

## **Effect of Moisture and Aging on Adhesive Strength**

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### ***Abstract:***

*In a place like the San Francisco Bay Area where we are always threatened by the possibility of suffering an earthquake, it is important to study the longevity of the adhesive patches that are commonly used to secure heavy and expensive laboratory equipment against sudden movement during earthquakes. Stainless steel adherends were used in concert with 3M VHB Double Coated Acrylic Foam Tape. This experiment tested two factors: time and ageing conditions. The ageing conditions were: 60°C, 60°C & 60% relative humidity under standard atmospheric conditions. Each set of specimens was left in their appropriate environment for a set time period. Interactions between the factors showed that the adhesives cured more fully and delivered higher shear strength. That is, for the test conditions specified, the durability of the adhesive was not compromised.*

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## **Introduction**

### ***What is an adhesive?***

An adhesive is a substance capable of holding material together by surface attachment.

The term adhesive includes cement, glue, paste, mucilage, etc. The materials being joined are called adherends.

### ***How does an adhesive work?***

To make adhesion possible, it is necessary to generate intrinsic adhesion forces across the interface. The magnitude and nature of those forces are very important.

There are four main mechanisms that could explain the adhesion process, these are:

- Adsorption Theory
- Mechanical Interlocking
- Diffusion theory
- Electronic theory

The adsorption theory is the most applicable theory but the other mechanisms are important too. Therefore, they will be briefly explained too.

#### **A. Adsorption Theory**

This theory states that the materials will adhere because of the interatomic and intermolecular forces that are established between the atoms and molecules in the surfaces of the adhesive and the adherend. These forces include:

- Secondary bonds
  - Van der Waals forces
  - Hydrogen bonds

- Primary bonds
  - Covalent
  - Ionic
- Donor acceptor interactions which are intermediate in strength between secondary and primary bonds (Acid-base interaction)

### **B. Mechanical Interlocking**

This theory proposes that the main source of intrinsic adhesion is the mechanical keying of the adhesive into the irregularities of the adhered surface.

### **C. Diffusion Theory**

This theory states that the intrinsic adhesion of polymer to a metal is due to mutual diffusion across the polymer/metal interface when certain metals are evaporated onto polymeric substrates.

### **D. Electronic Theory**

The electronic theory suggests that the electrostatic forces are created when the adhesive and adherend have different electronic bands structures. Then, there is some electron transfer on contact to balance Fermi levels which will result in the formation of a double layer of electrical charge at the interface. [1]

### ***Types of adhesives***

- A. Solvent based adhesives:** Adhesion occurs by action of the adhesive on the adherend. Solidification happens after the evaporation of solvent.
- B. Latex adhesives:** They are based on polymer latex and it is necessary that the polymers can flow and provide good surface contact on evaporation of water.

**C. Pressure-sensitive adhesives:** The adhesive flows by application of pressure.

When the pressure is removed, the viscosity of the polymer is high enough to adhere to the surface.

**D. Hot-melt adhesives or Thermoplastics:** They form good adhesives by melting, followed by cooling after the thermoplastic has filled surface voids.

**E. Reactive adhesives:** Low molecular weight polymers or monomers that solidify by polymerization reactions after application.

**F. Thermosets:**

- Unsaturated polyester, which has replaced lead for auto body repair.
- Polyurethanes, which are used to bond polyester cord to rubber in tires.
- Epoxy resins which are used in automotive and aircraft construction.

**G. Elastomers**

- Solutions of natural rubber used for laminating textiles
- Pressure sensitive tape like Scotch tape
- Synthetic rubber used instead of natural rubber [2]

### *Uses in industry*

The adhesives industry has found its place in many industries and will surely spread to many other fields.

**A. Building Industry**

- Ceramic, plastic and metal tile adhesives
- Plywood or wood paneling adhesives
- Cementitious materials adhesives

- Floor coverings
- Sinks and counter tops
- Roof adhesives
- Insulation
- Sandwich panels for prefabrication- type structures

#### **B. Electrical industry**

To be used in electrical equipment, an adhesive needs to have good electrical qualities, chemical resistance, moisture resistance, tracking resistance and radiation resistance. Adhesives are used to fabricate transformers, switch gear components, capacitors, microwave devices, motors, generators and insulators

#### **C. Electronic industry**

Electronic microchips may be mounted onto the printed circuit board using hot-temperature hardening acrylic adhesive.

