KEEP IT TOGETHER: AN EVALUATION OF THE TENSILE STRENGTHS OF THREE SELECT ADHESIVES USED IN FOSSIL PREPARATION

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Abstract.—The tensile strength of a select group of common fossil adhesives (50% w/w Paraloid B-72 in acetone, Paleobond PB100, and Devcon 2-Ton epoxy) was tested over a period of 9 months. Because the testing process is destructive, the tests needed to be standardized to draw valid conclusions, and because large sample sizes were desired for the most statistically accurate results, limestone adherends were used as a proxy for real fossil specimens. Paraloid B-72 in acetone at 50% w/w demonstrated long solvent retention and took several months to attain full strength. Although it was the statistically weakest of the three adhesives, it still required a significantly large force to be exerted (275.56 lbs/in^2; 19.37 kg/cm^2) in order for it to fail after only 3 days. If fossil specimens joined with Paraloid B-72 are provided with appropriate archival support, then the adhesive will slowly attain full strength. Devcon 2-Ton epoxy was stronger than the limestone adherends and created minor or major substrate failure in the majority of samples, indicating that its use be restricted only to specific situations, such as heavy specimens that cannot be supported externally. Paleobond PB100 samples failed on average between 535 and 636 lbs/in^2 (37.6 and 44.7 kg/cm^2) and generally demonstrated adhesive failure, with some minor substrate damage. Paraloid B-72 demonstrated substantial tensile strength in this study and has excellent long-term stability and reversibility. Therefore, Paraloid B-72 should be considered the default adhesive in the majority of fossil preparation practices.

Key words.—Adhesive, Conservation, Fossil, Paraloid, Preparation.

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Introduction

Adhesives are an integral component of fossil preparation and conservation. Adhesives provide structural support for fossil specimens when their surrounding rock matrix is removed during the preparation process. However, if the adhesive is stronger than the fossil adherend, physical stresses may cause new fractures to occur in the fossil rather than allow for failure of the adhesive join. The goal of fossil conservation should be to use materials that cause the least amount of damage to the fossil. Therefore, an ideal adhesive will be strong enough to bond but will fail cohesively at the bond line rather than in the fossil adherend. It is important to understand the variation in strength of different adhesives relative to the fossil needs when deciding which adhesive to use.

An ethic of minimum intervention is always preferred in the conservation of museum objects because no conservation material is completely removable. It is unknown what effects adhesives and consolidants may have on future methods of analysis. Preventive conservation methods are always preferable to interventive treatments. However, paleontological specimens usually require interventive treatments to prepare them for study or display. Extensive adhesion and consolidation of fragile specimens is often required. A lack of treatment during initial excavation could result in the deterioration of the specimen as it is exposed to environmental factors, namely, fluctuations in temperature and humidity. Subsequently, as the fossil undergoes preparation, the natural support of the rock matrix will be removed to reveal the fossil for study or exhibit, and the stabilization and reassembly of the fossil may be required, especially in heavily fractured specimens.

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A wide range of adhesives is available to fossil preparators, including solvent based adhesives such as Paraloid B-72 and reaction adhesives such as the cyanoacrylates and two-part epoxies. Of these adhesives, only Paraloid B-72 is well-tested with regard to its conservation properties. The potential of Paraloid B-72 as a structural adhesive (an adhesive that provides a bond for a considerable load for extended periods of time) has been investigated extensively.

A pioneering study by Stephen Koob (1986) found a 1:1 by weight solution of Paraloid B-72 in acetone to have a higher tensile strength than polyvinyl acetate or cellulose nitrate and that it increased in strength between 24 and 72 hours. The comprehensive study of adhesives by Jane Down et al. (1996) found Paraloid B-72 to be a medium strength (2–15 MPa) acrylic adhesive and one of the most chemically stable acrylic adhesives overall. In the same year, Vestergaard and Horie (1996) tested a 1:5 by weight solution of Paraloid B-72 in acetone as an adhesive for mastodon tooth adherends and found that it was weaker than cyanoacrylate and epoxy. Most recently Podany et al. (2001) conducted tensile and shear strength tests of Paraloid B-72 and found that while it was approximately one third as strong as epoxy in shear, it performed comparably to epoxy in tension (however, both the epoxy and Paraloid samples broke in the test substrate before the adhesive bond failed, so exact tensile strengths for the adhesives were not recorded). None of these studies investigated the change in strength of several adhesives over an extended period of time.

