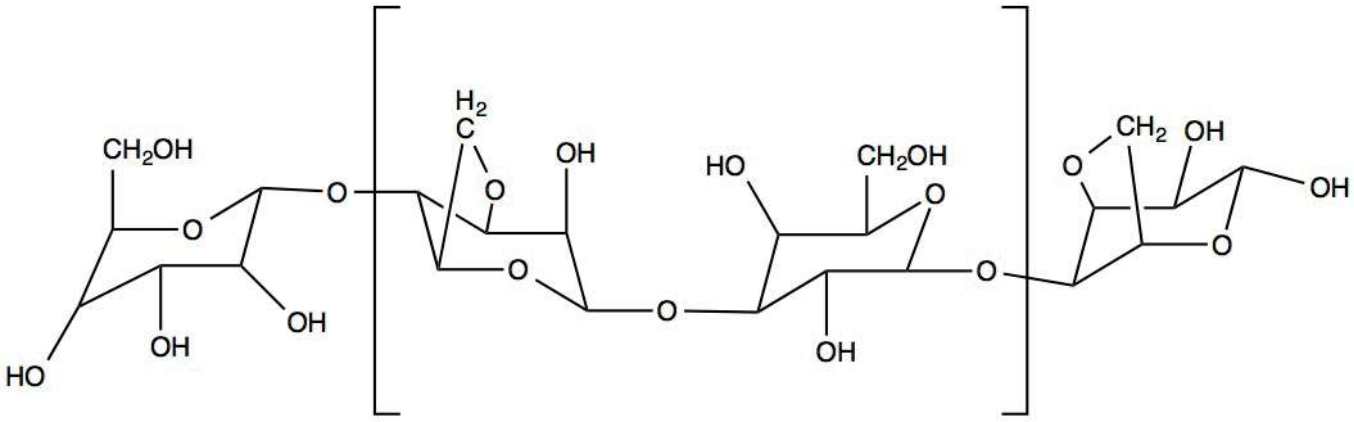

Fig. 1. The agarobiose monomer, β-(1,4)-(3,6)-anhydro-L-galactose and α-(1-3)-D-galactose.

Traditionally, the gels were extracted by freezing seaweed and then allowing it to thaw and dry. This produced flake or powdered agar (Armisen 1997). The process has since been commercialized, though the principles remain the same.

Rheology is the study of the flow of matter, and is important to understand in this context as it can be used to describe how the gel structure transforms as it changes phases from liquid to semisolid (fig. 2) (Labropoulos et al. 2002). Agarose gels transition from a dry powder to a liquid gel in the presence of water and heat. In its liquid state, agarose is a random coil; when it cools, the coil forms a double helix through the establishment of hydrogen bonds (fig. 2) (Labropoulos et al. 2002, 405). These double helixes crosslink and form a grid-like structure, thus creating the rigid gel (Arnott et al. 1974). This structure gives strength to the gels and forms the pores that allow for capillary action and diffusion to take place. This structure is crucial for the use of agarose gel in conservation, as it allows the gel to act as a poultice (Van Dyke 2004, 102).

2.2. AGAROSE GEL PROPERTIES AND PRODUCT CHOICE

A literature review has been used to identify some of the different types of agarose that have been used in conservation treatments and experiments. Table 1 provides a list of products that differ in price, properties, and sources. As these factors can affect treatment, an understanding of how and why agarose gels work is essential to ensure that the right product is selected for a given treatment, and obtained for an acceptable price.

The chemical companies surveyed for this paper listed up to seven properties associated with agarose gels. Three of these properties—grade, gelling temperature or transition temperature, and electroendosmosis—are considered below, as they may affect conservation treatments. In industry, agar gels are used differently, and as they are less pure, are not presented to the buyer with all of the same properties.

2.2.1. Grade

The trend seen in most conservation articles indicates a preference for molecular biology grade agarose (Campani et al. 1997; Warda et al. 2007; Pouliot et al. 2013). Agarose is used in the biological sciences for a range of different purposes including gel electrophoresis, a technique used to separate macromolecules, such as the nucleic acids that make up DNA, by size and electrical charge. As a result, molecular biology grade materials are purified to meet a specific standard, which is provided in the product literature. Purification involves the removal of agaropectin and other impurities that may restrict gelling capability or movement through the gel structure.

