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# Laboratory strength testing of pine wood and birch bark adhesives: A first study of the material properties of pitch

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ABSTRACT

Adhesives are an important yet often overlooked aspect of human tool use. Previous experiments have shown that compound resin/gum adhesive production by anatomically modern humans was a cognitively demanding task that required advanced use of fire, forward planning and abstraction, among other traits. Yet the oldest known adhesives were produced by Neandertals, not anatomically modern humans. These tar or pitch adhesives are an entirely different material, produced from a distinct, albeit similarly complex process. However, the material properties of these adhesives and the influence of the production process on performance are still unclear. To this end we conducted a series of laboratory based lap shear and impact tests following modern adhesive testing standards at three different temperatures to measure the strength of pine and birch pitch adhesives. We tested eight different recipes that contain charcoal as an additive (mimicking contamination) or were reduced by boiling (seething) for different lengths of time. Lap shear tests were conducted on wood and flint adherends to determine shear strength on different materials, and we conducted high load-rate tests to understand how the same material behaves under impact forces. Our results indicate that both pine and birch pitch adhesives behave similarly at room temperature. Pine pitch is highly sensitive to the addition of charcoal and further heating. Up to a certain extent, charcoal additives increase performance, as does extra seething. However, too much charcoal and seething will reduce performance. Similarly, pine pitch is sensitive to ambient temperature changes and it is strongest at 0 °C and weakest at 38 °C. Adhesive failures occur in a similar manner on flint and wood suggesting the weakest part of a flint-adhesive-wood composite tool may have been the cohesive strength of the adhesive. Finally, pine pitch adhesives may be better suited to resisting high-load rate impacts than static shear forces. Our experiments show that pitch production and post-production manipulation are sensitive processes, and to obtain a workable and strong adhesive one requires a deep understanding of the material properties. Our results validate previous archaeological adhesive studies that suggest that the manufacture and use of adhesives was an advanced technological process.

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## 1. Introduction

The use of adhesives for hafting in prehistory was a significant technological advancement (Ambrose, 2010; Koller et al., 2001; Lombard, 2007; Wadley, 2005; Wadley, 2010; Wadley et al., 2009; Wragg Sykes, 2015; Wynn, 2009). Three primary materials were used to make adhesives in prehistory: naturally sticky resins exuded from trees (Charrié-Duhaut et al., 2013; Helwig et al., 2014), a naturally sticky petroleum product known as bitumen (Boëda et al., 2008; Brown, 2016; Brown et al., 2014; Cârciumaru et al., 2012; Monnier et al., 2013), and manufactured tars or pitches produced from the destructive distillation (pyrolysis) of plant matter (Aveling and Heron, 1998; Grünberg, 2002; Koller et al., 2001; Mazza et al., 2006; Pawlik and Thissen, 2011). The earliest known adhesives are tars, dated to approximately 200,000 years ago, and were made from birch (*Betula* sp.) (Grünberg, 2002; Koller et al., 2001; Mazza et al., 2006; Pawlik and Thissen, 2011). Tar can be produced from any organic matter, and in recent times was more commonly made from pine (*Pinus* sp.) wood (Egenberg et al., 2002, Font et al., 2007, Hjulström et al., 2006, Robinson et al., 1987). The pyrotechnical challenges associated with tar production have placed it at the forefront of a debate on Neandertal cognition (Roebroeks and Soressi, 2016; Wragg Sykes, 2015), however little is known about the sensitivity of tar in relation to the production process. The laboratory performance experiments conducted here

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provide valuable data for understanding the material properties of tar-based adhesives, moving the discussion about Neandertal cognition and technical abilities forward.

Adhesives are used as a proxy to understand the technological and cognitive abilities of hominins (Ambrose, 2010, but see also Coolidge and Wynn, 2009, Villa and Soriano, 2010, Wadley, 2010, Wragg Sykes, 2015). This research has been dominated by compound resin/gum-ochre adhesives made by anatomically modern humans in Africa (Kozowyk et al., 2016; Wadley, 2005; Wadley, 2010; Wadley et al., 2009; Wadley et al., 2004; Wynn, 2009; Zipkin et al., 2014). In this scenario, it is hypothesised that the production and application of compound adhesives require advanced working memory, the ability to multi-task, an understanding of abstract terms (e.g. miscibility, stiffness, viscosity and tack) and fluid intelligence (as exemplified in transformative technology) (Wadley, 2013). The production of compound adhesives is complex and the end product does not resemble the individual ingredients. Moreover, the process is transformational and irreversible (Lombard and Haidle, 2012; Wadley, 2010; Wynn, 2009). Neandertal tar production, although different from compound adhesive manufacture, may have required similar cognitive abilities (Wragg Sykes, 2015). For example, the pyrolytic production process is a possible testimony to an understanding of abstract terms and fluid intelligence (Wragg Sykes, 2015) and is used to illustrate the technological abilities of Neandertals (Villa and Roebroeks, 2014).