This study investigates the tensile strength of three adhesives over a period of several months. This is critically important in paleontological collections; for when fossil specimens are prepared or receive conservation treatment they frequently remain in storage for years before being handled again.

Paraloid B-72 is often dismissed as an option for the repair of large and heavy specimens in favor of cyanoacrylates or epoxies, although it is used as the main adhesive and consolidant in many paleontology laboratories (Davidson and Brown, 2012). Two of the most common adhesive and consolidant substitutes are PaleoBond PB100 and Devcon 2-Ton epoxy. The preponderance of these adhesives in lieu of Paraloid B-72 as a structural adhesive may be because of doubts about its strength, because of difficulty manipulating it, or because the properties of other adhesives (such as the quick cure time of reaction adhesives) are preferred. Koob (1986) states “Criticisms often cited against the use of Paraloid B-72 and other acrylic resins include stringiness, poor adhesion, low tack and long setting-times.” Studies have shown that these issues can be overcome with good technique and practice (Koob 1986, Davidson and Brown 2012).

Strength is an important consideration when choosing an adhesive. If a join is made with an adhesive that is stronger than the fossil adherends, then the adherends rather than the adhesive will fail when subjected to physical forces. Handling for research, exhibit installation, and improper storage all create stress on a fossil. The force of gravity alone can be enough to cause a fossil to break under its own weight. Breakage can cause irreparable damage to the fossil and the loss of valuable scientific data. However, strength is not the only factor that should be taken into account when selecting the most appropriate adhesive from the three being evaluated. According to the Canadian Association for Conservation of Cultural Property and of the Canadian Association of Professional Conservators (CAC/CAPC) Code of Ethics, “ideally, the conservation professional shall use materials that can be most easily and most completely removed with minimum risk to any original part” (CAC/CAPC 2009). Reversibility and long-term stability are of paramount importance for fossils—these are natural objects that are

Table 1. Conservation properties of test adhesives.

Adhesive	Reversibility	Long-term stability
50% w/w Paraloid B-72 in acetone	Medium (Elder et al. 1997)	Good flexibility Resistant to yellowing Does not release volatiles (Down et al. 1996)
Paleobond PB100	Poor (Elder et al. 1997)	Degrades under alkaline conditions (Down and Kaminska 2006) Uncertain ageing characteristics (Down 2001).
Devcon 2-Ton epoxy	Nonreversible (Elder et al. 1997)	Yellows considerably in dark and light conditions, considered intermediate to unstable (Down 1984, 1986)

already millions of years old and can be expected to substantially outlast the materials used to preserve them. Any adhesive used on a fossil will potentially have to be removed at some point in the future; therefore, all conservation properties should be carefully considered. A summary of the conservation properties of the adhesives examined here is provided in Table 1.

This study is a specific evaluation of the tensile strength of Paraloid B-72 in comparison with PaleoBond PB100 and Devcon 2-Ton epoxy over a period of 9 months, and it was carried out in order to assist fossil preparators in making an informed decision when choosing the most appropriate adhesive.

Evaluated Adhesives

Three adhesives were evaluated:

A 50% w/w solution of Paraloid B-72 in acetone was tested. This is an ethyl-methacrylate copolymer that is supplied as clear beads that can be dissolved into a solvent (commonly acetone, ethanol, toluene, or xylene). It can be prepared to any desired concentration up to about 50% w/w for adhesive purposes and diluted for use as a consolidant. After application, the solvent evaporates, leaving only the acrylic copolymer behind. A major benefit of this adhesive is that it can be reworked after it has set by adding more solvent or warming it to near the glass transition temperature (Tg) of the polymer (see Davidson and Brown 2012). However, Paraloid B-72 may retain solvent for days, weeks, or even months depending on the material and size of the join, meaning that specimens must be handled carefully after use.

