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PAINTINGS

Stress-strain behavior of gelatin, sturgeon glue, and methylcellulose at fluctuating relative humidity

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Abstract
In this study, films of three common materials used in conservation—gelatin, sturgeon glue, and methylcellulose—were evaluated for dimensional change after 10 humid climatic cycles (53% and 75% relative humidity) under low force (0.1 N). Gelatin and methylcellulose formed stable films that expanded by an equal ratio upon humidification and contracted after drying. Methylcellulose showed the least dimensional change. The behavior of the two hydrocolloids could be compared to that of elastomers, whereas a very strong plastic deformation of up to 16.5% was observed for sturgeon glue during humidification. The experiment showed that sturgeon glue behaves like a hygroscopic thermoplastic and that its intermolecular bonds have a potential for rupture. As the cohesive strength of sturgeon glue at increased humidity is thus limited, this material is unlikely to fulfill the basic requirements of an adhesive. An alternative suggested by this research is methylcellulose, which is aging-resistant and exhibits a more stable and uniform behavior.

INTRODUCTION

In conservation, humidity may strongly influence material properties in different ways. Karpowicz (1989) and Zumbühl (2003) have shown that films of mammalian-collagen-based glue continuously contract during humidity cycles. The induced stresses can lead to the cohesive peeling of paint and to the formation of microcracks (Vandivere et al. 2019). This paper compares sturgeon glue, which is frequently used in painting conservation (Ackroyd et al. 2002), with gelatin with respect to tension build-up. Both were also compared with methylcellulose (MC), as a standardized and sustainable alternative material less susceptible to mold (Sindlinger-Maushardt and Petersen 2007) and more resistant to aging (Feller and Wilt 1990, Steger et al. 2022), under conditions of fluctuating humidity.

The three adhesives, i.e., sturgeon glue, gelatin, and MC, differed in their origin, purity, and composition but all are biopolymers and hydrocolloids (Phillips and Williams 2009) and can form gels (Almdal et al. 1992). Collagen-based proteins have long been used in conservation (Wiemers 1983, Ackroyd et al. 2002, Bischoff 2004). Gelatins are a purified and thus more reproducible binding agent than other collagen-based adhesives. They are mainly produced from mammalian proteins (Schrieber and Gareis 2007), specifically, porcine (type A) and bovine (type B) sources, and largely consist of glycine, proline, and hydroxyproline (Schrieber and Gareis 2007). Among gelatins' properties of interest are their defined viscosities (e.g., 6.67% approx. 1.8–9 mPa·s), bloom grade, pH, and molecular weight distributions (Schrieber and Gareis 2007). In general, gelatins exhibit good film-forming properties (Karim and Bhat 2009, 571), depending on the amount of water present in the film (Yakimets et al. 2005, Avena-Bustillos et al. 2011), the polymer concentration (Eysturskarð et al. 2009), and the composition of the molecular chains (Eysturskarð et al. 2010). Film formation by mammalian and warm-water fish gelatins is dependent on the temperature and the cooling rate. The cooler the drying condition (< 40 °C), the more stable and elastic the resulting films (Chiou et al. 2009, 329). Slow-cooled gelation just below the gelation temperature favors the formation of a highly ordered, semi-crystalline network (Haupt 2000, 97) with triple helical junction zones (Gómez-Guillén et al. 2002, Chiou et al. 2009). This network structure leads to a highly cohesive material (Flock 2018, Bridarolli et al. 2022). Film formation as a renaturation process was described by Harrington and Rao (1970, 3723).

Sturgeon glue, which for decades has been widely used in conservation, is not a standardized raw material, as there are natural and chemical variations depending on the source as well as differences in production. Therefore, it should not be confused with cold- or warm-water fish gelatins. In addition, product declaration is often insufficient and individual processing by conservators adds to further variability. The proteins obtained from the swim bladders of cold-water fish differ from those of mammalian gelatins (Eastoe 1957, Veis 1964, Schrieber and Gareis 2007, Alfaro et al. 2015), as they contain lower amounts of proline and hydroxyproline. Since the latter amino acids are mainly responsible for helix formation (Leunenberger 1991, Hickman et al. 2000, Bigi et al. 2004, Haug et al. 2004, Hao et al. 2009, Zhang et al. 2014), renaturation is reduced during film formation using sturgeon glue, resulting in a gel stability lower than that obtained with the same concentration of mammalian gelatin (Haug et al. 2004, Ninan et al. 2010, Zhang et al. 2014). Furthermore, the Bloom grade of cold-water fish proteins is less than 100 and no Bloom grade can be specified for sturgeon glue (Haupt 2000). The different mechanisms of film formation are an important determinant of the cohesive stability of the individual materials. However, in the commercial processing of sturgeon glue, the production conditions often are not specified and specifications regarding the origin of the source material vary considerably (Flock 2018, Soppa 2018, Bridarolli et al. 2022). The products may also contain various minor components with potential impact. For example, water-insoluble polymer elastin may account for ~1% of the product (Young and Lorimer 1961, Hickman et al. 2000, Florian 2007) and the presence of water-insoluble fatty residues (Hems and Curtis 2015) may reduce the cohesive properties of the film (Norris and McGraw 1964, Haug et al. 2004, Diaz et al. 2011). Finally, additional variability arises because glues are often individually prepared by conservators themselves, with differences in the extraction time and heat potentially resulting in different qualities of the final product (Haupt 2000).

