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Program in Art Conservation

Agar Bioplastic: Understanding Its Degradation and Exploring Its Viability as a Fill for Glass

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Abstract

While studies have shown its suitability and efficacy as a cleaning material, agar is often seen as a temporary material, not something where longevity is desired. In recent years agar has been explored more widely as a bioplastic, a class of materials becoming more ubiquitous every day which have even made their way into the art world. Museums with design collections have begun acquiring bioplastics and are coming face to face with the reality of how to care for these materials. There is little research into the long-term stability of agar in its dried form. This research aims to evaluate the stability of art made from agar by examining the long-term effects of a typical museum environment (controlling temperature and relative humidity) on lab and food grade agar, using artificial aging, visual observation, thickness measurements, colorimetry, Fourier Transform Infrared Spectroscopy (FTIR) and Pyrolysis Gas Chromatography-Mass Spectrometry (pyGC-MS). The results of this study will also inform whether agar is a viable material option for loss compensation of glass. After artificial aging, agar experienced significant and undesirable physical changes which were detectable both with and without instrumentation. Chemical changes were not detected by instrumentation. The notable changes in color and thickness of agar caused by aging would be problematic for artworks made of it, and if it were to be used as a conservation material.

Keywords: agar, bioplastic, glass fills, glass conservation, artificial aging, accelerated aging, colorimetry, FTIR, pyGC-MS

1. Introduction

Agar is a hydrocolloid derived from red algae of the Gelidium or Gracilaria families. The word agar comes from agar-agar, the Malay name for the algae from which the gel is produced. Agar comes in a variety of forms including powder, flakes and sheets. Dried agar, in any form, is insoluble in cold water but soluble in hot and dissolves readily in boiling water/upon heating. Agar has its origins in Japan and is generally believed to have been discovered by Tarazaemon Minoya in 1658 (Phillips and Williams 2009). Primarily used for culinary purposes, the use of agar spread throughout Asia and eventually reached Europe when Europeans in the East Indies learned to use this ingredient for making fruit jellies from the Malay people (Fisheries and Aquaculture Department of the Food and Agriculture Organization of the United Nations 1990). Agar was introduced in Europe in 1859 and was being used by Robert Koch as bacteriological culture medium by 1882 (Phillips and Williams 2009). From that point forward agar began being used not only in food preparation but also in science. Though agar is most commonly used as a thickening agent in foods and a solidifying component of bacteriological culture media it has been widely accepted as a rigid gel used for cleaning purposes across conservation disciplines. It is easily

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accessible, though varies in cost, simple to prepare and exhibits many qualities that are desirable for a conservation material.

In recent years agar has been explored more widely as a bioplastic alternative to traditional plastics. Bioplastics, or bio-based plastics, are plastics made from natural, raw materials rather than petroleum products. Bioplastics are becoming more ubiquitous every day and have even made their way into the art world. Many art schools are pushing students to experiment with these new and unique materials, testing their limits. With art students learning about these materials, it only makes sense that more bioplastic artworks are entering museum collections and will soon come under the purview of conservation. Museums with design collections such as the Philadelphia Museum of Art, MOMA and the Cooper-Hewitt have all begun acquiring bioplastics and are coming face to face with the reality of how to care for these materials. Aging behavior of bioplastics, even more specifically transparent bioplastics, has been studied but not extensively and there are no studies specifically about agar bioplastic in this context.

While many studies have shown its suitability and efficacy as a conservation cleaning material, agar is often seen as a temporary material that is used then discarded, not something where longevity is desired. There is extensive research into agar's physical properties, its rheology and gel strength, and mechanisms of gelation and decomposition; however, there is little research into its long-term stability, particularly in its dried form (Araki 1937, Tseng 1944, Mao et al 2017, Ouyang et al 2018, Zeece 2020).

Though applications of gels for cleaning in cultural heritage were first introduced for paintings conservation, all specialties have begun to incorporate gels in their figurative toolkit. Many innovative uses of agar gels, such as for stain reduction and electrochemical reduction of silver sulphide, have been explored in objects conservation (Scott 2012, Pouliot et al 2013, Joao et al 2017). While there is significant research into the applications of agar in the field of conservation there is only one study on the application of bioplastics in conservation practice, *Sustainable Future Alternatives to Petroleum-Based Polymeric Conservation Materials* by Shashoua et al, 2017. Their research addresses the use of bioplastics as adhesives and coatings in treatment and while this study is relevant it is not directly applicable to the aging of agar and its potential use as a treatment material. Further research into the use of bioplastics in art conservation is necessary.

There are a limited number of materials a conservator can choose from when filling losses in glass, and those typically used each have their own benefits and limitations. While conservators typically turn to epoxies or Paraloid B-72, there is interest in finding alternative materials with better working properties, chemical stability, and reversibility. Inspired by artists working with agar to mimic glass, such as Margaret Byrd and Yi Hsuan Sung, this study was designed to understand the degradation of agar bioplastic and thus its potential viability as a fill material for glass.

2. Materials

2.1 Agar Samples

Bioplastic samples of 4% (w/v) agar of two different purities, lab and food grade, were prepared for this study. Agar samples were prepared by adding agar powder to water, mixing, microwaving for 30 seconds on high, stirring and repeating this three times (see Table 1 for agar recipes). Twelve samples in total were prepared by casting agar into plasticine molds on sheets of glass, allowing them to fully dry for 7 days and then cutting them into uniform 1 x 1 ½ inch samples.

