Supplementary Material

Stick to it! Mechanical performance tests to explore the resilience of prehistoric glues in hafting

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***Additional Information on “Materials and Method”.***

## **Glue Details**

Seven glue types were included in the study with one glue type (resin) being used in five differing mixtures. Eleven glue mixtures were thus tested in total (Table 1). Both vegetal-based and animal-based (protein) glues were tested in addition to one synthetic polymer. The selection of glues has no intention to be exhaustive, but we do aim to provide a representative sample of glues that could have existed in the past on the basis of available raw materials and technology. Mineral-based adhesives such as bitumen are excluded from the study given their restricted geographic occurrence. Also special forms of glue, such as those based on casein, blood or exotic fruits are not considered. Birch tar is excluded from this study as its production process and the exact parameters used can lead to an important variation in its physical properties with little internal comparability as demonstrated in previous tests (cf. Kozowyk et al., 2017a) .

The selected vegetal-based adhesives are a mix of rosin and beeswax (30% of rosin and 70% of beeswax), a mix of pine resin and beeswax (70% of resin and 30% of beeswax) and resin/beeswax mixtures with varying proportions of a filler (ochre or charcoal). Rosin used in this experiment was purchased and consists of a distilled resin stripped of its oils. All other vegetal mixtures were traditionally made by an experienced experimenter (Ch. Lepers, TraceoLab, University of Liège), with resin collected directly from pine trees and beeswax collected directly from a local beehive. The use of resin with beeswax has been demonstrated by its molecular identification in Palaeolithic hafting remains in Italy (Degano et al., 2019), while the addition of mineral additives and other loading agents has also been documented archaeologically (e.g. (Rots et al., 2011; Wadley, 2005) and ethnographically (intentional addition: e.g., Dickson, 1981; non-intentional addition: e.g., Rots and Williamson 2004), justifying their inclusion in this test. As little reliable data exist on the possible influence of the amount of fillers used in the mixtures, it was decided to integrate identical resin/beeswax mixtures with two different fillers, either ochre or charcoal, and to vary the quantity of filler added to the mixtures. As a result, mixture RBO1 consists of 33% pine resin, 33% beeswax and 33% ochre; RBO2 of 42.5% pine resin, 42.5% beeswax, 15% ochre; and RBO3 of 46.25% pine resin, 46.25% beeswax, 7.5% ochre. Mixture RBC contains 45% pine resin, 45% beeswax and 10% ground charcoal.

The selected protein glues are bone glue (bovine), rabbit hide glue, sinew glue (bovine) and fish skin glue. Gelatinous protein-based adhesives such as these derive from collagen through a process of denaturation by hot water extraction (Skeist, 1990). All protein glues used in this test were purchased (Maison Laverdure, Paris). To ensure adhesive properties, a low heating temperature is maintained both through gelatine production by the vendor and the reheating process during the experiments. Protein glues are relatively simple to produce and could have been manufactured during the Palaeolithic. However, no archaeological evidence older than the Neolithic has yet been documented. The oldest evidence to date proves to be of bovine origin and was identified at Nahal Hemar Cave (Israel) with a radiocarbon date of 8210–7300 cal. BC (Solazzo et al., 2016). In Western Europe, the oldest collagen-derived adhesive (hide glue) was found on a wooden bow from the 4th millennium BC (Zurich, Switzerland) (Bleicher et al., 2015). Both examples benefitted from excellent preservation conditions, as protein glues rapidly decay (Kozowyk et al., 2020)(Cnuts, 2021).

The tested synthetic polymer concerns a modern thermoplastic glue (Ferr-L-tite). We chose to test this glue, obviously not archaeologically compatible, in reaction to a recent publication (Wilson et al., 2021) comparing modern synthetic glue to “traditional” organic adhesives like hide glue and pine rosin and their respective failure rate in order to validate the use of modern glue in archaeological experimentation under certain circumstances. We thus integrated this modern glue to further test these claims, as similar claims have been frequently made in the past, though rarely truly validated.

