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Archaeological Adhesives

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Summary

An adhesive is any substance that bonds different materials together. This broad definition includes materials used in everything from hafted stone tools to monumental architecture. In addition, the combination of bonding, plasticity, and insolubility meant that some adhesives were exploited for waterproofing and sealing of materials, as self-adhering inlays and putties, and as paints, varnishes, and inks. Adhesives have a history of at least 200,000 years. Throughout (pre)history and around the world, people used materials, including bitumen/asphalt, carbohydrate polymers such as starches and gums, natural rubbers, mortars, proteins (from casein, soy, blood, and animal connective tissue), insect and plant resins, and tars made from various barks and woods. Adhesives thus are very diverse and have widely varying properties: they can be tacky, pliable, elastic, brittle, water-resistant, fluid, viscous, clear, dark, and much more. They are a plastic *avant la lettre*. These properties can and were tweaked by mixing ingredients or by further processing. In the study of archaeological adhesives, their characterization is essential and this is best done with chemical and spectroscopic methods. When larger coherent samples as opposed to single finds are analyzed, adhesive studies can provide data on past technologies, socioeconomic organizations, and environments and raw material availability. Through sourcing and mapping of ingredients and adhesive end products, travel and transfer of materials and knowledge can be illuminated. Additionally, experimental reproductions provide data on technological aspects that otherwise are lost in the archaeological record. An archaeology of adhesives can reveal the transport networks, subsistence, mobility strategies, division of labor, and technological know-how that held societies together.

Keywords: glue, bonding, hafting, sealant, resin, tar, mortar, gum, rubber, ochre

Subjects: Archaeology

Introduction

Adhesive technology has a long and expansive history dating back about 200,000 years (Mazza et al. 2006). Both Neanderthals and Middle Stone Age humans created and used new materials by precisely blending disparate ingredients for a wide range of tasks. Adhesives made of materials such as birch bark and pine tar, bitumen, resin, gum, lime mortar, soybeans, rice, animal hides, and blood can be found in the archaeological record around the

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world. Artifacts from the Great Basin of the American Southwest clearly illustrate the diversity of ancient adhesive uses. Here, insect lac and tree resins were used to haft arrows, repair pottery, glue inlays, waterproof basketry, serve as a decorative paint, and create entire objects like game pieces and handles (Bisulca, Pool, and Odegaard 2017; Odegaard et al. 2014; Stacey, Heron, and Sutton 1998).

Though often overlooked, the technological and functional importance of adhesives cannot be underestimated. The “invention” of adhesive technology may have been subtle as prehistoric people identified natural sticky materials in the forms of processed starchy foods and resinous tree exudates. The use of these materials, however, quickly went beyond the simple collection of natural products. Striking to modern researchers is the deep understanding of material properties seen in the past, illustrated by the use of additives to improve adhesive properties and by the appropriation or manufacture of superior adhesive materials when available (e.g., Bisulca, Pool, and Odegaard 2017). Often, the collection, processing, or manufacture of adhesives follows specific protocols unique to a particular region, population, or material (Bakirtzis and Moniaros 2019; Binford 1984; Loewen 2005).

Adhesives can be molded into any desirable shape, making them the first known plastic materials. With these plastic properties, adhesives can create a regular smooth joint even when separate pieces are irregular and do not fit well. Moreover, some adhesives were used on their own (e.g., to create game pieces and figurines). The freedom to form any desired shape had lasting implications on how hominins interacted with and perceived their worlds, possibly shaping their minds in the process (cf. Overmann and Wynn 2019). Some adhesives have a natural shine and a translucent quality, providing a smooth, glossy finish. Different adhesives also have a number of ephemeral qualities, such as distinct smells and tastes. For example, resins such as frankincense and myrrh are commonly used in incense and perfumes, whereas some tars and gums may have been chewed like modern-day chewing gum (Al-Harrasi et al. 2019; Jensen et al. 2019) and some were used in food and beverage preparations (Langenheim 2003; McGovern 2013). Though difficult to illuminate in archaeological research, these properties must have guided the material use and choices by humans in the past.

Mechanisms, Classification, and Identification

An adhesive is any substance that bonds different materials together. Adhesives can be differentiated from one another by their materiality, bonding potential, material properties, function, or source. This definition is useful as it includes a broad range of materials from resin in hafted stone tools to cement in monumental architecture.

The application of adhesives to the surface of an adherent is termed “wetting.” When the adhesive hardens or “cures,” it creates a bond with the adherent surface. In (pre)history, adhesives were used to hold multiple components together, such as a hafting arrangement, inlays, or broken sherds (Figure 1). Adhesives used for bonding were also applied in the

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waterproofing and sealing of materials, as self-adhering inlays and putties, paints, varnishes, and inks. The focus here is on adhesives that bond materials, but occasionally their versatility is illustrated with examples from these other categories.

A wide range of materials have natural tacky qualities, such as fresh tree resin, but adhesives are also manufactured. Here, we recognize three processes: chemical reaction (e.g., two-component adhesives like cement), distillation or pyrolysis of biomass (e.g., birch bark and pine tar), and hydrolysis of animal matter (e.g., hide glue). Sometimes, the base material requires further improvement because it is either too brittle or too soft for the intended function. In such a case, additives like ochre and beeswax may be added to manipulate the adhesive's material properties.

Modes of Adhesion

Understanding the mechanisms of adhesion is useful when trying to optimize bonding. For example, surface roughening may increase mechanical interlocking, and surface cleaning may do the same for chemical bonding (Mahoney 1990; Packham 2003). Adsorption is the main adhesion mechanism. Here, the interaction of van der Waals forces of the liquid adhesive and the solid adherent causes interfacial attraction or physical bonding of molecules and ions (Comyn 2005; Gent and Hamed 1990; Schultz and Nardin 2003). In chemical and electrostatic bonds, materials swap or share electrons or transfer electrons, respectively (Comyn 2005; Ebnesajjad and Landrock 2014). As a result of this mechanism, protein glues adhere well to wood (Bye 1990; Conner 2001). Interdiffusion and interlocking/mechanical modes mean that the molecules of the adhesive or adherent diffuse into the other material and that adhesives lock into cavities and pores of the adherent (Comyn 2005; Gent and Hamed 1990; Mahoney 1990; Packham 2003; Schultz and Nardin 2003). This mechanism also implies that some surfaces, like a very polished and dense material, may be harder to fasten with adhesives than rough or porous surfaces.

Why Use Adhesives?

Adhesive bonding has clear advantages over mechanical fastening, like pegs, and fibers. First, adhesives can more effectively distribute and absorb stress, making them useful in building construction (Cagle, Lee, and Neville 1973; Skeist and Miron 1990) and high-load applications such as projectiles. Adhesives allow dissimilar materials, such as stone and wood (or metal and glass), to be joined. They allow the bonding of big to small materials, such as in jewelry, and the bonding of thin materials like book pages, inlays, gold foil, and mosaics. Furthermore, because many adhesives function as sealants, they can be used to close and waterproof objects like pottery lids and baskets. Another advantage of adhesives is that small adhesive failures are easily repaired (Cagle, Lee, and Neville 1973; Skeist and Miron 1990).

Although the presence or absence of adhesive technology sometimes is used as a marker for complex technological know-how (Wragg Sykes 2015), adhesion is not always the best solution. Many adhesives are sensitive to degradation. Some, such as sugar-based adhesives,

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are not ideal in environments where humidity is high, and others, such as rosin, are brittle at low temperatures. Because glues perform poorly under peel and cleavage loads, adhesives are unsuitable in joints where they are exposed to these (Cagle, Lee, and Neville 1973; Skeist and Miron 1990). Furthermore, some composite tool technology requires movement of the joint. Therefore, in circumstances such as arctic marine hunting with harpoons (Wells, Renouf, and Rast 2014), adhesives may have been intentionally avoided.

Classification and Identification

There are several ways to categorize adhesives. In archaeology, they are generally organized by chemical composition or origin. Categorizing adhesives this way gives us information on what materials were available to prehistoric people. In industry, adhesives are also categorized by their material properties, which is informative about the technological process. The method of curing, along with adhesive application, solubility, and suitability for different adherents, is also frequently used to classify adhesives. Adhesives can cure in a number of ways, such as when they lose their solvent or carrier through evaporation (e.g., gums). Hot-melt adhesives (e.g., pine resin) set when the material cools and solidifies. Curing can also happen through *in situ* chemical reactions such as polymerization (e.g., linseed oil) or hydration and hydrolysis (e.g., cement). Finally, pressure-sensitive adhesives, such as on tape, never fully cure and remain tacky (Comyn 2005; Skeist and Miron 1990).

Before identification, archaeological adhesives generally are first observed with the naked eye or an optical microscope. Under the microscope, resin sometimes appears glass-like and translucent and may show surface cracking (Langejans and Lombard 2015). The presence of starch grains might point to a starch-based glue. Native starch granules of different species can be identified by using optical light microscopy. However, because gelatinization is part of the glue production method, most grains will probably have become unidentifiable (Crowther 2012; Torrence 2006). Similarly, with structural binders like limestone- and gypsum-based plasters, optical microscopy and thin-section analysis constitute the first stage of research (Artioli 2010b, 249). Although plasters may have a comparable morphology, their composition and pyrogenic production can still be different. Overall, the morphology of organic and inorganic remains on tools is an unreliable means for specific identification (Koller and Baumer 2010; Monnier, Ladwig, and Porter 2012), and additional study of the context and characterization of adhesive remains is required to confirm their authenticity and nature (Aveling and Heron 1998; Langejans 2011; Monnier et al. 2013; Yates et al. 2015).

