Understanding adhesives through the treatment of a 19th century mahogany chair

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This paper serves as an exploration of the principles of conservation science and its practical application relating to the adhesive failure treatment of an 19th century mahogany chair. The treatment was carried out at West Dean College by this paper's author in the 2018-2019 academic year (Nov-Jan). The purpose of this document is to provide a practical understanding of basic principles of polymer adhesives and how they impact decision making on live objects. It is intended to be accessible for individuals of all levels so that it can be used by its author as an educational tool for staff and volunteers who are interested in better understanding their work.

Introduction

In November 2019 work began on a 19th century mahogany bar back chair (pictured right) by a furniture conservation student at West Dean College. The initial assessment of the chair revealed that while most of the structure was stable, the front proper right joint (front right if you are sitting in the chair) where the leg and the front cross piece meet had split and broken in multiple places. Figures 2 and 3 show the outline of where an adhesive had been used previously to repair the damage. The conservation student noted that the timber pieces were misaligned, leaving in some places a 2mm gap filled with the polymer adhesive. The adhesive itself was brittle and failing to adhere to the timber properly. These factors resulted in an unstable leg.

Figure 1. Mahogany bar back chair

In furniture work, there are a number of adhesives that are used, and it is not always apparent to the general furniture owner or repairer as to what has been used previously, what can be done about it, and why adhesives behave the way they do. This paper looks at the nature of polymer adhesives to better understand the science behind their behaviour. It discusses the basic structure of polymers, the theories of adhesion, and physical properties of polymer adhesives in their liquid and solid states. It then follows the process the student went through to analyse and remove the adhesive from the timber surface, exploring concepts of solvency, and glass transition temperature.
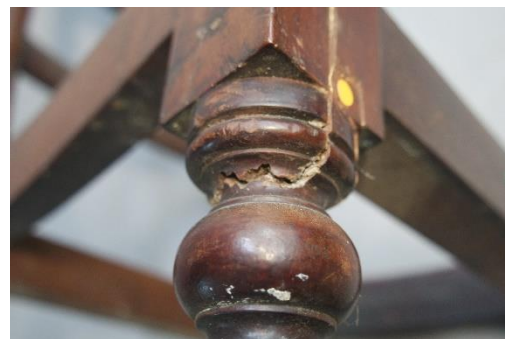

Figure 2 Adhesive filled gap from front

Figure 3 Adhesive filled gap from underside

Inherent properties of polymer adhesives

Before discussing in-depth the characteristics of adhesive failure in this mahogany chair, it is important to understand the basic properties of polymer adhesives. This first section will explore the physical and chemical properties common to all polymers starting with a general definition of polymers and an understanding of how they operate. It will then discuss polymer adhesives common to woodwork, their bonding properties, and common ways in which they fail.

What is a polymer? The short answer is that a polymer is a chain of smaller molecular parts called monomers.

Understanding Polymers

Hydrocarbons (specifically alkanes) are a good place to start in understanding the properties of these chains. *Figure 4* shows a single methane molecule, which consists of one carbon atom bonded to four hydrogen atoms. *Figure 5* shows an ethane molecule which consists of two carbon atoms bonded to six hydrogen atoms in a very similar structure to the methane molecule. *Figure 6* shows a hexane molecule with six carbon atoms bonded to fourteen hydrogen atoms. The basic structure of the molecules is similar, consisting entirely of carbon atoms bonded with hydrogen atoms. It is the number of carbon atoms in the chain which gives them their varying characteristics and establishes methane, ethane, and hexane as different alkanes (Horie, 2010).
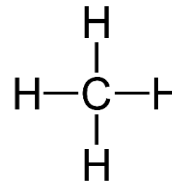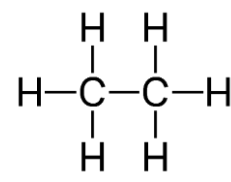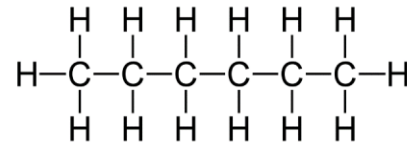
Figure 4 Methane   **Figure 4 Ethane**

Figure 4 Hexane

There are a few qualities we can determine early on about these three molecules. Size and weight are two of them. Because the hexane molecule is a longer chain, consisting of a greater number of atoms, it is a larger and heavier molecule than ethane. Another quality of note is their capacity for entanglement. As they are strings or chains in their structure, the longer the string the more likely it is to become physically entangled with other similar molecules (Down, 2015). As Hexane is a longer chain, it is more likely to entangle with other hexane molecules than either ethane or methane. These factors contribute to why methane and ethane exist at room temperature as gasses and hexane exists at room temperature as a liquid. It takes far more energy to get a hexane molecule to spread out enough to become gaseous.

