# An Investigation of Strength of Adhesives & Single Lap Adhesive Joints at Various Temperatures and Filler Concentrations


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# An Investigation of Strength of Adhesives & Single Lap Adhesive Joints at Various Temperatures and Filler Concentrations

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A thesis submitted in partial fulfillment of the requirements for the degree of

MS Mechanical Engineering

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*This work is dedicated to my parents, my brother & sisters who have always loved me unconditionally and whose excellent examples have taught me to work hard for the things that I aspire to achieve.*

# Abstract

The research presents the behavior of the mechanical properties of epoxy adhesives & adhesively bonded single lap joints made using same epoxy adhesives (LY-556 /AD 22962). Research has shown that various factors impact the overall strength of epoxy adhesives & adhesively bonded single lap joints and their strength can be improved through various techniques and processes. One of the effective methods is addition of filler material to adhesive. Cork powder is a versatile natural raw material and being used as a crack stopping filler for enhancing the strength of adhesives & adhesively bonded single lap joints. However, this behavior of cork powder changes with change in filler concentration. Present study is focused on observing the variation in mechanical properties of epoxy adhesive & adhesively bonded single lap joints at different temperatures and different concentrations of cork powder and their effect on mechanical properties of adhesives & adhesively bonded single lap joints. An experimental investigation is conducted to study the strength of LY-556 epoxy adhesives & adhesively bonded single lap joints with Aluminum 5052 adherends at different temperatures and different cork filler concentration. The adhesives & single lap joints are tested under tensile testing at ultimate testing machine. The temperature ranges from 25 degrees, 50 degrees, 75 degrees and 100 degrees and the cork powder concentration for each temperature are 0.25wt.%, 0.5wt.%, 0.75wt.% and 1wt.%. It is observed that for different temperature and for each concentration, the strength of adhesives reflects an increase in toughness with addition of cork powder while overall tensile strength & modulus decreases with increase in cork powder & temperature. Single lap joints shows similar behavior trend so that he highest failure strength is observed at room temperature and at 0.5wt.% cork powder concentration and minimum strength is observed at 100 degrees (close to glass transition temperature) and at 1wt.% concentration. The type of failure is changes from mix mode failure to cohesive failure as temperature and cork powder changes from low to high.



***Keywords:*** *adhesives, single lap adhesively bonded joint, cork powder concentration, temperature, strength*

# Table of Contents

# List of Figures

# List of Tables

# Chapter 1: Introduction

## 1.1 Motivation

From the beginning of aircraft design, it has been witnessed that adhesives were utilized in joining the wooden pieces of the very first aircraft of history. Different wooden elements were initially used in designing of aircrafts but due to low strength, it did not provide the required strength as compared to solutions like metal structure. Metallic structures were able to fly at higher altitude with lower fatigue effects due to higher strength. However, metallic structure joints required to be joined through fasteners and mechanical hardware (bolts, rivets, welds etc.) while wooden joints could be joined through various adhesives. Due to addition of metallic structure and hardware, weight to fuel consumption ratio became much more along with higher cost effects [1].

A method of combining materials called adhesive bonding involves which adherend (structural element to be joined) is bonded through an adhesive which is placed between the adherend surfaces. When the adhesive solidifies between adherend layers, it produces an adhesive bond which provides great strength and reduces chances stress singularities in the joining structures [2].The primary characteristic in an adhesively bonded joints is the overall mechanical strength of the joint and it directly effects other properties of the joint. Moreover in order to avoid structural damage, joint failure need to be reduced for which an improved stiffness is required. Therefore, it is appropriate to use adhesive bonding as an alternative of metal fastening. Apart from weight reduction in the joint (up to 10 – 30% than mechanical joints), adhesive joints provide an increased stiffness, better sealing properties, resistance to degradation through corrosion and improved fatigue strength [3]

## 1.2 Adhesives

Adhesive is a substance that is used to adhere / bond two distinct materials together. Adhesive behavior is quite difficult to identify since it is categorized in so many ways, such as by source, chemical properties, application area, physical formulation, modes of application and temperature settings, thermosetting, thermoplastic, heat / pressure sensitivity. Adhesives are further classified into two categories based on their functions: structural adhesives and non-structural adhesives [4].