Agar, on the other hand, is usually marketed for microbiology as a growth medium for bacteria and does not require the same degree of purity.

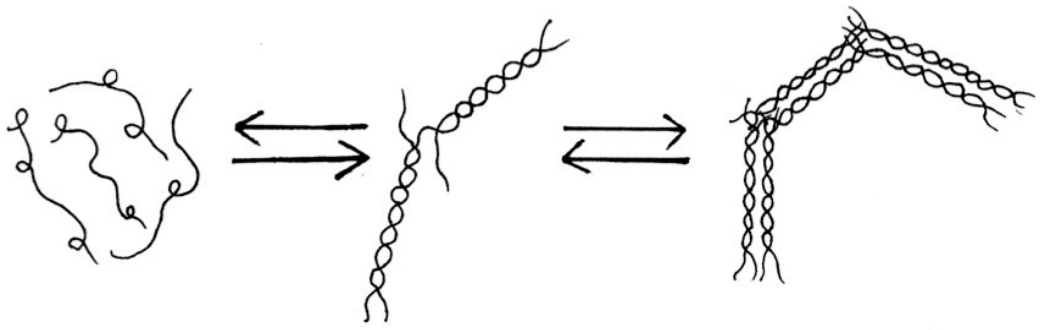

Fig. 2. The gelling phases of agarose from left to right: agarose in its liquid phase, the double helix forming and crosslinking as the gel cools, the crosslinked helixes forming the rigid gel.

Table 1. Properties of Agar and Agarose used in Conservation

Grade	Gelling Temp	Melting Temp	EEO	Use	Cost* *As of 2014	Source
IBI Molecular Biology Grade	36°C	88°C	0.005–0.13	pH, Conductivity, Cleaning (Wolbers, pers. comm.)	$111.00/100g	Universal Medical
IBI Molecular Biology Grade, Low Melt Point	24–28°C	65.5°C	0.12	Enzymes (Wolbers, pers. comm.)	$295.00/100g	Universal Medical
Agarose LE Molecular Biology Grade	36°C	88°C	0.13	Cleaning (Shaeffer and Gardiner 2013)	$104.00/100g	Benchmark Scientific
A9539 Molecular Biology Grade	36°C	N/A	0.09–0.13	Research, Testing (Schmitt 2014)	£151.00/100g	Sigma Aldrich
Fluka Agarose 05068	34–37°C	N/A	0.23–0.27	Testing, Cleaning (Campani et al. 2007)	Discontinued	Sigma Aldrich
Seakem LE Agarose	34.5–37.5°C	90°C	N/A	Aging, Testing (Warda et al. 2007)	$137.00/100g	Cambrex Bio Science
Fluka Agar 05040	35°C	70°C	N/A	Testing, Cleaning (Campani et al. 2007)	$43.90/100g	Sigma Aldrich
VII-A A0701 Low Melt Point	26°C	65.5°C	0.12	Wolbers' Workshop for Textiles, Berlin, 2013	£727.00/100g	Sigma Aldrich
Fluka Agarose A4679	34–38°C	N/A	.09–0.13	Replacement for Fluka 05068 as recommended by Sigma Aldrich	£253.50	Sigma Aldrich

2.2.2. Gelling and Melting Temperature

An important property to consider for certain types of treatments is gel hysteresis, or the difference between the melting and gelling temperatures (Armisen 1997). In general, agarose melts above 80°C and gels at around 35°C (Armisen 1997). For most uses, including general cleaning treatments and the measurement of pH and conductivity, the heat needed to melt the agarose and the point at which the liquid begins to thicken are irrelevant, so any product can be selected.

Heat sensitive additives however, especially enzymes, may benefit from the use of a modified, low-melting-point gel, as their lower melting temperature will not denature the enzyme proteins as they are

added to the liquid gel solution. Low-melt gels can be used in the same conditions as a standard grade agarose. Despite the versatility of low-melt agarose, the price is usually more than double that of the standard agarose (table 1), and for this reason it may be more expedient to instead use the standard gel and add the enzyme as it cools.