**Table 1. Tested glue types and number of samples per contact material (RBC= resin-beeswax-charcoal; RBO =resin-beeswax-ochre).**

The glues were prepared by either hydration only (fish skin glue) or heating (other glues) to make them fluid enough to permit an application to the samples. The glues needing limited heating (sinew glue, hide glue, bone glue) were indirectly heated by placing the container in a water bath to avoid brutal temperature changes. Resins were gently heated on a stove, while Ferr-L-Tite was heated with a heat gun according to the advice of the manufacturer. Once in a liquid state, the glues were applied to join a flint blank to an organic material (“tester”) to reproduce the kind of contact experienced in hafting. Both components were glued together in a single lap joint.

1. **Sample production and details**

## **Lithic blanks**

An experienced knapper (Ch. Lepers, TraceoLab) pressure-knapped all blanks from fine-grained flint (Harmignies, Belgium). A single type of stone was selected in order to limit our variables. It cannot be excluded that a wider selection could lead to varying results. The samples were flaked rather than sawn to guarantee comparability with archaeological implements and to maintain structural properties of the surface. The straightest and flattest laminar blanks presenting a minimum of 12.5 mm width and 25mm length were selected for the experiment in order to ensure that the contact zone between the stone tool surface, the glue and the organic testers was as large as possible.

## **Organic testers**

Three organic materials commonly used as handles were opted for: dried pinewood, dried bone and dried antler. The testers present themselves as strips or tabs of wood, bone or antler. Bone and antler were sawn and roughly sanded to shape them and create a flat surface to receive the adhesive. Bone was glued on the compact or cortical bone part, while antler was glued on its medullar part. These three materials do not have the same roughness, which could influence the general resilience of the composite samples.

## **Composite (glued) samples**

Several identical composite sets were made in order to guarantee reproducibility and permit statistical analysis. Five identical samples were aimed for, with some exceptions in the case of breakage or inconsistent results. Indeed, some samples lead to very disparate results in which case more samples were added to allow further analysis. Other samples broke inappropriately but when remaining samples already showed very consistent results, an addition of new samples was not deemed necessary.

For each tester, the sample surface receiving a coat of glue has identical dimensions: 2.54cm in length, 1.27cm in width. The organic part of the testers is always 0.6 cm thick. Flint samples were produced so their dimensions would cover the entire area provided by the organic sample.

The glued samples were then left to dry for 24 hours indoors, while pressure was applied by clamps. This step intended to prevent samples from falling apart before the glue had dried. After the initial 24 hours drying period, if deemed necessary, we applied more glue on certain samples with visible gaps between their two constituting parts to ensure that the contact area was completely filled with glue and that the experiment would not suffer from an occasional irregularity in the flint implement. The samples were then clamped again and left to dry for another 24 hours.

# **EXPERIMENTAL SETUP**

## **Universal test bench and environment**

The experiments were conducted with a SHIMADZU (Autograph AG-S-X) test bench. All samples were tested with the same device at room temperature (i.e. 18°C). Consistency in temperature was considered essential as previous experiments had shown that the hardness of resin-beeswax mixtures may vary depending on the temperature with colder conditions increasing its brittleness and warmer conditions its flexibility (see also Coppe, 2020). The temperature was thus monitored and kept constant throughout the experiment in order to ensure comparability of the results.

## **Procedure**

Samples were unclamped one hour before bench testing. Some samples fell apart as soon as clamps were taken off and this glue thus proved unsuccessful. All other samples were subsequently mounted on the test bank. As mentioned, three possible forces can be considered in the context of tool use: flexion (e.g., scraping tools), shearing (e.g., knives) and impact (e.g. projectiles). Integrating all these forces in one study would necessitate a broad spectrum of tests with numerous samples. Therefore, the study was restricted to evaluating the performance of the glue types under shear-compression force as a first important step. Impact and flexion forces will be incorporated in follow-up studies.