### 1.2.1 Structural Adhesive

Such materials perform and exhibit exceptional strength in critical stress areas. Their major role is to keep structures together and make them strong enough to withstand heavy loads and function normally for longer durations [5]. Table 2.3 displays these different categories of adhesives.

Table 1.1 Types of adhesives

| Adhesive Material | Modifier |
|---|---|
| Epoxy | |
| Modified (or alloyed) epoxy | Toughener, nylon, phenolic, polysulfide, resorcinol and phenol formaldehyde, melamine, and urea-formaldehyde |
| Modified (or alloyed) phenolic | Nitrile, vinyl, neoprene |
| Polyaromatics | |
| Polyester | |
| Polyurethane | |
| Anaerobic | |
| Cyanoacrylate | |
| Modified acrylic | |
| Neoprene (chloroprene) | |
| Nitriles (acrylonitrile-butadiene) | |
| Polysulfide | |

### 1.2.2 Non-Structural Adhesives

These adhesives only need to hold structures in place without withstanding extreme loads; they are not intended to bear heavy loads [6]. Similar to pressure-sensitive tapes, this category is sometimes referred to as "holding adhesives."

The structural adhesives further divides epoxy into two categories:

- One has low viscosity
- Other is solids with a high melting point

For high strength, high impact resistance, and toughness, only low viscosity adhesives of these types are utilized in aircraft.

### 1.2.3 Adhesive Selection

There are numbers of criteria for selecting the adhesives which fulfill over specific requirement as shown below [7].