As agar bioplastic typically contains glycerin, which imparts flexibility to the material, three samples of each grade of agar were prepared with the addition of glycerin, and three were prepared without. Benchmark Scientific molecular biology grade agar powder was used as the lab grade material, Living Jin agar-agar powder as the food grade material and Beauty 360 pure glycerin was used in samples containing glycerin.

4% (w/v) Agar	4% (w/v) Agar with glycerin
• 30ml distilled water • 1.2g agarose	• 30ml distilled water • 1.2g agarose • 0.5g glycerin

Table 1. Agar Recipes

3. Methods

All sampling and analysis was conducted in the Scientific Research and Analysis Laboratory at Winterthur Museum. Exposed surface samples were taken from each agar sample to conduct analysis. All analysis was conducted on controls as well as aged material samples for comparative purposes.

3.1 Visual Examination

Visual examination was performed on each sample before and after artificial aging and analysis. Photographs were taken using an iPhone 11.

3.2 Artificial Aging and Interval Sampling

Once the samples were prepared, and selectively sampled, they were laid flat on an metal tray and placed inside an ESPEC test chamber to undergo artificial aging to evaluate the effect of temperature and relative humidity on the agar over time. Aging was conducted following parameters used in analysis of paper degradation, mimicking a museum setting. Samples were subjected to 80°C and 65% relative humidity for a total of 21 days. These settings were adapted from the methodologies used by Šelih et al. (2007), and Wiggins et al. (2019). The settings on the oven were ramped down to ambient temperature and relative humidity over the course of 3 hours to open the door and collect interval samples. One interval sample of each material was collected prior to aging (Day 0) as well as after 3, 7 and 21 days of exposure in the ESPEC chamber. Samples were collected by cutting off a small piece at the lower right corner with clean scissors and stored in vials. Once samples were collected, documented and placed back inside the ESPEC, the oven settings were ramped back up to 80°C and 65% relative humidity over the course of 3 hours to continue the aging process.

3.3 Thickness Measurements

Thickness measurements were taken using a Starrett 3732XFL-1 Electronic Micrometer without output to evaluate shrinkage or swelling of the material.

3.4 Colorimetry

Colorimetry measurements were taken before and after artificial aging using a Konica Minolta CR-221 chroma meter. The chroma meter was calibrated before taking measurements for this study. Colorimetry uses the CIELab color space to measure color and calculate color differences in 3-dimensional space. L*, a* and b* represent three values used to quantify color. L* represents lightness from black to white, a* corresponds with red to green and

b* corresponds with blue to yellow. These values are used to calculate Delta- E which represents the overall color change measured.

3.5 Fourier Transform Infrared Spectroscopy (FTIR)

Samples were analyzed before and after aging by FTIR (Fourier-transform infrared) microspectroscopy, an instrumental technique that permits the general classification of natural organic materials (such as waxes, proteins, oils, polysaccharides, and resins) and the more specific identification of synthetic resins, inorganic pigments, and natural minerals. Sample material was acquired with a stainless-steel scalpel and the aid of a stereomicroscope and then placed directly on a diamond cell. The material was rolled flat on the cell with a steel micro-roller to decrease thickness and increase transparency. The sample was analyzed using the Thermo Scientific Nicolet 6700 FT-IR with Nicolet Continuμm FT-IR microscope (transmission mode); data was acquired for 128 scans from 4000 to 650cm^{-1} at a spectral resolution of 4cm^{-1}. Multiple scrapings of the sample were taken from the bulk sample and multiple spectra were taken from different areas within each scraping. Spectra were collected with Omnic 8.0 software and analyzed in this program with various IRUG and commercial reference spectral libraries.

3.6 Pyrolysis- Gas Chromatography - Mass Spectrometry (py-GC-MS)

Samples were analyzed by py-GC-MS with no chemical derivatization. Samples were placed into a 50μL stainless steel Eco-cup fitted with an Eco-stick and placed into the pyrolysis interface where it was purged with helium. The Frontier Lab EGA/PY-3030D double-shot pyrolyzed system was interfaced to an Agilent Technologies 7820 gas chromatogram equipped with 5975 mass selective detector (MSD). A J&W DB-5MS Agilent 19091S-433 capillary column was used for separation (30m × 250μm × 0.25μm) with helium carrier gas set to 1.2 mL/minute. Samples were thermally desorbed from 30°C to 600°C at 10C/min in a helium gas protected environment. These parameters were adapted from Ouyang et al. (2018). The split injector was set to 280°C with a split ratio of 20:1 and no solvent delay (9.26 psi). The GC oven temperature program was 43°C for zero minutes then ramped at 10°C/minute to 325°C, followed by a three minute isothermal period, for a total run time of 38.2 minutes. The MS transfer line was at 280°C, the source at 230°C and the MS quad at 150°C. The mass spectrometer was scanned from 33-600amu at a rate of 2.59 scans per second.

4. Results

4.1 and 4.2 Visual Examination, Artificial Aging and Interval Sampling

A visual difference in the samples before and after aging, as well as at the various intervals, was very evident as seen in Figure 1. The lab grade samples which were initially clear, turned a light amber color, and the food grade samples, which were initially a pale yellow, turned a deep warm amber color.