It is important to note here that hexane is not actually a polymer of methane. Methane cannot polymerise (form chains), because methane is what is referred to as a completely saturated hydrocarbon (Horie, 2010). This means that every potential space for covalent bonding has been filled and is stable. Ethene, or ethylene (see *Figure 6*), on the other hand is capable of being polymerised. While very similar to ethane in that it consists entirely of carbons bonded with hydrogen atoms, it has two fewer hydrogen
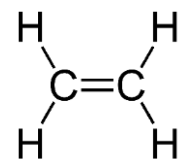
Figure 5 Ethene (Ethylene)

atoms to ethane. Instead of bonding with three hydrogens each, the carbon molecules are double bonded with one another. This double bond can be split, and more ethene monomers can be attached to create longer chains. It is the ethene (or ethylene) molecule that is polymerised to produce long chain hydrocarbon polymers such as paraffin and microcrystalline waxes[1] (Horie, 2010).

Figure 7 shows a short section of a very common polymer adhesive, polyvinyl acetate (or PVA). As illustrated, the section shows two monomers within the chain, but that polymer chain could contain thousands of vinyl acetate monomers.
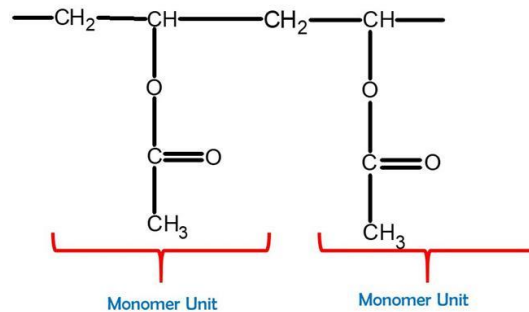
Figure 6 Polyvinyl acetate chain segment consisting of two vinyl acetate monomers (Duluth Labs, 2017)

These polymer chains are not long straight lines in real life. They curl up in three dimensions. Sometimes they bond together in regular crystalline structures, and sometimes they flow around each other in irregular amorphous structures. Sometimes, like in the case of many waxes, they do both (Down, 2015). They can also branch off in multiple directions, and those branches can join with other polymer strands in a process called crosslinking (Horie, 2010).
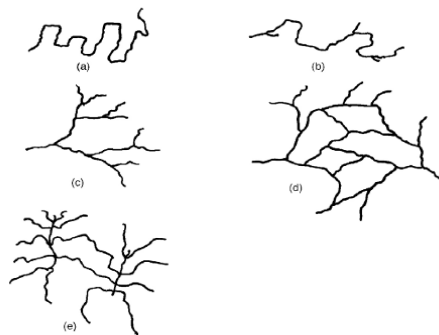
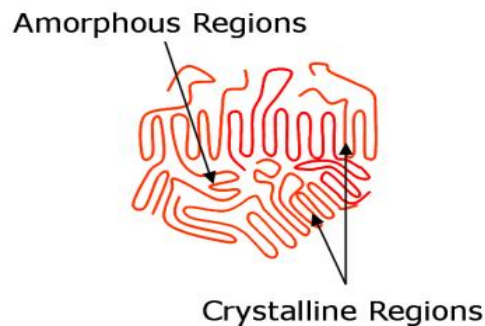**Figure 8 Examples of polymers branching and crosslinking (Horie, 2010)**

Figure 8 Semi crystalline polymer structure (UNSW, n.d.)

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[1] The number of monomers in a single chain is referred to as the degree of polymerisation (DP). Polymers are often assessed by their molecular weight, but this is not a precise measurement of DP, only an indication (Horie, 2010).

Adhesive polymers in their liquid and solid states

In almost all cases, a polymer adhesive will be applied in some variety of liquid or gel state and left to solidify either through physical or chemical processes. Polyvinyl acetate (PVA), hide glue, and epoxy resin are all adhesives used in furniture work which are handled in this way. However, they all differ in their mechanisms for doing this. Table 1 outlines the differences between how some common adhesives are often formed in their liquid state and how they harden to form a solid state.