PaleoBond PB 100 is an ethyl cyanoacrylate reaction adhesive. When applied to a surface it reacts with moisture and starts to polymerize (Davidson and Alderson, 2009). Polymerization is very fast, and therefore the working time for this adhesive is under a minute.

Devcon 2-Ton epoxy is a two-part epoxy adhesive. Equal amounts of both the resin and hardener are thoroughly mixed together to create a reaction and then applied to the join surface. Proper preparation of two-part epoxy is vital otherwise it will not perform as intended. Incorrect mixing may prevent the two components from reacting entirely, causing issues such as not attaining full strength and tackiness. Insufficient time for all the components to react is the reason that 5-minute epoxies are not recommended (Davidson, 2009). Devcon 2-Ton epoxy has a working time of about 10 minutes.

The purpose of this study was to compare the strength of the adhesives over time. Each of the three chosen adhesives was tested 3 days, 3 weeks, 3 months, 7 months, and

Table 2. Tensile testing results.

Sampling time category	50% w/w Paraloid B-72 in acetone		PaleoBond PB 100		Devcon 2-Ton epoxy	
	N	Mean lbs/in^2 ± SE	N	Mean lbs/in^2 ± SE	N	Mean lbs/in^2 ± SE
3 days	52	275.55830 ± 18.073	43	535.1627 ± 23.8959	24	835.6051 ± 23.9701
3 weeks	59	454.1778 ± 21.9833	57	597.7786 ± 17.888	47	776.3143 ± 18.7524
3 months	52	481.3075 ± 20.9196	57	568.9203 ± 18.2076	39	751.6155 ± 29.2552
7 months	33	611.5545 ± 32.9394	33	549.0287 ± 17.2139	25	839.7997 ± 30.9229
9 months	33	536.8736 ± 28.0548	32	636.4430 ± 21.3812	22	717.6044 ± 31.1763

9 months after sample preparation. Thirty to 35 test samples were prepared for each sampling time category. This number decreases where the sample broke in the adherend or was faulty in some way. The 3 day, 3 week, and 3 month sampling time categories were tested in subsequent years and have doubled the sample size. Thirty-five samples were prepared for the 7 and 9 month categories, as they were only tested once. The number of samples is recorded in Table 2; Figure 1 illustrates the construction of the samples used in this study.

