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ORIGINAL ARTICLE

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

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Abstract
Known preserved glue remains are relatively rare, but they witness a broad range in glue types that may have existed in the past, varying in terms of the main component and additives. However, archaeological experimentation rarely mirrors that variety, leading to a certain lack of information on the behaviour and interest of several traditional glues, notably animal-based glues. Through mechanical bench testing (shear–compression), this paper aims to document the resilience of 11 different glues and mixtures in combination with three different organic substrates and flint, in order to reproduce a realistic hafting system. Results highlight that glue performance depends on the hafting materials used, thereby demonstrating that the choice of an appropriate glue depends on the hafting arrangement envisaged. The results also indicate that no glue outperforms all others or witnesses universal applicability. We therefore argue that glue use and performance should be evaluated from the perspective of the complete tool within a particular use context and environment. Only such an integrated perspective permits to reflect on the role of glue in past tool technologies, including its manufacture, composition and use, and on the significance of glue with regard to human behaviour and its evolution.

KEYWORDS
animal glue, additives, adhesive, bench test, experimental archaeology, glue, mechanical testing, ochre, resin

RESEARCH CONTEXT

Despite being rarely preserved for the Palaeolithic period, adhesives have been important in current debates on prehistoric technologies. A major trend in the recent literature deals with the use of glues, especially specific glue mixtures (i.e., resin with ochre) or birch tar, as a proxy to evaluate hominin cognition (Gowlett, 2016; Niekus et al., 2019; Wadley, 2010; Wadley et al., 2009), (See Schmidt et al., 2019, for counter-arguments). The addition of ochre to resin has been argued to be an intentional process to influence adhesive properties (Wadley et al., 2009), while birch tar production has been argued to be a demanding process requiring great control over hearth temperatures and a lot of investment for a rather limited amount of adhesive (Kozowyk, Soressi, Pomstra, & Langejans, 2017), though recent publications bring nuance to this claim (Koch & Schmidt, 2021). In addition, experiences gained from experiments performed up to now somewhat diverge in the evaluation of the complexity (and efficiency) of the production process (Cnuts et al., 2018; Schmidt et al., 2019), and one may question whether the discussion on what glue would be the most complex to produce is the most fruitful direction to take if one wants to understand the use of glue, its variation, the technical properties guiding its choice and its role in technological evolution.

Several experiments with glue have been performed, either with regard to glue used in the hafting of stone tools (e.g., Rots, 2010), glue production (Kozowyk, Soressi, Pomstra, & Langejans, 2017; Osipowicz, 2005; Wadley, 2005) or glue performance tests (Gaillard, 2016; Kozowyk, Poulis, & Langejans, 2017; Le Bouder & Yau, 2019; Wilson et al., 2021). In spite of that, several biases or problems can be identified with regard to the current body of available experimental data, part of which we intend to remedy in this study.

First, most experiments have made use of a limited range of glue types, being either those found archaeologically (birch tar, resin with differing mixtures) or modern glues, which has led to a general bias towards certain types of glue, though a broad range of potential glues exists and these are not only vegetal based, but may also be based on mineral or animal components.

Second, while mechanical performance tests have been performed in the past (Kozowyk, Poulis, & Langejans, 2017; Wadley, 2005; Wilson et al., 2021; Zipkin et al., 2014), these tests have again only concerned a rather narrow range of glue types and the results—while informative—cannot be transferred to archaeological contexts because they focused on joining one type of material only (wood glued on wood, flint glued on flint (Kozowyk, Poulis, & Langejans, 2017). The performance of glue when joining two identical and mostly organic materials (i.e. wood) cannot be directly compared to the prehistoric situation where stone tool hafting necessitates gluing a mineral component (flint, quartzite, quartz, etc.) to an organic one (e.g., handles). Not every glue can be expected to adhere as effectively to every contact material, and it is therefore important to evaluate the performance of a certain glue type in combination with a range of contact materials in order to make informed technical choices in experiments and try to understand past technical choices. This is particularly important in the case of projectile experiments, as the degree of adhesion will have a fundamental role in fracture formation (Coppe & Rots, 2017; Rots & Plisson, 2014).