Table 1. Liquid states and hardening processes of basic polymer adhesives

	Polyvinyl Acetate (PVA)	Polyvinyl Butyral	EVA (Hot Glue)	Cyanoacrylate ("Superglue")	Epoxy Resin
	Thermoplastic				Thermosetting
Liquid or Gel State	Emulsion in liquid	Dissolved in a solvent	Applied in semi-fluid molten state	Not yet polymerised cyanoacrylate monomers	Exists in liquid state at room temperature when polymer chains are not yet interlinked
Solidifying Process	Dispersion and evaporation of liquid and particle coalescence	Evaporation of solvent	Cooling	Polymerises in chemical interaction with moisture	Chemical interaction between epoxy and hardener causes polymer chains to crosslink into one large molecule

What can be seen in this chart are five different ways in which the polymer is able to be applied in a liquid state and five ways in which those polymers solidify. The first four substances are thermoplastic materials, and the fifth is a thermosetting material. This impacts how they exist in both their solid and liquid states (Down, 2015).

PVA and PVB both have some similarities in their application and solidification processes (mostly by transfer within a separate liquid and the removal of that liquid). EVA, instead of being emulsified or dissolved in a separate liquid, is heated up past its glass transition temperature (explained below) to a more gelatinous state, which makes it easier to apply. Cyanoacrylate rapidly undergoes polymerisation through chemical interaction with moisture (usually in the air or on the surface of the material) after being applied.

Epoxy is a thermosetting material, and it hardens through a chemical interaction with another substance referred to as a catalyst[2]. This process is not reversable, and as such thermosetting and crosslinking materials can be very difficult to remove (Rivers & Umney, 2007).
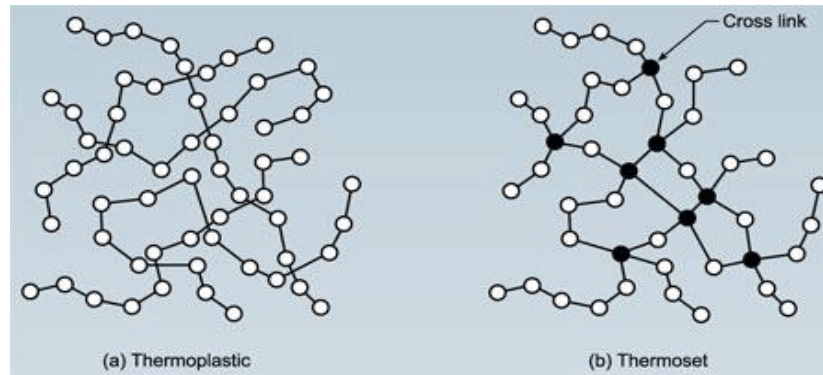

Figure 9 Thermoplastic vs thermosetting (Recycled Plastics, n.d.)

An important adhesive category in furniture not shown above is protein-based animal adhesives. These adhesives are thermoplastic and are traditionally both heated past their glass transition temperature and simultaneously dissolved in a liquid (water). In their hardening process they go through both phases of solidifying through cooling and evaporation of their solvent (Williams, 2015).

Glass transition temperature

As mentioned above, polymers and glassy materials have something referred to as a 'glass transition temperature'. The glass transition temperature is a term for the point where an amorphous polymer will transition from a solid brittle state into a flexible or rubbery state. As the material warms up, the molecular chains will begin to expand and move around one another more easily. At a certain heat, the polymer chains become less entangled and they begin to flow past one another[3]. The glass transition temperature is a terminology for this point (Horie, 2010).

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[2] Commercially referred to as a 'Hardener' for epoxies.

[3] As an example, plexiglass (polymethyl methacrylate) is often shaped, bent, and curved by heating it up past its glass transition temperature to a point where it is flexible, and then letting it cool below its glass transition temperature after shaping is complete (Dorning, 2018).
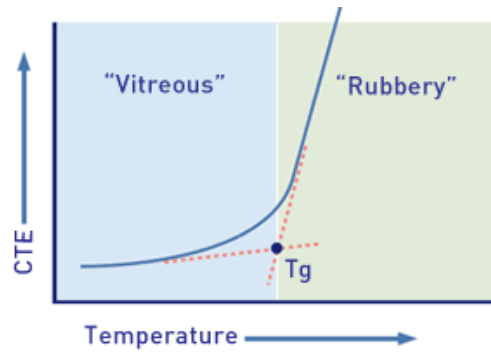
Figure 10 Glass transition temperature (Tg) (Master Bond, n.d.)

Figure 10 demonstrates how this glass transition temperature is determined. As it shows, there is not one single point where the material changes instantly, but there is a noticeable degree of change for each polymer around a certain point that can be expressed through the extension of the pre-transition trajectory on the graph and the post-transition trajectory (Dorning, 2018).

The glass transition temperature of a polymer will be impacted by the size of the polymer chains and level of branching and crosslinking, as well as other internal bonds between molecules (Down, 2015).