#### **D. Automobile industry**

Many different types of adhesives are used in the automobile industry. The main uses are:

- Bonding brake linings and transmission bands.
- Bonding door weather strips and trim materials
- Brake-shoe bonding
- Bonding of glass to metal

- Bonding of metal body sections, etc.

#### **E. Aircraft industry**

- Join of load-bearing components in aircraft.
- Problems with stress concentration around rivets was reduced
- Much cheaper than rivets
- Great saving in weight

#### **F. Aerospace industry**

Adhesives have been found which can withstand the extremes of heat and cold encountered during a space voyage.

#### **G. Medical and Dental industries**

- To adhere caps, braces, etc...
- Superglues are used to close up incisions instead of surgical stitches

#### ***Advantages and disadvantages***

Some of the advantages that the use of adhesives can offer are:

- The ability to join dissimilar materials
- The ability to join thin sheet –material efficiently
- An improved stress distribution at the joint, which provides a good dynamic fatigue resistance to the bonded component.
- It is the most convenient and cost effective technique
- Increase in design flexibility
- An improvement of the appearance of the bonded structure

- An improvement in corrosion resistance

However, there are a few disadvantages:

- It is difficult to reach long service-life from adhesive joints. Sometimes, environment affects adhesive joints causing failure.
- The upper service temperatures that adhesives can withstand are limited.
- The strength and toughness in tension or shear are relatively low compared to many metals
- Non destructive test methods for adhesive joints are limited compared to those used with other fastening methods

### ***Experimental Motivation***

It would be important to remark that some of the adhesive failure causes are referred to environmental effects like moisture and temperature. Adhesives find many applications in joining load-bearing members. In a place like the San Francisco Bay Area where we are always threatened by the possibility of suffering an earthquake it is important to study the longevity of the adhesive patches that are commonly used to secure heavy and expensive laboratory equipment against sudden movement during earthquakes. Therefore, the effect of moisture, temperature and time on the adhesive strength was determined in this experiment using 3M acrylic adhesive, type 4950. Type 4950 is a multi-purpose adhesive, and a common adhesive use to fasten down laboratory equipment.

## ***Background***

Adhesives are one of the oldest joining techniques. Egyptians used resinous adhesives to bond ceramic vessels at least 6000 years ago. Adhesives derived from bitumen and tar pits were used as mortar by the builders of the Tower of Babel. A biblical example of the knowledge of adhesives is recorded in the twenty-second chapter of Ecclesiastes. It is indicated here that the proper selection of an adhesive must be made to be compatible with the substrate being joined. In fact, engineers are still struggling with problems like that. However, there was a little advancement in adhesive technology until the twentieth century. Great strides were made during the World War II due to military requirements. It could be said that the adhesive industry has grown at a rapid rate since the fifties and it continues to grow these days.

In explaining failure of adhesive joints, it is important to know the difference between design deficiency and processing deficiency.

- Design deficiency

If the fracture of the adhesive produced residues on both faces, it is called cohesion failure. This type of failure is normally caused when the adhesive contains voids and obvious defects. Refer to Figure 1 for illustration of cohesion and adhesion failures.

- Processing deficiencies

This kind of fracture is characterized by interfacial failure of the bond. There will be areas where the adhesive remains in only one of the adherend surfaces, with the matching surface being free of adhesive. This

type of failure is known as adhesion failure and it is usually caused by inadequacy of the surface preparation used when the bond was formed. [3]



Figure 1: An illustration of adhesive and cohesive failures

There are three basic principles for design of adhesive joints.