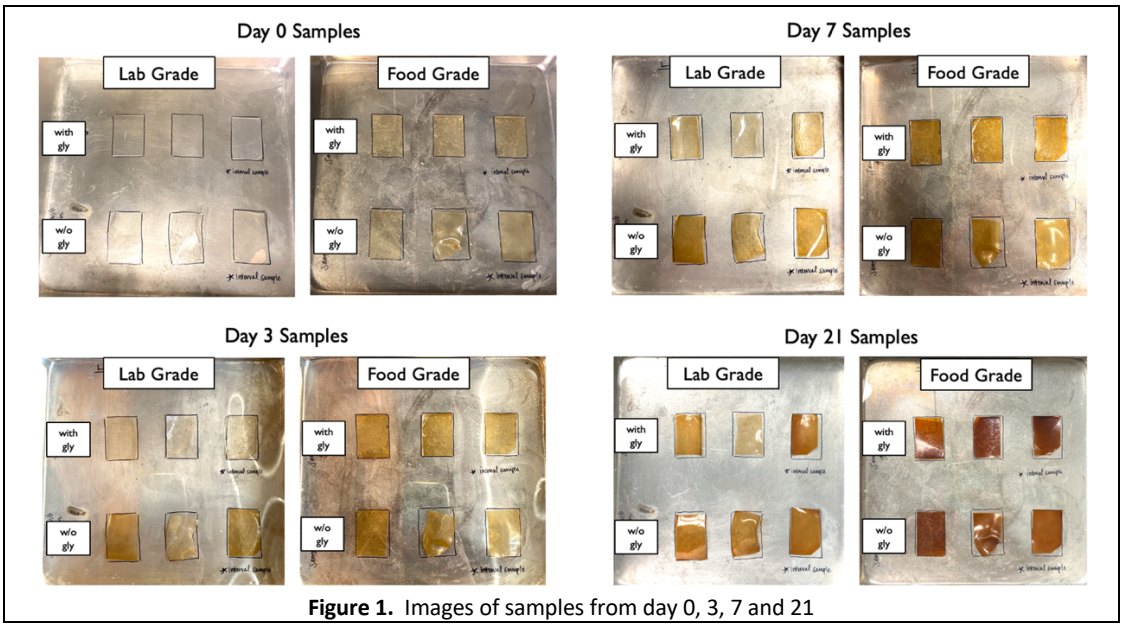


Figure 1. Images of samples from day 0, 3, 7 and 21

4.3 Thickness Measurements

Thickness measurements of all samples are presented in Table 2. Each sample was measured three times to obtain an average measurement. The values for percent change in thickness were graphed to convey the degree of dimensional change over the course of aging, as seen in Figure 2. The dimensional changes caused by prolonged exposure to elevated temperature and relative humidity could not be measured within the scope of this study but can be seen in Figure 3. The conditions in the oven led to wrinkling, curling of edges and corners and overall distortion of the samples.

Thickness Measurements of Agar Samples				
Sample	**Thickness (mm)**			**Average Thickness**
	Measurement 1	**Measurement 2**	**Measurement 3**	
Day 0 - LG w gly	1.053	1.025	1.051	1.043
Day 0 - LG w/o gly	0.37	0.471	0.361	0.4
Day 0 - FG w gly	0.54	0.477	0.564	0.527
Day 0 - FG w/o gly	0.31	0.314	0.32	0.315
Day 3 - LG w gly	0.748	0.726	0.716	0.73
Day 3 - LG w/o gly	0.379	0.337	0.325	0.347
Day 3 - FG w gly	0.41	0.422	0.397	0.409
Day 3 - FG w/o gly	0.254	0.334	0.292	0.293
Day 7 - LG w gly	0.52	0.577	0.556	0.551
Day 7 - LG w/o gly	0.325	0.311	0.312	0.316
Day 7 - FG w gly	0.371	0.331	0.288	0.33
Day 7 - FG w/o gly	0.239	0.273	0.239	0.25
Day 21 - LG w gly	0.522	0.523	0.503	0.516
Day 21 - LG w/o gly	0.313	0.298	0.302	0.304
Day 21 - FG w gly	0.244	0.233	0.254	0.244
Day 21 - FG w/o gly	0.244	0.233	0.233	0.237

Table 2. Thickness measurements of agar samples before and after aging.

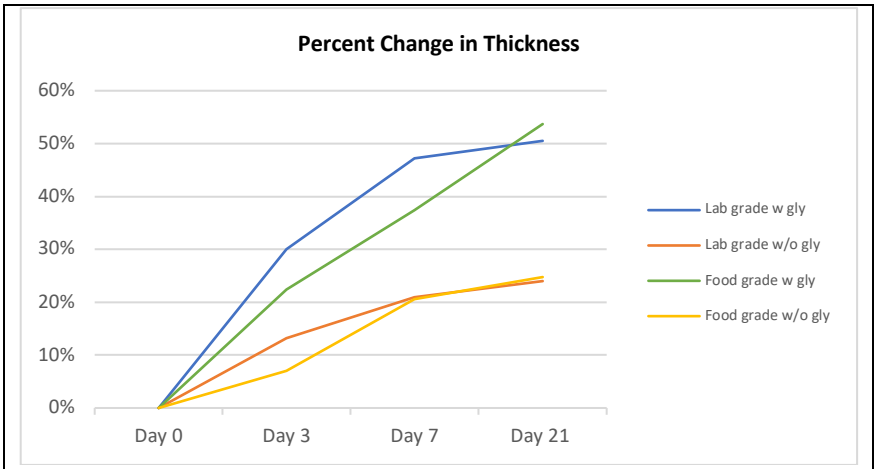
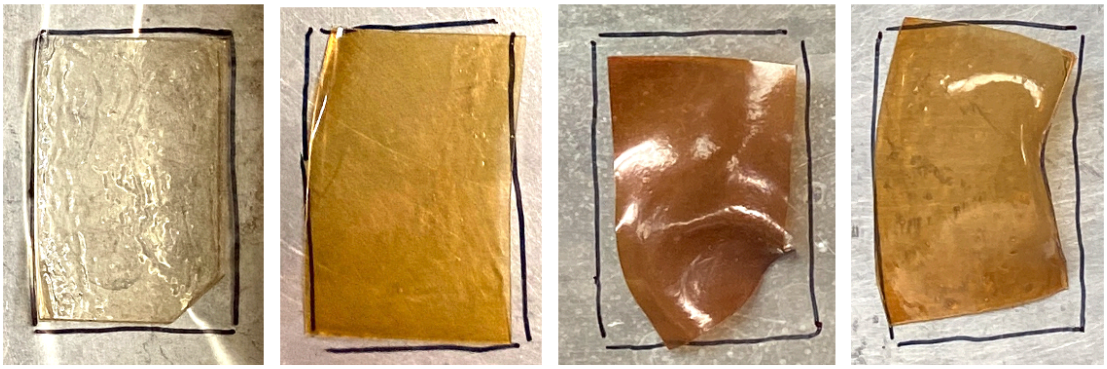