A Note on Creep

While the polymer chains flow past each other more rapidly and easily once the material has surpassed its glass transition temperature (some examples shown to the right), there is still a capacity for the polymer chains to move around slowly over a long period of time. This slow movement or 'cold flow' of a polymer is referred to as creep. Stress and strain on an adhesive in a joint can increase the rate of creep and encourage it (Horie, 2010)*.*

Adhesive	T_g (°C)	Set state at 22°C
silicone rubber	-112–125	rubbery
Lascaux 360 HV	-28	rubbery
Mowilith DMC2	10	rubbery
poly(vinyl acetate)	28	semi-glassy, semi-flexible
Paraloid B-72	40	semi-glassy, semi-flexible
poly(vinyl butyral)	49	semi-glassy, semi-flexible
poly(methyl methacrylate)	105	glassy/rigid
epoxy resin (aliphatic amine hardeners)	100–150	glassy/rigid

Figure 9 (Down, 2015)

Mechanisms of adhesion

The exact mechanisms behind adhesion are not completely understood or agreed upon, but there are a few theories for understanding the different ways a substance might adhere to another (Down, 2015). For the purposes of this paper, the table below has been produced to categorise some of the different bonds and methods of adhesion which may be present in polymer adhesion.

Table 2. Categories of some bonds associated with adhesion **(Horie, 2010) (Down, 2015)**

	Intramolecular Bonds	Intermolecular Bonds	Mechanical	Interdiffusion
Description	Primary chemical bonds which are associated with bonding atoms together within a molecule. Relies on the sharing or exchanging of electrons.	Secondary bonds which are associated with bonds between molecules. Weaker than intramolecular bonds. Relies on relationships of electron charges between molecules and atoms.	Physical bond, whereby the solid hardened mass of interwoven polymer molecules keys in with pores, grooves, and other physical attributes of the adherent	The intermingling, entanglement, and diffusion of molecules of two substances between one another.
Examples	• Covalent Bonding • Ionic Bonding	• Hydrogen Bonding[4] • Dipole forces • Dispersion forces	• Polymer penetration of keyed or textured surfaces	• Inter-polymeric diffusion (ie. layers of shellac mixing in with one another.

Cohesive and adhesive failure

As per the mechanisms above, it is understood that an adhesive substance has both adhesive and cohesive properties. Adhesion is the bond between the substance and another material, while cohesion is the bond between a substance and itself. If one were to pull on the top and bottom of an Oreo, there are 3 general ways in which it could come apart (illustrated in *Figure 11)*.

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[4] Hydrogen bonding can also be intramolecular, but for simplicity in explanation it has been placed in the intermolecular category in this table.
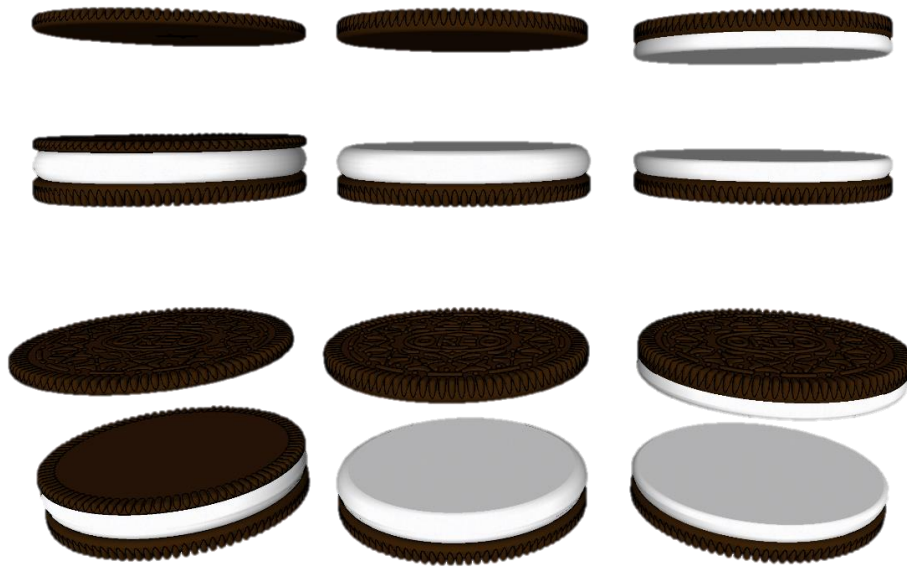
Figure 11 Adhesion and cohesion failure in Oreo biscuits[5]

1. The biscuit could break: This would be a cohesive failure in the 'substrate'.
2. The white stuff inside could separate cleanly from the chocolatey biscuit: This is a failure in the adhesion between the 'adhesive' and the 'substrate'.
3. The white stuff could tear in half: This would be a failure in the cohesion of the 'adhesive'.

