

## Hygric properties of gelatine-based adhesives

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**Abstract:** *Hygric Properties of Gelatine-Based Adhesives.* Studies about wood adhesives have been carried out for several years in the Institute for Building Materials ETH Zürich. Several investigations about adhesives that have been published are [2, 3, 4, 5, 6]. In this work, three types of gelatine-based adhesives (animal glues) which are hide, bone and fish glue were investigated. As a comparator, one type of synthetic adhesive one-component moisture-curing polyurethane adhesive (1C-PUR) was investigated simultaneously. The focuses of this work are on moisture transport and sorption properties of the adhesives. Therefore, two experimental tests diffusion test and sorption test were performed. In the results from diffusion test, it is found that animal glues have a very less vapor resistance against moisture. Which means that water can easily diffuse through the animal glues. The results of sorption test shows a higher moisture content inside animal glues compare to synthetic glue. It recorded for a very wet condition (20°C/95% RH) the moisture content reach up to approximately 60% for animal glues and only 3% for synthetic glue.

*Keywords:* wood adhesive, gelatine-based adhesive, animal glue, moisture transport, sorption properties.

### INTRODUCTIONS

In this work, the investigations are carried out in the field of wood adhesives. This is a part of bigger project focused on the studies about wood for cultural heritage objects. Based on the following project, the wood adhesives, such as hide glue, bone glue and fish glue are studied. All of these adhesives are animal glues, gelatine-based adhesives and they are usually used for wooden cultural heritage objects. Although, several studies about wood adhesives are already available in the literature, but most of them are only focus for synthetic adhesives [2, 4, 5, 6]. Only a few studies have ever been published for the gelatine-based adhesives [3]. Therefore, the properties of gelatine-based adhesives are not yet well known and not yet well documented. This work is expected could be able to provide a further knowledge about the gelatine-based adhesives.

The objects using gelatine-based adhesives, such as music instrumental objects and old cupboards usually stored in the museum without a controlled climate condition. In the museum, the materials are exposed to a range of unstable moisture condition. Based on data recorded by Musée d'ethnographie de Genève, during a rainy season the relative humidity of the room could increase up to 70%, but during the dry season, the humidity decrease until 15%. This range of humidity will affect the moisture content inside the material and it will also changes the properties of the material. This condition could effect for both the woods and the glues. This study is taken into account the moisture transport and sorption properties inside the adhesives.

### MATERIALS

As it is mentioned before, the current work is focusing on an investigation for animal glues. Three type of animal glues that are used in this work are hide glues (dry cubes, Kremer Pigmente), bone glue (dry pearls, Kremer Pigmente) and fish glue (liquid, Kremer Pigmente). They are well known for their traditional uses. The resulting bound could be really strong when they are used properly. However, they have a thermal and moisture limitation. Under a high temperature or moisture, the resulting joint made by using animal glues could be easily

break. In the other hand, this property of the material could be very useful. A joint can be easily separated by applying heat or moisture and mistakes could be corrected.

Animal glues like hide and bone glues are usually sold in dry form. In this condition, the animal glues have an unlimited shelf time. The dried glue should be dissolved into warm water first before using it. Once the glues dissolve, they are not storable anymore. In liquid condition they are very vulnerable to microorganisms, such as fungus, etc. The animal glues also stink. Nevertheless, because they are natural materials, they are non-toxic and environmental friendly materials. Moreover, a little practice is needed to be able to work properly with animal glues. They have a very short open time, the amount of time the glue remains liquid and workable. Animal glues' open time is usually less than a minute. This makes it quite difficult to work with. Heating the pieces to be glued and gluing in a warm room might also be another option to prolong the open time.

On the other hand, the synthetic glue 1C-PUR glue sold in a ready to use form. The 1C-PUR used in this work is 1C-PUR L1275.000 Geislich Ligamenta. This synthetic glue does not have thermal and moisture limitation, It can be used in almost all conditions.