Third, glues can be used for multiple purposes and the stress they are submitted to in use is not necessarily the same. This means that the functional context is an essential element when studying glues. Some glues show a strong resilience as a contact adhesive (being used in a single joint contact situation), while others only prove effective as a mastic, when used to seal an object in a groove or slot or protect bindings from moisture, for instance. Classifying adhesives into one of these two groups can be challenging. Some glues are well resistant to impact forces, while others are not, or their performance can be improved by adjusting the glue mixture. Beeswax, for instance, has often been used to make glues more pliable and resistant to shocks (e.g., Dickson, 1981). The production process itself may also influence the mechanical properties of glue: if resin is overheated, it loses its adhesive properties (a phenomenon that benefitted from an early description in the Natural History of Pliny the Elder) (Sherwood et al., 2020), while also the performance of birch tar is influenced greatly by the temperatures that it was exposed to during production (Niekus et al., 2019). Moreover, glues are not stable and their properties vary depending on the environmental conditions under which they are used. A certain glue or glue mixture that performs well in cold conditions may need to be reconsidered for a use in more temperate or warm conditions. We also observed this experimentally (see, e.g., Coppe, 2020).

Fourth, if one wants to consider appropriately the functional context of glues and the effectiveness of glue in tool use, one needs to test the resistance of different glue types with regard to a range of possible forces. 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).

In spite of past efforts, the current experimental basis for properly understanding prehistoric glue use is thus still rather weak and this study intends to further strengthen the experimental basis by filling a number of existing gaps identified above, with a particular focus on accommodating the bias towards resin and tar and on resolving part of the remaining problems in mechanical performance tests.

MATERIALS AND METHODS

Glues

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 is not 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, Poulis, & Langejans, 2017).

The selected vegetal-based adhesives are a mix of rosin and beeswax (30% rosin and 70% beeswax), a mix of pine resin and beeswax (70% resin and 30% beeswax) and resin/beeswax

TABLE 1 Tested glue types and number of samples per contact material

Glue type	Bone	Wood	Antler	TOTAL
Fish	6	5	5	16
Bone	5	5	5	15
Hide	5	5	5	15
Sinew	5	4	5	14
Resin–beeswax	6	6	5	17
RBC	5	5	5	15
RBO1	5	5	5	15
RBO2	5	5	5	15
RBO3	5	5	5	15
Rosin–beeswax	5	5	5	15
Ferr-L-Tite	5	5	5	15
TOTAL	57	55	55	167

RBC, resin–beeswax–charcoal; RBO, resin–beeswax–ochre.

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 & 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 consisted 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 contained 45% pine resin, 45% beeswax and 10% ground charcoal.

The selected protein glues were bone glue (bovine), rabbit hide glue, sinew glue (bovine) and fish skin glue (see Supporting Information for more details on glues and glued samples).

The tested synthetic polymer was 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.

Composite (glued) samples

Three organic materials commonly used as handles were chosen: dried pinewood, dried bone and dried antler. The testers were strips or tabs of wood, bone or antler. These were glued to laminar flint blanks. 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 (see Supporting Information for details).

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 therefore monitored and kept constant throughout the experiment in order to ensure comparability of the results.

Procedure

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 (Figure 1). The maximum force data were recorded by the test bench program Trapezium X in N (see Supporting Information for settings and further details on procedure) and later on recalculated to obtain a value in N/mm^2.

The degree of cohesion was evaluated once bench testing was complete on the basis of the broken sample. The amount of residual adhesive on both contact materials was scored using a point system, with a total of four points distributed between the mineral (flint) and organic contact material (wood, bone, antler) depending on the quantity of glue remains (Table 2).

RESULTS

During the tests, 29 out of a total of 196 samples broke inappropriately and could not be further analysed. These samples were either not perfectly secured in the test bank due to which they broke under flexion stress instead of shear–compression, or the tester materials broke before the glue could break. These unsuccessful samples were reproduced in all but one case scenario: when results within the batch of samples was already highly consistent (e.g., sinew glue/wood batch); unsuccessful samples were not replaced. The new samples received a different inventory number from the original sample, but for the sake of simplicity the original, unsuccessful

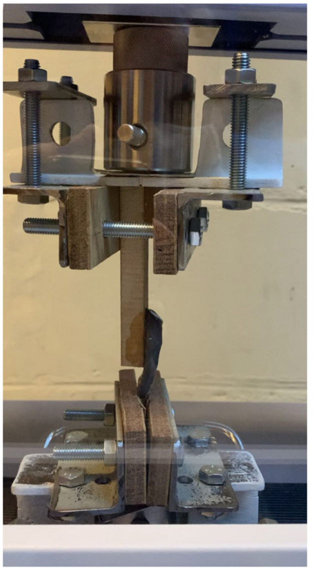

FIGURE 1 Experimental setup. A sample is mounted onto the test bench

TABLE 2 Point system permitting evaluation of the adhesion degree of the glue to each of the contact materials