2.2.3. Electroendosmosis (EEO)

Electroendosmosis (EEO) is the movement of liquid under the influence of an electric current (Agarose Matrix 2011). When powdered agarose is hydrated to form a gel, soluble counter cations will dissociate from charged side chains such as sulfates that have not been removed during purification. In the context of gel electrophoresis, EEO occurs when the applied electrical current causes these cations to move through the gel in the opposite direction of the molecules being separated, which can skew results through internal convection (Sigma Aldrich 2014). Because of this, low EEO gels are needed for electrophoresis. For conservation, this property has less effect, as there is no electric current being applied.

3. GLASGOW RESEARCH

The agarose gel study undertaken at the University of Glasgow (Schmitt 2014) aimed to identify ideal working standards on common textile fibers through the manipulation of the basic working properties. It examined three main questions:

- What effect does the type of textile fiber have on the rates of diffusion and capillary action of agarose gel poultices at given concentrations?
- How does the concentration of the agarose gel poultice affect/influence its working properties?
- Does the depth of the agarose gel affect its effectiveness as a poultice?

It was decided to test agarose rather than agar, as it had been used in the majority of published conservation treatments consulted (Van Dyke 2004; Stockman 2007; Warda et al. 2007; Ellis 2009; Sahmel et al. 2012; Pouliot et al. 2013; Shaeffer and Gardiner 2013). The agarose product chosen was one that is available worldwide through Sigma Aldrich at a median price point (table 1). Agar and preparation methods using solutions other than water were not included in this study in order to limit the number of variables tested.

Three plain-weave fabrics of similar thread count were chosen for testing: cotton lawn, silk habotai, and wool delaine. Fabrics were scoured and cut into equal-sized squares. These textiles were stained using Parker Quink, a water-soluble fountain pen ink, using a stencil to maintain consistent size and shape (fig. 3). Based on the physical properties of agarose discussed above and trends in existing literature (table 2), three concentrations of agarose gel—1% w/v, 2.5% w/v, and 4% w/v—were chosen for testing. Two depths of gel were also tested. 0.5 cm was chosen based on the most commonly used gel depth described in conservation literature and 0.3 cm based on the observation of the free-form gel depth that occurs when agarose is poured on a flat surface (Van Dyke 2004; Sahmel et al. 2012).

Agarose was weighed out according to the concentration being made. Deionized water was then added and the solution was placed on a hot plate and heated to 85°C, or until the solution was clear and no powder was visible in the beaker. The liquid gel was then poured into macaroon molds to form the individual gel disks. After about half an hour, the gels had set and cooled, and could be removed from the molds.

Fig. 3. Application of ink onto fabric using a brush and stencil.

Table 2. Summary of Trends in Conservation Treatments Utilizing Agarose or Agar

Author	Medium	Gel Concentration	Gel Depth	Time Applied	Treatment
Van Dyke 2004	Paper	1.2% Agarose	0.5–0.8 cm	N/A	Enzymes
Stockman 2007	Paper	1.2% Agarose	N/A	N/A	Enzymes
Warda et al. 2007	Paper	1% Agarose	N/A	1–4 min.	Adhesive removal (no enzymes)
Pouliot et al. 2013	Ceramics	2-4.5% Agarose	N/A	N/A	Cleaning
Campani et al. 2007	Ceramics	1.5% Agarose 2% Agar	1 cm	90-160 min.	Residue investigation
Sahmel et al. 2012	Textiles (Wool)	1% Agarose (4% for Tests)	~0.5 cm	15 min. to 60 min	Cleaning
Shaeffer/ Gardiner 2013	Textiles (Linen)	1% Agarose (4% for Tests)	N/A	1 hr.	Cleaning
Ellis 2009	Textiles (Cotton)	1% Agarose	N/A	N/A	Enzymes
Younger/ Duffus 2014	Textiles (Cotton)	2.25% with Agarose 3.5% with Agar	N/A	2 hr.	Cleaning
Gulotta et al. 2014	Stone	3% Agar	Painted onto surface	30 min.	Cleaning