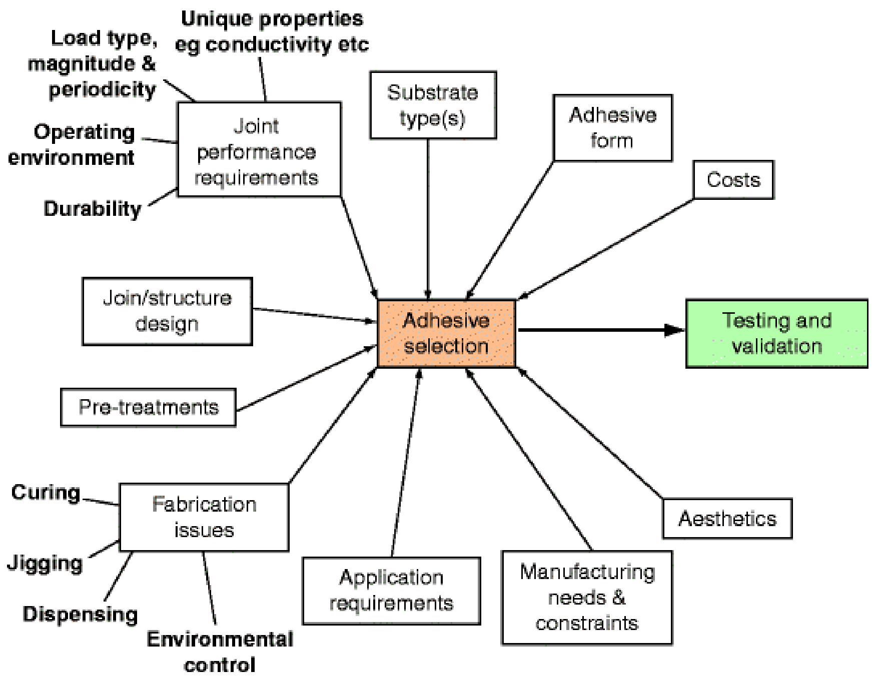



Fig. 1.1 Important Criteria to consider when selecting adhesives

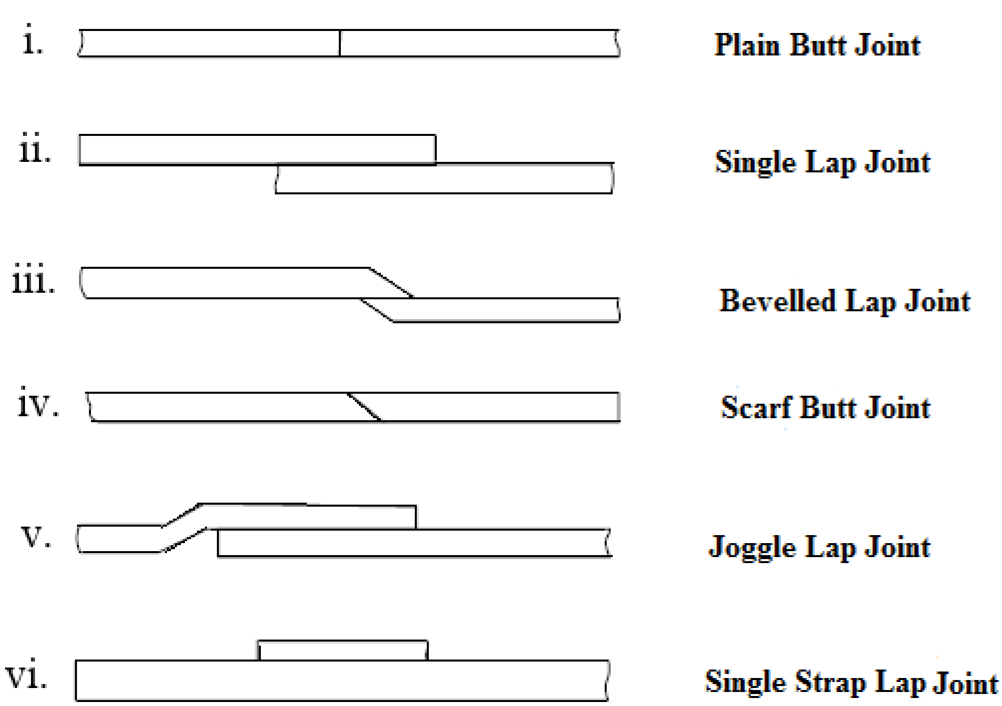
## 1.3 Adhesive Joints

Adhesive bonding can provide one or more of the following benefits, depending on the type of adhesives used, joint configuration, application area:

- The capacity to combine several similar and dissimilar materials, metals and composite, whose composition, mechanical and physical properties may vary (thin / thick sheets and foils joined where other fastening methods would cause distortion or disfiguration of geometry). [8]
- The removal of uneven surface contours brought on by mechanical fasteners like screws / bolts, improving the appearance of the completed joint assembly [9].
- The more consistent distribution of loading stresses over the whole adhesively bonded area with reduction of stress concentrations, which are more likely to appear with mechanical hardware. This facilitates the bonding processes for much thinner adherends which can be easily joined without damage to strength and by saving cost and overall weight. [10]

- No additional fasteners contribute to weight reduction and no holes are to be drilled which contributes in structural integrity of load bearing members [11].
- Adhesive joints have a benefit that they have an ability to bond heat sensitive materials which can be possibly damaged by conventional joining methods such as brazing / welding.
- These joints offer an improved sealing / protection properties against environmental factors such as moisture and chemical exposure, electric charge, thermal, or acoustic damage, and also lower corrosion chances through hardware / fasteners between dissimilar metals [12].
- Adhesive joints offer simpler assembly processes and hence contribute to reduction in additional costs incurred to hardware requirements and in turn providing weight saving through a single bond between structural adherends [13].

Adhesive joints have wide areas of application and based on structural requirements, there are various kinds of adhesive joints. Some of the examples of such joints are single lap joints, tapered joints, double lap joints, strap joints etc. Figure x below shows a few examples of the similar type of adhesive joints.