Reagent Testing

The presence of a compound sometimes can be demonstrated when a specific material/reagent is added and when this leads to unique chemical reaction. A reagent can be a chemical compound, antibody, and enzyme. A positive reaction can be marked by a color change or enzymatic activity. Examples are *o*-toluidine reagent testing, where the polysaccharides from starch- and mucilage-based adhesives can be distinguished from other sugars (Bisulca, Odegaard, and Zimmt 2016; Fullagar et al. 2015), and picro-sirius red for

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collagen detection (Stephenson 2015). Amylase is used to identify starch (Hardy 2009) and there are a range of immunological antibody tests for proteins from different sources (Gernaey et al. 2001). Gas chromatography mass spectrometry (GC-MS) and spectrometry has largely replaced reagent testing as the former gives more specific results and the methods are less destructive. However, regent testing is still used because it is generally simple and inexpensive.

Separation

GC-MS is the most frequently used and most precise method to characterize organic compounds in unknown archaeological samples. It allows the identification of different organic components in a mixture. Components or groups of components that are unique to specific materials, such as lupeol and betulin in birch bark tar, can serve as biomarkers for characterizing materials (Evershed 2008). As such, GC-MS can, for example, differentiate resins from different species of pine or amber from sub-fossilized and fresh resins by establishing the degree of polymerization (Seyfullah et al. 2018; Stacey, Cartwright, and McEwan 2006). Moreover, different tar production processes and tar fractions can be identified by comparing the chemical composition and ratios of specific markers (Egenberg, Holtekjølen, and Lundanes 2003; Regert 2004). For example, the presence or absence of betulinic acid and certain fatty acids and diacids can be used to help differentiate between tar produced in a single pot from tar produced using a double-pot *per descensum* method (Rageot et al. 2018). From some European Neolithic sites, there is already evidence for a standardized production process for birch tar; but at other sites, birch tar was produced at a small/individual scale (Regert 2004).

In the GC part of the analysis, different molecules are separated and exit the GC column at different times and in a characteristic order depending on their molecular weight and chemical character. In the following MS part of the analysis, the now-separated components are ionized and identified on the basis of their molecular weight (Giorgi 2009; Schilling 2010). Compounds are identified by comparing the mass spectra to known libraries and experimental data. The downsides of this method are that it is destructive and generally cannot be done *in situ*. In addition, the interpretation of spectra can be complex for materials that lack obvious biomarkers or characteristic molecules and for degraded samples with little molecular integrity.

A wide range of chromatography–mass spectrometry methods is available and they differ in how the samples are carried through the chromatographer, how the sample is ionized, and how the MS results are analyzed (Giorgi 2009). Which method to use largely depends on the nature of the material and the guiding results from infrared (IR) analysis. A cutting-edge example is proteomics for the characterization of protein compounds in adhesives and varnishes. With methods like liquid chromatography-matrix-assisted laser desorption ionization-time of flight/time of flight-mass spectrometry (LC-MALDI-TOF/TOF-MS), species-specific peptides resulting from enzymatic hydrolysis of the sample are characterized (Dallongeville et al. 2016; Hendy, Welker, et al. 2018; Kuchova, Hynek, and Kodicek 2009; Orsini, Parlanti, and Bonaduce 2017).

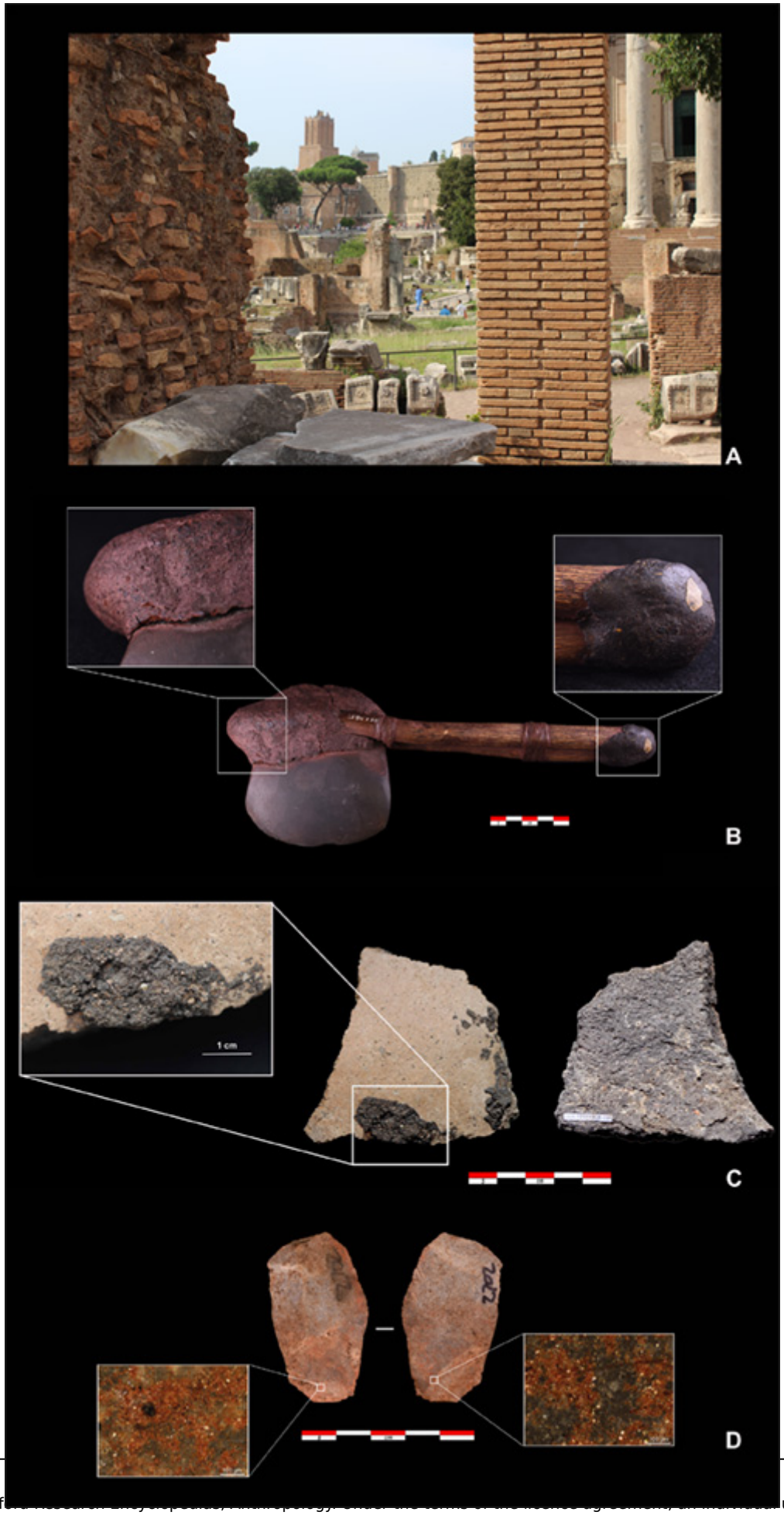
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Spectroscopy

In archaeology, spectroscopy is one of the most frequently used sets of analytical methods. It includes techniques like Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy (Raman), scanning electron microscopy (SEM), and x-ray fluorescence (XRF). In spectroscopy, the interaction between radiation and matter provides information on elements present or their chemical bonds in molecules or both. When a sample is exposed to method-specific electromagnetic radiation, the type, frequency, and amount of radiation that is emitted, absorbed, and scattered can be measured. The peaks in a resulting spectrogram act as "fingerprints" that can be linked to molecular functional groups, crystalline structures, or heavier elements or a combination of these. Depending on the method applied in archaeology, spectroscopy is used to analyze inorganic materials, such as pigments (SEM, XRF, XRD, and Raman), and organic materials (FTIR, nuclear magnetic resonance [NMR], and Raman) (Artioli 2010a). Spectrographic methods generally characterize one material or element. They rarely provide information on the molecular association or origins of those elements. Therefore, it can be difficult to distinguish material information from background data (e.g., iron oxide from ochre residues and iron oxide that is part of the stone tool). For the same reason, these methods are not well suited for mixtures as they generally provide a broad characterization. On the other hand, spectroscopic methods are generally non-destructive and some can be used to characterize *in situ* samples. They also provide complementary data for the organic chemistry (GC-MS), which is valuable because many adhesives contain inorganic additives.

Characterization of archaeological materials, and mixtures in particular, can be a challenge, and a range of techniques is generally required. For example, natural rubber consists mainly of the isomeric polymer (a long-chain molecule) 1,4-polyisoprene and (to a lesser extent) of terpenoids, phenolics, alkaloids, proteins, and fatty acids (Hurley 1981; Langenheim 1990). NMR is often used to identify 1,4-polyisoprene in ancient materials (Hosler, Burkett, and Tarkanian 1999), and a combination of IR and Raman spectroscopy helps to differentiate isomeric crystalline forms of polyisoprene (Haider 2012). FTIR and GC-MS can identify organic compounds, such as proteins, methylene, and methyl groups (Hosler, Burkett, and Tarkanian 1999; Ley-Paredes et al. 2018). Correct identification can aid in the sourcing of adhesives; it also allows behavioral inferences. In Pleistocene archaeology, for example, adhesive production is considered a technologically complex task, which in turn may be cognitively demanding (Wadley, Hodgskiss, and Grant 2009). However, major differences in the processes are involved in the manufacture of different adhesives. Therefore, accurate material identification is paramount to understanding the technological and potentially cognitive complexity of different hominins.