All samples underwent a shear compression force until their glue broke. If one or both contact materials broke first, or if too much elasticity was observed as for example in the case of an insufficiently dried adhesive, the sample was rejected from the study as the force required to break the composite sample cannot be accurately determined. The rejected samples were recreated and tested once again in order to replace the missing data. The maximum force data were recorded by the test bench program Trapezium X in N (see Annex for settings) and later on recalculated to obtain a value in N/mm².

1. **RESULTS – General Table**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **ID** | **Haft material** | **Gluing Specifics** | **mineral substrate remains** | **organic substrate remains** | **Resilience (N per cm²)** |
| EXP114/167 | Antler | 70 % resin 30% wax | 1 | 3 | 74,43 |
| EXP114/168 | Antler | 70 % resin 30% wax | 2 | 2 | 0 |
| EXP114/169 | Bone | 70 % resin 30% wax | 1 | 3 | 101,67 |
| EXP114/170 | Bone | 70 % resin 30% wax | 1 | 3 | 115,43 |
| EXP114/171 | Bone | 70 % resin 30% wax | 1 | 3 | 67,83 |
| EXP114/172 | Wood | 70 % resin 30% wax | 2 | 2 | 74,24 |
| EXP114/177 | Wood | 70 % resin 30% wax | 1 | 3 | 101,76 |
| EXP114/178 | Wood | 70 % resin 30% wax | 1 | 3 | 24 |
| EXP114/179 | Wood | 70 % resin 30% wax | 1 | 3 | 48 |
| EXP114/180 | Wood | 70 % resin 30% wax | 1 | 3 | 54,4 |
| EXP114/181 | Wood | 70 % resin 30% wax | 1 | 3 | 98,24 |
| EXP114/41 | Bone | 70 % resin 30% wax | 2 | 2 | 181,23 |
| EXP114/43 | Bone | 70 % resin 30% wax | 1 | 3 | 172,67 |
| EXP114/44 | Bone | 70 % resin 30% wax | 1 | 3 | 200,64 |
| EXP114/71 | Antler | 70 % resin 30% wax | 1 | 3 | 175,67 |
| EXP114/74 | Antler | 70 % resin 30% wax | 1 | 3 | 129 |
| EXP114/75 | Antler | 70 % resin 30% wax | 1 | 3 | 189,57 |
| EXP114/151 | Antler | bone | 2 | 2 | 79,65 |
| EXP114/152 | Bone | bone | 2 | 2 | 90,88 |
| EXP114/153 | Wood | bone | 2 | 2 | 192,96 |
| EXP114/154 | Wood | bone | 2 | 2 | 142,4 |
| EXP114/155 | Wood | bone | 2 | 2 | 205,44 |
| EXP114/156 | Wood | bone | 2 | 2 | 48,96 |
| EXP114/30 | Wood | bone | 2 | 2 | 195,2 |
| EXP114/56 | Bone | bone | 2 | 2 | 77,67 |
| EXP114/57 | Bone | bone | 2 | 2 | 117,85 |
| EXP114/58 | Bone | bone | 1 | 3 | 112,96 |
| EXP114/59 | Bone | bone | 2 | 2 | 104,33 |
| EXP114/86 | Antler | bone | 1 | 3 | 260 |
| EXP114/87 | Antler | bone | 2 | 2 | 239,47 |
| EXP114/89 | Antler | bone | 2 | 2 | 268,74 |
| EXP114/90 | Antler | bone | 1 | 3 | 246 |