0	No adhesion at all; no glue remains on this contact surface
1	Adhesion with little to no visible remains on the contact surface
2	Even distribution of adhesive remains on both contact surfaces
3	Adhesion of most of the visible remains on this contact surface
4	All adhesive remains are situated on this contact surface

Points are attributed per contact material, with a total of 4 points attributed per sample in the format *X*–*X* (0–4, 1–3, etc.).

samples are not included here in the original table of samples as no further analysis could be performed on them. A total of 167 successful samples (Table 3) was thus produced, to which a few samples were added in one specific case: when results proved highly varied for one adhesive/contact material batch, one or two samples were added to the tests to increase sample size and to verify the validity of the results.

Ferr-L-Tite failed to adhere to flint in most cases, with only three samples adhering long enough to be secured in the test bench and submitted to the test. The others showed no adhesion, and thus had a resulting breakage maximum force of 0 N/mm^2.

The results were examined per contact material and per glue type. Given that for hafting purposes both the durability of a joint and its reliability are essential factors, both aspects were considered on the basis of the maximum force required to break up the joint and the range of variation in the results. In addition, the adhesive cohesion to the contact surfaces was evaluated but only for samples that could be successfully tested and for which a breakage maximum force could be recorded.

Maximum force comparison

In general, protein glues tend to perform better with either substrate when used in a single lap joint, and higher values for their resilience were reached (Figure 2). Fish glue, in particular, performs systematically better than the other glues, reaching a superior median for each substrate, but it is also the most unstable glue, with the greatest range of variation. Nevertheless, both vegetal and protein glues overlap in resilience, and the most resilient glues vary in function of the organic substrate. Vegetal mixtures perform distinctly differently depending on their loading agents or lack thereof in combination with different substrates. Ferr-L-Tite proved unfit for simple lap joint adhesion on nearly every composite sample and was not included further in what follows.

Evaluation according to organic contact materials

When results were compared between the different organic contact materials, independent of the glue type used, differences between batches were not important. All glues combined, no organic contact material showed a distinguishable difference in resilience in comparison to the others. This shows that all considered organic materials are in theory relevant hafting materials and that, all glue types combined, the nature of the organic contact material is not a determining factor in the resilience of the composite assembly.

However, variations in resilience were noticeable when results for each of the three organic testers were broken down per glue type (Figure 3). For any given glue, we observed a different behaviour and resilience depending on whether the contact material was bone, antler or pine wood, thereby demonstrating that certain combinations perform better than others. For each glue–substrate combination, the stability of the results was also reviewed. The interquartile range (IQR) obtained in the box plot diagrams was used to determine the stability in performance of each glue/substrate combination. Lower values indicate little variation in results, while higher values indicate a broader range of variation and thus less stability in performance.

With bone—the least porous material used in this experiment—fish glue showed the highest median values in terms of resilience, but showed little stability. The second-best combination with bone proved to be resin mixed with beeswax without loading agents. In terms of values, it was the less stable vegetal glue of the selection in combination with bone. It was followed by hide glue and RBO3. The remaining protein glues followed: first sinew and then bone glue. Surprisingly, bone did not combine well with bone glue despite their molecular chains being similar

TABLE 3 Experimental data. This table contains the resilience results for each sample, and cohesion results on both flint and organic substrate

ID	Haft material	Gluing specifics	Mineral substrate remains	Organic substrate remains	Resilience (N per cm^2)
EXP114/167	Antler	70% resin 30% wax	1	3	74.43
EXP114/168	Antler	70% resin 30% wax	2	2	0
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
EXP114/169	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
EXP114/41	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
EXP114/172	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
EXP114/151	Antler	Bone	2	2	79.65
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
EXP114/152	Bone	Bone	2	2	90.88
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
EXP114/153	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
EXP114/182	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

(Continues)

TABLE 3 (Continued)