1. To design the joint such that the adhesive is always stronger than the unnotched strength of the adherends
2. To ensure that the overlap length is sufficient to enable the adhesive shear stress to decay to near zero to make the joint resistant to creep and load rate effects
3. The adherends surfaces must be:
  - Free of contamination
  - Sufficiently chemically active to enable formation of chemical bonds between the adhesive and the adherends
  - Resistant to environmental deterioration in service, especially by moisture. [4]

When an adhesive failure occurred, it may be difficult to assess if the failure of the bonded joint was due to the environment. It becomes necessary to analyze if the failure

comes from the adhesive, the interfacial regions or the adherend. Most of the experience in determining joint failure modes has come from examining the fracture surfaces of joints after failure. From these examinations it has been found that degradation of the interface between the adherend and the adhesive is a major cause of failure. These studies also reveal that there is a change in the joint failure mode due to the effects of aging and moisture. The main change is from cohesive failure to a failure in the interfacial region of the joint. This change in failure mode is evidence that the aging environment may change the physical and mechanical properties of the adhesive and the adhesive/adherend interface. [6]

## **Procedures**

### ***Sample Acquisition***

- 150 Stainless steel plates were prepared. These plates were to have the dimensions of 3"x 1" x 1/8".
- Adhesives were provided by a local 3M distributor for use in the research. The adhesive was 3M VHB Double Coated Acrylic Foam Tape, type 4950.

### ***Sample Preparation***

- Each of the stainless steel plates had to be cleaned with an alcohol pad in order to remove any debris or grease from the surface.
- Once the Stainless steel plates were cleaned the alcohol needed to evaporate before the adhesives were to be attached.
- The adhesive patches were then cut into 1" x 1" squares.
- The adhesive patches were then attached to the Stainless steel plates.

- Pressure was then applied perpendicular to the adhesive surfaces for a minimum of 20 minutes to allow the adhesive to bond well.

### ***Oven & Environmental Chamber Preparation***

- The oven temperature was set at  $60 \pm 15^\circ\text{C}$ .
- The Environmental chamber was also set at  $60 \pm 15^\circ\text{C}$  and at 60% relative humidity.
- Using a simple DOE setup, we were able to set up an experiment utilizing weeklong intervals to monitor the changes to the experiment.

### ***Sample Insertion Into the Instron***

- The Instron model 4202 series IX was used for all testing.
- The prepared final specimen was then inserted into the Instron machine with spacers. Spacers were added for the purpose of removing the cross shear from the testing process.
- Sample was sheared at a crosshead speed of 5mm/min.

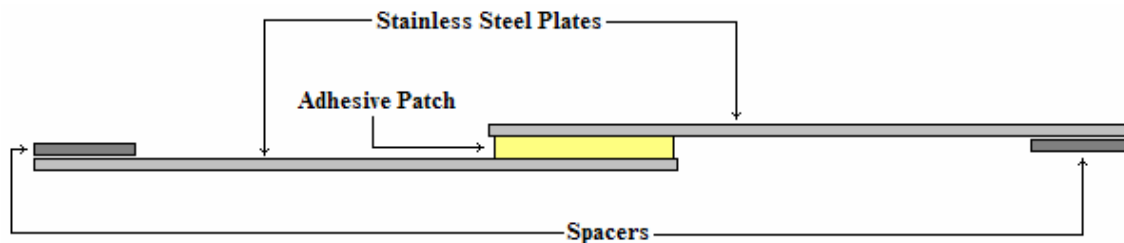


Figure 2: Sketch of the adhesive/adherend system.

## Results

As in accordance with the goals of this project, the effect of aging through heating and humidity exposure was investigated. The investigation focused on two primary factors: time and aging conditions. The responses of the factors were measured through lap shear. Table 1 served as an experimental outline and as a data input table. Time was broken down into four increments: 0-week, 2-week, 4-week, and 6-week. At the end of each increment, lap shear testing was performed on the specimens. Aging conditions were divided between the use of heat and heat in combination with humidity.

Table 1: Experimental Outline

		Aging Conditions	
		60 °C	60 °C/60%RH
Time	0-Week		
	2-Week		
	4-Week		
	6-Week		

The 0-Week row served as the control group for this experiment. With the lap shear performed, the analysis of the data was done with the common statistical analysis of variance (ANOVA). In this case, the variances of time and aging conditions were used in analyzing the durability of adhesive being tested. To facilitate the ease of this analysis, the statistical software, MINITAB version 13.3, was chosen for this purpose.