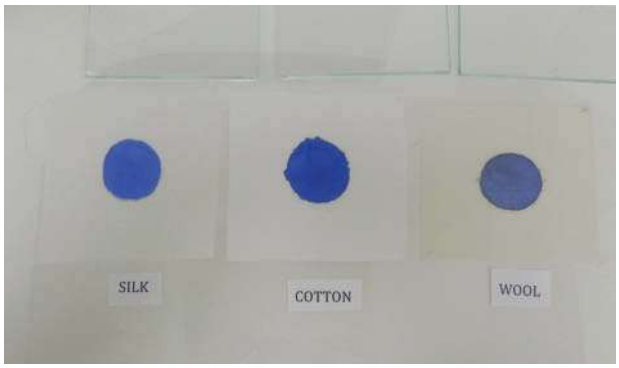

Fig. 4. Examples of fabric prior to gel application. Fabrics from left to right: silk, cotton, wool.

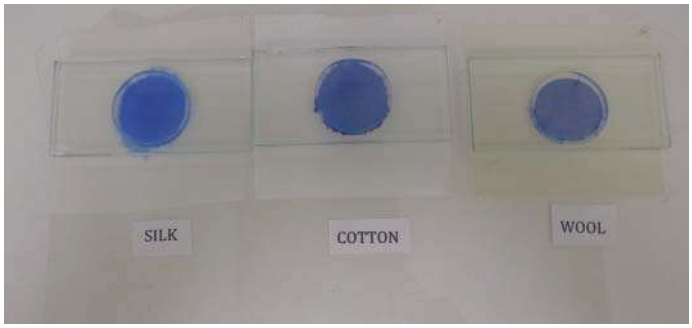

Fig. 5. Examples of gels applied to fabric samples. Fabrics from left to right: silk, cotton, wool.

Gels were placed on the textiles on top of the ink stains and lightly pressed into place; glass weights were also placed on top to increase surface contact (figs. 4-6). This system was left in place for an hour. The gels were then examined and photographed, and the following information was recorded:

- the degree of contact as evidenced by the fabric imprint on the gel,
- how much and how evenly the stain was absorbed into the gel matrix, and
- the increase in the diameter of the stain.

The procedure was repeated four times for each fiber type, gel concentration, and gel depth to achieve statistically significant results.

3.1. DEFINITION OF TERMS

Wicking is the lateral movement of water or solvent through a substrate, that may carry with it any solubilized substances. Wicking is dependent on the amount of liquid released, the properties of the substrate through which it is moving, and the environmental conditions in which it is happening. Tidelines represent the point at which the solvent or water evaporates, leaving residual soluble matter in concentrated form. For this project, the extent and nature of the tidelines and ring marks were used as a guide to understand the amount of water released. The term “ring mark” or “ringing” is used here to describe the overall stain caused by ink residue being

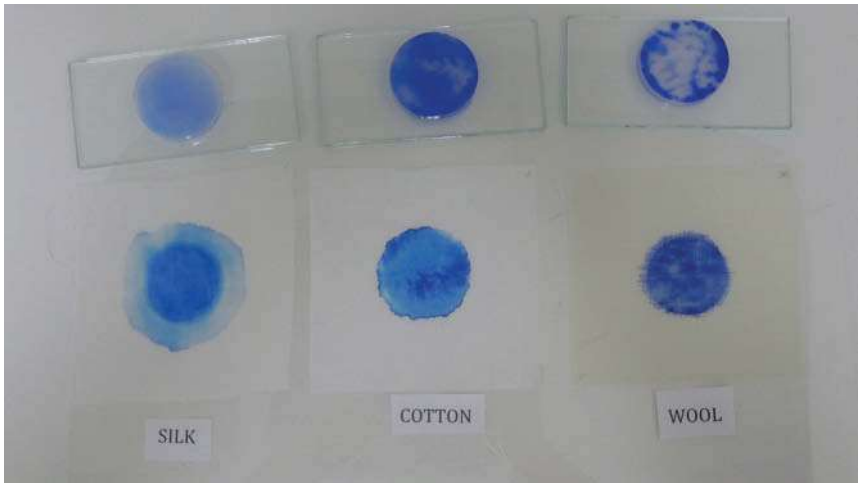

Fig. 6. Examples of fabric and gels after tests. Fabrics from left to right: silk, cotton, wool.