ID	Haft material	Gluing specifics	Mineral substrate remains	Organic substrate remains	Resilience (N per cm^2)
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
EXP114/157	Antler	Fish	1	3	460.59
EXP114/158	Antler	Fish	2	2	95.41
EXP114/61	Antler	Fish	1	3	305.33
EXP114/64	Antler	Fish	2	2	290.77
EXP114/65	Antler	Fish	1	3	190.15
EXP114/159	Bone	Fish	2	2	280
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
EXP114/01	Wood	Fish	2	2	144
EXP114/03	Wood	Fish	2	2	128
EXP114/160	Wood	Fish	2	2	424
EXP114/161	Wood	Fish	2	2	340.8
EXP114/162	Wood	Fish	2	2	315.84
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
EXP114/81	Antler	Hide glue (rabbit)	1	3	207.52
EXP114/85	Antler	Hide glue (rabbit)	2	2	276.33
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
EXP114/166	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
EXP114/146	Antler	Resin/charcoal	1	3	128
EXP114/147	Antler	Resin/charcoal	2	2	109.12
EXP114/148	Antler	Resin/charcoal	1	3	111.41

(Continues)

TABLE 3 (Continued)

ID	Haft material	Gluing specifics	Mineral substrate remains	Organic substrate remains	Resilience (N per cm^2)
EXP114/149	Antler	Resin/charcoal	2	2	166.08
EXP114/150	Antler	Resin/charcoal	2	2	141.91
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
EXP114/106	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
EXP114/131	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
EXP114/111	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
EXP114/91	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
EXP114/136	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
EXP114/116	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
EXP114/100	Wood	Resin/ochre 2	2	2	112.64
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

(Continues)

TABLE 3 (Continued)

ID	Haft material	Gluing specifics	Mineral substrate remains	Organic substrate remains	Resilience (N per cm^2)
EXP114/99	Wood	Resin/ochre 2	1	3	105.6
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
EXP114/121	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
EXP114/101	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
EXP114/197	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
EXP114/66	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
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

(Continues)

TABLE 3 (Continued)

ID	Haft material	Gluing specifics	Mineral substrate remains	Organic substrate remains	Resilience (N per cm^2)
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

and being expected to create a solid bond. Resin and beeswax mixtures in combination with bone resisted best if unloaded or with a small amount of ochre. A small concentration of charcoal did not offer an impressive median value, but some samples reached values similar to RBO3.

With wood, fish glue also performed best—far above the median values of other glues, but with great variation in measurement values. It was followed by bone glue, which was far more stable in its performance. The RBO3 (7.5% ochre) mixture came next, outperforming both hide and sinew glue, which both offered stable but unimpressive results. Resin and beeswax adhesives used on wood performed better when loaded with a small amount of ochre than with none, and did worse when loaded with charcoal, even in small concentrations. The rosin and beeswax mixture reached third place in regard to vegetal glues.

With antler, the most porous material among the tested contact materials, fish glue was shown to be the most resilient glue once more, but still showed an important variation in performance. It was followed by bone glue, which reached its highest median value and also proved very stable in its performance, with a reduced range of variation in the results. Resin and beeswax with no loading agent came third according to the median value, but their range of variation overlapped with the other remaining glues. Hide and sinew glues were not the most resilient in combination with antler and proved to be quite unstable. Vegetal glues—rosin and beeswax, and resin and beeswax excepted—showed a very stable performance. Resin and beeswax mixtures were most resilient without any loading agent, with a small proportion of charcoal, or with a high proportion of ochre (RBO1).

Evaluation according to glue type

Combinations can either be considered successful and resilient from the perspective of a given organic substrate, for which a glue can then be selected, or from the perspective of a certain glue, in which case the substrate that would best fit the glue can be considered. The force required for sample failure varied more between different glues than was the case for different organic substrates, and glue type should thus be presented as a more influential variable. However, this factor on its own does not explain all variability. Ferr-L-Tite proved unfit for simple lap joint adhesion on nearly every contact material and is not considered further here.

In general, we observed that protein glues reached higher maxima in terms of resilience but did not always perform consistently. Vegetal glues produced results at lower values but tended to be very stable. These tendencies varied and could be contradicted with specific combinations. The addition of ochre or charcoal influenced the performance of resin and beeswax on certain substrates, but this influence remained limited and only proved to enhance adhesion when used in small concentrations.