Tar is made by heating biomass under reducing conditions and experiments confirm that wood tar production (Kurzweil and Todtenhaupt, 1990; Piotrowski, 1999; Todtenhaupt and Kurzweil, 1996; Voß, 1991) and birch bark tar production (Czarnowski and Neubauer, 1991; Groom et al., 2013; Palmer, 2007; Schenck and Groom, 2016; Schenk, 2011; Weiner, 1988) are sophisticated processes. Both can be made using aceramic technology (without pots), similar to what might have been available during the Palaeolithic (Itkonen, 1951; Schenck and Groom, 2016). To produce tar, organic material must be heated to a high enough temperature, under sufficiently reduced environments, and it must be collected without allowing it to burn or become over-saturated with ash, soil, or other contaminants (Pawlik, 2004). When tar is produced it may still need further refinement before it is suitable to use as an adhesive. This may be in the form of additional heat treatment to evaporate and remove the more volatile liquid components (water, methanol, acetic acid) rendering what is more accurately described as 'pitch' (Egenberg et al., 2003). Alternatively, the tar may be thickened with an additive, such as charcoal, in a similar manner to ochre and gum (cf. Wadley, 2005). Experimental reproduction of tar resulted in contamination with plant products and fire by-products including charcoal (Kurzweil and Todtenhaupt, 1990; Osipowicz, 2005; Pawlik, 2004; Pomstra and Meijer, 2010). Although a current theoretical framework details the complexities of Palaeolithic tar production (Wragg Sykes, 2015 and refs therein), post-production processes during prehistory are unknown, and it is unclear how sensitive the performance of pitch adhesives are to refinement with heat or to contamination. As with other natural adhesives, we know little about the adhesive performance of tar under different circumstances. Insight into these issues may help reveal prehistoric choices and add to the existing cognitive framework.

Here we present a first attempt to understand the effect of post-production manipulation on shear strength and impact resistance of wood and bark tar pitches. We explore adhesive strength in relation to tree species, climate, substrate material and force/activity. Pine tar is more ubiquitous in later periods than birch tar (Surmiński, 1997), and it might be that these two adhesives had different (additional) functions. It is possible that one is stronger (capable of withstanding higher maximum stress) than the other, and therefore more or less preferred. To this end we conducted lap shear and impact tests on pine and birch tar pitch. Experiments were also conducted to understand the influence of post-production refinement and manipulation. In these tests charcoal was added in set increments to mimic increased charcoal contamination. This will help us understand how clean the production of pitch needed to be and if the intentional or accidental addition of charcoal would be beneficial. Similarly, we tested tar in different stages of reduction. Prehistoric tar was used under variable environmental circumstances and it is possible that one of the attractions of this adhesive over resin was that it performed well under a wide temperature range (Kozowyk et al., 2016). We therefore tested tar for strength under different temperatures. Some adhesives may perform better on specific adherends or substrate materials. Standard strength tests generally use aluminum and wood adherends; we added flint to understand how tar strength on wood and flint compare. Finally, different force load-rates were at work in different prehistoric tasks and an adhesive may react differently to one than another. Prehistoric peoples may have selected adhesive materials based on these differences. We therefore compare the strength of tar under two different forces: quasi-static lap shear and impact.

## 2. Materials

### 2.1. Pine pitch, birch pitch, and charcoal

Tar is a dark coloured viscous liquid produced through the pyrolysis or gasification of biomass (Betts, 2000; Collin and Höke, 2005; Purevsuren et al., 2004). Tar can also be obtained from coal (Collin and Höke, 2005), or occur naturally as a material commonly known as bitumen or asphalt (Betts, 2000). When tar is in a liquid state, containing higher percentages of volatiles, it is referred to simply as 'tar'. The term 'pitch' or 'tar pitch' refers to the more viscous, semi-sold or solid fraction of tar (Betts, 2000; Collin and Höke, 2005; Legasse, 2012). Pitch is also sometimes confusingly employed to refer to natural resin exudates collected from conifers (Gibby, 1999; Loewen, 2005), although this is more of a colloquial use of the term (Langenheim, 2003) and will be avoided here.

The two states, tar and pitch, may have different functions. Historically, fluid tar materials were used for waterproofing and preserving wooden roofs and boats (Bonaduce and Colombini, 2004; Connan and Nissenbaum, 2003; Prehn, 1991) and more solid pitch-like varieties were used as fixatives and for caulking ships (Egenberg et al., 2003). Prehistorically, tars could have served as a waterproof coating to protect sinew, raw-hide, or vegetable fibre bindings from moisture (Rots, 2013) and pitches could have been used as the bonding agent itself (Grünberg, 2002; Koller et al., 2001; Pawlik and Thissen, 2011). Although there is no precise classification that separates 'tar' from 'pitch', we will use the word 'tar' from here on to refer to the unrefined material obtained through the pyrolysis of woody plant materials, being in a liquid state at room temperature. 'Pitch' will be used to refer specifically to the refined fraction of tar that has been reduced to a semi-solid or solid at room temperature.

To control the material properties and to conduct a reproducible experiment we used commercially available pine tar, otherwise known as 'Stockholm tar' as our primary ingredient. Because birch bark tar is not commercially available we produced it using the 'two pot' method (Hansen, 2007; Kurzweil and Todtenhaupt, 1990; Piotrowski, 1999) in an open fire with metal containers. This method is quite refined, and produces a liquid tar with little charcoal contaminates. Both the pine and birch tar were reduced to pitch by boiling over a hot plate until they appeared solid at room temperature (cf. Egenberg et al., 2002).

To test the influence of production-related contamination we added commercially available powdered charcoal. This is pure charcoal made from beech (*Fagus* sp.) and ground into a fine powder (<30 μm). Without the use of ceramics or metal containers to isolate the tar end-product from fire by-products, it is probable that charcoal would be a leading contaminant. There are other materials that could and probably did contaminate adhesives, including plant material from the bark or wood, soil, sand, or ash (Pawlik, 2004), but charcoal is perhaps the most significant and is thus the one we have chosen to test here.

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