The use of MCs is well established in paper conservation but much less common in painting conservation (Ackroyd et al. 2002), although the potential of MCs in conservation is receiving increasing attention. Several examples of MC applications have been published over the last several decades (Bauer and Koller 1965, Brachert 1965, Bosshard Van der Brüggen and Bosshard Van der Brüggen 1974, Baker 1984, Oess 1995, Döll 1997, Wuntschek 1997, Mercier 2003, Hoppmann and Schubert 2005, Sindlinger-Maushardt and Petersen 2007, Genton 2014, Soppa and Léchenne 2017, Gaasch 2019, Soppa et al. 2020, Murgia and Soppa 2022, Soppa et al. 2022). MCs are a simply structured polysaccharide based on glucose with different degrees of polymerization and substitutions. They contain segments with increased methylated hydroxyl groups (R-O-CH_3) and with a larger number of OH groups (Tavera Quiroz et al. 2013, 2918). The degree of substitution in commercial MC is typically 1.7–2.2, producing a semiflexible polymer that is water soluble at low temperatures (Coughlin et al. 2021). MCs have unique film-forming properties, with more highly methylated segments responsible for the self-assembly of the polymer chains. Unlike proteins, MCs form a strong gel at temperatures above 50 °C. The sol-gel transition occurs upon heating and continues to be a subject of intense research

(Schmidt et al. 2020, Coughlin et al. 2021, Niemczyk-Soczynska et al. 2022). During this transition, multiple polymer strands combine to form a stable network made up of fibrils with a diameter of around 15–20 nm and a length depending on the degree of polymerization.

Gelatin, sturgeon glue, and MCs have high cohesion at 50% relative humidity (RH) but lose strength at higher humidity (Debeaufort and Voilley 1997, Bridarolli et al. 2022). From a conservation point of view, a highly relevant question, explored in this study, is how does the behavior of the three hydrocolloids compare under conditions of fluctuating relative humidity?

MATERIALS AND METHODS

The effects of the origin and composition on the hygric properties of gelatin, sturgeon glue, and MC were investigated by preparing films of each one that were then tested during 10-day sessions. Strain tests at constant stress load over 10 repetitive climate cycles in 10 days (i.e., 1 cycle per day) were performed according to Zumbühl (2003) under identical conditions for each film type. The following adhesives were tested: (i) as a standard gelatin, a medium Bloom (180 Bloom, type A) porcine gelatin (Roth, art. no. 4274.1), based on its broader range of molecular chain lengths (Schrieber and Gareis 2007) and thus a higher similarity to sturgeon glue (Haupt 2000, Geißinger and Krekel 2007) than type B (bovine) gelatins; (ii) sturgeon glue, prepared as described below from sturgeon bladder (*Acipenser baerii* Brandt) and purchased from Störleim-Manufaktur (http://www.stoerleim-manufaktur.de); (iii) as a MC, cellulose ether A15LV (batch no. F293I2K001, Dupont), chosen in order to obtain a concentration and film thickness comparable to that of the protein adhesives.

All aqueous adhesive solutions were prepared as concentrations of 12% by weight in demineralized water. The gelatin solution was prepared by swelling the grains for 2 h until they were transparent and then dissolving them at a temperature of 55–65 °C for about 15 min. Sturgeon glue was prepared by 12 h of swelling, followed by 2 h of heating at 68 °C and filtration. The sturgeon glue film was heated only briefly, until it dissolved (40–50 °C). MC was prepared according to the hot/cold method (https://www.youtube.com/watch?v=0XsMNdYF7iY) and left overnight at 4 °C (Soppa 2018).