Protein glues

Fish glue performed best when combined with a wooden substrate, then with antler and finally with bone. Its range of variation in performance was generally wide and the highest maximum

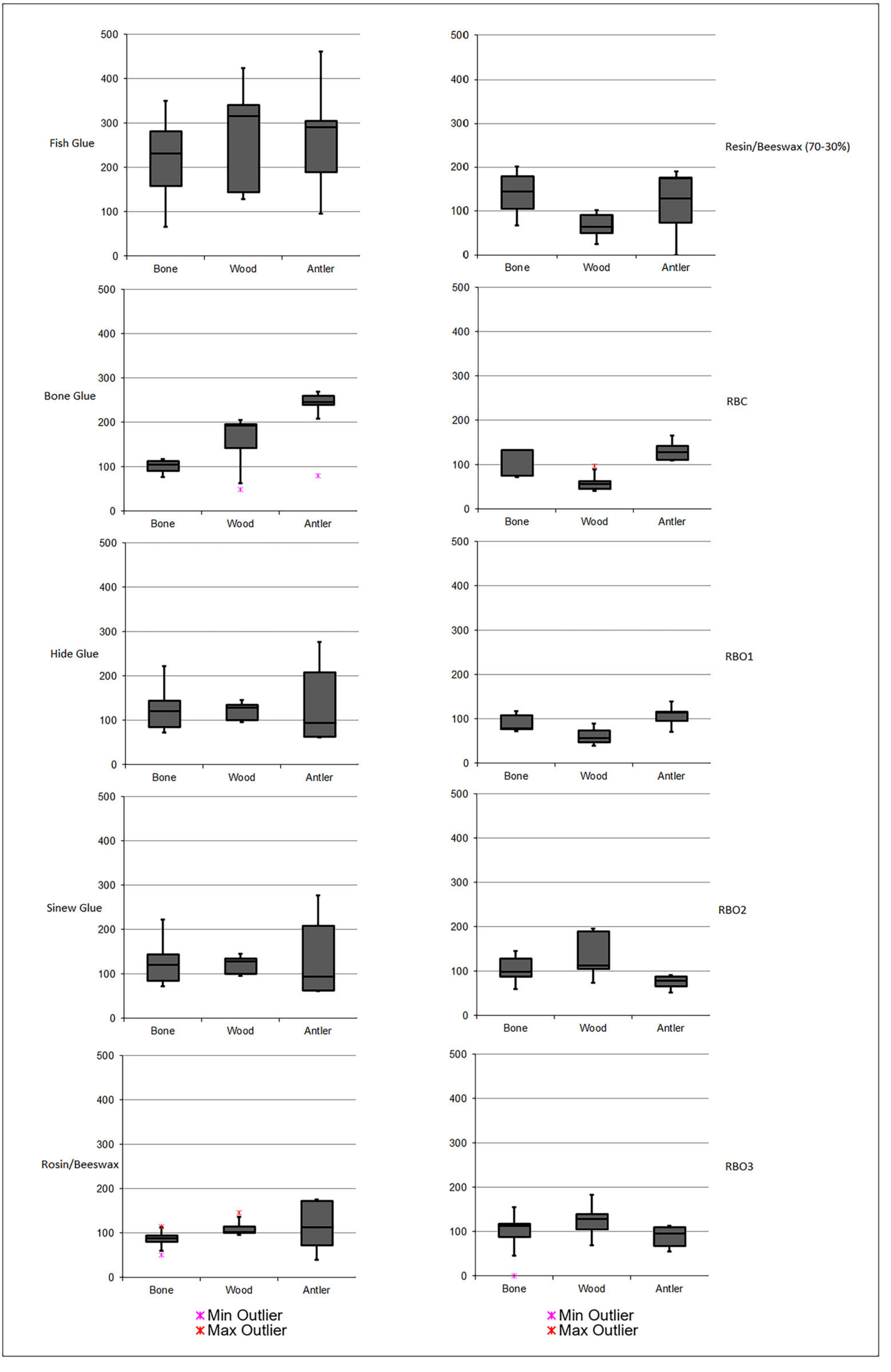

FIGURE 2 Box plots comparing the resilience of different glue/substrate combinations