## TESTS AND RESULTS

Two tests have been done to understand the hygric properties of the gelatine-based adhesives. These two tests are diffusion test and sorption test. To performed the test, the adhesive films of the glues as the test's samples were produced. For producing the films, the glues were casted on a flat plastic foil with a certain thickness. After a certain time, the glues will be fully cured (completely dry) and form a thin layer of films. At this rate, they are ready to use and can be further cut into the shape needed for the tests. The resulting dry thickness of adhesive films from each type of the glue is measured approximately 0.1 mm. The films will be further cut into a certain shape depending on the test purpose. For diffusion test, the films are cut into circular shape with diameter 60mm. Whereas for sorption test, no special shape is needed. In the following part, each test will be discuss in detail.

### Diffusion Test

The diffusion test procedure is performed according to [1]. Based on the norm, two test methods were performed, wet and dry cup method in normal climatic condition 20°C/65%. To ensure the reliability of the results, five samples were tested for each test method. The main different of both methods is the cups were filled with water in the wet cup test to simulate the moisture transport from the cup through the adhesive. Whereas in the dry cup method, the cups were filled with silica gels to simulate the moisture transport into the cup. For this test, the fully cured adhesive films were cut into a circular shape with diameter 60 mm. To strengthen the edges of the films, rings made from acrylic glass with inner diameter 52 mm, outer diameter 60 mm and thickness of 5 mm were glued in the both size of the adhesives films. To start the test, the adhesive films clamped with rings on both sides were place to cover the mouth of every cup. Then, a special rubber band was used to seal the gap between the cups and the ring, this will ensure that the moisture can only transport through the adhesive films. Gradually the mass of each cup is recorded. Therefore, mass changed per time of each sample is recorded. Moreover, the diffusion parameters can be calculated. The diffusion coefficient and the water vapor resistance factor of the adhesives as the results from this test are shown in table 1.

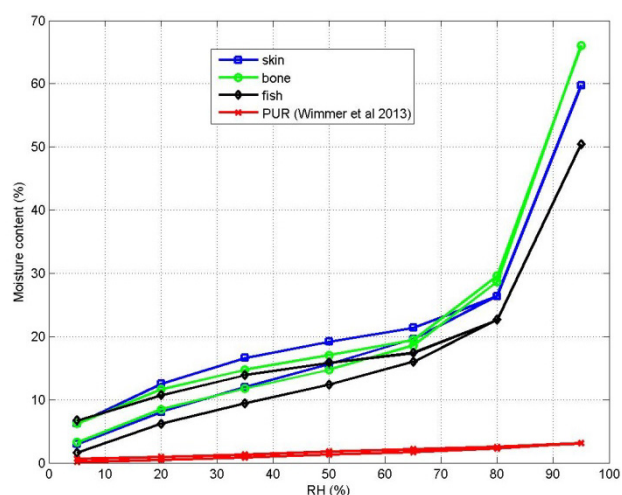
**Table 1.** Diffusion parameters

Adhesive	Dry Cup		Wet Cup	
	D [m <sup>2</sup> /s]	$\mu$ [-]	D [m <sup>2</sup> /s]	$\mu$ [-]
<b>Skin</b>	$2.388 \cdot 10^{-12}$	711.6	$1.292 \cdot 10^{-12}$	199.7
<b>Bone</b>	$1.449 \cdot 10^{-12}$	1134.6	$1.323 \cdot 10^{-12}$	154.7
<b>Fish</b>	$1.823 \cdot 10^{-12}$	1076.7	$1.785 \cdot 10^{-12}$	177.7
<b>PUR</b>	$2.859 \cdot 10^{-12}$	8497.8	$1.506 \cdot 10^{-12}$	6928.0