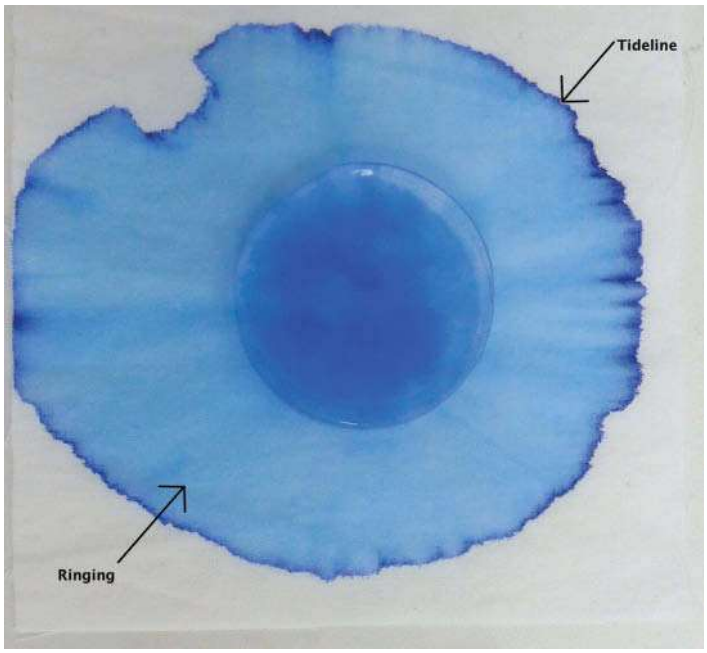

Fig. 7. Example of ringing and tidelines.

drawn along the fibers from the outward movement of water from the gel into the substrate (fig. 7). "Tideline" is used to describe the concentrated line of staining along the outer edge of the ring mark (fig. 7).

3.2. RESULTS

3.2.1. Silk

Compared to the other fiber types, silk showed the most water movement through the fabric at all concentrations. Wicking carried the ink outside the original dimensions of the stain in all tests (figs. 8-10). This movement resulted in ringing and a faint tideline, as the moisture from the gel wetted out much of the textile, emphasizing the challenges of controlling wicking in silk fabrics. Decreases in gel depth and increases in concentration reduced this movement; however, the wicking was still comparatively extensive when gels were applied to silk versus when they were applied to cotton and wool.

3.2.2. Cotton

Cotton exhibited less wicking of the ink through the textile than silk (figs. 8-10). Tidelines were prevalent in all the tests. Lateral spread could be controlled by changing concentration and depth and ensuring good contact between the gel and the textile. For example, a balance between the rates of diffusion and capillary action was achieved at a concentration of 2.5% when there was enough surface contact, as evident by the limited wicking into the textile (fig. 9). The changes visible from test 1–2 to tests 3–4 show how the application of light finger pressure on the gel prior to applying the weight enhanced contact and thus capillary action.

3.2.3. Wool

Wool's hydrophobic nature reduced the movement of the ink through the textile as its resistance to water limited the degree of wicking (figs. 8-10). This property allowed changes in depth and concentration to have a greater effect on the results of these tests.

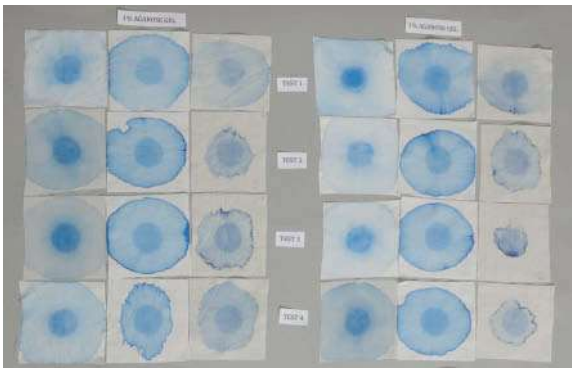

Fig. 8. Results of 1% gels on fabric substrates. Movement of ink through fabric samples. 0.5 cm gel samples are on the left and 0.3 cm gel samples on the right. Fabrics are laid out from left to right, always in the order of silk, cotton, and wool.