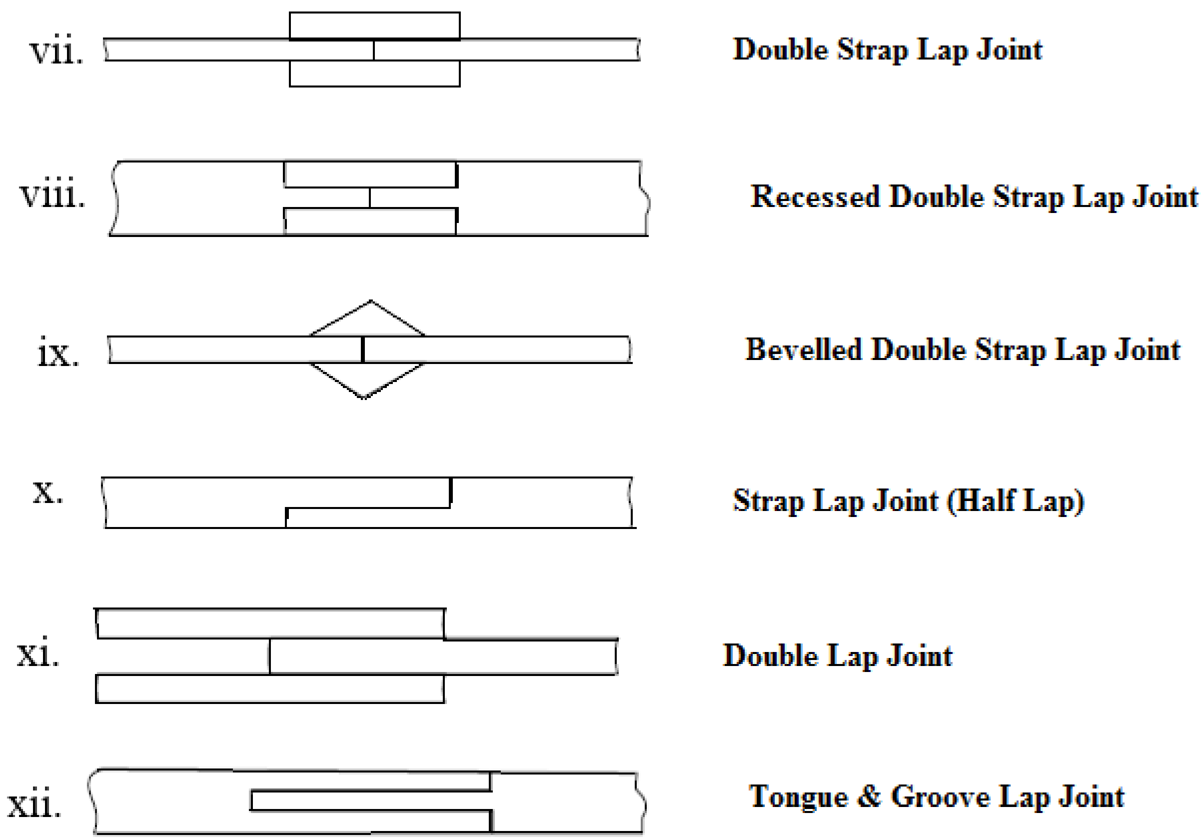



Fig. 1.2 Different types of Adhesive Joints

Various joint configurations are utilized by researchers & engineer based on the application area of the joints. In this regard, single-lap joints (SLJs) are broadly used in major structural elements and load bearing members of various assemblies since these joints have a high strength and provide great support with a very simple geometry (overlap bond between two adhering materials). Single lap joints are a common choice utilized for assembling and joining of both similar and dissimilar materials. Due to simple geometry, application advantages and high strength of single lap joints, these are most commonly utilized joints in many structural areas and critical load bearing members of complex structures and numerous research / experiments are being conducted on adhesives & SLJs to study their behavior and study the methods to improve strength of adhesives & SLJs [14]. The strength of adhesives & SLJs depends on various factors and also the magnitude to which these factors are varied, these factors can be: -

- Adhesive bonding region (joint overlap length)
- Choice of joint adherends

- Joint thickness
- Curing conditions of adhesive
- Surrounding temperature during testing
- Use of nano / micro fillers in adhesives