| EXP114/182 | Wood | Ferr-L-Tite | 1 | 3 | 16,32 |
| EXP114/183 | Wood | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/184 | Wood | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/185 | Wood | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/186 | Wood | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/187 | Bone | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/188 | Bone | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/189 | Bone | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/190 | Bone | Ferr-L-Tite | 1 | 3 | 50,56 |
| EXP114/191 | Bone | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/192 | antler | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/193 | antler | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/194 | antler | Ferr-L-Tite | 1 | 3 | 16 |
| EXP114/195 | antler | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/196 | antler | Ferr-L-Tite | 0 | 4 | 0 |
| EXP114/01 | Wood | Fish | 2 | 2 | 144 |
| EXP114/03 | Wood | Fish | 2 | 2 | 128 |
| EXP114/157 | Antler | Fish | 1 | 3 | 460,59 |
| EXP114/158 | Antler | Fish | 2 | 2 | 95,41 |
| EXP114/159 | Bone | Fish | 2 | 2 | 280 |
| EXP114/160 | Wood | Fish | 2 | 2 | 424 |
| EXP114/161 | Wood | Fish | 2 | 2 | 340,8 |
| EXP114/162 | Wood | Fish | 2 | 2 | 315,84 |
| EXP114/31 | Bone | Fish | 1 | 3 | 66,72 |
| EXP114/32 | Bone | Fish | 2 | 2 | 282,33 |
| EXP114/33 | Bone | Fish | 2 | 2 | 349,44 |
| EXP114/34 | Bone | Fish | 2 | 2 | 149,82 |
| EXP114/35 | Bone | Fish | 2 | 2 | 183 |
| EXP114/61 | Antler | Fish | 1 | 3 | 305,33 |
| EXP114/64 | Antler | Fish | 2 | 2 | 290,77 |
| EXP114/65 | Antler | Fish | 1 | 3 | 190,15 |
| EXP114/163 | Antler | Hide Glue (Rabbit) | 2 | 2 | 61,76 |
| EXP114/164 | Antler | Hide Glue (Rabbit) | 2 | 2 | 62,31 |
| EXP114/165 | Antler | Hide Glue (Rabbit) | 2 | 2 | 93,93 |
| EXP114/166 | Wood | Hide Glue (Rabbit) | 2 | 2 | 128 |
| EXP114/21 | Wood | Hide Glue (Rabbit) | 1 | 3 | 96 |
| EXP114/22 | Wood | Hide Glue (Rabbit) | 1 | 3 | 100,8 |
| EXP114/23 | Wood | Hide Glue (Rabbit) | 2 | 2 | 145,6 |
| EXP114/24 | Wood | Hide Glue (Rabbit) | 1 | 3 | 134,4 |
| EXP114/51 | Bone | Hide Glue (Rabbit) | 1 | 3 | 143,67 |
| EXP114/52 | Bone | Hide Glue (Rabbit) | 1 | 3 | 121,45 |
| EXP114/53 | Bone | Hide Glue (Rabbit) | 1 | 3 | 221,45 |
| EXP114/54 | Bone | Hide Glue (Rabbit) | 1 | 3 | 85,33 |
| EXP114/55 | Bone | Hide Glue (Rabbit) | 1 | 3 | 72,33 |
| EXP114/81 | Antler | Hide Glue (Rabbit) | 1 | 3 | 207,52 |
| EXP114/85 | Antler | Hide Glue (Rabbit) | 2 | 2 | 276,33 |