The averages of the each of the sample group were plotted as shown in Figure 3. With ANOVA, a determination was made objectively to see if there were differences among the sample groups. Following that there were two factors being investigated in this experiment, a two-way ANOVA method was chosen to analyze the data. The analysis method took into consideration that the factors are crossed instead of being nested; and

these factors were fixed and not random. The factors were considered crossed because each level of the factor time was associated with each level of the factor aging condition. The two factors were fixed because a decision was made in advance that time and the stated aging conditions were more critical in assessing the durability and reliability of the adhesive. That is, a choice was made not to select experimental factors at random. One of the primary requirements for a successful analysis with ANOVA was that the data has to be normally distributed. The data collected didn't fall into that category. Therefore, transformation of the data was needed in order for ANOVA to be used. The method chosen for the data transformation was the Box-Cox transformation with lambda equal to zero. Figures 4 and 5 showed the before and after effect with Box-Cox transformation of the data.

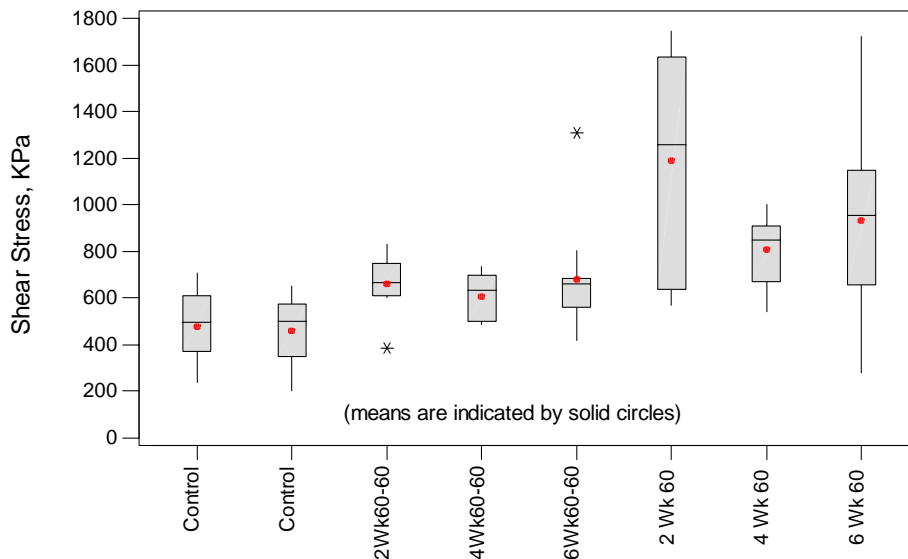


Figure 3: A box plot of the experimental data by sample group. The group average is indicated by the solid dot. The box and extension line illustrates the spread of the data.

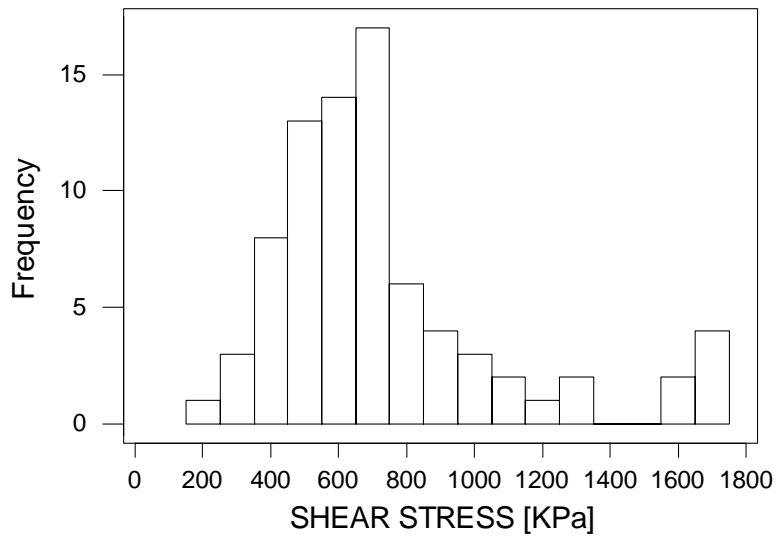


Figure 4: A histogram illustrating the shape, spread and center of the raw data.