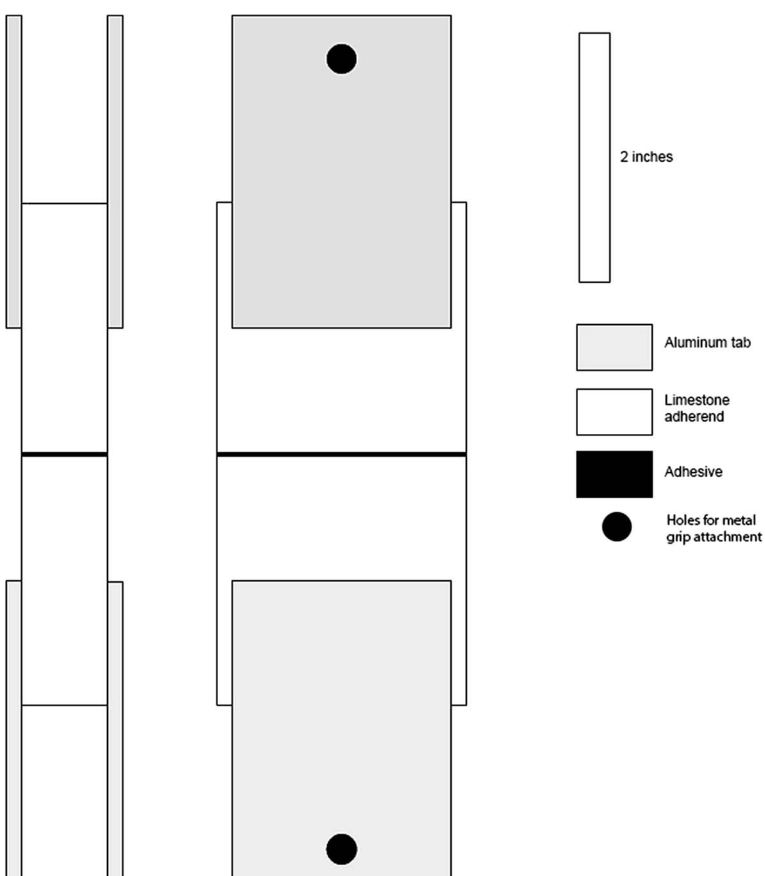

Figure 1. Tensile testing sample, side and front views.

Sample Preparation

Tensile testing samples were prepared using modified ASTM standard D 2094-00 (Standard Practice for Preparation of Bar and Rod Specimens for Adhesion Tests). This standard is designed for metal or plastic adherends that can be drilled through for attachment to a tensile testing machine. Drilling through the limestone adherends led to breakage, so an aluminum tab system was used to attach these samples to the testing machine. Samples consisted of two limestone adherends measuring approximately 2 × 2 × ¾ inches (5.08 × 5.08 × 1.905 cm) butted against each other axially. A fossiliferous limestone floor tile was chosen as a proxy for fossil bone for several reasons. A large sample size was desired to produce the most statistically accurate results, which would have been difficult to source from original fossil material. Previous studies have all had small sample sizes and not investigated change in strength over more than 3 days. Sample preparation and testing are both destructive processes, and using Alberta fossil material on this scale would be difficult because it is protected under the Alberta Historical Resources Act. Finally, the tests needed to be standardized to draw valid conclusions, and a dense, stable substrate was required for this. It would not be practical to use fossil specimens because it would be impossible to standardize the test. Because the samples were both fossiliferous and capable of being replicated and standardized, the limestone was selected to be representative of very large, dense, heavily permineralized fossil bone.

The adherends were cut by hand using a diamond saw, creating minimal variation in size. The surface area of the join was measured on each adherend to ensure maximum possible accuracy when calculating the tensile load at failure. The join surface was sanded smooth, washed with high-pressure water, and air-dried before being adhered. The substrate was not porous, and no consolidation was done to the join surface because it would pool at the surface. With porous fossils, sometimes preconsolidation of a porous bond area is necessary to prevent starvation of adhesive in the join. The sides of the adherends were also sanded to remove any polish from the floor tile. Aluminum tabs were adhered to the sides on the top and bottom of each adherend with Devcon 2-Ton epoxy, and custom metal grips were made in order to connect the samples to a universal testing machine. A 0.3-ml amount of adhesive was applied by syringe to the adherends and spread evenly over the bond surface. The test samples were placed upright with the long axis perpendicular to the workbench and left to adhere under gravity.

Tensile Testing

Samples were tested at the University of Alberta's Department of Mechanical Engineering using a Materials Testing Systems universal testing machine set to add 20 lb of tension per second. Testing took place over the span of 3 years, and several sampling time categories (3 day, 3 week, and 3 month) were tested twice in subsequent years to increase the sample size.

Results

Results were analyzed in Systat v.12 (2007). Analysis of variance (ANOVA) analyses were run with a Bonferroni correction (Table 2).

Figure 2 illustrates the tensile strength of the three adhesives over all time sampling categories.