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Figure 1. Examples of different adhesives. *A*, Roman concrete at the *Forum Traiani* in Rome (Italy). In the background, Trajan's Market (100–110 CE) and the Tower of the Militia (13th century CE). The walls and the pillars in the foreground are built in *opus latericius*, which was the predominant construction technique in the Imperial Era. In the brick wall on the left, the facing is missing and the concrete core (*opus caementicium*) is clearly visible. *B*, Ethnographic Australian aboriginal axe hafted with spinifex resin adhesive mixed with ochre and plant fibers. *C*, Pre-Columbian ceramic sherd with black adhesive remains relating to pottery repair, from the La Cajita site in Colombia, dating to about 1230 CE. *D*, Dorsal and ventral view of a weathered experimental flint tool with ochre stains resulting from the ochre-loaded hafting adhesive (Kozowyk, van Gijn, and Langejans 2020). Photos courtesy of Maria Pina Garaguso (*A*), Paul Kozowyk (*B*), and Alessandro Aleo (*C*, *D*).

Archaeological Adhesive Materials

Archaeological remains of adhesives are found worldwide and what people used depended on their needs, the availability of materials in their environment, their technological knowledge, and their socioeconomic worlds (Figure 1). As adhesives finds are rare for the Middle and Upper Paleolithic worldwide, research here often is limited to reports of very old and rare single finds. Other time periods and regions have a deeper tradition of adhesive research (e.g., bitumen in Mesopotamia) and as a result these adhesive materials are well studied.

Bitumen/Asphalt

Bitumen/asphalt (sometimes referred to as naphtha) is a black viscous semi-solid to solid material originating from crude petroleum. Millions of years ago, layers of zooplankton and algae were caught in sedimentary rock. Under high pressure and high temperatures, these organics are transformed into bitumen, water, gas, and oil. These materials surface when the sealing rocks are damaged, creating pools of bitumen and gas wells (Forbes 1955; Schwarcz and Rink 2000). Some bitumen deposits naturally lost their volatile components and formed bituminous rock in the sedimentary rocks (Forbes 1955, 25). Both pools and rocks occur as outcrops worldwide and were mined in the past.

Bitumen, a mixture of oxidized hydrocarbons, can be characterized with GC-MS, generally by sterane and terpene biomarkers. Steranes are polycyclic alkanes derived from eukaryote steroids. The most important terpene group is geohopanoids, which derived from bacterial cholesterol surrogates (Connan 1999). The composition of bitumen is unique to its origin and can be geographically sourced using the biomarker composition and $\delta_{13}C$ isotope values, analyzed with gas chromatography/combustion-isotope-ratio mass spectrometry (GC/C-IRMS) (Brown et al. 2014; Schwartz and Hollander 2016). Anthropogenic heating to cure or recycle bitumen creates characteristic and identifiable polyaromatic hydrocarbon markers, such as fluoranthene and pyrene (Connan 1999; Schwartz and Hollander 2000). The mining,

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production, transport, and trade of bitumen have been studied extensively through chemical analysis (Brown et al. 2014; Connan et al. 2020; Faraco et al. 2016; Schwartz and Hollander 2000, 2016; Wendt and Lu 2006).

Bitumen is known from archaeological records in Europe, Southwest Asia, North Africa, the Americas, Japan, and Southeast Asia (Argáez et al. 2011; Brown 2016; Connan and Van de Velde 2010; Connan et al. 2020 and references therein; Cosac et al. 2013; Habu 2004; Jansen 1989; Wendt and Cyphers 2008). In Mesopotamia, Late Woodland Pennsylvania, and Olmec Mesoamerica, it was collected from asphaltic crude oil wells, some of which were formally cased. In Mesopotamia, the mining may have been assisted by a windlass (Forbes 1955; Selsor et al. 2000; Wendt 2009). Floating bitumen was collected from the Dead Sea by simply drawing it onto boats. Here and elsewhere, containers, leaved branches, bunches of reeds, and linen towels were also used to collect bitumen (Forbes 1955). Bitumen also seeps from marine deposits and washes ashore where it can be readily collected, such as during the Late Neolithic of the California Channel Islands (Brown 2016). Rock asphalt, sedimentary rocks that contain 5% to 20% bitumen, can be heated to extract bitumen, as was done in Antiquity using the double-pot method. Bitumen from rock asphalt contains a characteristic fraction of fine mineral particles (Forbes 1955). Bitumen is water-insoluble and therefore was often used in waterproofing construction and containers. It has antibacterial properties and is relatively resistant to decay; generally, it preserves well.

When liquid bitumen was collected, it would have required additional treatment, depending on its intended function. Prior to the invention of distillation, around 1200 CE in Mesopotamia, natural evaporation and heating gave bitumen the right viscosity (Forbes 1955). Additives such as minerals, plant fibers, waxes/oils, and resins sometimes were added to liquid bitumen (Brown 2016; Connan and Van de Velde 2010; Schwartz and Hollander 2000, Selsor et al. 2000; Wendt 2009 and references therein). Waxes and oils lower the melting point of the material, potentially saving time and fuel during application. Tempered bitumen also cures more quickly than fresh bitumen, and additives increase the strength and volume of the material (Brown 2016; Forbes 1955; Wendt 2009). Processed bitumen was stored in balls or cakes and in jars. Bitumen can be recycled and reused, but after extensive reheating, it becomes too hard and too brittle to use (Schwartz and Hollander 2000). When containers are waterproofed, liquid bitumen is poured into a container and a pebble is added. As the container and pebble are turned, the bitumen is spread over the surface (Brown 2016; Forbes 1955).

Neanderthals in Syria used bitumen as an adhesive to haft stone tools 70,000 years ago (Boëda et al. 2008). The use of bitumen as a glue to haft stone tools is also reported for modern humans and its use continues well after the start of the Neolithic (Connan et al. 2008). Bitumen is also used to cement bricks in monumental architecture in Mesopotamia and as a glue for jewelry inlays and mosaics and to repair ceramic objects (Connan 1999; Connan et al. 2004; Forbes 1955). In Mesopotamia and California, it was used as an ornamental paste and paint, and bitumen was used as a material on its own to sculpt (Brown et al. 2014; Connan et al. 2004; Forbes 1955 and references therein). Bitumen collected in the oil fields along the coast of the Tohoku district in Japan was traded and used between 3300 and 300 BCE to haft

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harpoons and to mend broken pottery (Tanaka 2000). Another major application is waterproofing of boats, drainpipes, walls, roofs, pottery, and baskets (Brown 2016; Connan and Van de Velde 2010; Connan et al. 2004; Wendt and Cyphers 2008). Bitumen has well-known anti-inflammatory properties and has a wide range of medical applications, from Upper Paleolithic dental filling to skin treatment in Antiquity (Nissenbaum 1999; Oxilia et al. 2017).

Carbohydrate Polymers

Polysaccharides or sugars are nature's most abundant polymers and the main component of carbohydrate glues. Plant starch, plant mucilage, and gum are used mostly as adhesives but this group also includes some micro- and marine organisms like red algae agar (Baumann and Conner 2003; Mathias, Grédiac, and Michaud 2016). All carbohydrate adhesives are large polar macromolecules with a high molecular weight. This feature provides the material with its adhesive strength (Baumann and Conner 2003; Mathias, Grédiac, and Michaud 2016). Generally, carbohydrate adhesives are water-soluble, making them relatively easy to work with. This feature also allows the reuse of glued objects. However, it does leave them prone to microbial and mechanical decay.

Carbohydrate polymer adhesives can be identified through their specific polysaccharides (Colombini and Modugno 2009). Reagent testing (Bisulca, Odegaard, and Zimmt 2016; Fullagar et al. 2015) and GC-MS (Bonaduce et al. 2007; Guasch-Ferré et al. 2020; Kuchova, Hynek, and Kodicek 2009) are most often used for characterization. Other methods like Raman and micro-FTIR are less specific but will allow the distinction from other adhesive groups like proteins and resins (Brambilla et al. 2011). The presence of starch grains might indicate a starch glue.

Gum

Natural gums are polysaccharides that originate from plant exudates and seed coatings; they provide protection when a plant is damaged, or they stop seeds from germinating too early. Gums are generally a tacky light to dark yellow/orange soft material that darkens over time and becomes more viscous, eventually hardening completely. Visually, gum appears similar to resin. However, owing to the sugars, gum is soluble and resin is insoluble in water.

Exudate gums can be collected from natural or artificial wounds in trees like *Acacia* sp. (gum Arabic), *Astragalus* sp. (gum tragacanth), and many fruit trees. In seed gums, like guar gum (*Cyamopsis tetragonoloba*), polysaccharides first must be extracted through grinding, after which they are brought into solution. The production process for other gums, like agar obtained from red seaweed (*Rhodophyceae* sp.), is more laborious and includes cycles of washing, hot water extraction, filtration, gelation, freeze-thaw processes, drying, and grinding (Imeson 2009).

In Italy, gum may have been used in an adhesive mixture also containing beeswax and ochre on stone projectiles dating back to 45,000 to 40,000 years ago (Sano et al. 2019). In New Mexico, gum remains dated between 700 and 1100 CE were found on bows but their precise

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function is unclear (Bisulca, Pool, and Odegaard 2017). Gum exudate, particularly from fruit trees, is a well-known binder in Antique Mediterranean wall painting and stucco (Brecoulaki et al. 2012; Guasch-Ferré et al. 2020 and references therein; Newman and Serpico 2000) as well as Mayan wall paintings (Sheets 2017 and references therein). Gum, and sugar in general, increases the cohesive strength and traction resistance in lime (Rampazzi et al. 2016). Prickly pear or nopal mucilage (*Opuntia* sp.) improves water and stress resistance, and the plasticity of lime mortar (Rodriguez-Navarro et al. 2017), and was used as an additive in the stucco floors of Teotihuacan (Montes et al. 2004). Although archaeological evidence for the use of seed gums is lacking, their use is documented historically (Imeson 2009).