It is also likely to have some combination of the above. Often there will be both adhesive and cohesive failure across the material, whereby some of the adhesive remains intact and some of it detaches (Down, 2015).

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[5] Special thanks to Harry T. Morris for producing this model. Copyright 'Harry T. Morris Furniture' 2019

Adhesive failure and treatment of the 19 C mahogany chair
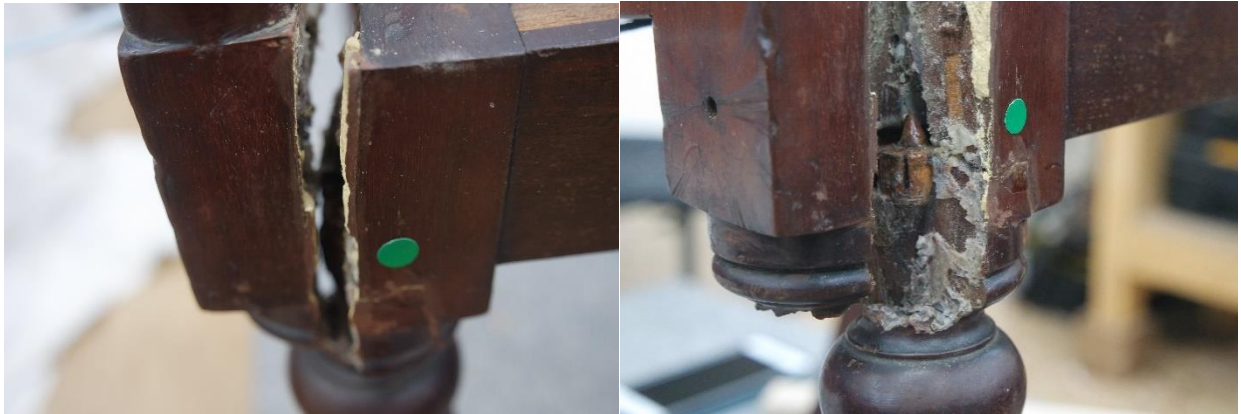
Now that some groundwork for understanding polymer adhesives has been laid out, this paper will begin to approach the damaged chair leg of the 19th C mahogany chair, discussing the issues discovered by the conservator and the treatment methods utilised. It will focus primarily on the scientific elements of the process and as such will not cover all aspects of the treatment.

The adhesives discovered within the broken timber leg of the mahogany chair presented a few immediate problems to the conservator. The unknown thick white adhesive between the largest vertical split in the timber showed a combination of cohesive and adhesive failure, although mostly cohesive failure. An iron screw had also been added to the broken leg, which once removed revealed that the extent of the adhesive failure ran the entire vertical length of the break.

Due to the 2mm thickness of the polymer adhesive, it is understandable that the cohesive failure is likely to be due to the brittle nature of the polymer in its solid state. Strength and flexibility tend to be adverse to one another in terms of properties a polymer will have, and while the adhesive is strong, it has limited flexibility (Down, 2015). This was tested in the adhesive on the chair through mechanical force with a porcupine quill. While the material was difficult to crush, it snapped apart with minimal force. With the build up being so thick, the brittle nature of the polymer allows greater likelihood of a cohesive failure. Amorphous polymers become more brittle as they age, particularly if kept below their glass transition temperature (Eric F. Hansen, 1991). This would increase the chances of a break in such a thick build up.

Identifying the adhesive

It was determined by the conservator and their advisor that to stabilise the structure of the chair the bulk of adhesive would need to be removed so that the timber pieces could align accurately, and a new adhesive could be applied with a minimal gap between the timber pieces. In order to have the best

success in removing the adhesive, given the different ways that polymer adhesives operate, it would ideal to identify the substance.