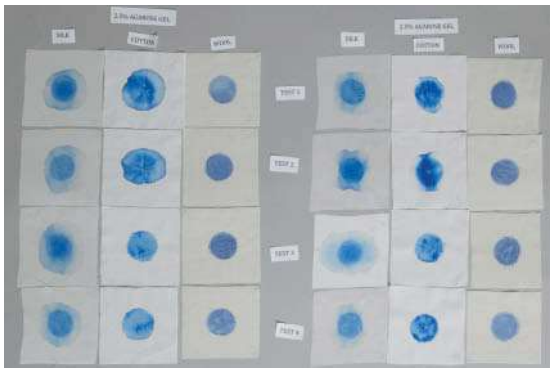

Fig. 9. Results of 2.5% gels on fabric substrates. Movement of ink through fabric samples. 0.5 cm gel samples are on the left and 0.3 cm gel samples on the right. Fabrics are laid out from left to right, always in the order of silk, cotton, and wool.

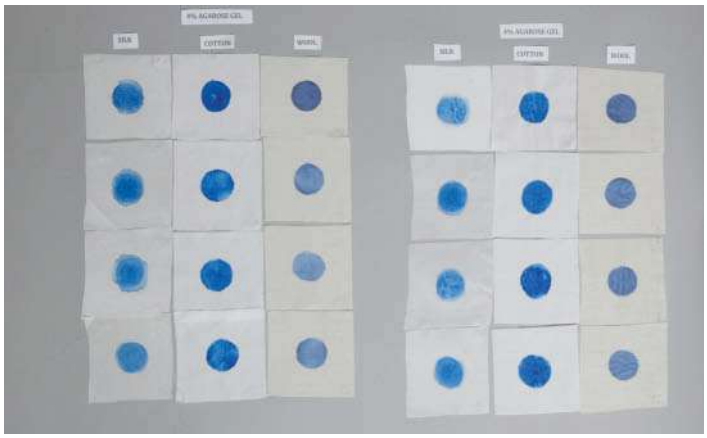

Fig. 10. Results of 4% gels on fabric substrates. Movement of ink through fabric samples. 0.5 cm gel samples are on the left and 0.3 cm gel samples on the right. Fabrics are laid out from left to right, always in the order of silk, cotton, and wool.

3.2.4. Absorption of ink

The amount of ink absorbed into the gels differed for each textile and concentration. Concentrations of 2.5% and 4% exhibited more absorption of ink than the 1% gels. The uptake was, however, unevenly distributed through the gels (figs. 6, 11). Ink uptake was usually associated with points of good contact between the gel and the textile. Fiber-specific information was difficult to gauge from the gels, as the amount of ink applied to each fabric differed and thus the results across fiber types are statistically nonviable. Based on the color of the gel, with darker gels indicating a higher concentration of ink, 2.5% gels absorbed the most ink. As this is a cross concentration comparison including all fiber types, this conclusion has merit. 2.5% gels were able to solubilize the ink and maintain a balance of capillary action without flooding outward into the textile.

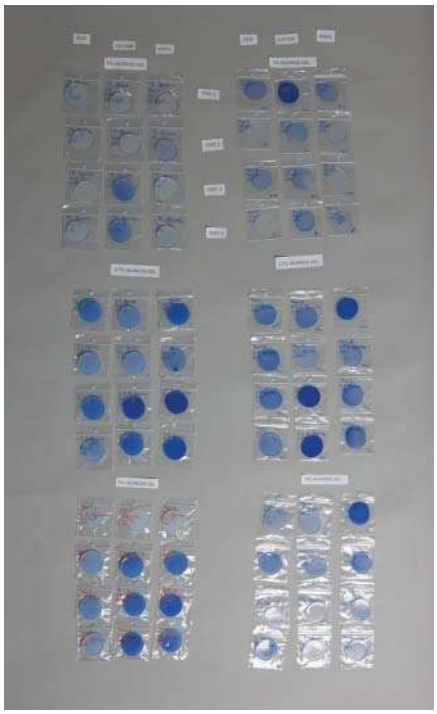

Fig. 11. Movement of ink into gels. 0.5 cm
gel samples are on the left and 0.3 cm gel samples on the right.
Order mirrors that of the fabric samples.