Starch

Starch glues are generally a white and opaque tacky viscous liquid. Plant starches are the energy storage organelles of plants and are most abundant in storage organs like tubers and roots. The granules contain tightly packed polysaccharides in crystalline regions with straight-chained and aligned molecules; these regions are linked by amorphous areas of unaligned molecules. To function as a glue, the molecules need to be exposed and thus the granules must be opened, a process known as gelatinization. In the past, this was probably achieved through heating (~50–85°C), although alkali and acid treatments and oxidation have the same effect (Baumann and Conner 2003; Crowther 2012; Gott et al. 2006). During gelatinization, the crystalline structures are lost, but on curing the linear molecules align with one another and harden. Starch glues are water-soluble and bond well with wood and metals. Fillers, particularly minerals like clay, are used to control and limit the curing time as well as the glue penetration into the substrate (Baumann and Conner 2003).

Carbohydrate adhesives from grains like rice and maize but also from orchid bulbs were often used on paper and papyrus and the binding of books and codices (Berdan, Stark, and Sahagún 2009; Martínez Cortés 1970; Wiedeman and Bayer 1983). Sticky-rice soup is a well-known additive in historic Chinese mortars, increasing strength and durability (Zeng, Zhang, and Liang 2008).

Latex and Natural Rubbers

Natural rubbers are made from latex exuded by plants. Latex is a white milky liquid emulsion composed of complex mixtures of terpenoids, phenolics, proteins, and alkaloids (Hurley 1981; Langenheim 1990). Natural rubbers are classified using their main isomeric compounds: cis-1,4-polyisoprene and trans-1,4-polyisoprene (Takeno et al. 2008). Vulcanization occurs when cis-1,4-polyisoprene cross-links with non-isoprene compounds, creating a molecular chain structure that has high tensile and tear strengths and great durability (Wei et al. 2019). Natural rubbers based on trans-1,4-polyisoprene are hard, show good resistance to biological degradation, and have non-conducting properties (Takeno et al. 2008). Other main (non-isoprene) compounds in natural rubber are proteins and lipids; these reduce curing time and decrease adhesion (Smitthipong et al. 2014). The isomeric compounds in natural rubbers are

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characterized with ^{13}C NMR (Lambert, Wu, and Santiago-Blay 2005) and IR and Raman spectroscopy (Haider 2012). GC-MS and FTIR are used to identify the organic compounds (Ley-Paredes et al. 2018).

Several plant families, generally tropical ones, produce latex. The trans-1,4-polyisoprene rubbers originate mainly from *Palaquium gutta* and *Manilkara bidentata* (Takeno et al. 2008). The cis-1,4-polyisoprene rubber production relies on several species of which *Hevea brasiliensis* is the most suitable for commercial use (Mooibroek and Cornish 2000; Venkatachalam et al. 2013). The exact function of latex is unclear; it can be an energy reserve, toxic metabolite excretion (Greve 2000), or a defense mechanism when tissue is damaged (Agrawal and Konno 2009).

Several traditional processing methods have been documented in South America and Asia. Production generally starts with incising the bark of a tree, after which latex flows into a recipient. Latex quality and yield vary between trees. After collection, the water in *Hevea brasiliensis* latex is evaporated over a fire (Ernst 1976). *Palaquium gutta* in South Asia is felled before being incised so the latex coagulates in the cuts and then is scraped off and formed into a ball. After immersion in hot water, the latex becomes plastic again and is cleaned and shaped for storing (Vander Laan 1927). However, the material is brittle and prone to cracking without processing (Hosler, Burkett, and Tarkanian 1999; Tarkanian 2003). An early example of such processing comes from Mesoamerican technology, where *Castilla elastica* latex was mixed with the juice from the *Ipomea alba* vine. After about 15 minutes of stirring, the sulphur from the vine juice cross-linked with the polyisoprene, and the latex solidified into a white viscous mass (Hosler, Burkett, and Tarkanian 1999). This vulcanized rubber is less brittle and more shock-absorbent than natural latex (Tarkanian and Hosler 2011).

In South Africa, a resinous latex from *Euphorbia tirucalli* mixed with beeswax was found on an about 42,000-year-old stone tool (La Nasa et al. 2020). This naturally toxic material is a well-known additive of ethnographic hunting poisons (Van Wyk and Gericke 2000). Latex in Mesoamerica was used as salves for ulcers and sores, as chewing gum, in medical drinks, burnt as incense, used in waterproofing, as a binder in body and other paints, as material for figurines, suppositories, flasks, and sandals, and to haft blades onto handles (Lattermann 2013; Tarkanian and Hosler 2011). In South Yucatan, Mayan engineers included natural rubber in mortars for the construction of the Principal Building at the Witzinah archaeological site (Ley-Paredes et al. 2018), most likely to improve the mortars' plasticity and strength.

Most of the evidence of ancient rubber from processed latex comes from Central America. Here, rubber balls were used in the game ōllamaliztli 3600 years ago (Ponciano Ortíz, Ma. del Carmen Rodrígez, and Jorge 2017). *Ipomoea alba* was probably added to induce coagulation of *Castilla elastica* latex, improving the material properties of the rubber (Hosler, Burkett, and Tarkanian 1999). Historically, natural rubber was collected and used by indigenous people in Central, South, and North America (Snow 2015; Tarkanian 2003) and the Caribbean (Curet, Newsom, and deFrance 2006).

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Rubber was a valued commodity, as exemplified by a natural rubber dating to 120 and 235 CE found in a closed tunnel under the Temple of the Feathered Serpent in Teotihuacan. *Castilla elastica* does not naturally occur here, and rubber was traded over several hundred kilometers (Gómez-Chávez et al. 2017).

Mortars

Mortar refers to any generic mixture of a solid binder and aggregates, suspended in water. Mortars are generally used to bind construction materials but were also applied in waterproofing and tool hafting (Cagnana 2000). Early mortars consisted of clay, bitumen, and unheated limestone or gypsum. Mortar composition varies depending on the base materials, geographic location, and function. As a result, the slurry can have a range of colors and textures.

Mortars can be defined through their hardening mechanism and the specific binders that instigate this process. Hardening through water evaporation in clays and limestone is the simplest process. Mortars can also harden through a reaction between calcium carbonate and carbon dioxide from the air, a chemical reaction between calcium sulphates and water, and finally by a chemical reaction between alumino-silicate binders when wetted (Artioli 2010b). In addition to binders, a wide range of fillers and other additives, including plant gums and fibers, sticky-rice soup, and volcanic ash, were used in the past. These additives influence the hydration and crystallinity, improving worktime, workability, strength, and flexibility (Artioli 2010b; Rampazzi et al. 2016).

The mineral composition and phases of mortars and plasters are characterized with optical microscopy, petrographic analysis, and SEM- and XRD-related methods. XRF, inductively coupled plasma MS, and FTIR are used to characterize the non-crystalline organic components. Because they contain microcrystalline and highly atomic disordered calcite, pyrogenic lime mortars can be differentiated with FTIR from non-heat-treated mortars (Artioli 2010b and references therein; Friesem et al. 2019).

Complex mortars are produced by heating limestone (800–900°C), gypsum (100–200°C), or alumino-silicate (1450 °C) binders to create a reactive material (Hughes and Valek 2003). To prevent fracture formation during curing and to increase durability, the composite generally is mixed with inert aggregates like sand. Fine aggregates with a particle size of less than 1 mm create mortars; when larger aggregates are added, it creates the construction material generally termed concrete. To produce a hydraulic mortar that sets under water, reactive aggregates containing silica and aluminum such as silica glass, volcanic ash, pumice, fine-crushed bricks or pottery, and metallurgical slag can be added to limestone and dolomite before heating (Artioli, Secco, and Addis 2019; Borsoi et al. 2019).

Portland cement is produced by heating limestone and alumino-silicate–containing clays at very high temperatures. This is subsequently powdered and mixed with gypsum to produce a highly reactive mixture called clinker. When this is mixed with water, a series of chemical

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reactions take place that create an interlocked grid of crystalline and amorphous phases that give the material a high mechanical resistance (Artioli, Secco, and Addis 2019 and references therein; Moropoulou, Bakolas, and Anagnostopoulou 2005).

Lime and gypsum mortars are archaeologically and historically the most common. Cement mortars are a more recent invention, appearing first during Roman times and again later in the 18th-century (Artioli 2010b). By 16,000 years ago at Lagama North VIII in the Levant, lime plaster was used to haft stone microliths (Bar-Yosef and Goring-Morris 1977) and similar use of lime-based adhesives is documented at other Natufian sites (Friesem et al. 2019). From the Paleolithic to Neolithic transition onwards, lime plasters were produced in the Levant to cover domestic surfaces and surfaces in mortuary contexts. However, not all mortars found are completely pyrogenic; they are part untreated calcium carbonate, which generally was mixed with local soil, dung, burnt bone, and plant remains. These additives created a durable material that was simpler to make than a similar volume of quick lime (Friesem et al. 2019). At the site of Gesher, a mud plaster containing silt, iron oxide, quartz, and vegetal material was used to haft a flint crescent around 11,000 years ago (Shaham, Grosman, and Goren-Inbar 2010). About 4000 years ago in Poland, a calcite putty was used to hold an ornament together (Rumiński and Osipowicz 2014). Gypsum and lime plasters were used in Pharaonic Egypt in monumental architecture for construction, decoration, and coatings (Coppola, Taccia, and Tedeschi 2013; Regourd et al. 1988). The use of mortar with hydraulic proprieties dates to the Late Bronze Age of Cyprus, where fine-crushed ceramic fragments were added to the lime paste (Theodoridou, Ioannou, and Philokyprou 2013). These artificial reactive materials were replaced mainly with Santorini volcanic sand by Greeks (Moropoulou, Bakolas, and Anagnostopoulou 2005).