A sample of the polymer was taken to a Fourier Transform Infrared Spectrometer (FTIR)[6]. The FTIR machine observes the molecular response to different wavelengths of infrared light. The chart below shows the amount of infrared light that was transmitted back off the surface of the material at different wavelengths (Dorning, 2018).
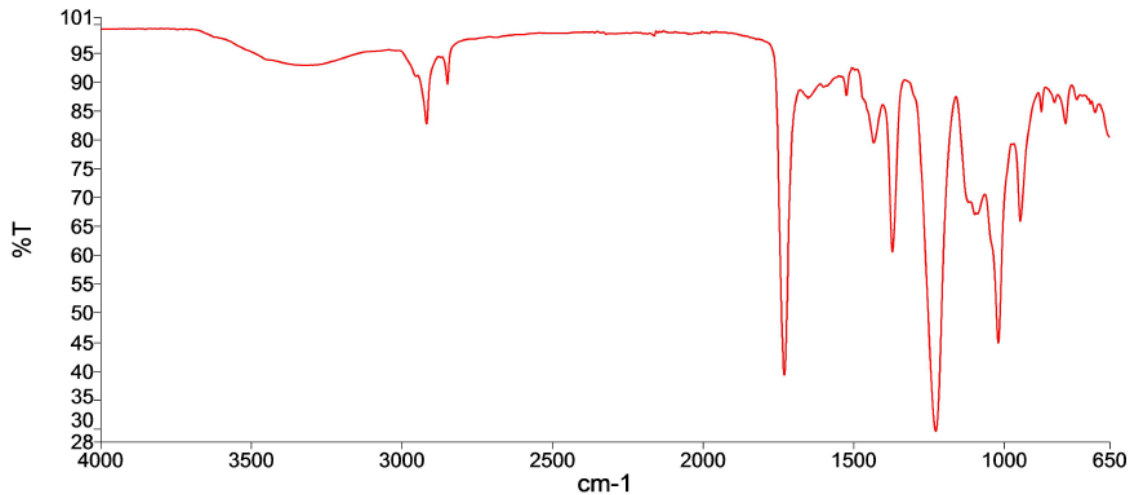

Figure 10 FTIR Transmittance reading from unknown substance found on chair

This kind of reading does not tell exactly what the substance is, but it can be compared with readings from known materials. While certain atoms will absorb different wavelengths of light (for instance in the reading above the two spikes near the 3000 mark suggest some Oxygen/Hydrogen groups and the large spark at around 1750 suggests the presence of carbon), the reading is not precise enough to get a complete breakdown of the substance (Dorning, 2018). When this reading was compared with other known readings in the West Dean College database, its closest match was to UHU All Purpose adhesive, a commercial polyvinyl acetate homopolymer (Jane L. Down, 1996). However, there was some noticeable variation and the scan also compares highly with other PVA/EVA copolymers[7]. As such, it was determined likely to be a PVA copolymer, and would be treated as such.

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[6] There are other analytical methods which can be used to help begin to determine the adhesive, including hyperspectral imaging, visual analysis, contextual analysis, solvent testing, UV light, and even burning samples.

[7] A copolymer is a polymer where two or more different types of monomers are combined in a chain. (Horie, 2010)
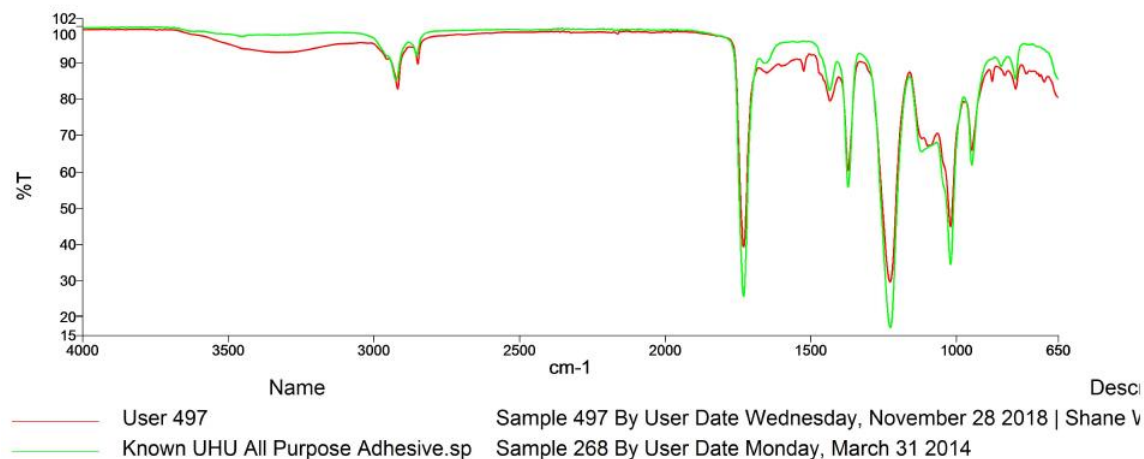
Figure 11 Comparison between FTIR scan of unknown adhesive and known UHU All Purpose adhesive

Adhesive removal methods

In removing the adhesive from the chair, the conservation student implemented several informed approaches:

Mechanical: Given the brittle nature of the polymer in its hardened state, as observed above, the first approach was mechanical. By applying force to the adhesive, it could be chipped apart and removed. However, a risk is run in this method. If the adhesion strength is greater than the cohesion strength in the timber, the timber could break away with the chipped adhesive (Rivers & Umney, 2007). Steam was applied to the surface to soften the timber and thus weaken the adhesive bonds. Mechanical bonding in particular could be reduced by a softening of the timber structure and physically releasing the polymer.
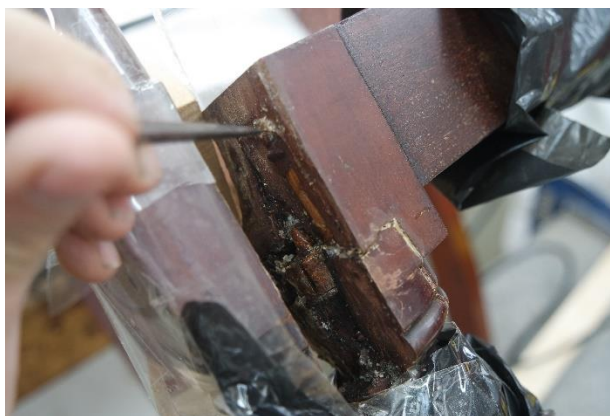

Figure 12 Mechanical removal of adhesive after application of steam

Additionally, the water from the steam can form a layer between the adhesive and the adherend disrupting hydrogen bonding and other intermolecular forces and displacing the adhesive from the surface (Edge, 1992).

Solvent/Chemical: Knowing that the material was likely to be a PVA or PVA copolymer, the conservator was able to consult a solubility chart in order to trial different solvents for instances where mechanical removal was too risky. Uncertain of the adhesive, pieces of the polymer were placed in containers with different solvents. Three were known to be compatible with PVA.

Solvents tested were acetone, Enliven nail varnish remover (contains acetone), and Nitromors (acetone and methanol in a gel) as known solvents, and isopropanol as a known unlikely solvent for comparison. The samples were left for two days at room temperature. The polymer softened in the Nitromors, varnish remover, and acetone but did not completely dissolve. This suggests that increased entanglement due to branching of the polymer or crosslinking reduced the solubility of the polymer. The softening allowed the conservator to remove the adhesive from the exposed timber surfaces due to the solvent's impact on mechanical and intermolecular bonding. This worked successfully to remove adhesive from exposed areas. However, in one instance two timber pieces were still firmly joined but needed to be separated to finish the treatment.
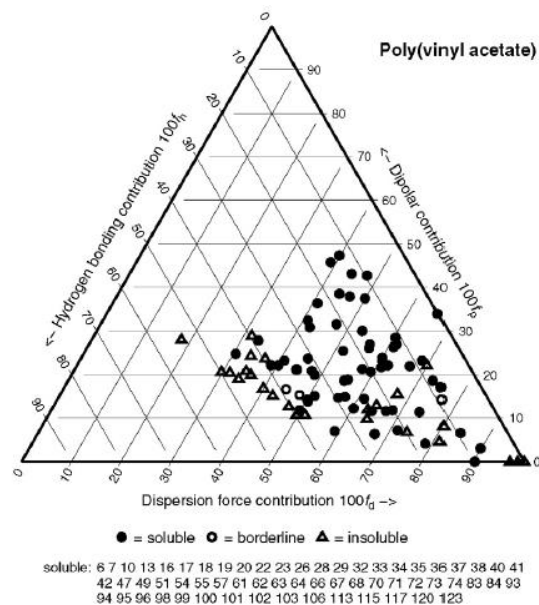

Figure 13 Teas Chart for polyvinyl acetate. Teas charts sort solvents based on three distinct properties: Dipole dipole, dispersion, and hydrogen bonding. Usually substances were similar qualities are more likely to mix. (Horie, 2010)

Heat: In order to separate the two firmly adhered pieces, heat was applied using a soldering iron and wire rod inserted into a hole drilled through the adhesive. The intention to heat the polymer above its glass transition temperature and allow the two pieces to be mechanically separated. After five minutes at an unknown temperature increase, the conservator began to mechanically force the timber pieces apart. The adhesive elongated but retracted to its original position once force was no longer applied. This too is suggestive of crosslinking behaviour in the polymer. If it was not crosslinked, the polymer chains would flow into a new position when above glass transition temperature and stay there once cooled. However, because the polymers are covalently linked together, they can move and shift more easily when above glass transition temperature but will not shift into a new position. The conservator used the heat again with force to elongate the adhesive, and then was able to cut the elongated polymer with a scalpel to separate the pieces.

Figure 14 Application of heat into adhered join using soldering iron and wire.