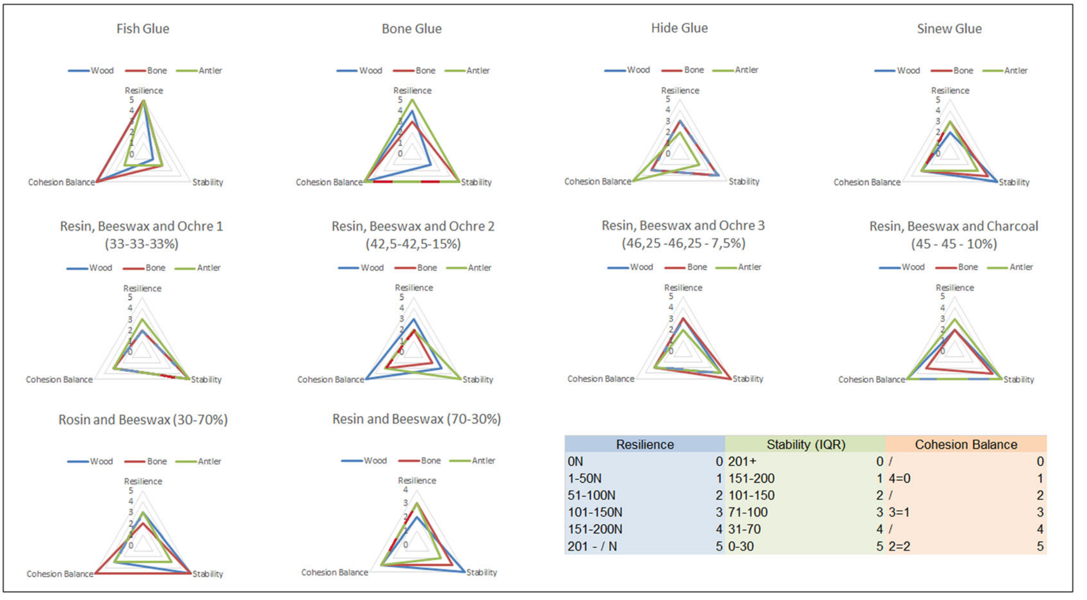

FIGURE 3 Radar graphs of the performance of different glue–substrate combinations according to resilience, stability and cohesive balance. The initial results are calibrated on a 0–5 index to facilitate visual comprehension (see chart legend at the bottom of the figure for calibration details)

values were obtained when combined with antler. This variation in results cannot be explained by a more important roughness of the contact material that could have created more contact surface for the glue to work with. It can be presented as a very resilient glue in a single lap joint system when submitted to shear–compression, but its performance tended to be variable in comparison to some other glues.

Bone glue is one of the most resilient protein glues but behaved very differently depending on the organic substrate. It combined best with antler, and its resilience decreased significantly with wood and again with bone. It proved to be a stable glue with a small range of variation in the results for each substrate.

Hide glue showed overlapping general results for all three organic substrates. Its highest median value was reached when combined with wood, then bone and, finally, antler. Nevertheless, the highest maxima were obtained with antler, but this combination also led to the widest range of variation in the results. By contrast, a combination with wood offered very stable results with limited variation, while a combination with bone tended to show a slightly wider range of variation.

Sinew glue was the least resilient protein glue and often shown to be less resilient than vegetal glues. It was most resilient when combined with bone if based on the median values, and this combination showed great stability. A combination with antler offered the highest maximum values in terms of resilience but the range of variation in the results was too wide, with little consistency, and the median value was thus much lower. Finally, combinations of sinew glue and wood were not very resilient and showed little consistency in the results. It was the least resilient combination of the entire set of experiments in this study, Ferr-L-Tite excluded.

Vegetal glues

The rosin and beeswax mixture (30–70%) adhered the strongest to antler, but with an important variability in results. Wood was the second to best combination and showed stable results, as did a combination with bone.

Resin mixtures (all combined) performed best with antler, then bone and, finally, wood. Their resilience was, however, quite similar and offered stable results. This observation was not systematic when resin mixtures were studied individually (see Supporting Information). The resin and beeswax mixture (70–30%) was most resilient when combined with bone and showed stable results. Its combination with antler was second best but, again, this combination led to a wider variation in results. Wood was the least resilient combination. RBO1 (33% ochre) offered stable results with all three substrates, but was more resilient with antler, then bone and, finally, wood. Its general performance remained generally low compared to the other resin mixtures with lower concentrations of a loading agent. RBO2 (15% ochre) and reached higher resilience than RBO1 generally, but also show that a lower concentration of ochre combined better with a wooden and bone substrate but did not combine well with antler. Results show a narrow range of variation for both antler and bone, and a wider range of variation for wood. RBO3 (7.5% ochre) was one of the most resilient vegetal glues and the most universal resin mixture with additives, as its performance was stable, with a narrow range of variation for all three substrates. It reached the highest maximum values in terms of force until breakage when paired with wood, bone coming in second and antler last.