| EXP114/106 | Wood | Resin/charcoal | 2 | 2 | 96,96 |
| EXP114/107 | Wood | Resin/charcoal | 1 | 3 | 63,04 |
| EXP114/108 | Wood | Resin/charcoal | 2 | 2 | 41,28 |
| EXP114/109 | Wood | Resin/charcoal | 2 | 2 | 45,76 |
| EXP114/110 | Wood | Resin/charcoal | 3 | 1 | 56,96 |
| EXP114/126 | Bone | Resin/charcoal | 2 | 2 | 133,57 |
| EXP114/127 | Bone | Resin/charcoal | 1 | 3 | 76,16 |
| EXP114/128 | Bone | Resin/charcoal | 1 | 3 | 76,16 |
| EXP114/129 | Bone | Resin/charcoal | 1 | 3 | 133,57 |
| EXP114/130 | Bone | Resin/charcoal | 1 | 3 | 71,65 |
| EXP114/146 | Antler | Resin/charcoal | 1 | 3 | 128 |
| EXP114/147 | Antler | Resin/charcoal | 2 | 2 | 109,12 |
| EXP114/148 | Antler | Resin/charcoal | 1 | 3 | 111,41 |
| EXP114/149 | Antler | Resin/charcoal | 2 | 2 | 166,08 |
| EXP114/150 | Antler | Resin/charcoal | 2 | 2 | 141,91 |
| EXP114/111 | Bone | Resin/ochre 1 | 0 | 4 | 117,76 |
| EXP114/112 | Bone | Resin/ochre 1 | 1 | 3 | 76,8 |
| EXP114/113 | Bone | Resin/ochre 1 | 1 | 3 | 71,64 |
| EXP114/114 | Bone | Resin/ochre 1 | 2 | 2 | 107,69 |
| EXP114/115 | Bone | Resin/ochre 1 | 1 | 3 | 78,22 |
| EXP114/131 | Antler | Resin/ochre 1 | 1 | 3 | 70,59 |
| EXP114/132 | Antler | Resin/ochre 1 | 1 | 3 | 116,15 |
| EXP114/133 | Antler | Resin/ochre 1 | 0 | 4 | 139,2 |
| EXP114/134 | Antler | Resin/ochre 1 | 2 | 2 | 114,39 |
| EXP114/135 | Antler | Resin/ochre 1 | 1 | 3 | 95 |
| EXP114/91 | Wood | Resin/ochre 1 | 1 | 3 | 48 |
| EXP114/92 | Wood | Resin/ochre 1 | 1 | 3 | 89,92 |
| EXP114/93 | Wood | Resin/ochre 1 | 1 | 3 | 73,92 |
| EXP114/94 | Wood | Resin/ochre 1 | 1 | 3 | 40 |
| EXP114/95 | Wood | Resin/ochre 1 | 1 | 3 | 56,32 |
| EXP114/100 | Wood | Resin/ochre 2 | 2 | 2 | 112,64 |
| EXP114/116 | Bone | Resin/ochre 2 | 1 | 3 | 88,32 |
| EXP114/117 | Bone | Resin/ochre 2 | 1 | 3 | 145,26 |
| EXP114/118 | Bone | Resin/ochre 2 | 1 | 3 | 60,19 |
| EXP114/119 | Bone | Resin/ochre 2 | 1 | 3 | 98,08 |
| EXP114/120 | Bone | Resin/ochre 2 | 1 | 3 | 128,22 |
| EXP114/136 | Antler | Resin/ochre 2 | 1 | 3 | 88 |
| EXP114/137 | Antler | Resin/ochre 2 | 1 | 3 | 91,43 |
| EXP114/138 | Antler | Resin/ochre 2 | 0 | 4 | 79,03 |
| EXP114/139 | Antler | Resin/ochre 2 | 1 | 3 | 66,56 |
| EXP114/140 | Antler | Resin/ochre 2 | 0 | 4 | 52,44 |
| EXP114/96 | Wood | Resin/ochre 2 | 2 | 2 | 195,52 |
| EXP114/97 | Wood | Resin/ochre 2 | 3 | 1 | 190,08 |
| EXP114/98 | Wood | Resin/ochre 2 | 2 | 2 | 73,6 |
| EXP114/99 | Wood | Resin/ochre 2 | 1 | 3 | 105,6 |