Conclusion

Using a combination of mechanical, solvent, and thermal methodologies, the conservation student was able to successfully remove all of the unknown adhesive from the 19th century mahogany chair. Understanding the reasoning behind why these methods worked, including disruption of adhesive bonding, dissolution of the adhesive, and increasing flexibility in the adhesive through heating it above a glass transition temperature, helped to inform and streamline the process in a relatively safe manner for the object. These techniques worked because of inherent properties in polymer adhesives, largely stemming from their nature as long molecular chains.

In understanding the failure of the previous adhesive, and the nature of polymers and polymer degradation, the conservator will be able to make an informed decision as to how to adhere the components of the chair in a more stable manner that will withstand degradation and the forces regularly applied to a chair.

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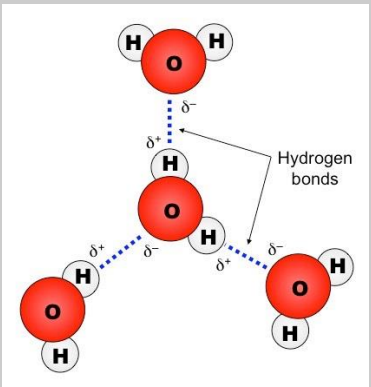Thornton, J., 2005. *Adhesives and Adhesion,* Buffalo: Buffalo State University.

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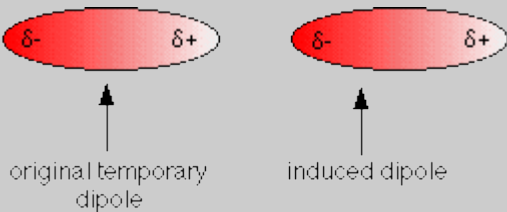[Accessed 22 January 2019].

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Appendix 1

Further details on adhesive bonding

Table 3. selection of bonds in adhesion

Covalent Bonding	The sharing of electrons between two atoms. When the outer layer of electrons surrounding an atom is not full (2 electrons in the first ring, and 8 in subsequent rings), atoms can bond with one another by sharing electrons until that outer ring is full.
Ionic Bonding	The exchange of electrons between two atoms. It is similar to covalent bonding, except that instead of sharing the electrons, one atom gives up an electron to the other. This will cause both atoms to become either positively or negatively charged depending on whether they picked up the negatively charged electron or not. Positively and negatively charged atoms (called ions) attract one another.
Hydrogen Bonding	A unique bond created when hydrogen atoms are bonded to highly electronegative atoms (specifically oxygen, nitrogen, and fluorine) and come in close contact with atoms in another molecule which have a pair of electrons in their outer shell not actively engaged in intramolecular bonding. These are referred to as the 'hydrogen bond donor' and 'hydrogen bond acceptor' respectively. The electron of the hydrogen will be pulled towards the electronegative atom in its own molecule, making it positively charged, while the non-bonding pair of electrons on the hydrogen bond acceptor are negatively charged. This bond is very strong in comparison with other dipole forces and van der Waals forces. **Figure 15 Hydrogen bonds in water (Holbrook, 2018)**
Dipole Forces	Dipole forces are caused by a polarity within a molecule. As demonstrated in hydrogen bonding, within a molecule there is potential for electrons to be found more towards one end of the molecule than the other. This happens when electronegative atoms (normally having more than 4 electrons in its outer shell) and electropositive atoms (normally having fewer than 4 electrons in its outer shell) are intramolecularly bonded. The electrons from the electropositive atom are drawn more towards the electronegative atom. This gives the entire molecule a polarity, with one side being more positively charged, and the other side being

	more negatively charged. The positive side of the molecule will be attracted to the negative side of nearby molecules and vise versa.
Dispersion Forces *(Van der Waals forces)*	Dispersion forces are similar to dipole forces but involve uncharged atomic interaction. While the molecular polarity described in Dipole forces are considered permanent, dispersion forces are an attraction between temporary dipole forces. Atoms have electrons moving in shells around a nucleus, but they tend to move in chaotic patterns. However, there is a chance that at any given point, the electrons may be moving more towards one side of the atom than the other. This will give that atom a temporary polarity. When that happens, another atom within proximity may have its electrons pulled towards the negatively charged side of the first atom. Each atom, although not ionic or containing any permanent polarity, will interact in a polar bond due to the electronegativity and positivity caused by the electrons in relation to their nucleus. What may continue to happen is that they will switch polarity in relation to one another back and forth as their electrons continue to orbit the nucleus. This 'dance' keeps them bonded together, although fairly weakly. **Figure 16 dispersion forces (Anon., n.d.)**