Figure 2. Graph of percent change in thickness of samples over the course of 21 days.



3a. *Wrinkling* **3b.** *Curling of edges & corners* **3c.** *Overall distortion* **3d.** *Overall distortion*

Figure 3. Details of samples exhibiting dimensional distortion after aging.

4.4 Colorimetry

The color change exhibited by the samples before and after aging was perceptible without instrumentation; however, it was important to confirm this quantitatively and to compare the color values obtained. The initial colorimetry measurements of each sample and the overall color change (ΔE) are presented in Table 3. The ΔE values were calculated using the formula below and graphed to convey the rate of color change over the course of aging, as seen in Figure 4.

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

Colorimetry Measurements of Agar Samples					
Sample	**Interval**	**L***	**a***	**b***	**ΔE From Day 0**
Lab grade agar with glycerin	Day 0	81.76	-109.81	140.97	-
	Day 3	82.26	-106.91	141.84	3.07
	Day 7	77.19	-96.58	133.09	16.06
	Day 21	62.52	-71.68	107.81	54.07
Lab grade agar without glycerin	Day 0	89.05	-118.27	153.54	-
	Day 3	77.99	-96.16	134.48	21.16
	Day 7	78.43	-98.13	135.24	29.21
	Day 21	59.17	-60.63	102.04	82.87
Food grade agar with glycerin	Day 0	82.36	-107.54	142.01	-
	Day 3	77.37	-95.83	133.41	15.36
	Day 7	73.74	-88.37	127.15	25.74
	Day 21	55.23	-62.5	95.23	70.38
Food grade agar without glycerin	Day 0	84.64	-108.04	145.94	-
	Day 3	80.88	-100.38	139.45	8.14
	Day 7	79.38	-98.15	136.87	14.41
	Day 21	57.83	-64.7	99.72	68.8

Table 3. Colorimetry measurements (L*, a* and b* values) of agar samples before and after aging, and ΔE values.

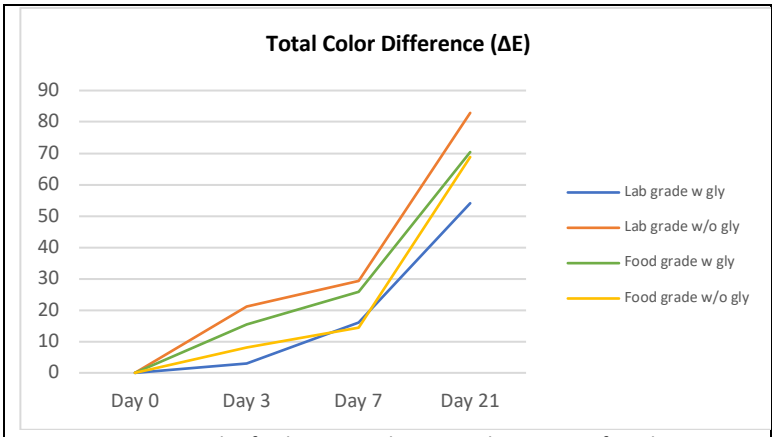
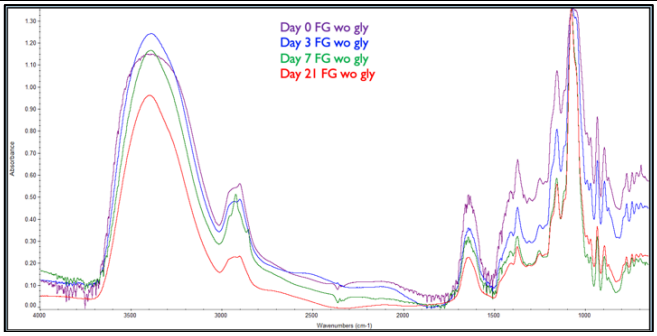
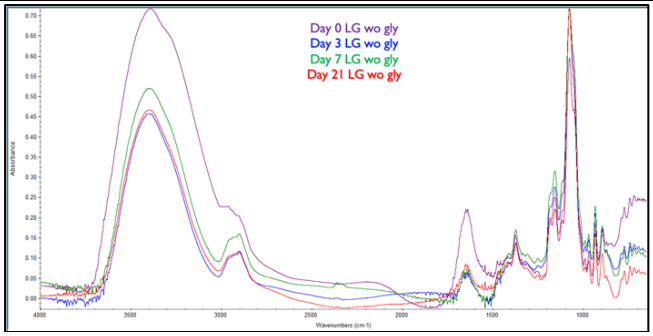


Figure 4. Graph of colorimetry data over the course of 21 days.