| EXP114/101 | Wood | Resin/ochre 3 | 1 | 3 | 128,64 |
| EXP114/102 | Wood | Resin/ochre 3 | 1 | 3 | 139,84 |
| EXP114/103 | Wood | Resin/ochre 3 | 1 | 3 | 104,96 |
| EXP114/104 | Wood | Resin/ochre 3 | 3 | 1 | 183,36 |
| EXP114/105 | Wood | Resin/ochre 3 | 2 | 2 | 69,76 |
| EXP114/121 | Bone | Resin/ochre 3 | 2 | 2 | 88,64 |
| EXP114/122 | Bone | Resin/ochre 3 | 1 | 3 | 112,32 |
| EXP114/123 | Bone | Resin/ochre 3 | 1 | 3 | 155,13 |
| EXP114/124 | Bone | Resin/ochre 3 | 0 | 4 | 0 |
| EXP114/125 | Bone | Resin/ochre 3 | 1 | 3 | 117,44 |
| EXP114/141 | Antler | Resin/ochre 3 | 1 | 3 | 66,96 |
| EXP114/142 | Antler | Resin/ochre 3 | 1 | 3 | 113,08 |
| EXP114/143 | Antler | Resin/ochre 3 | 1 | 3 | 109,12 |
| EXP114/144 | Antler | Resin/ochre 3 | 1 | 3 | 95,04 |
| EXP114/145 | Antler | Resin/ochre 3 | 2 | 2 | 55,65 |
| EXP114/197 | Wood | Rosin (30%)+Beeswax (70%) | 1 | 3 | 144,96 |
| EXP114/198 | Wood | Rosin (30%)+Beeswax (70%) | 1 | 3 | 102,72 |
| EXP114/199 | Wood | Rosin (30%)+Beeswax (70%) | 1 | 3 | 100,16 |
| EXP114/200 | Wood | Rosin (30%)+Beeswax (70%) | 1 | 3 | 96 |
| EXP114/201 | Wood | Rosin (30%)+Beeswax (70%) | 1 | 3 | 114,88 |
| EXP114/202 | Antler | Rosin (30%)+Beeswax (70%) | 1 | 3 | 112,64 |
| EXP114/203 | Antler | Rosin (30%)+Beeswax (70%) | 1 | 3 | 172,16 |
| EXP114/204 | Antler | Rosin (30%)+Beeswax (70%) | 1 | 3 | 39,68 |
| EXP114/205 | Antler | Rosin (30%)+Beeswax (70%) | 2 | 2 | 72,64 |
| EXP114/206 | Antler | Rosin (30%)+Beeswax (70%) | 1 | 3 | 176 |
| EXP114/207 | Bone | Rosin (30%)+Beeswax (70%) | 2 | 2 | 93,44 |
| EXP114/208 | Bone | Rosin (30%)+Beeswax (70%) | 2 | 2 | 49,92 |
| EXP114/209 | Bone | Rosin (30%)+Beeswax (70%) | 1 | 3 | 87,68 |
| EXP114/210 | Bone | Rosin (30%)+Beeswax (70%) | 2 | 2 | 113,92 |
| EXP114/211 | Bone | Rosin (30%)+Beeswax (70%) | 1 | 3 | 80 |
| EXP114/173 | Wood | Sinew Glue | 1 | 3 | 44,48 |
| EXP114/174 | Wood | Sinew Glue | 2 | 2 | 56,96 |
| EXP114/175 | Wood | Sinew Glue | 2 | 2 | 153,6 |
| EXP114/176 | Wood | Sinew Glue | 1 | 3 | 54,4 |
| EXP114/36 | Bone | Sinew Glue | 1 | 3 | 76 |
| EXP114/37 | Bone | Sinew Glue | 2 | 2 | 68,67 |
| EXP114/38 | Bone | Sinew Glue | 1 | 3 | 111,04 |
| EXP114/39 | Bone | Sinew Glue | 1 | 3 | 115,69 |
| EXP114/40 | Bone | Sinew Glue | 1 | 3 | 113,23 |
| EXP114/66 | Antler | Sinew Glue | 1 | 3 | 104,31 |
| EXP114/67 | Antler | Sinew Glue | 1 | 3 | 69,45 |
| EXP114/68 | Antler | Sinew Glue | 1 | 3 | 163,38 |
| EXP114/69 | Antler | Sinew Glue | 1 | 3 | 196,33 |
| EXP114/70 | Antler | Sinew Glue | 1 | 3 | 50,78 |