The use of mortars as a structural binder widely increased during Roman times (Figure 1A). Roman engineers optimized the use of hydraulic mortars, mixing them with artificial pozzolan materials (crushed ceramics) for terrestrial structures (cisterns, baths, fountain, and aqueducts). For structures continuously exposed to water, they used a mixture of lime plaster and highly reactive volcanoclastic materials, mainly volcanic ashes from Pozzuoli, Italy (Artioli, Secco, and Addis 2019; Cagnana 2000; Hughes and Valek 2003). The standardized production of high-quality hydraulic mortars and cement rapidly disappears after the end of the Roman Empire. This process was rediscovered during the Renaissance and further developed during the 18th and 19th centuries until the adoption of modern cement (Artioli, Secco, and Addis 2019; Hughes and Valek 2003).

In Central America, lime mortars were used in Mayan monuments as structural binders and for decorative purposes (Hansen, Rodriguez-Navarro, and Hansen 1996). In India, lime plasters were used from at least the fourth millennium BCE and their use increased toward the end of the first millennium BCE (Artioli, Secco, and Addis 2019; Singh, Vinodh Kumar, and Waghmare 2015). The production of lime mortars in China started in the third millennium BCE. There, distinctive inorganic–organic lime mortar mixes, like sticky-rice lime mortar, were developed to improve strength, water resistance, and durability (Zeng, Zhang, and Liang 2008).

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Protein-Based Adhesives

Protein-based glues get their adhesive strength from the internal cross-links when the heavily coiled chains of amino acids are broken. When this happens, the highly polar groups on the amino acids are freed and can interact with polar adherends, such as wood (Bye 1990; Conner 2001). The preparation differs somewhat per source material. Protein glues are identified with antibody identification methods, such as ELISA (enzyme-linked immunosorbent assay), fluorescence methods, GC-MS, and proteomics (Giuffrida, Mazzoli, and Pessione 2018; Hendy, Warinner, et al. 2018; Kuchova, Hynek, and Kodicek 2009).

Protein glues bond well with wood and paper and were widely used in historic paper- and woodworking (e.g., Lambuth 2003; Oh 2019; Van der Werf et al. 2017; Van Geel et al. 2018). They were common paint binders (e.g., Chambery et al. 2009; Ma et al. 2017). However, their poor preservation probably explains why we find so few protein glues in the archaeological record (Kozowyk, van Gijn, and Langejans 2020).

Animal Glues

In animal glue, collagen proteins are obtained from animal hides, bones, and connective tissues. To break the internal coiled structure of the large proteins, they are heated in water to about 60°C. Temperatures higher than about 79°C will lead to a rapid loss in glue strength (Schmidt 1991). Animal glue is best applied as a thin film and when allowed to dry slightly before connecting the adherends. Collagen-based animal glues deform when heated and are more sensitive to water compared with other protein glues. This, however, allows the bond to be realigned after rewetting. In addition, water resistance may be invoked by heating the glue for a short time at temperatures over 100°C (Brandis 1990). Animal glues are insoluble in oils, waxes, and alcohol. Fish glue, like sturgeon bladder glue, is produced using methods similar to animal glue production. These glues can be made in conventional cooking vessels (Piezonka et al. 2020), which may complicate the accurate identification of production vessels. All protein glues are prone to microbial decay and generally have a short shelf-life (Lambuth 2003). Historically, there exist numerous unique uses for animal glues manufactured in specific ways or from different raw materials. For example, “mouth glue” is a type of animal glue produced to avoid any unpleasant taste, so that it can be softened for application by chewing and mixing with saliva (Duhamel du Monceau 1771).

Animal glue, probably made from cattle or sheep hides (or both), was used to glue cherry bark decorations on a 5000-year-old Neolithic yew bow from Switzerland (Bleicher et al. 2015). In Ancient Egypt, animal glue formed part of a plaster named gesso, used as a ground layer for painted or gilded decorations on wood. A glue ingot dating back to late second millennium BCE indicates that glues were prepared on a large scale and stored prior to use (Lucas and Harris 2012). Republican Roman legionary shields were made from three layers of beech, glued together with animal glue. In some cases, a protective leather outer layer was glued to the wooden shield. It is likely that the described leather covers were needed to protect the sensitive animal glues from the elements. In the Levant, the addition of bitumen to this plywood adhesive may have improved its water resistance (Buckland 1978; Feugère 2002; Sim

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and Kaminski 2012; Stiebel 2007). Fish glues were used in Hellenistic and Roman medicine (Scarborough 2015) and as binders in artwork, manuscripts, and polychrome objects (Dallongeville et al. 2013). Bovine, rabbit, and fish glues were used to secure gilt work in 18th-century Italy (Dallongeville et al. 2011). In a royal tomb of the Western Han dynasty (206 BCE–8 CE) in China, hide glue was found binding the leg and body of a terracotta horse (Wei, Ma, and Schreiner 2012). Protein glues were often used to secure inlays, particularly in woodworking. Example are the 3500-year-old bone inlays in a wooden shaman staff in China (Rao et al. 2015) and the use of either cow hide or soybean glue on 18th-century Japanese lutes with ivory, pearl, boxwood, and sandalwood inlays (Nakamura and Naruse 2016). Ethnographically, fish glue is known to have been used in arctic regions to bond wooden composite bows together (Lepola 2015).

Casein, Soybean, and Blood Adhesives

In blood, casein, egg, and soybean adhesives, the main ingredients are very large and globular protein molecules consisting of amino acids. These adhesive types are generally a sticky liquid. In order to obtain the casein protein from milk, a weak acid like vinegar can be added or milk can be left to sour. The curds that separate from the whey are casein. The casein is washed, dried, and ground and then dissolved in water before proteolysis. For soy-based adhesives, relying on a mix of 18 soy proteins, the beans are dried, ground, and rehydrated. Blood glues, consisting mainly of albumin and globulin proteins, prior to industrial drying, were processed in fresh liquid form (Lambuth 2003). An alkaline material, like hydrated lime or sodium hydroxide, is added to the dissolved casein, soy, or blood proteins. The hydrolysis reactions break the cross-links between the chains of the amino acids (Bye 1990; Conner 2001). Protein glues can be hot- or cold-cured and are strongest when pressure is applied. As the water evaporates, the glues harden, and through chemical reactions at room temperature the proteins become insoluble.

At Border Cave in South Africa, casein was found as a binder, possibly in a plaster-type glue, in a 49,000-year-old ochre crust attached to a stone tool (Villa et al. 2015). At Nahal Hemar Cave in Israel, about 10,000-year-old baskets may have either stored, or been coated in a medicinal collagen–plant protein mixture (Solazzo et al. 2016). Protein glues make good binders; for example, blood likely was added to increase the bond strength in some complex Chinese Qing dynasty lime mortars used in wood constructions (Fang et al. 2014). Blood was used in the arctic for gluing the seams of wooden bowls together during manufacture, to repair soapstone objects, and together with oil to strengthen and seal ceramic vessels. It was so favored that dried blood was kept on hand to be used whenever needed (Harry et al. 2009).

Resins

Resins are exudates secreted by plants and insects generally for protection. Resins are water-insoluble and consist of hydrocarbons like terpenoids, toluene, and waxes (Colombini and Modugno 2009; De Silva et al. 2009). When FTIR is used, resins can be differentiated into broad groups based on terpenoids and phenolic compounds (for plant resins) and polyesters

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and waxes (for insect resins). The three main characterizing groups for plant resins are diterpenoids, triterpenoids, and aromatic esters/phenolics (Martín Ramos et al. 2018). GC-MS can be more specific regarding species, production methods, and stages of refinement (Colombini and Modugno 2009; Derry 2012). Before application, resins are softened by heat or dissolved in oils. Extensive heating removes volatiles and hardens the material. Sometimes, resins then are mixed with plasticizers like beeswax and animal fat to make them less brittle.

Insect Resin

Insect resins or lacs are excreted by Keriidae insect family members as they forage the branches of trees. Lac-producing insects are known from Southeast Asia, the desert regions of the southern USA, and northern Mexico (Bisulca, Pool, and Odegaard 2017). Insect resins consist of polyesters and waxes, and the chemistry is specific to the insect species and plant host.

To produce insect resin, branches with lac are collected and the resin is scraped off (Stacey, Heron, and Sutton 1998). In India, the “sticklac” is crushed, screened, winnowed, washed, and dried. The now “seedlac” is further purified by hot-sieving it in a dense cloth near a coal fire, creating shellac (Sharma 2017). The wax content of sticklac is higher than that of shellac and this may leave sticklac somewhat more plastic. In historic times, shellac was refined further through bleaching. Insect resin can be applied as a varnish by rubbing it onto a surface. It can also be heated and applied in a semi-molten state (Derry 2012; Stacey, Heron, and Sutton 1998).