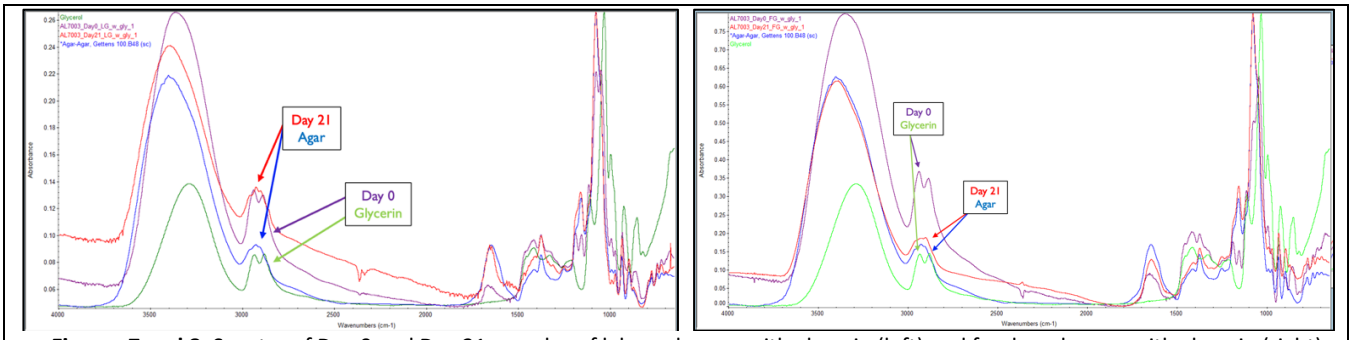
4.5 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR produced the results shown in Figures 5, 6, 7 and 8. The results did not vary between samples with glycerin and samples without, or between food and lab grade samples. The spectra in Figures 5 and 6 come from samples of agar without glycerin from days 0, 3, 7, and 21 for each grade of agar. The spectra across the various intervals look the same, there are no new peaks and no peaks have disappeared. Figures 7 and 8 show spectra from day 0 and day 21 samples of lab and food grade agar with glycerin compared to the reference spectra of glycerin and agar.





Figures 5 and 6. Overlaid FTIR spectra of food (left) and lab (right) grade agar samples from days 0, 3, 7 and 21.



Figures 7 and 8. Spectra of Day 0 and Day 21 samples of lab grade agar with glycerin (left) and food grade agar with glycerin (right), compared to reference spectra of glycerin and agar.

4.6 Pyrolysis- Gas Chromatography - Mass Spectrometry (py-GC-MS)

Py-GCMS did not produce any meaningful results. The pyrogram (Figure 9) shows peaks due to column bleed; the major ion in the mass spectra of the peak is m/z=207 of siloxanes from the column being released due to degradation from age and heating of the GC oven.

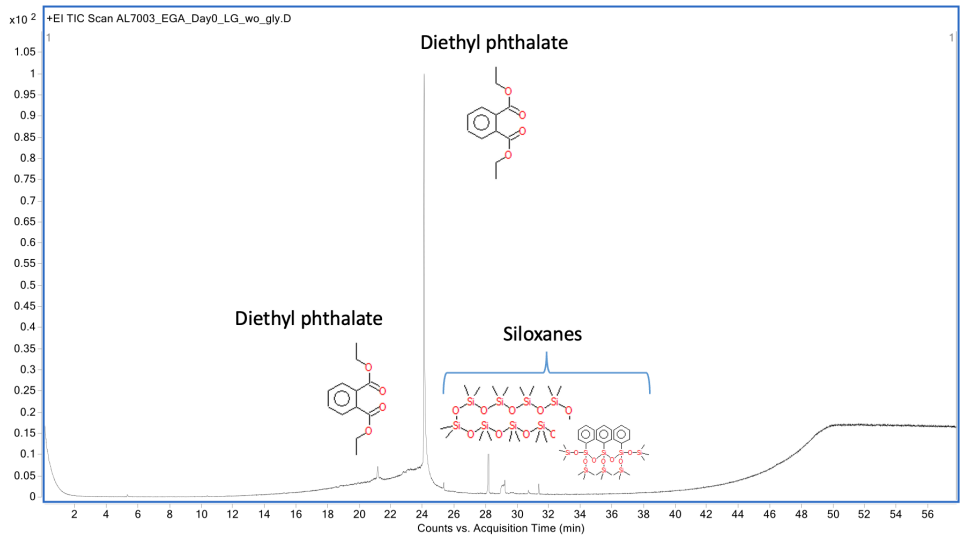


Figure 9. GC-MS pyrogram showing peaks due to column bleed and environmental contamination of plasticizer, not detection of agar sample.

5 Discussion

Agar clearly undergoes a color change upon prolonged exposure to elevated temperature and relative humidity (Figure 1). The color change or browning exhibited by the samples is likely due to caramelization of the polysaccharides which occurs at high temperatures. Based on the colorimetry measurements taken and the overall color change calculated (ΔE), the agar samples experienced a detectable and quantifiable color change (Table 3). The ΔL* values indicate the samples became darker, the Δa* values indicate the samples became redder and the Δb* values indicate the samples became bluer. Color change steadily increased over time with increased exposure to high relative humidity and temperature (Figure 4). There were no sudden jumps or significant plateaus in the rate of change over the course of the 21-day study.