Keeping in view, all the above stated and other factors, various techniques & testing procedures have been thoroughly developed by researchers so that the response of adhesives & adhesively bonded SLJs in terms of overall strength and toughness [15]. In this interest, both analytical and experimental researches have been widely conducted throughout the years. Many computational analysis models have also been designed to study the effects of above stated factors. In literature, a comprehensive research data is presented regarding the crucial factors that enhance cohesive & adhesive properties of an adhesive and these factors can impact the strength of a joint in such a way that strength might increase or decrease, or its stress concentration will vary [16]. Methods to increase the strength of adhesives & in turn the strength of adhesively bonded SLJs through variation of certain elements in both adhesives & adherends has been developed. Some techniques to study the mechanical behavior of adhesives & joints are reviewed in Chapter 2.

## 1.4 Application Areas of Adhesive Joints

Adhesively bonded joints have become a popular choice in comparison to conventional mechanical joints in various engineering fields (aerospace, civil, structural, marine etc.) since they are a lightweight solution with lesser fabrication cost and improvised damage tolerance. The application of these joints in structural components has significantly increased because adhesive joints have higher structural integrity in comparison to mechanical joints since there is no provision for stress concentrations which contribute to deterioration of structural integrity of a joint [17].


Fig. 1.3 State-of-the-art adhesive bonding applications on Airbus A380

A greater benefit offered by adhesives is that a large variety and types of adhesives are available based on area of application (commercial, aviation, automotive etc.) [18]. Due to their promising characteristics and high processing potentials, there has been an increased utilization of epoxy resins in various industries and structural application areas [19]. Adequate results of adhesion to several materials have been reported in epoxy resins due to their polar nature. Moreover, epoxy adhesives are distinguished by their adhesion response and chemical resistance and hence weighted with better properties than most other adhesives [20]. Another key benefit reported by many researchers is that epoxy resins is that they have a diverse variety of resins and multiple option of curing agents and the option of altering the properties of epoxy adhesives give a chance for selection of the best suited composition for the bonded material and the operating conditions [9,12,16–18].

Adhesively bonded joints have a wide application area because of significant mechanical advantages such as reduced weight, reduced cost effects, higher coefficient of stiffness, higher strength to weight ratio in comparison to conventional joining methods like mechanically fastened joints [21]. Surface treatment process of adherend surface during the preparation of adhesively bonded joints is a deciding factor in strength of the joint [22]. The mechanical strength of a joint is the key characteristic of any adhesively bonded joint. Moreover, material adhesion phenomena is a complex factor along with the strength measurement. Adhesive joints' strength can be measured through multiple testing methods. Despite being a superior alternative to mechanically fastened joints, adhesive joints have different regime of complications such as strength of adhesive joints is directly dependent of thermal expansion coefficient of joined material and hence temperature effects in adhesive joints are more instrumental than mechanical joints.

## 1.5 Characteristics of Adhesives & Adhesively Bonded Joints

In theory, studies show that the overall failure loads of an adhesive & adhesively bonded joint are significantly impacted by the physical properties of selected adhesives such as thermosetting, thermoplastic, heat-activated, synthetic and natural adhesives and the adherends (metallic / composite) in a joint, also the type of joint (such as double strap, tapered scarf, single lap), selection of appropriate surface treatment processes as per selected adherend, thickness of adhesive layer used in the bonding region of joint & adherends thickness, overlap length of joints, testing methodologies, mould preparation for adhesive samples, curing setup, calibration of testing equipment, surrounding environment, testing temperatures, moisture and handling of researcher. Nonetheless, some factors are usually constrained in actual applications and a small tolerance is available to change these factors. Based on the existing limitations, an interesting area with need of research is to optimize the behavior of adhesives under variation of different factors & study the response of these adhesives in the form of adhesively bonded joints so that it is possible to sustain higher loads and improve overall toughness & load transfer ability of these adhesives & adhesively bonded joints in numerous mechanical applications [23], [24].

The mechanical strength & failure modes of different adherends in single lap joints has been documented by observing the effect of joint overlap length and thickness of joint adherends on the strength and failure mode of adhesives & adhesively bonded single lap joints. Based on experimental data, it has been noticed that the shear strength behavior is largely affected by overlap length of joints as compared to joint thickness [25].