Insect resins often were used as a varnish on wooden objects, gildings, and paintings in historic Europe and eastern Asia (Colombini and Modugno 2009; Stacey, Heron, and Sutton 1998). In the American Southwest, prehistoric Hohokam used insect resins to glue jewelry inlays and haft arrows and repair and glue pottery. It is likely that they started trading resin with neighboring Mollogong and Ancestral Pueblo people from around 1000 CE. From around this time, Mollogong people preferentially used insect resins over tree resins for arrow hafting (Bisulca, Pool, and Odegaard 2017).

Plant Resin

Plants secrete resins to protect themselves from herbivores, dehydration, pathogens, and environmental damage caused by events such as fires and storms (Colombini and Modugno 2009; Seyfullah et al. 2018; Stacey, Cartwright, and McEwan 2006). Plant resins consist of volatile and non-volatile terpenoids, including resin acids, and/or phenolic compounds that are lipid-soluble and water-insoluble as well as other sometimes characteristic compounds like toluene in spinifex resin (Colombini and Modugno 2009; De Silva et al. 2009; Langenheim 2003).

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Resins are collected directly from plants, which may be purposefully wounded to induce secretion. Many plants produce resins, archaeologically most notably conifers of the *Pinaceae* and deciduous trees of the *Burseraceae* families. Some specific examples are Scots pine (*Pinus sylvestris*), Norway spruce (*Picea abies*), balsam fir (*Abies balsamea*), European larch (*Larix decidua*), yellowwood (*Podocarpus elongatus*), gumbo limbo (*Bursera simaruba*), and frankincense (*Boswellia sacra*) (Langenheim 2003). The production of resin from mastic trees (*Pistacia lentiscus*) is well known from historic times and has clear harvesting conventions. Similar practices are known from, for example, North and East Africa (Al-Harrasi et al. 2019; Bakirtzis and Moniaros 2019 and references therein; Tadesse, Desalegn, and Alia 2007). The trunks and branches are scored several times per week to induce resin flow. After the mastic has dried, it is collected, sieved, and sorted from soil and dust and finally washed and completely dried (Bakirtzis and Moniaros 2019). Native North Americans collected spruce resin and processed it by dropping it in boiling water for waterproofing canoes, a job that has been recorded ethnographically as being conducted primarily by women (Waugh 1919). The resin floated and separated from large impurities, after which it was skimmed off. Fat, charcoal, and sometimes ochre were added to spruce resin to make it less brittle (Adney and Chapelle 1964). Ethnographic accounts indicate that South and Central American resins sometimes were “cooked” to obtain turpentine and pitch (Sahagún in Stacey, Cartwright, and McEwan 2006, 233).

A variety of ways of collecting, processing, and using resins in Australia exist depending on the plant species, technical knowledge, and local culture (Matheson and McCollum 2014). Among the Alyawara in Central Australia, resin was collected by thrashing spinifex grass (*Triodia* sp.) with a stick against a large flat rock, collecting the resin dust on a sheet of bark, and gently heating it over a fire and rolling it with heated rocks into a solid resin tablet (Binford 1984). Spinifex (*T. pungens and T. irritans*) may have been the preferred plant sources of resin in prehistoric Australia (Bradshaw 2013; Parr 1999; Pitman and Wallis 2012). However, other resin sources, including *Erythrophloem chlorostachys*, *Myoporum platycarpum*, *Callijtrus collumellaris*, and *Xanthorrhoea* sp., were used when available (Bradshaw 2013; Langenheim 2003; Parr 1999) (Figure 1B). The specific botanical origins of different resins in Australia can be used to determine the trade and exchange networks of past peoples (Matheson and McCollum 2014).

Fossil resins generally are older than 40,000 years and result from the loss of essential oils and the natural polymerization of resin acids. Sub-fossil resin or copal or copalite dates back to the Quaternary; it is not to be confused with fresh South and Central American “copal” resins, which are mostly *Bursera* sp. excretions. Amber is older and completely polymerized (Case et al. 2003; Seyfullah et al. 2018). Fossil resins are very hard and are more difficult to dissolve and soften than fresh resins. They are generally used in varnishes and less as an adhesive (Colombini and Modugno 2009). Amber was a prized material around the globe and was part of many trade networks. For example, the trans-European network that flourished between 1550 and 1100 BCE included Nordic amber along with Cornish tin, South German copper, Carpathian salt, and Mediterranean wine and olive oil (Kristiansen and Suchowska-Ducke 2015).

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Resins are perhaps the most widespread adhesives in prehistory; 64,000 years ago, *Podocarpus* sp. resin, sometimes loaded with ochre and possibly quartz and burnt bone, was used to haft stone tools in South Africa (Charrié-Duhaut et al. 2009; Villa et al. 2015). Neanderthals (Degano et al. 2019) and European Mesolithic hunter-gatherers (Croft et al. 2018) used resins to haft stone tools. North American Paleoindians used resins mixed with ochre and fossil resins to haft tools and repair canoes (Helwig et al. 2014; Kurtz 1997; Tankersley 1994). In Borneo, bone and stingray spine points were hafted with fibers and probably a resin-based adhesive around 10,000 years ago (Barton et al. 2009). In Argentina, projectile points dated to between 3900 and 1900 BCE showed hafting residues associated with resins from *Cercidium praecox* (Cattáneo et al. 2018). In pre-Columbian Central and South America, fresh "copal" resins were used as inlays, to create figurines, as an adhesive in mosaics and to repair pottery, and for embalming. Also, in ceremonies it was used as incense, either to burn or as an offering (Kaal et al. 2020 and references therein; Stacey, Cartwright, and McEwan 2006).

European Iron Age people used resin to waterproof and repair pots (Rageot et al. 2016). In Asia, resins were used as an adhesive and sealant to coat ceramic jars and seal lids onto pots and as a caulking in boats (Burger et al. 2011; Gianno et al. 1987). In Australia, resins were used 3000 years ago and possibly as far back as 40,000 to 50,000 years ago for a number of different tasks, including hafting stone tools, waterproofing baskets, caulking boats, and making ornamental items (Allen and Akerman 2015; Bradshaw 2013; Gamage et al. 2012; Maloney et al. 2015; Powell, Fensham, and Memmott 2013). Chinese and Japanese lacquers, often referred to simply as saps, are a complex resinous blend consisting mostly of phenolics, gum, and water obtained from lacquer trees (*Anacardiaceae*), specifically *Toxicodendron vernicifluum* and *Rhus verniciflue* (Langenheim 2003; Wu et al. 2018). Known primarily for its use as a surface finish or paint binder in later times, lacquer was also used to repair a Neolithic canoe and to fix broken ceramics at the 8000-year-old site of Kuahuqiao, China (Wu et al. 2018).

Tar

Tars are a black or dark brown viscous liquid. They are formed by the thermal decomposition of cellulose, hemicellulose, and lignin in plant material, which produces aromatic hydrocarbons, acetic acid, methanol, ketones, and phenols (Collin and Höke 2005). Tars are complex mixtures with at least 200 different components that vary depending on the material source and production method (Betts 2000; Kurt and Işık 2012). Tars are water-insoluble and oil- and terpene-soluble. Pitches often refer to the solid or semi-solid portion. Confusingly, pitch also is used to refer to tapped resin from certain tree species, such as the "pitch pine," and the term "tar" is preferred. Tars get their name from the source material that is used to create them (for example, coal tar, birch bark tar, and wood tar).

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Birch Bark Tar

Like other tars, birch bark tar is a viscous black-brown liquid containing a complex mixture of hydrocarbons. It is produced through the controlled heating or destructive distillation of bark from trees of the genus *Betula*. FTIR and GC-MS are the main characterization methods. Here, triterpenoid molecules with a lupane skeleton, which are present in raw birch bark are identified. These molecules include betulin, betulinic acid, lupeol, and their derivatives betulone, lupenone, lupa-2,20(29)-diene, and lupa-2,20(29)-dien-28-ol (Aveling and Heron 1999; Colombini and Modugno 2009). Depending on the preservation, the presence or absence of fatty and triterpenic acids, deriving from the suberic polymer of the bark, sometimes can be used to differentiate tar production methods (Rageot et al. 2018).

A number of different techniques may have been used to produce tar in the past. As only a few historic tar production structures are known, prehistoric reconstructions rely on ethnographic and experimental data (Itkonen 1951; Kozowyk et al. 2017 and references therein). Primarily, birch tar is produced by heating bark in a closed or reduced oxygen environment, although it can also be collected by condensation on a flat, overhanging rock surface (Schmidt et al. 2019). In pre-ceramic societies, a reducing environment can be created by placing the bark under a smoldering pile of ash and embers, inside a small pit in the ground, or enclosed in an earthen oven-like structure (Kozowyk et al. 2017). After the advent of ceramics, tar could be produced more easily by either single- or double-pot production techniques (Rageot et al. 2018).

At the Italian site of Campitello Quarry, birch bark lumps were found attached to flint flakes dating to about 200,000 years ago (Mazza et al. 2006). Similar finds from the Paleolithic show that Neanderthals used birch bark tar to back flint flakes, making them easier to handle (Niekus et al. 2019), possibly also attaching them to wood handles (Koller, Baumer, and Mania 2001). Birch bark tar was used as an adhesive for hafting tools and repairing pottery vessels, as a waterproofing agent, and sometimes for decorative elements across Europe throughout the Mesolithic (Aveling and Heron 1998), Neolithic (Lucquin, March, and Cassen 2007; Mitkidou et al. 2008; Osipowicz et al. 2020; Pesonen 1994; Regert 2004; Urem-Kotsou et al. 2002; Van Gijn and Boon 2006), Iron Age (Regert 2004; Regert et al. 2003; Reunanen, Holmbom, and Edgren 1993), Roman period (Charters et al. 1993; Regert et al. 2019), and Middle Ages (Stacey et al. 2020). Birch tar appears rarely outside Europe despite its useful material properties which allow it to remain effective at low temperatures and be reused multiple times (Kozowyk and Poulis 2019). However, it was identified in a ceramic vessel from northern China (dating to 4000 to 3500 years ago), where it may have been used on composite tools (Rao et al. 2019). It is unclear why birch tar has not been found in, for example, North America, where birch trees were used extensively by Paleo-Indians.