All samples shrank upon aging but to varying degrees (Figure 2). Shrinkage across all samples was to be expected as agar is a water-based material, so exposure to extreme temperature would naturally drive off water present in the samples. The samples with glycerin shrank more than those without, though not significantly, and this change was not noticeable to the naked eye. It is likely that the samples with glycerin shrank more than those without as they contained more water to begin with and thus had more water to lose during aging. No consistent trends were found between the thicknesses of food and lab grade agar, suggesting that purity does not correspond to degree of shrinkage. Exposure to increased temperature and relative humidity caused visible dimensional changes to agar which was observed but not measured or evaluated analytically in this study (Figure 3).

Structural indicators of chemical change were not clearly detectable by FTIR. When overlaid, the spectra of samples taken from the various intervals all look very much the same, whether they are food or lab grade, or made with or without glycerin (Figures 5 and 6). A slight change in band shapes was observed; whoever, no additional peaks developed over the course of aging and no peaks disappeared from the spectra. While agar is undergoing an obvious physical change upon aging, there is no chemical change occurring, or at least not one readily detectable by FTIR. A small change was detectable by FTIR but only for samples containing glycerin. When comparing the spectra of both the lab and food grade agar with glycerin samples to the reference spectra of glycerin and agar we found that samples from Day 0 look similar to glycerin while samples from Day 21 look like agar. This suggests that while the agar in the samples did not degrade to a degree detectable by FTIR, the glycerin in the samples did as it was detectable at day 0, and not at day 21 (Figures 7 and 8). It is unclear why or how the glycerin is disappearing. The boiling point of glycerin is 85°C, which is higher than the temperatures reached during the experiment, so evaporation cannot explain the disappearance. An understanding of why the glycerin disappeared over the course of aging requires further research.

As illustrated by the GC-MS pyrogram, we were unable to detect any degradation products from the agar samples analyzed, and only detected compounds associated with column bleed and plasticizers present in the ambient environment (Figure 9). By using the parameters set forth by Ouyang et al (2018) we hoped to separate and detect the agar degradation products identified in the paper. We found this paper did not provide sufficient information regarding the separation methodology/parameters used so we attempted this using our standard method and were unsuccessful. While this is likely due to the method we used, it is also worth noting that agar requires exceedingly high temperatures to degrade which were not reached in this study, so it is likely that the agar would not degrade to a degree that could be detectable by GC-MS, even if we had the correct method. With the right methodology/parameters for separation, GC-MS would likely be the best option for detecting a chemical change in the agar samples.

Conclusion

Artificially aged agar was analyzed using visual observation, thickness measurements, colorimetry, FTIR and py-GCMS to understand its degradation and see if the physical and chemical changes it undergoes are detectable with analytical instrumentation. While results from observation, thickness measurements and colorimetry were conclusive, those from FTIR and GC-MS were not. While certain tools can be used to confirm changes observed visually through changes in measurement before and after aging, at this point we cannot state that degradation of agar is always detectable/identifiable by FTIR or GC-MS.

Agar experienced significant physical changes upon aging which were detectable with and without various types of instrumentation. The notable changes in color and thickness of agar caused by aging would be problematic for artworks made of the material, as well as if it were to be used as a conservation material on objects.

While this study confirmed that agar exhibits undesirable properties upon aging, we're left wondering how it compares to other materials used by artist and conservators. New and innovative materials like agar need to continue to be researched for the benefit of artists, conservators and collections care people charged with their preservation. The hope is that this study will be repeated with traditional as well as innovative materials being used in art and conservation.

Acknowledgments

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Appendix I – Additional Data

Additional FTIR spectra obtained during this study using parameters outlined in Methods section 3.5.