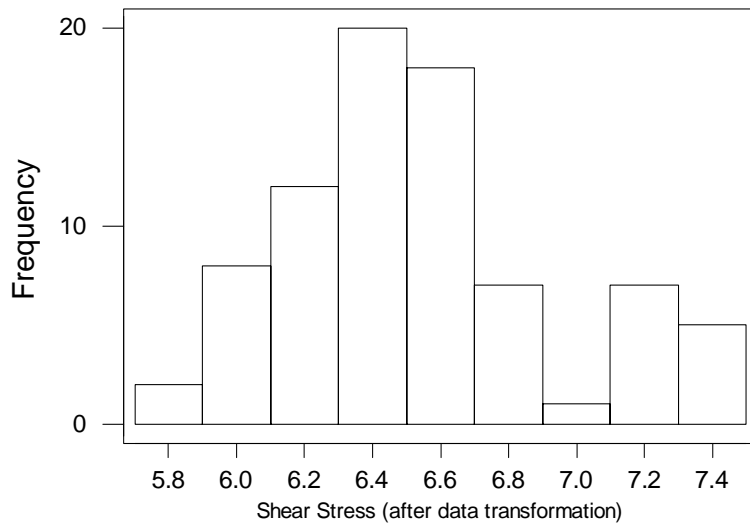


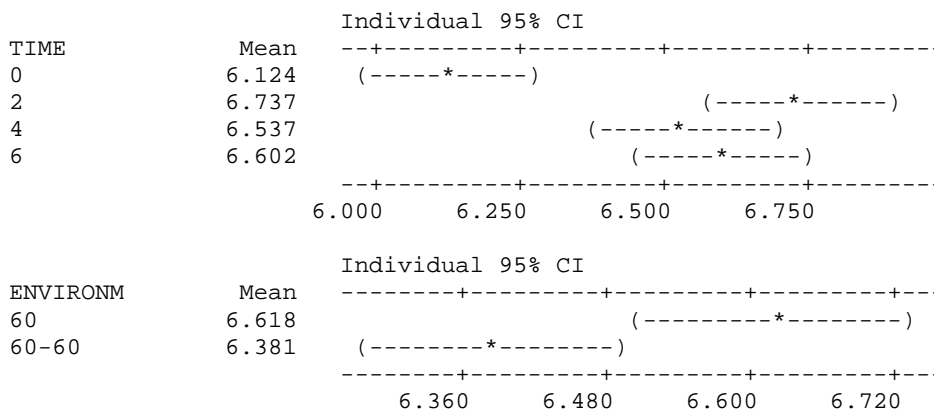
Figure 5: Same as Figure 4. The shear stress data were transformed with Box-Cox transformation method to fit data into a normal distribution.

With the basic requirement for ANOVA fulfilled, the effect of time and aging conditions on adhesive durability was assessed. Table 2 provided the summarized result from the ANOVA. Among the different columns in Table 2, only column with  $p$ -values were used for final assessment of the experimental factors. With alpha of 0.05 or confidence level of 95%, the significance of the  $p$ -values in Table 2 was interpreted. Since all the  $p$ -values were all less than 0.05, time, aging condition, and their interaction had an effect on the adhesive durability. Table 3 illustrated these effects.

Table 2: Two-way balanced ANOVA results (with data from Box-Cox transformation)

Source	DF	SS	MS	F	P
TIME	3	4.188	1.396	11.78	0.000
ENVIRONM	1	1.123	1.123	9.47	0.003
Interaction	3	0.995	0.332	2.80	0.046
Error	72	8.533	0.119		
Total	79	14.839			

Table 3: A breakdown of ANOVA results (with data from Box-Cox transformation)



Although all the  $p$ -values are less than alpha of 0.05, the interaction between time and aging conditions had a significant effect on adhesive durability. It was not observed that

by exposing the adhesive in normal indoor environment, the adhesive strength would increase. It was observed time and aging conditions played a role in adhesive durability in this particular experiment.