Adhesive films were produced using a film applicator (Erichson, width: 13 mm; wet film thickness: 300 µm) on a rigid Melinex foil. MC was applied at room temperature. Since slow gelation just below the gelation temperature favors the formation of a highly ordered, semi-crystalline network (Haupt 2000, 97; Gómez-Guillén et al. 2002; Chiou et al. 2009), the protein glues were cooled slowly before they were applied with the applicator. The films were dried for 14 days at 20 ± 1 °C and 50 ± 3% RH. The film thicknesses are summarized in Table 1.

The mechanical properties of the films were measured in a specially manufactured PMMA climate chamber. The mechanical tests were performed using a tensile testing machine (Zwick 1120, test expert) at a constant force of 0.1 N. Following preconditioning of the films at 53% RH (saturated salt

solution, magnesium nitrate, $Mg(NO_3)_2$ 6 H_2O), 10 climate cycles were run. Each climate cycle lasted 24 h and consisted of humidification for 9 h at 75% RH using a saturated NaCl solution followed by drying for 15 h at 53% RH, at a constant temperature of 21 ± 1 °C. The measurement interval was 6 min, unless movement was registered by the measuring instrument. A data logger with a measurement interval of 2 min was placed in the climate chamber. A minimum of two 10-day experiments were run per adhesive. A third replicate was run to confirm the results in the case of heterogeneous materials. Deformation of the binder films was documented photographically after the climate cycles.

Table 1. The thickness and width of the adhesive films

Adhesive	Thickness in µm	Width in mm	A in mm²
Gelatin	20.2 ± 0.6	14.3 ± 0.4	0.2860
MC	17.5 ± 0.5	13.3 ± 0.1	0.2328
S-glue1	20.3 ± 1	14.9 ± 0.15	0.3025
S-glue2, 3	18 ± 1	16.7 ± 0.1	0.3006

RESULTS

The variations in volume and mechanical properties under changing climatic conditions are important parameters in the evaluation of an adhesive or consolidant. In this context, the tested binding media show characteristic properties. During humidification, these materials expand due to their rapid swelling, and during drying the films contract again due to the desorption of water. A general observation in this study, however, was that, after the first cycle, the length of the film was no longer the same, due to internal structural changes. This observation suggested two processes in conflict: If the film was plasticized by water absorption, it irreversibly stretched. If, on the other hand, a reorganization of the molecular structure occurred during the humid cycle, due to an improved molecular mobility and structural organization, then a smaller film dimension resulted after drying. In this study, the final assessment was based on the overall result upon completion of the 10 cycles over the 10-day experiment and whether a trend could be identified.

Gelatin and MC appeared to expand and shrink equally and regularly, except during the first cycle (Figure 1). Following the first three cycles, the dimensional fluctuation leveled off. Thereafter, the dimensional change during water absorption and desorption within a climatic cycle was almost in equilibrium. This behavior corresponded to the properties required of an ideal adhesive. The sorption capacity and thus the swelling rate varied between the different materials. Our data are consistent with those obtained in published studies of water vapor permeability (MC = 0.312 gmm/m2hkPa [Donhowe and Fennema 1993], warm-water fish gelatin = 1.31 gmm/m²hkPa, mammalian gelatin = 1.88 gmm/m²hkPa [Avena-Bustillos et al. 2011]). Both the MC and the gelatin films showed some shrinkage after 10 cycles (Tables 2, 3), by 0.35% and 1.2%, respectively. A similar behavior of gelatin was documented previously, such that our results are consistent with those reported by Zumbühl (2003) and Karpowicz (1989). The behavior of sturgeon glue, on the other hand, was highly plastic even at