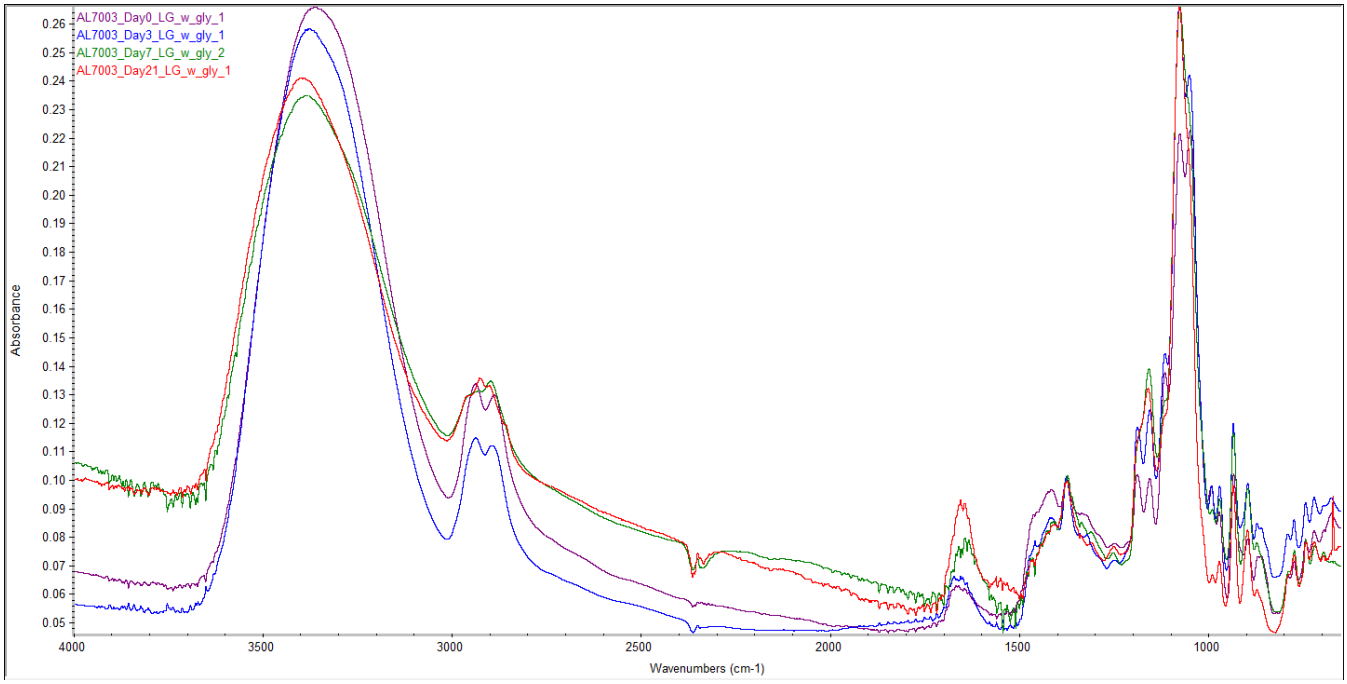

Figure 1. Overlaid spectra of lab grade agar with glycerin from days 0, 3, 7 and 21

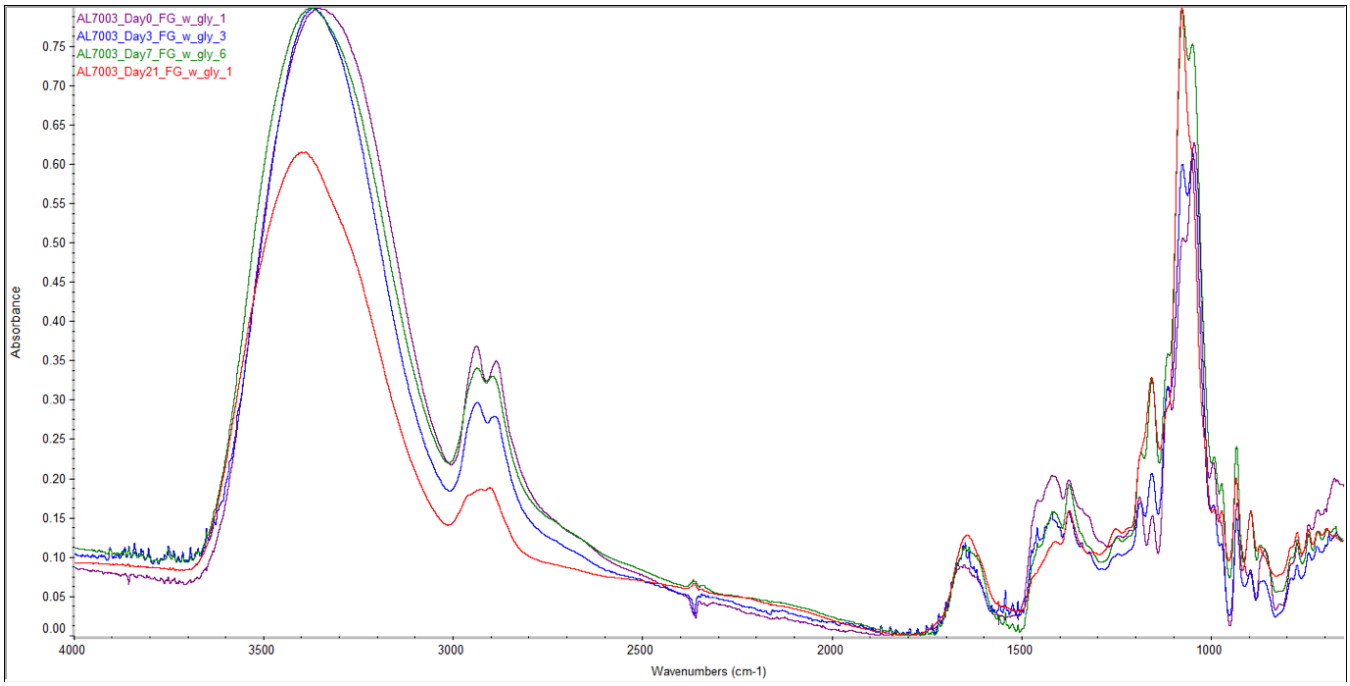

Figure 2. Overlaid spectra of food grade agar with glycerin from days 0, 3, 7 and 21