The adhesive bonding of aluminum adherends signifies a major advantage and a useful alternate to conventional mechanical joining techniques, such as bolting & welding of joints. Adhesives provide a uniform joining and adhesive bonding of joints provides a more uniform stress distribution and reduces any chances of stress singularities, also adhesives can join smaller components without any complexities, can avoid or diminish the corrosion chances between dissimilar materials, and there is no requirement of holes that can initiate cracks in the structure. The characteristics of an adhesively bonded joint differ with the type of material used for joining, adhesive / cohesive properties of the adhesive used, surface cleaning & preparing prior to joining etc. The area adjacent to the interface between adherend and the polymer adhesive is the essential parameter in enhancing the mechanical properties of the joint. It has been recorded in research that debonding phenomena is mostly interfacial or in close proximity to interface, and clearly associated to interphase structure. Weak boundary layers play a significant role in influencing the fracture stresses of adhesives & adhesively bonded joints. The fracture strength behavior of various adhesive lap joints has been used as testimony for the existence / non-existence of weaker boundary layers.

The impact of low / high temperature on the strength & fracture behavior of epoxy adhesives & adhesive joints are also been investigated in some research papers. Studies found that the temperature values before the glass transition temperature (Tg) depict quite higher strength and modulus of adhesive joint but at that point their ductility is comparatively reduced. On the contrast, at the temperature values above Tg, adhesives demonstrate a much more flexible and tougher behavior but at the same time, tensile strength & modulus will reduce. Hence, it has been found in multiple studies that the temperature greatly influences adhesive bond properties of epoxy adhesives & adhesively bonded single lap joints.

Adhesive strength and the strength of single lap joints is increased by addition of various concentration of natural fillers such as cork particles. Cork powder acts like a crack stopper and fills the empty intercellular spaces in the adhesive and hence it hinders the growth of any cracks in the adhesive. It is hence established that cork particle improves the fracture toughness of epoxy adhesives and adding cork particle along with surface treatment of adherends has lower strain energy rate as compared to the adhesive with cork particle and without surface treatment in case of epoxy adhesives.

## 1.6 Adhesive Test Methods

In mechanical engineering, testing materials is essential for determining how they will behave under various specimen forms, stresses, and environmental conditions. However, when examining adhesive joints, testing must take into account a number of factors, including:

- Mechanical behavior of adhesives & adhesive joints under various loading cases (tensile, shear, peel, impact, fatigue etc.)
- Adhesive response, weather the failure is cohesive / adhesive
- Impact of temperature on strength of adhesives & adhesive joints
- Study impact of adhesive parameters (curing conditions, addition of fillers etc.) on adhesives & adhesive joints strength, toughness etc.

Society of American Engineers (SAE) provides some tests (Aerospace Recommended Practices i.e. ARP) for assessing the characteristics of adhesives & adhesives joints between metal / metal or metal / composite, these practices are mentioned below:

### 1.6.1 Tensile Test

It is a standard test for determining the adhesive's & joints' tensile strength & tensile modulus by applying a normal force on the specimens. Tensile tests have the benefit of demonstrating elastic and inelastic behavior of adhesives & joints, which helps calculate adhesive structure's fracture energy, strain rates and failure loads [26].

### 1.6.2 Shear Test

Because shear force is the primary source of in operation failures, shear tests are frequently used to evaluate adhesives & joints behavior. The stress distribution along the bond is not constant during tensile testing. While several factors, such as adhesive thickness and adherend stiffness, influence the shear test results, it is higher than normal in the bond line [27].

### 1.6.3 Peel Test

It is used to measure ductile adhesives' resilience to intensely localized stresses. Peeling force is always exerted along the front line. The load transmission is maximum in such testing process [28].

### 1.6.4 Fatigue Test

The performance of bonded structures under cyclic tensile loading cannot be explained by static tensile testing. An ASTM fatigue test is used to explain this phenomena. Through this testing procedure, three types of this loading are also recognized (entirely reversed, repeating, and fluctuated). Failure cycles is noted after test, and data is displayed in the form of an S-N graph. The fatigue life of the bonded joint against various cycles is essentially shown in this S-N graph [29].