Wood Tar

Wood tars are generally distilled from trees from the *Pinophyta* or conifer order that are naturally rich in resin, although in some cases tar is collected as a biproduct of charcoal manufacture using deciduous trees (Ciesla 2002). FTIR and GC-MS can be used to identify

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wood tar through the presence of stable compounds originating in wood and bark, which are formed during tar production and are absent in natural resins. Examples are products of the thermal decomposition of lignin, oxygenated benzenes, other aromatics like retene, hydroxyls, and methyl esters, and the absence of oxidized resin acids/colophony markers (Bailly et al. 2016; Egenberg et al. 2002; Font et al. 2007).

Much of what is known about making tar comes from literary and historic sources (cf. Bent 1698; Langenheim 2003 and references therein). Archaeological evidence includes funnel-shaped pits found in Sweden dating to the Roman Iron Age (Hjulström, Isaksson, and Hennius 2006) and oven and pottery sherds related to tar production and collection from Medieval Germany (Beck et al. 1998). Tar production became more widespread from the 15th century onwards, reaching its height in the 18th and 19th centuries in eastern Europe and the Baltic and consisting later of more brick kilns (Surmiński 1997). In the Białowieża forest in eastern Poland, the density of 18th-century wood tar kilns is estimated to range between 2 and 6 per 100 km^2 (Samojlik et al. 2013).

Wood tar production systems can be either autothermic or allothermic. In autothermic processes, the raw material is also the fuel that generates the heat needed to distil tar. These systems generally consist of a pit or ditch with a container or channel at the bottom to catch the tar. The pit is filled and capped, often with conifer wood, which is set on fire and then covered with sods. Inside the tar pit, the hot wood smolders and tar forms. To prevent tar from charring, it is produced in a reducing environment; however, autothermic distillation requires some oxygen to keep the wood smoldering. Traditional/historic examples are Scandinavian dales and tar graves and Turkish kurna; the latter system dates back to at least the late Roman Period (Egenberg et al. 2002; Hjulström, Isaksson, and Hennius 2006; Kurt, Suleyman Kaçar, and Isik 2008).

Allothermic production uses an external fuel supply to heat wood held in an isolated container, such as a small ceramic or metal pot, an oven-like earthen structure, or a large brick or metal oven or kiln (Beck 1996; Kozowyk et al. 2017; Surmiński 1997). In central-southern France during the Roman period, tar was produced in a “dolium,” a ceramic container consisting of an upper compartment to house the wood and a lower compartment to collect the tar (Loewen 2005). Oxygen supply is limited inside the container, ensuring that tar is formed and collected instead of combusting. Kilns also allowed for the collection of more wood alcohol and turpentine, which normally is lost through evaporation in tar pits (Kunnas 2007).

Wood tar was used to impregnate fish nets, coat or waterproof ceramics, repair broken pottery, haft arrowheads, and caulk ships (Pietrzak 2012 and references therein). Tar was produced from the yellowwood tree (*Podocarpus elongatus*) at Border Cave around 43,000 to 40,000 years ago. Pine wood tar dates to the Late Neolithic in Greece, where it was used to glue and waterproof ceramic vessels around the start of the fourth millennium BCE (Urem-Kotsou et al. 2018). Wood tar has been identified on an archaeological ink stick of the Eastern Jin period (317–420 CE) in China (Wei et al. 2012) and in a mummy embalming fluid from 2500 years ago at Deir el-Bahari, Egypt (Koller et al. 2003).

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The most common application of wood tar was waterproofing and caulking of ships and boats. An early example dates to about 2500 years ago in Israel, where it was found on keel and hull planking (Connan and Nissenbaum 2003). Pine wood tar has also been identified as caulking on Etruscan shipwrecks and the English Tudor ship the *Mary Rose* (Robinson et al. 1987). Tar was such an important commodity during the 15th to 19th century for the trans-Atlantic shipping industry that the French government brought expertise, thus transferring knowledge, from the Baltic to France and Canada to boost production (Loewen 2005).

Additives

Additives are materials mixed with adhesives to modify properties such as plasticity and rheology, to inhibit oxidation and degradation, and to increase volume to lower costs. Wax was used as a plasticizer in Stone Age hafting adhesives in South Africa (La Nasa et al. 2020). Ground (burnt) bone was used by Stone Age people in Africa and Europe (Bradtmöller et al. 2016; Charrié-Duhaut et al. 2013). Plant fiber was added to Levantine Epi-Paleolithic mud mortar and Roman cement, and sticky-rice soup was added to Chinese mortar. These additives, like lignin products added to modern cements, increased strength and workability (Moropoulou, Bakolas, and Anagnostopoulou 2005; Yang et al. 2009). Calcium carbonate was added to animal glues used in the 2000-year-old Han dynasty terracotta army (Wei, Ma, and Schreiner 2012); and volume fillers, such as sand, are known from Australian spinifex hafting adhesives (Dickson 1981). Furthermore, many additives serve a combination of functions.

Beeswax

Beeswax is a natural wax or lipid synthesized by honeybees (*Apis* sp.) for the construction of their honeycombs. Most productive are *Apis mellifera* and *A. cerana* (Tulloch 1980); however, many other members of the Apidae family also produce wax. The major components found in beeswax are alkanes, alkenes, free fatty acids, monoesters, diesters, and hydroxy monoesters (Hepburn, Pirk, and Duangphakdee 2014), and wax generally is identified with GC-MS techniques (Garnier et al. 2002; Heron et al. 1994). Beeswax has a melting temperature of about 66 to 71°C and remains relatively soft at room temperature. It is used as a plasticizer and rheology modifier to improve workability and soften otherwise brittle materials (Kozowyk and Poulis 2019; Kozowyk, Langejans, and Poulis 2016).

Bee hunting and keeping for honey and wax are well known from (pre)history and ethnography (Crane 1999), but the use of wax as (part of) an adhesive is not widely documented as the chemical differentiation from natural waxes can be difficult. However, at Border Cave in South Africa, lumps of beeswax and beeswax additives in a hafting adhesive were dated to 43,000 to 42,500 years ago (d'Errico et al. 2012; La Nasa et al. 2020). Roughly contemporaneous beeswax may be present in resin-based adhesives from Fosselone Cave in Italy (Degano et al. 2019). Beeswax has also been identified as being mixed with pine resin and birch bark tar during the European Paleolithic and Early Iron Age (Degano et al. 2019; Rageot et al. 2016; Regert 2004). It is likely that, by the Neolithic, the honeybee was being widely exploited (Roffet-Salque et al. 2015) and the range of uses expanded. For example,

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beeswax figures are found in Australian rock art (Nelson et al. 1995; Watchman and Jones 2002) and beeswax was used in a Neolithic dental filling (Bernardini et al. 2012). Beeswax was used in the lost-wax method in metal casting (Bray 1978; Quezada-Euan et al. 2018), such as for golden objects from the pre-Columbian burial site at El Caño, Panama (Kaal et al. 2020).

Fats and Oils

Fats and oils are water-insoluble storage compounds used by organisms to provide energy for cell growth (Firn 2010). The general structure of fats and oils consists of triglycerides composed of a glycerol backbone joining three fatty acids. Generally, terrestrial animal fats (tallow) are more saturated and contain primarily palmitic, oleic, and stearic acids, making them solid at room temperature. Plant oils have relatively low levels of saturated fatty acids and are composed predominantly of palmitic, oleic, and linoleic acids and are liquid at room temperature (Pollard and Heron 2008). Drying oils are further distinguished from non-drying oils and the former harden through surface oxidation (Petrie 2000). Drying oils can be recognized by their high content of dicarboxylic acid in relation to long-chain acids (Colombini and Modugno 2009).

Fats and oils have a wide variety of uses, including waterproofing boats and animal skins (Adney and Chapelle 1964) and serving as a binder for paint (Rudner 1983) and a flux in solder (Fell 1982). They are also versatile additives in adhesives by, for example, lowering the melting point and decreasing viscosity (Pollard and Heron 2008). Animal fats were mixed with birch bark tar for repairing a ceramic vessel in Roman Britain (Dudd, Evershed, and Gibson 1999) and used in hafting during the Mesolithic in Europe (Leito, Kriiska, and Vahur 2011). Holocene, and possibly Middle Stone Age, hunter-gatherers in Africa used fat in combination with *Podocarpus* sp. resin for hafting (Charrié-Duhaut et al. 2013, 2016). Drying oils can also be the main constituent of an adhesive when the bond is subject to low levels of stress, such as the linseed oil–based adhesive used for gilding decorations at the Takht-e Soleyman Palaces in Iran (Doménech Carbó et al. 2010).

Ochre

Ochre to refers to earth pigments rich in iron oxides. The main ingredient of “red ochre” is hematite (Fe_2O_3), and “yellow ochre” consists of limonite, an assortment that includes hydrated iron hydroxide ($FeO(OH)\cdot nH_2O$). Hematite can be artificially produced by heating goethite (“brown ochre,” FeOOH) at relatively low temperatures (280–350 °C) (de Faria and Lopes 2007; Sajó et al. 2015). Ochre generally is identified with XRF and Raman spectroscopy.