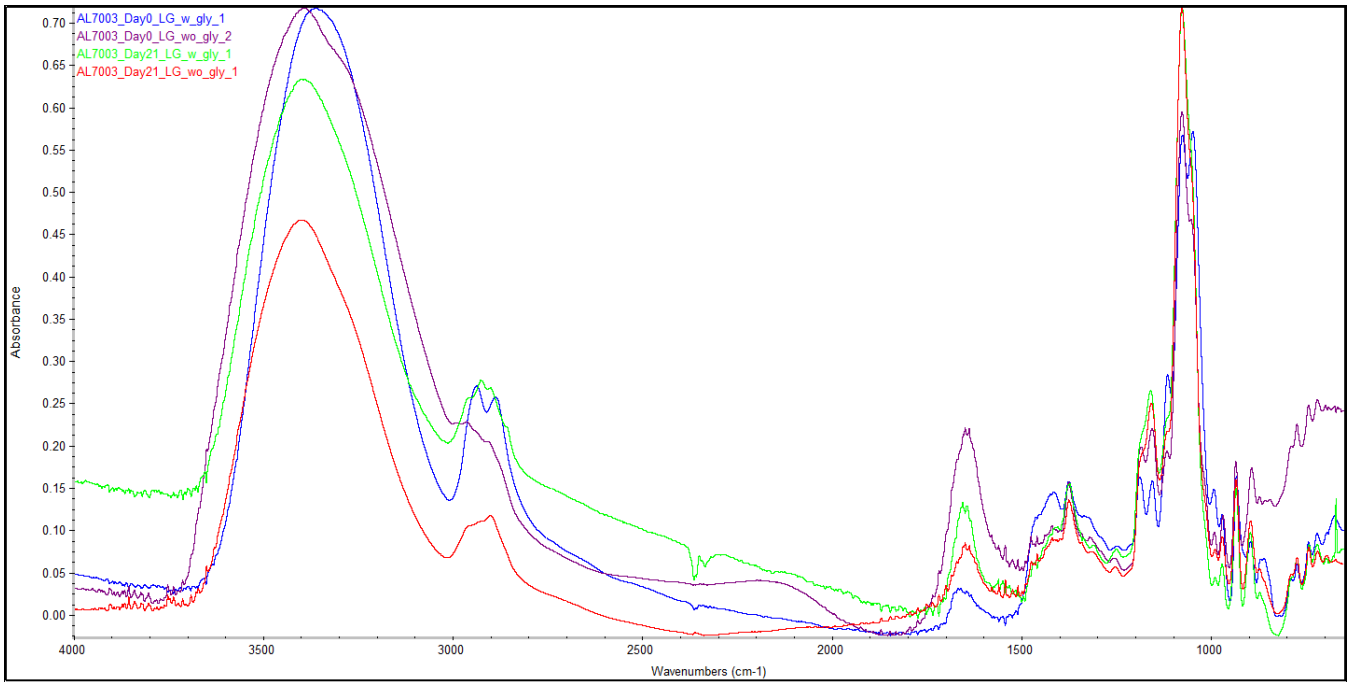

Figure 3. Overlaid spectra of lab grade agar samples with and without glycerin from days 0 and 21

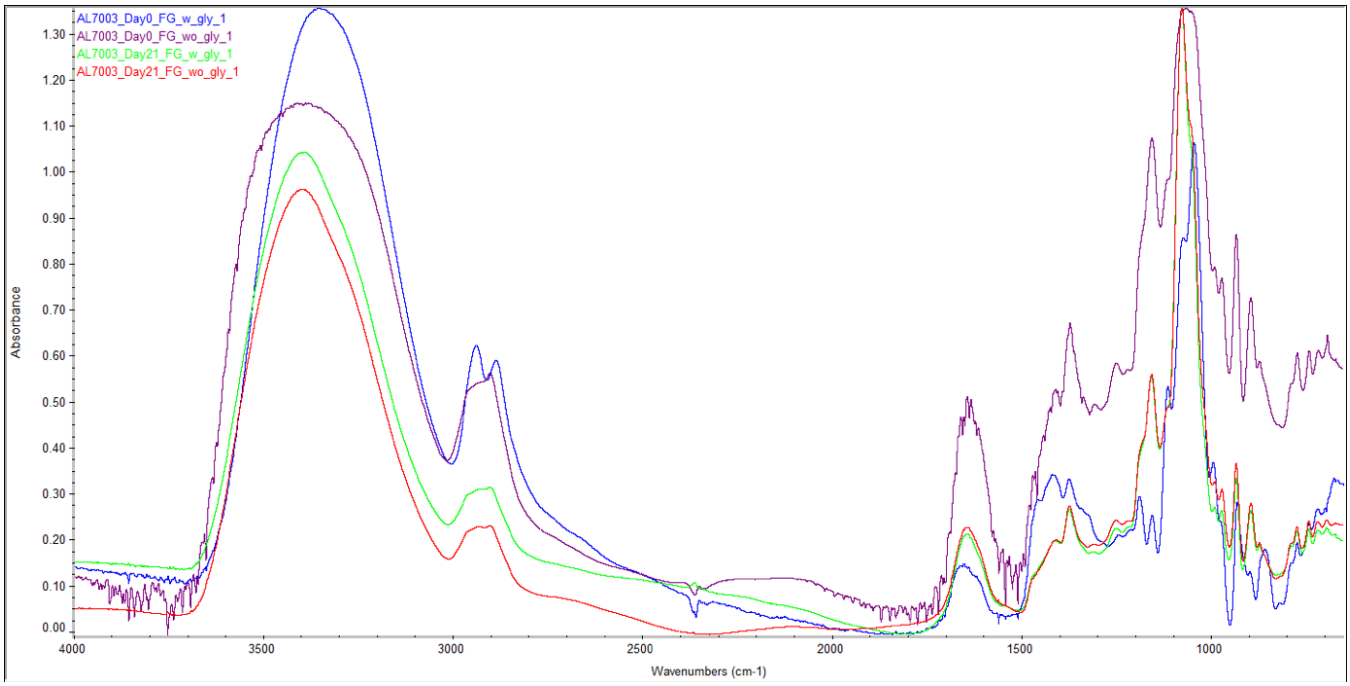

Figure 4. Overlaid spectra of food grade agar samples with and without glycerin from days 0 and 21