### Sorption Test

Sorption test is used to measure the moisture content inside the materials conditioned under a certain climate. In this work, a sorption isotherms are performed for each sample at 20°C. Sorption isotherms of the adhesive films was performed using DVS Advantages apparatus (EMPA, Dübendorf, CH). This machine could accurately determine the sorption at constant temperature and with a wide range of preset relative humidity (RH). The accuracy of the balance used during the test is up to  $5 \times 10^{-7}$  mg. For the test, any arbitrary shape of the

adhesives films could be used. The films broken into small pieces were placed inside the DVS chamber with the total mass approximately 20 mg. The test was begun by pre-drying the material (40°C) for 1 day. Then, the test was started from 20°C/5% RH and gradually increase with a step of 15% humidity until 20°C/95% RH (adsorption). After that, the RH was gradually decrease with a step of 15% humidity until reach back to 20°C/5% RH (desorption). In each step of humidity, DVS kept it until the material reach a constant mass (less than 0.001 % mass change in 30 minutes). The results are the moisture contents of the material at certain climate condition. The test results are shown in table 2. Furthermore, the results are plot in a graph (fig. 1) to easily see the comparison between each adhesive.



**Figure 1.** Sorption curves

**Table 2.** Moisture content of the adhesives

Adhesive		RH						
		5%	20%	35%	50%	65%	80%	95%
<b>Skin</b>	Adsorption [%]	2.95	8.02	11.98	15.63	19.55	26.39	59.78
	Desorption [%]	6.22	12.52	16.64	19.19	21.41	26.39	
<b>Bone</b>	Adsorption [%]	3.25	8.46	11.77	14.7	18.54	28.61	66.05
	Desorption [%]	6.2	11.66	14.77	17.03	19.49	29.69	
<b>Fish</b>	Adsorption [%]	1.6	6.17	9.42	12.39	15.96	22.69	50.39
	Desorption [%]	6.73	10.68	13.9	15.84	17.44	22.61	
<b>PUR [6]</b>	Adsorption [%]	0.12	0.48	0.83	1.32	1.67	2.28	3.14
	Desorption [%]	0.62	0.97	1.33	1.82	2.17	2.53	

## CONCLUSIONS

The water vapor resistance factor ( $\mu$ ) resulted from diffusion test shows that the animal glues have a very low resistance against moisture compare to synthetic glue especially for wet cup test. It is recorded that the  $\mu$  factor of animal glues in the dry cup test are approximately 10 times lower than synthetic glue. In the wet cup test the  $\mu$  factor of animal glues are drastically decrease up to 35 times lower than synthetic glue. During the wet cup method, the animal glues films are really soft indicating a high moisture content in the material and causing a rapid transport of the moisture. This results is supported by sorption test (table 2). It is shown that at 20°C/95% RH, the moisture content of animal glue are extremely high, it is recorded up to 66% for bone glued. In fact, it is impossible to maintain the dry form of the animal adhesive films conditioned in 20°C/95% RH. Since in this high humidity the films are really wet and started to become gels.

## OUTLOOKS

For further studies, mechanical test and creep test for identifying the material parameter under a short and long term loading of the adhesive should be performed. Furthermore to fulfill the main project, two types of wood will be further studies as well. The woods are cherry (*Prunus Avium L.*) and walnut (*Juglans Regia L.*), these are the woods that are usually used for wooden cultural heritage objects. In the end, the combination between woods and adhesive, such as glued joint or plywood will be experimentally tested as well. The studies will be also continued to a numerical modeling of the material. Numerical modeling is done using the material properties obtained from experimental test. Afterward, various things will be modeled numerically including the experimental tests themselves to validate the model.

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**Streszczenie:** *Właściwości higroskopijne klejów opartych na glutynie.* Praca obejmuje badania trzech typów kleju glutynowego – skórniego, kostnego i rybnego. Jako materiał porównawczy wybrano jednoskładnikowy klej poliuretanowy (1C-PUR). Badania skupiały się na przewodności wilgoci i własnościach sorpcyjnych klejów. PRzeprowadzono testy dyfuzji i sorpcji, wykazujące że kleje pochodzenia zwierzęcego stawiają bardzo niski opór przed penetracją pary wodnej - woda łatwo dyfuzuje przez te kleje. W bardzo wilgotnym środowisku (20°C/95% WW) wilgotność klejów glutynowych osiąga 60%, przy wilgotności kleju syntetycznego tylko 3%.

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