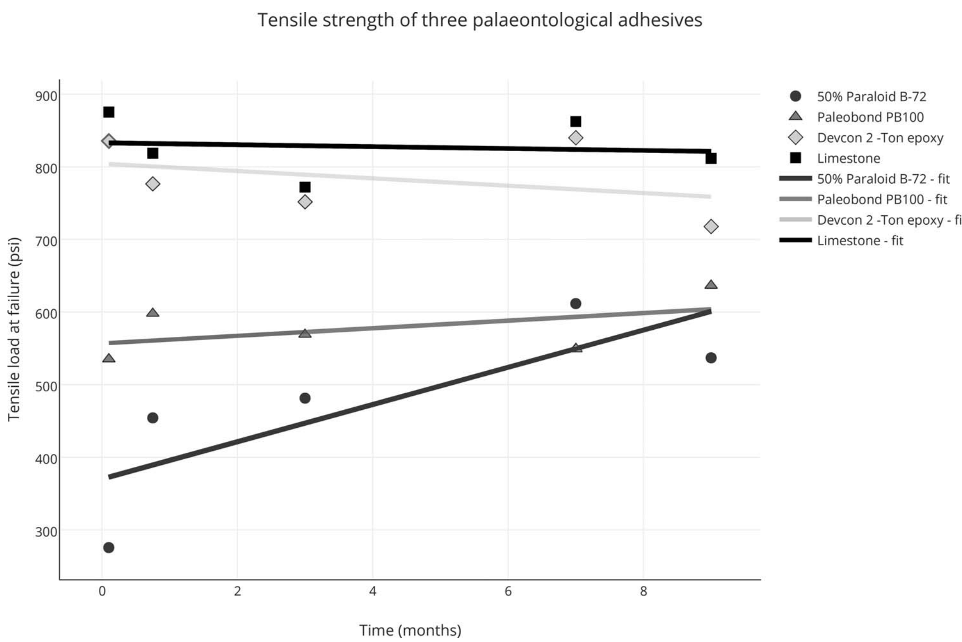

Figure 2. Tensile strength of the three adhesives over all time sampling categories.

Paraloid B-72

The weakest of the three adhesives was 50% Paraloid B-72 in acetone, which was to be expected from previous studies and personal communications with experienced fossil preparators. It was weaker than Paleobond PB100 in all but one case (ANOVA, $P < 0.05$ except at 7 months where $P = 0.295$), but the issue of strength corresponds to the length of time it takes for the solvent to evaporate. The 3 day samples were noticeably more flexible during testing than the 3 week samples, and the strength and reduction of flexibility increased over time up to 7 months. In samples up to 3 weeks, the adhesive visibly stretched as the sample was pulled apart, demonstrating cohesive failure (flow) due to solvent retention (see Figure 3 for 4 types of bond failure). Most of these samples have been classified as cohesive failures of the adhesive (see Table 3). Samples older than 3 weeks made an audible "pop" at their breaking point and generally displayed either adhesive or minor substrate failure. This demonstrates a significant increase in tensile strength during this time ($P = 0.00$), indicating that the solvent release was very slow in all test samples. This retention would vary according to the size of the join and the material adhered. Koob (1986) used pennies as adherends and observed an increase in strength of 9.5 times between 24 and 72 hours.

Despite the extended solvent retention, the day three Paraloid B-72 samples failed at a mean tensile load of 275.558 lbs/in^2 (19.37 kg/cm^2), which is a substantial amount of force to exert on a fossil. The implication of this is that fossils joined using Paraloid B-72 may require several days for the adhesive to set before handling can occur, depending on the size of the join and the nature of the material. Although this can be inconvenient, the extra time required is a reasonable compromise for the benefit of a reversible, stable adhesive. Fossil specimens should be safely supported when being repaired or stored to allow for strength to be attained as the solvent evaporates. Whenever possible all fossils should be stored in archival supports such as plaster support jackets (see Jabo et al., 2005,