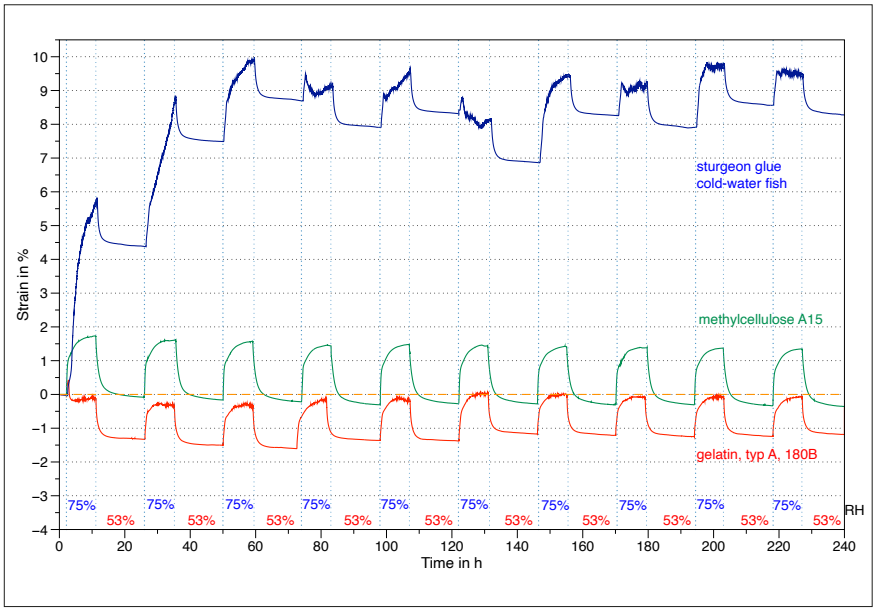

Figure 1. Strain-stress graph for gelatin (red), methylcellulose (green), and sturgeon glue (blue) after 10 cycles of 9 h humidification (75% RH) and 15 h of drying (53% RH)

Table 2. Relaxation or shrinkage after 9 h humidification (in %) during each of the 10 cycles

Adhesive	0	1	2	3	4	5	6	7	8	9	10
Gelatin	0	−0.13	1.0	1.17	1.46	1.2	1.43	1.27	1.29	1.31	1.31
MC	0	1.73	1.7	1.75	1.68	1.78	1.72	1.69	1.7	1.69	1.69
S-glue1	0	5.5	4.42	2.41	0.43	1.78	−0.12	2.64	1.04	1.89	0.94
S-glue2	0	8.2	−0.52	−1.7	1.14	0.46	1.96	6.02	1.76	0.04	−2.94
S-glue3	0	16.5	-	-	-	-	-	-	-	-	-

Table 3. Shrinkage (in %) after 15 h of drying during each of the 10 cycles

Adhesive	0	1	2	3	4	5	6	7	8	9	10
Gelatin	0	−1.2	−1.18	−1.27	−1.21	−1.22	−1.23	−1.3	−1.32	−1.3	−1.25
MC	0	−1.81	−1.79	−1.8	−1.76	−1.76	−1.72	−1.72	−1.7	−1.72	−1.71
S-glue1	0	−1.12	−1.31	−1.21	−1.22	−1.31	−1.32	−1.24	−1.39	−1.24	−1.26
S-glue2	0	−1.38	−1.44	−1.44	−1.58	−1.58	−1.28	−1.46	−1.54	−2.0	−1.3
S-glue3	0	-	-	-	-	-	-	-	-	-	-

a very low strain of 0.1 N, whereas replicates of the sturgeon glue films showed varying and inconsistent properties. The first sturgeon glue film (S-glue1) underwent strong deformation, up to 10%, within three humidity cycles. Thereafter, the dimensional change leveled off at around 8%, with shrinkage and expansion phases appearing across cycles. The effect within individual cycles was erratic. A systematic behavior was also not discernible in the second sturgeon glue film (S-glue2) while after 3 h of humidification the third film stretched by 16% and after another 5 h it ruptured at 16.5% elongation.

The behavior of the three materials after 9 h of humidification was examined in greater detail (Table 2). In the case of gelatin, a maximum value of 1.3% was reached on average after 4 h of humidification. The absorption rate of MC was slower (~ 6–7 h) and averaged at 1.75%. Sturgeon glue films, however, exhibited a highly variable behavior with inconsistent results, as the three replicates reacted strongly and unpredictably (Tables 2, 3).

DISCUSSION

The material properties of adhesives are largely determined by their drying-related structures. During gelatin film formation, a stable network is formed in which the amorphous polymer chains are held together by highly ordered crystalline junction zones. The renatured domains are stable at room temperature. At high humidity, however, the molecular mobility allows the crystalline triple helixes to continue to grow, so that the material becomes increasingly crystalline (Zumbühl 2003), until, in the end, a very hard and brittle film is present. In the case of MC, gelation is still being studied in detail (Schmidt et al. 2020, Coughlin et al. 2021, Niemczyk-Soczynska et al. 2022). Our findings suggest that, similar to gelatin, MC reorganized into a more ordered structure. In MC films, the stabilizing sites are assigned to fibrils with semicrystalline structures. However, the films tested here were not formed over the gel phase and thus did not have a high density of fully formed fibrils, although enough crystalline segments to stabilize the film and generate an elastomeric system were presumably present. The MC fibrils remained largely stable over the climate cycles, with a stability comparable to gelatin. The minimal shrinkage of the film over 10 climate cycles indicated an increasing organization of the fibrils. Similarities between gelatin and MC were also seen in the binder films after 10 climate cycles (Figure 2), as both films remained flat, with few changes observed.