The addition of ochre to an adhesive affects the rheology, increases strength, and decreases tack to improve workability (Kozowyk, Langejans, and Poulis 2016; Wadley 2010). Although there is ample evidence that ochre was a highly valued pigment used for symbolic purposes, early evidence from South Africa demonstrates that ochre was used selectively depending on the type of stone tool, perhaps to alter adhesive characteristics in response to different adherend surfaces (Lombard 2007). Ochre pieces were probably ground, crushed, or scraped

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to a powder when used as additives. The powder then was mixed with various substances (plant gum/resin, animal fat, wax, and animal products) to produce compound adhesives but also paint, body lotions, and cosmetics (Hodgskiss and Wadley 2017; Villa et al. 2015).

From 200,000 years ago onwards, the number of ochre finds increased in Africa. In contemporaneous Europe, Neanderthals also used ochre (Roebroeks et al. 2012; Wadley 2015). Although numerous sites provided worked ochre pieces, ochre powder, and ochre-processing residues on grindstones, tools, and shells (Dayet et al. 2013; Henshilwood et al. 2011; Hodgskiss 2013; Hodgskiss and Wadley 2017; Rosso, Pitarch Martí, and d'Errico 2016), evidence for its use as an additive is scarce. Ochre stains on tools generally are accepted as preserved remains of ochre-loaded hafting adhesives (Figure 1D). These compound adhesives thus may have a long history of use; the earliest observations are from 120,000 years ago from North Africa (Rots, Van Peer, and Vermeersch 2011; Schmidt et al. 2015). Moreover, ochre stains on tools are quite common in the Paleolithic of Africa, Europe, the Levant, and North America (Dayet et al. 2017; Dayet et al. 2019; Henry and Shen 1995; Leroi-Gourhan and Allain 1979; Lombard 2006; Shaham, Grosman, and Goren-Inbar 2010; Wadley 2015 and references therein; Wojcieszak and Wadley 2018). Chemical analysis on stone tools from Paleo-Indian North America, Middle Stone Age South Africa, and Aurignacian Europe confirms that resins were mixed with ochre to create compound adhesives (Bradtmöller et al. 2016; Helwig et al. 2014; Rots et al. 2017).

Quartz

Quartz (SiO_2) is a tectosilicate and highly stable mineral, commonly found in igneous, metamorphic, and sedimentary rocks, particularly sandstone (Cleary and Conolly 1971; Götze 2009). This common mineral is found from epithermal to alpine systems, in hydrothermal veins, and as residual mineral in soils and sediments (Anthony et al. 2001). Quartz properties depend on the environmental and thermodynamic conditions during mineralization. Sometimes, quartz crystal can incorporate ions of Al, Ti, Ge, Fe, H, Ag, Cu, and P, considerably changing its structural, electrical, and optical properties (Götze 2009). Quartz is characterized by using petrographic methods.

Quartz sands can be added as a loading agent in adhesive mixtures, where it influences the rheology and acts as a filler. Experimental studies suggest that quartz grains, particularly in the size range of silt and clay, improve adhesive shear strength (Zipkin et al. 2014). A hafting adhesive on a quartz flake dated to at least 56,000 years ago from Diepkloof Rock Shelter (South Africa) shows the presence of quartz crystal and bone fragments (Charrié-Duhaut et al. 2013; Miller, Goldberg, and Berna 2013). Archaeological mortars used in hafting and construction in the Anatolian peninsula (Miriello et al. 2011), North Africa (Gliozzo et al. 2009), Central America (Goodall et al. 2007; Hansen, Rodriguez-Navarro, and Hansen 1996), South America (Zanchetta et al. 2020), and Asia (Ma et al. 2018) often include quartz sands.

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What Adhesives Reveal

Adhesives were, and are, used around the globe in domestic contexts, in large-scale construction, and in weapons for hunting and warfare. The reason for using a specific adhesive can be related to the access, availability, and abundance of raw materials and to needs, knowledge, and sociocultural practices (Doelman and Cochrane 2012). These constraints can offer insights into the larger context of the adhesive find, such as mobility, trade, environment, and technology. To unravel these constraints and larger contexts, adhesive finds first must be characterized. Methods like ancient DNA analysis and enzymatic digestion of plant gum sugars coupled to MALDI-TOF MS and MS/MS analysis may further hone the characterization of adhesive materials *and* their makers (Granzotto et al. 2019; Jensen et al. 2019). The chemical make-up (GC-MS) and crystalline structures (spectroscopy) of adhesives are imperative for the identification of ingredients and their sources. However, to unlock the potential of adhesives as an information source, these analyses are best applied to large archaeological and comparative assemblages.

For example, there is ethnographic evidence that spinifex and other resins were collected and traded across Australia for use in various functions (Aiston 1929; Pitman and Wallis 2012 and references therein). Although many of the resin-producing species are region-specific and despite the presence of large museum collections, these ethnographic accounts on trade remain mostly anecdotal and lack details (Powell, Fensham, and Memmott 2013). There are also cases where adhesive production appears to be organized along gender lines where women make spinifex adhesives and men apply them to their tools (Binford 1984). A detailed chemical study on the sourcing of botanical, ethnographic, and archaeological resins, possibly coupled with ancient DNA, will lead to a wealth of information on trade, division of labor, function, and prestige (Parr 2002).

A parallel example comes from Europe, where historical information shows the interconnectedness between pine tar, war, espionage, and colonialism/empire-building (Loewen 2005), but material analysis is lacking. Owing to international trade and the rise of colonialism, the European tar demands for ship-building increased greatly from the 16th century onwards. Dutch merchants encouraged clear cutting in the Baltic and using the dead, resinous rich wood to produce vast amounts “total tar.” This impacted the tar market and resulted in further tensions between France, England, and Holland at the time of the Anglo–Dutch Wars of the late 17th century (Wilson 2012). To compete, the French and English attempted to transfer and partly replace Swedish tar technology from the Baltic to their North American colonies and parts of France. France’s attempt at purchasing the skills of Swedish tar makers resulted in the accidental hiring of a double agent, who nearly ruined French tar manufacture (Loewen 2005). A detailed chemical analysis would provide data on the location, source, and production methods of tars from this age. It would fine-tune historic knowledge and shed new light on past events. Heritage conservation would also benefit because the exact recipe for tar used on wooden Nordic buildings, mainly churches, is lost (Lindblad, Fredrikson, and Källbom 2021). This historic case study and other well-known examples, such as Mesopotamian bitumen and East African “copal” (Crowther 2012; Forbes 1955; Schwartz

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and Hollander 2016), emphasize the importance of natural polymers in the past. It is not unthinkable that similar large networks and politics were involved in the Neolithic trade of birch tar in the Mediterranean (Rageot et al. 2021), the Olmec bitumen trade in Mesoamerica (Wendt and Cyphers 2008; Wendt and Lu 2006), and also in deeper prehistory.

Adhesives can also be characterized in terms of material performance through field testing of their production and use and laboratory testing of properties, such as strength, durability, and viscosity. Such large-scale experimental datasets are lacking for most adhesives yet are highly informative about prehistoric technological knowledge and research biases. For example, field testing (Gaillard et al. 2016; Wadley, Hodgskiss, and Grant 2009) and standardized laboratory testing (Kozowyk, Langejans, and Poulis 2016; Zipkin et al. 2014) showed that ochre has a functional role, increasing adhesive strength and workability, and that the recipes for effective compound adhesives are narrow and adhesive makers must have worked meticulously and skillfully. Experimental archaeology has provided much-needed technological data on the production complexity of birch tar by Neanderthals (Blessing and Schmidt 2021; Kozowyk et al. 2017; Osipowicz 2005; Ossendorf et al. 2019; Schenck and Groom 2016). It has also shown that birch tar both preserves and performs better than other materials (Kozowyk and Poulis 2019; Kozowyk, van Gijn, and Langejans 2020) and that Paleolithic ochre stains indeed may be the preserved remnants of ochre-loaded adhesives (Figure 1D) (Kozowyk, van Gijn, and Langejans 2020).

Conclusion: An Archaeology of Adhesives

Adhesives come in a multitude of forms with widely varying properties, a plastic *avant la lettre*. They can be tacky, pliable, water-resistant, elastic, brittle, fluid, viscous, clear, dark, and much more. Almost all of these properties can be tweaked by mixing ingredients or by further processing each adhesive, and often several properties are exploited in one application. Archaeology has shown us how these materials have been altered and exploited in all forms throughout human history. For example, resin was used to waterproof *and* reinforce baskets (Bisulca, Odegaard, and Zimmt 2016), it was mixed with ochre and possibly fat for hafting stone tools (Helwig et al. 2014), and it could be distilled to make incense for trade and ceremony (Stacey, Cartwright, and McEwan 2006). Few materials are as uniquely versatile in the archaeological record or the present day.

Moving beyond the study of single objects, adhesives offer insights into past societies and tell a story about technology and ingenuity: how Mesopotamian bitumen became a preferred construction material for monumental architecture and bituminous roads, which helped build, maintain, and expand the influence of empires; how pine forests of the Baltic provided much of the tar needed for the trans-Atlantic shipping industry from the 17th to 19th centuries; and how northern Neanderthals living in small and mobile groups maintained and transmitted technological knowledge. An archaeology of adhesives reveals far more than the materials alone. It can reveal the transport networks, subsistence, mobility strategies, division of labor, ancient knowledge, and technological skill that holds societies together.

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