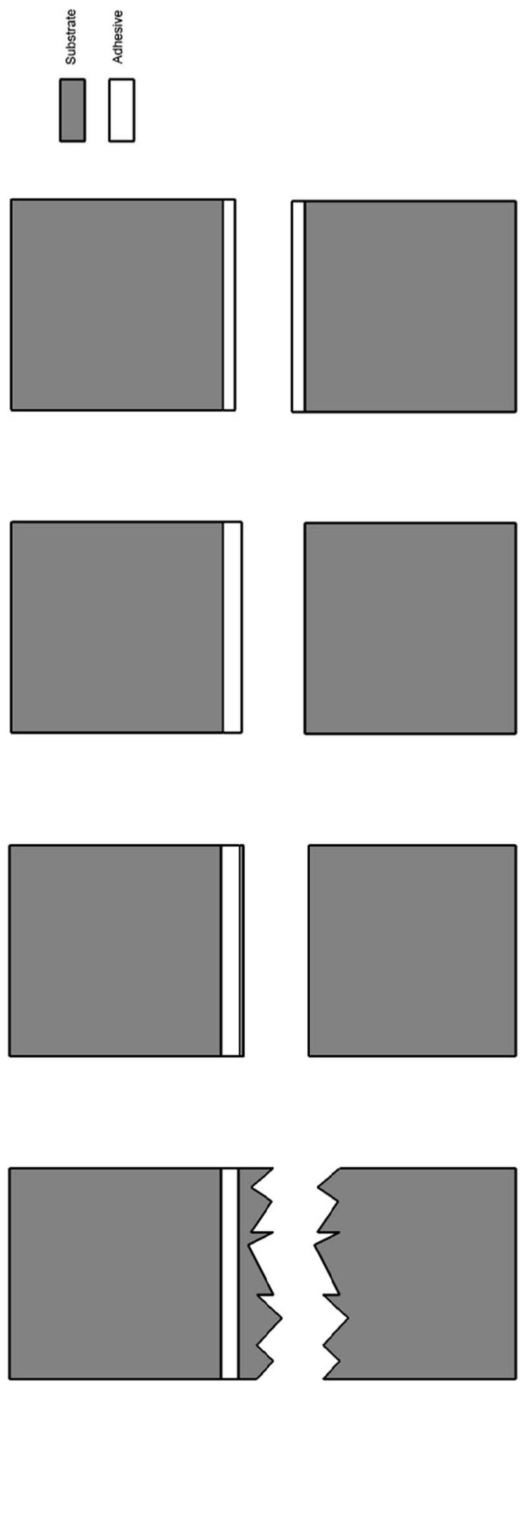

Figure 3. Four types of bond failure.

Table 3. Percentages of each failure type within each time sampling category.

Sampling time category	Failure type (%) Cohesive	Adhesive	Minor	Major
3 day				
Paraloid	75	25	0	0
Cyanoacrylate	0	100	0	0
Epoxy	0	0	42	58
3 week				
Paraloid	30	52	7	11
Cyanoacrylate	3.5	65.5	3.5	27.5
Epoxy	0	22	22	56
3 month				
Paraloid	3.7	55.6	29.6	11.1
Cyanoacrylate	4	77	15	4
Epoxy	0	8.3	58.3	33.3
7 month				
Paraloid	27	33	33	7
Cyanoacrylate	0	91	6	3
Epoxy	0	3	41	56
9 month				
Paraloid	11	0	80	9
Cyanoacrylate	0	39.4	48.5	12.1
Epoxy	0	0	15.2	84.8

for a description of jacketing methods) or polyethylene foam lined boxes to reduce the impact of physical stresses on them.

PaleoBond PB100

PaleoBond PB100 was the intermediate in strength of the three adhesives and yielded consistent averages for all sampling time categories. There was no significant difference between sampling time categories at a 95% confidence interval with the exception of between 3 days and 9 months ($P = 0.014$). The consistency of the results should be expected, as this is a reaction adhesive and does not depend on either the evaporation of a solvent (as with Paraloid B-72) or the correct mixing of two different substances (as with Devcon 2-Ton epoxy). PaleoBond may be a good choice in terms of strength for this limestone substrate. However, this adhesive is difficult to reverse and has unknown long-term stability (Table 1).