3.2.5. Contact

1% gels indicated better surface contact on all textiles, as exhibited by the weave imprint of the surface of the gel. This contact decreased as concentration increased. 4% gels exhibited the least contact, with very little fiber imprint visible on the surface of the gel. 2.5% gels fell between these two results: good contact was achieved, though the imprint of the weave was not completely visible across the entire surface of the gel. Issues with contact were observed after the first two test cycles of 2.5% gels. At this point, light finger pressure was applied to the gels prior to weighting, and both contact and capillary action were greatly improved (fig. 9).

4. DISCUSSION

4.1. THE EFFECT OF FIBER TYPE

Fiber type is one of the most important factors affecting the diffusion of moisture through a fabric. In this research it was seen to influence the rates of diffusion and capillary action of the gels tested. For silk, a strongly hygroscopic fiber, the fabric absorbed moisture from the gel preferentially even at 4%, leading to wicking and tidelines at the point of evaporation. This would suggest that when using agarose gels on silk, it would be beneficial to consider the application of a barrier to limit lateral movement of the gel solvent into the textile. This has been successfully achieved on wool using cyclododecane (Sahmel et al. 2012).

For wool, the rates of diffusion and capillary action appeared to be balanced at concentrations of 2.5% and 4%, as results for these tests exhibited minimal wicking of moisture into the textile. This suggests that agarose gels might be considered for use independently, i.e., without a barrier layer, on wool and other similar fibers.

It is, however, important to stress that there are many other factors which impact the rates of diffusion, such as fiber processing, fabric structure, the presence of finishes, soiling, and staining. It was out of the scope of this research to examine the effect of these variables, but they would be necessary considerations in any practical application.

4.2. THE INFLUENCE OF CONCENTRATION

Across all substrates, 1% gels showed extensive ringing and tidelines, with very little absorption back into the gel; it is therefore suggested that 1% gels are not ideal for use on textile substrates without a barrier. It might be argued that a 2.5% gel would be overall more effective in most treatment situations, reducing risk and achieving better capillary exchange.

In general, the results of these tests indicated that gels of a higher concentration (2.5% or 4%) that had good contact with the substrate resulted in strong capillary action and limited preferential diffusion into the fiber. The 2.5% gels offered the best contact with the textiles; their flexibility allowed them to bend and conform to the weave structure, encouraging the formation of a system of diffusion and capillary action imperative to poultice treatments. The rigid 4% gels could not achieve contact with the textile to the same degree, limiting the overall effectiveness of diffusion and capillary action.

It is also important to consider that a gel of higher concentration cannot be used with some additives such as enzymes, as the small pore size would be too restrictive. Furthermore, the property of wettability must be considered when forming gels with any additive or cleaning product, as their addition may change the surface tension of the water within the gel and hence the wetting out of the substrate.

4.3. THE EFFECT OF GEL DEPTH

Decreasing the depth of gel reduced the volume of water held within the gel matrix, thus further limiting the movement of water through the textile substrate. Reducing the depth of a gel could therefore aid in controlling a poultice on more hydroscopic materials. This variable can be used in conjunction with concentration to achieve ideal contact and capillary action without flooding the substrate.

4.4. LIMITATIONS OF THE RESEARCH

The results of the experimental phase broadly fulfilled the aims of the research. However, it is important to acknowledge that the scope of the testing was limited and that the observations made would benefit from further, more targeted investigation. The fabrics tested were new and although they were consistent in terms of weave count, they were not identical. Only water was tested as a solvent. Although this provides a baseline standard that helps in understanding how diffusion and capillary action work under the conditions tested, further experimental work into the use of other solvents or additives would be useful. Length of treatment, the use of barriers, and the influence of textile condition on the application method and results are all areas of further research.

5. CONCLUSION

There is great potential for the use of agarose as a rigid gel poultice in textile conservation. This project has contributed to the body of knowledge about the physical and chemical properties of agarose, and through preliminary testing, has indicated how agarose gels work on different textiles at different depths

and concentrations. Although there is much scope for further research at an academic level, and it is always necessary to undertake extensive object-specific testing, it is hoped that the information gathered during the project will be of benefit to conservators who are considering using agarose.

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SOURCES OF MATERIALS

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GELLING IN THEORY AND PRACTICE: AN EXAMINATION OF AGAROSE GELS IN TEXTILE CONSERVATION

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