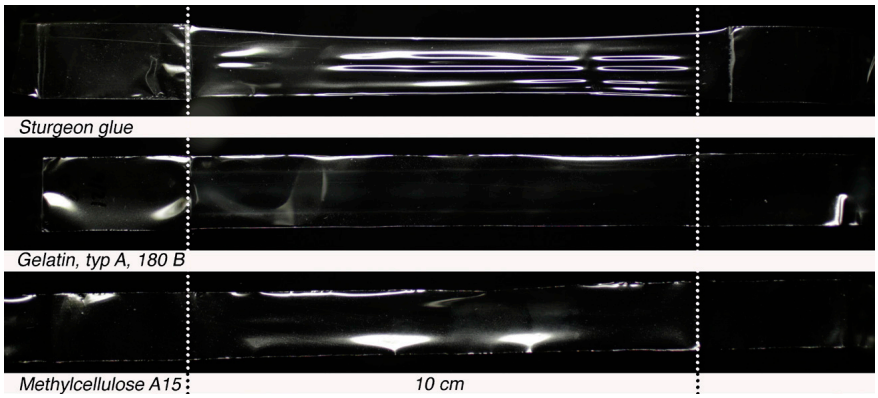

Figure 2. Sturgeon glue, gelatin, type A, 180 Bloom and methylcellulose A15 after 10 cycles consisting of 9 h of humidification (75% RH) and 15 h of drying (53% RH)

By contrast, network formation during the drying of sturgeon glue was not comparable to that of gelatin. This can be attributed to the different amino acid sequences, such that helix formation in sturgeon glue is strongly limited and stabilizing crystallites are formed only to a limited extent at room temperature. Due to its mainly amorphous structure, sturgeon glue can thus be described as a thermoplastic structure with physically entangled polymer chains. At high humidity, the polymer chains can readily slide past each other, so that the plasticized material can stretch many times. While there may be sites that suggest renaturation upon humidification, these are not stable enough to withstand re-humidification, thus resulting in sequential as well as irreversible deformation (Figure 1). This was observed in the longitudinally and transversely warped sturgeon glue film (Figure 2).

The different origins of fish gelatins strongly influence the properties of the respective films. The gelatins of cold-water fish have significantly less proline and hydroxyproline than mammalian gelatins, and those of warm-water fish similar proportions of these amino acids (Gilsenan and Ross-Murphy 2000, Avena-Bustillos et al. 2011). Thus, like mammalian gelatins, gelatins of warm-water fish are able to form a stable film network with crystallites (Soppa et al. 2023). The gel strength of warm-water fish gelatin ranges from 230 up to 460 Bloom (Kasankala et al. 2007, Akita et al. 2020). Warm-water fish gelatin is suitable for bonding, while cold-water fish gelatin (the purified counterpart of sturgeon glue) is preferred for the consolidation of powdery paint. These differences evidence the importance of providing information on the fish species in sturgeon glues used by conservators, as film formation behavior will greatly impact the applicability of these materials. Unfortunately, this information is often lacking in the data sheets.

CONCLUSION

The adhesives investigated show very different film properties related to the film formation process during drying as well as distinct behaviors under varying climate conditions. The results of our study showed that MC and gelatin behave like elastomers, a property that recommends their use as adhesives in conservation. Sturgeon glue, however, undergoes very strong plastic deformation, up to 16%, during humidification and high reactivity during climate cycles, even at a high concentration of 12% and a very low strain load of 0.1N/≈0.3 mm^2. Since sturgeon is a cold-water fish, its glue has fewer proline and hydroxyproline amino acids than mammalian gelatin, resulting in weaker cohesion for the same chain length. Nevertheless, the enormous deformation was surprising. The irregularity observed within the moisture cycles highlighted the heterogeneity and weak cohesion of sturgeon glue.

These findings are a cause for concern, considering the large number of heritage objects treated with sturgeon glue and stored in unstable climate conditions. While MC is often assessed negatively based on fears that it loses significant cohesion at high moisture levels, according to our results this is not the case. In fact, the material properties of MC make it an ideal adhesive for conservation, since, among other advantages, such as product standardization and content declaration, this binder exhibits stable, uniform strain and shrinkage behavior. The applicability of MC should thus be optimized, and its long-term behavior at other degrees of polymerization, concentration, and drying at fluctuating relative humidity should be further investigated to increase its acceptance by conservators. Overall, as the goal is to achieve sustainable conservation measures without inducing mechanical stress to the original substance, a more predictable, slowly reacting, and chemically stable glue able to replace sturgeon glue is desirable. MC is a good adhesive for many applications. However, the high viscosity of the solution can be a disadvantage in some situations. In this case, cold-water fish gelatin is a good alternative to sturgeon glue.

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