Devcon 2-Ton Epoxy

Many of the samples adhered with Devcon 2-Ton epoxy experienced major or minor substrate failure. These results indicate that this epoxy would be an undesirable choice of adhesive for joining this substrate, as it is preferable for a join to fail cohesively when subjected to stress rather than in the substrate, therefore causing minimal damage. The density and stability of fossils is highly variable, and many of them would break under a lower tensile load than the limestone slab used here, which broke at 700–1000 lbs (318–454 kg). It follows that Devcon 2-Ton epoxy may not be an ideal choice for adhering fossils in general. The adhesive often remains on one side of the join and peels off the first few millimeters of the limestone on the other side of the join, or a new break forms directly beside the old one. This is the worst kind of break because the adhesive is then incredibly difficult to remove in order to fit a fossil back together, and some loss of fossil bone is likely from the break. For particularly large and heavy specimens it may be the only reasonable choice, in which case the use of a barrier layer of Paraloid B-72 could be used to promote reversibility (Podany et al. 2001).

Physical Damage

The tensile test samples failed in one of four ways: (1) major substrate failure, where a break occurred in the adherend and the bond area remained intact, or both adherends experienced major breakage around the bond area; (2) minor substrate failure, where the adherend broke in the substrate adjacent to the adhesive bond line, taking a thin layer of limestone with it; (3) adhesive failure, where the adhesive separated from the adherend with no damage; or (4) cohesive failure, where the adhesive pulled away from itself but remained on both sides of the adherend. If this is considered in terms of fossil material, the ideal type of failure would be cohesive, as this does not damage the fossil.

The epoxy samples showed the highest incidence of major substrate failure. This reflects the similar tensile strengths of the epoxy and the limestone. If this were a fossil, this kind of failure would result in significant damage. The Paleobond PB100 samples generally displayed adhesive failure, with the adhesive remaining on opposite sides of the adherends. However, those characterized as adhesive failures often had a very thin layer of bone attached in some areas. This does represent damage to the join surface, but the join surface remained smooth, meaning that in a fossil substrate the damage would have been negligible. Paraloid B-72 showed cohesive failure in 75% of samples at 3 days, causing no damage to the limestone adherends. However, the increase in strength over time as the acetone evaporated coincided with an increase in surface damage to the adherends, creating minor substrate damage in the later time sampling categories.

Discussion

Study Limitations

Although this study was designed to give fossil preparators a better idea of the capabilities of some commonly used adhesives, there are some limitations of the study that should be taken into consideration when evaluating these results. First, only tensile strength was evaluated here, and there are other forces that act on a fossil. Shear and bending tests may yield very different numbers than those reported here (see Podany et al. 2001 for shear/tensile difference). Second, these tests measure the maximum load placed on a join within a short time span (approximately 1 minute), and do not investigate the effects of a lesser load over an extended period of time.

These tests demonstrated that Devcon 2-Ton epoxy creates major substrate failures, even within an adherend as strong as the limestone used here. The preservational state of fossil material is highly variable, but many fossils are weaker and more fractured than this substrate. This suggests that epoxies should be used only when necessary and only with heavy, well-consolidated specimens that cannot be provided with suitable external support.

PaleoBond PB100 did not create major substrate failures in many test samples and in fact showed the least surface damage of all three adhesives. In most cases the adhesive appeared to detach from the adherend with minimal surface damage, although a very thin layer of adherend was often attached. Many other samples showed minor substrate failure occurred at the bond line. In a fossil this would result in some bone loss at the join.

Paraloid B-72 demonstrated slow solvent release in these samples that resulted in full strength only being attained after several months. This is reflected in the types of failures seen in each sampling time category, changing from cohesive at 3 days, to adhesive, and finally to minor substrate damage in the later categories. These results suggest that 50% w/w Paraloid B-72 in acetone is a strong enough adhesive for the vast majority of fossil preparation applications. Although solvent may be retained for weeks or even months in larger joins, such as those of our test samples, placing a specimen in proper archival support will protect it from damage as it continues to attain strength.

Every adhesive has a very particular situation that calls for its use, but the evidence would suggest that the particular situations that appropriately call for cyanoacrylate and epoxy are far smaller than the actual number of situations in which these adhesives are used. This is especially true when considering the negative characteristics of these adhesives, which include poor reversibility, questionable long-term stability, and excessive strength for most applications in paleontology.

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