## **Discussion**

The factors for this experiment were selected to resemble real world conditions. For indoor environment, temperature and humidity are the primary factors affecting adhesive joints. The amount of fluctuation for these two environmental conditions will depend on whether the indoor environment is air-conditioned or not. In addition, time in combination with these environmental conditions will affect the performance of the adhesive joint. Due to the time restraint of this experiment, the amount of time to age the test specimens was limited to six weeks maximum. In this regards, no deterioration was observed on the adhesive joints. Referring to Figure 3, the average shear stress actually increased with the aging conditions for the time specified. Through a t-test comparison with a confidence level of 95%, the average shear stress,  $982\pm 155$  kPa, for the sample group heated at  $60\text{ }^{\circ}\text{C}$  was higher than the average shear stress,  $651\pm 58$  kPa, for specimens exposed to  $60\text{ }^{\circ}\text{C}/60\%$  RH. This observation was not surprising given that other experiments have shown water affects both the adhesive and the adhesive/adherend interfacial zone [5]. The shear stress for the control samples was determined to be  $470\pm 59$  kPa. This shear stress value was close to data (550 kPa) provided by the product manufacturer.

For the time allotted to this experiment and the experimental factors selected, the adhesive durability did not show signs of deterioration. On the contrary, there was indication that the adhesive strength increased with exposure to 60 °C environment, see Figure 3. This increase in adhesive strength was in line with manufacturer data indicating that there can be an increase in adhesive strength with exposure to elevated temperature (66 °C).

The spread in the adhesive shear stress data could be attributed to surface preparation of the adherends. This observation was based on the joint failure mode, which was mostly adhesive failure. With good adherence, a cohesive failure may be produced. More rigorous cleaning methods for the adherend surface were not chosen because of the objective to resemble real world conditions. Hence, a practical and cost effective cleaning like isopropyl alcohol rubbing of the adherends were used in preparing the adhesive surfaces. It had been found, however, surface pre-treatment like etching, solvent degreasing enhance the strength of adhesives [1]. It is well known that surface pre-treatments do have an effect on adhesive strength; in this regards, the effect of surface pre-treatments on adhesive strength will require a separate experiment.

## **Conclusion**

Within the limitation of this experiment, few conclusions can be drawn. These conclusions are:

- No deterioration of the adhesive strength was found after environmental exposures of 60°C heat and/or 60°C/60% relative humidity.

- A longer experimentation time for each of the controlled factors would deliver a more comprehensive array of data
- More varying of the testing factors, (ie. Varying the Relative Humidity and Temperature incrementally) to give the data a wider spread.

## **Acknowledgments**

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## Appendix A

Lap Shear Data:

Time, wk	60 °C Soak		60 °C/60% RH Soak	
	Shear Strength, kPa	Failure Mode	Shear Strength, kPa	Failure Mode
0	535	adhesive	545	adhesive
	705	adhesive	522	adhesive
	627	adhesive	549	adhesive
	375	adhesive	252	adhesive
	400	mostly adhesive	481	adhesive
	629	adhesive	204	adhesive
	456	adhesive	645	adhesive
	541	adhesive	379	adhesive
	325	adhesive	651	adhesive
	370	adhesive	381	adhesive
2	1678	adhesive/cohesive	703	adhesive
	689	adhesive	730	adhesive
	639	adhesive	664	adhesive
	1744	adhesive/cohesive	616	adhesive
	1615	adhesive/cohesive	832	adhesive
	1259	adhesive/cohesive	797	adhesive
	624	adhesive	601	adhesive
	1575	adhesive/cohesive	622	adhesive
	1671	mostly cohesive	385	adhesive
	570	adhesive	666	adhesive
4	1001	adhesive	606	adhesive
	884	adhesive	487	adhesive
	687	adhesive	679	adhesive
	778	adhesive	506	adhesive
	895	adhesive	699	adhesive
	624	adhesive	487	adhesive
	951	adhesive	734	adhesive
	851	adhesive	658	adhesive/cohesive
	849	adhesive	527	adhesive
	543	adhesive	695	adhesive
6	874	adhesive	676	adhesive
	1124	adhesive	557	adhesive
	1224	adhesive	1307	adhesive/cohesive
	1080	adhesive	661	adhesive/cohesive
	1722	adhesive/cohesive	420	adhesive
	739	adhesive	682	adhesive
	1036	adhesive	803	adhesive/cohesive
	834	adhesive	507	adhesive/cohesive
	281	adhesive	566	adhesive
	414	adhesive	622	adhesive