RBC adhered best to antler, while maintaining great consistency in the results. It performed second best with bone, with a wider range, and performed worst with wood, and this result proved highly consistent.

Adhesive cohesion on contact surfaces

In nearly all samples the link of the flint surface to the glue was the weakest point in the composite system and broke first. This was not an unexpected result, as organic materials are naturally rougher and thus offer more surface area for contact. Since the same flint type was used for every sample and the contact surface for each flint tester had been measured, it can be excluded that variation in flint testers caused variation in the maximum force values up until breakage. This permits us to conclude that the compatibility between the organic substrate and the glue type was the main driving factor. The limitless variety of stone types is, however, not explored in this paper, in order to limit our variables. We cannot exclude that roughness of a mineral could have an impact on the resilience of a glued system.

An appreciation of the cohesive balance of the glue is also considered (Table 2). If the glue fails with a 2–2 cohesive distribution ratio, cohesion is considered balanced and obtains an index of 5. A 3–1 distribution ratio indicates partial balance and results in an index of 3, while a 4–0 ratio results in an index of 1. This cohesive balance is not systematically symptomatic of a higher resilience or stability. It only aims to describe the glue's behaviour in relation to both mineral and organic substrates within a fused system.

In general, the best-performing composite combinations reach their maximum force while the glue stays cohesive on both contact surfaces (2–2 cohesive ratio). Nearly all fish glue samples presented a cohesive fracture with every substrate. Bone glue on antler, as well as RBC on antler, presented a cohesive fracture of the glue and all performed well in resilience. However, resin/beeswax and antler also performed well but did not present a cohesive fracture of the glue. Results obtained with wood substrates seem to confirm our hypothesis, as fish glue, bone glue and RBO2 all presented cohesive fractures. These three glues did reach the highest maxima for maximum force with wood (the median was not used for RBO2 in this case, but the highest maximum was). From another point of view, the best combination for an RBO2 mixture was a wooden substrate. Bone substrates do not allow a similar conclusion, as the 2–2 cohesive ratio only repetitively presented itself with rosin and beeswax. The latter was not the most resilient combination but did perform second best if considered from the perspective of the glue type. This should be investigated in more detail in the future as the limited number of samples did not allow for a systematic study of the plastic behaviour of our experimental glues.

DISCUSSION

The use of tripartite samples permits more realistic reproduction of hafted tools and a more reliable evaluation of their mechanical behaviour; it also places earlier hypotheses in a different light. Indeed, Kozowyk, Poulis, and Langejans (2017) tested both flint-on-flint samples and wood-on-wood samples using birch tar as a glue, and they observed cohesive failure on both sample types. They concluded that the glue was the weakest point of a composite sample and that it would also fail cohesively. Our results challenge this interpretation by showing differing behaviours depending on the glue type and organic substrate used, which demonstrates the importance of realistic mechanical testing.

In this experiment, vegetal glues generally tend to be outperformed by some protein glues. This can be explained by the fact that not all glues are suitable for the same situations, or for a single lap joint. Protein glues have been used in a long history of crafts and are known to perform well in single lap joint contacts. Ferr-L-Tite is a perfect example of a glue that did not perform well, because it is meant to be used as a sealant. It could very well also be the case for most vegetal glues, even though they prove to adhere well to both mineral and organic parts of the composite samples and to show various levels of resilience through testing. The experimental work presented here serves as a first reference for further research on different hafting systems, such as inserts, that could examine the resilience of glue–contact material combinations in more detail.

Our results show that an increase in the roughness of the organic contact material is not a sufficient factor on its own to ensure a better performing glue or a more robust composite arrangement. Similarly, while the glue type and mixture influence the resilience of a system, these are also not influential enough on their own to explain all variability. Therefore, careful combination of a substrate and glue is key to the resilience of a composite system. The presence and concentration of loading agents can greatly affect the resilience of a composite system, and combinations involving vegetal mixtures can be strengthened if the loading agent and quantity are chosen accordingly. These results imply that in experimental archaeology multiple combinations of glue types, glue mixtures and substrates should be considered depending on the specific case study and its objectives. Depending on whether stability in performance and thus reliability of the hafting system or the overall resilience of the hafting system is the key factor, suitable combinations may differ. If one takes the example of projectile weapons, combining antler points or hafts with bone glue to keep lithics to place can be expected to produce stable results. If stability in performance is not of interest, but high resilience is preferred, one could choose to use fish glue. When using a vegetal-based adhesive, the use of additives should be considered and, if used, the concentration and nature of additives are best adapted to the raw material of the haft. For instance, the performance of a resin mixture on wood is influenced by the concentration of ochre. RBO1 (33% ochre) performs poorly on wood in comparison with RBO3 (7.5% ochre). It does, however, show slightly better results than with no ochre at all, but the difference is too small to justify the use of ochre for enhancing the system's performance. Similarly, the addition of ochre appears to broadly improve the consistency in performance of the glue mixture. While these results lend some ground to the intentional addition of ochre for improving glue performance as argued by others (e.g., Wadley et al., 2009), the evidence is still rather weak, suggesting that other, non-functional parameters are at stake as well.

Controlling the different variables and parameters that intervene with regard to glue use leads to more controlled experiments and reproducible results, notably in projectile experiments in which hafting is a key parameter that also impacts fracture formation (Rots & Plisson, 2014). The hafting of barbs indeed shows similar conditions to our mechanical experiment, as it is laterally mounted and thus should be subjected to shear upon impact, providing a good case study. The hafting of barbs has failed recurrently in many projectile experiments (e.g., Chesnaux, 2009; Pétillon et al., 2011; Tomasso et al., 2018), and failure rates could perhaps be reduced by choosing a more suitable glue in line with the different weapon components used. Pétillon et al. (2011), for instance, glued a percentage of the flint insert antler points with a resin–beeswax and ochre mixture. Our results have shown that a resin, beeswax and ochre mixture does not perform best with antler and flint, and that a bone glue could have led to a stronger composite system and perhaps less failure during the shooting experiment. Of course, in the case of weapon systems, failure of the hafting bond can also be intentionally sought if the shooter wishes that the barbs detach upon impact to create greater haemorrhage (Rots & Plisson, 2014). Glue is thus a versatile product and its use can be easily adapted to a range of needs and materials, as also suggested on the basis of archaeological ochre mixtures found on stone-backed microliths (Lombard, 2007).

Raw material availability is a determinant factor in the choice of a specific glue, but also functional considerations likely played an important role. Few studies take these functional considerations into account, even though the importance of flexion or compression with a particular task may drastically differ and the glue should thus be chosen in agreement with the exerted pressure. Scraping tools may thus benefit from the use of another glue or glue composition than adzing tools or projectiles. Also, the environmental conditions in which a glue is used play a role, as glues react differently to humidity and temperature, and a glue that functions well on land may not be suitable in aquatic environments. Fishing tools, for instance, obligatorily require a water-resistant glue. We thus emphasise the importance of considering all functional and environmental aspects of using glues when experimenting and studying archaeological glue remains in order to fully exploit its potential in improving our understanding of technological evolutions and their implications.

CONCLUSION

Many hafting systems rely on the use of glue, and an in-depth knowledge of glue types, possible recipes and their mechanical performance is thus essential when trying to understand when hafting appeared, and how hafting systems varied between regions and through time. Despite the fact that the mechanical properties of glue are crucial in understanding its use in hafting and its influence on the choice of hafting system, little to no mechanical tests had yet been performed to examine the properties of different glue recipes and their performance in joining stone and organic materials together. Different experimental studies have, of course, contributed partial data, in particular regarding vegetal-based mixtures, but previous mechanical studies have systematically focused on one-component tests—that is, joining two parts of the same material together. While such studies provide useful data, they are not directly relevant for understanding how different glue types performed and may have been used in interaction with particular hafting designs. Our results show that not all glue types perform equally well and that the organic material used in combination with these glues matters. The use of specific concentrations of additives in an adhesive mixture can also radically change the strength of the composite system. The glue type, mixture and organic substrate are insufficiently influential on their own to justify the variations in resilience, but the importance rests within how they are combined. Overall, it cannot be said that protein and vegetal glues adhere better to vegetal or animal-based substrates—each situation must be considered on its own. While only single lap joints were considered here, further research should examine other directions of pressure, and further explore how glue performance can be improved in order to understand what composite systems work best depending on the functional constraints of the task.

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