### 1.6.5 Impact Test

Impact testing examines the adhesives & adhesive joint's capacity to absorb energy. This energy (force) is applied to the specimen over a very brief period of time. Several techniques are utilised to conduct this test, including the pendulum or gravity impact test and compressed air drop test. According to ASTM standards, these tests assess how rapidly an adhesive / adhesive joint becomes sensitive to an applied loading conditions [30].

## 1.7 Problem Statement

The mechanical characteristics of structural adhesives, such as toughness, can be enhanced by adding particles (nano or micro). In addition to their great strength and stiffness, structural adhesives are also renowned for their poor ductility and toughness. The goal of this thesis is to increase the failure loads of brittle epoxies and SLJs as well as to identify a desired amount of cork powder and temperature condition to improve overall strength of SLJs. The suggested approach addresses both of these issues since testing are carried out for both epoxy adhesive and single lap adhesively bonded joints under varied temperature settings. Epoxy resins are reinforced by the addition of cork powder. This natural reinforcement offers excellent impact energy absorption and provides improved thermal properties. The cork particles behave as impediments to the transmission of the crack and hence contribute in improving the toughness of epoxy adhesive.

Additionally, this approach would permit the usage of cork powder, a product that the cork business hasn't yet looked at. The effective usage of this particle for reinforcing epoxy adhesives & adhesive joints would open chances to utilize natural particles like cork with many potential benefits.

## 1.8 Aims and Objectives

This study's primary goal is to examine how cork particles can be used as a reinforcement material for fragile adhesives under varied temperature conditions. Details are highlighted below:

- To investigate the fundamental mechanical properties (tensile strength & of the selected adhesive with and without cork powder
- To determine the best combination of cork powder and adhesive, i.e., the concentrations to improve adhesive toughness
- To study the effect of cork powder on adhesively bonded single lap joints and compare the behavior of adhesives & adhesive joint configuration
- To assess the effect of temperature on the strength and failure load capacity of adhesives & the study the impact on adhesively bonded joints
- Prediction of strength variation with cork powder and temperature variation and analyze combined effect of both factors (if any)
- To compare failure loads phenomena in adhesives & adhesively bonded single lap joints

## 1.9 Research methodology

The following methodology was used to accomplish the goals of this MS thesis:

- Over the last few decades, a number of latest techniques for making structural adhesives more durable have been created. As a result, literature study of techniques for enhancing the toughness of adhesives with addition of various nano / micro particles was conducted. The primary techniques for toughening adhesives are summarized with a focus on cork particles.
- High temperatures make the adhesive more ductile but weaker and more prone to creep, whereas low temperatures make the adhesive more brittle (lower strain to failure). Response of various adhesives to varying temperatures was studied in literature review.
- At various temperatures, the mechanical characteristics of the glue reinforced with microscopic cork particles were evaluated. The choice of tensile tests as a method for assessing the impact of cork particle concentration and different environmental temperatures conditions.

- Tensile tests were conducted to assess the influence of the cork amount (0, 0.25, 0.5, 0.75, and 1 wt percent) at various temperatures (25, 50, 75, and 100°C) in order to comprehend this effect. It was feasible to make some inferences regarding the impact of the amount of cork on the ductility of the glue by combining the effects of these two parameters.
- Tensile tests of adhesive dog bone shaped specimens were performed to observe the influence of cork particles concentration & temperature on adhesive intrinsic and the response of adhesive after application in adhesive joint was studied.
- Tensile experiments on single lap joints are being tested to check the strength on the basis of two different parameters. All the experiments are done on either different temperatures (25, 50, 75, 100°C) or cork powder concentration (0, 0.25, 0.5, 0.75 and 1 wt%) on aluminum adhesive joints.
- Ultimate testing machine is being used to test the strength of epoxy adhesives & single lap joints under tensile testing.

Full factorial design of experiment for present work is as shown:

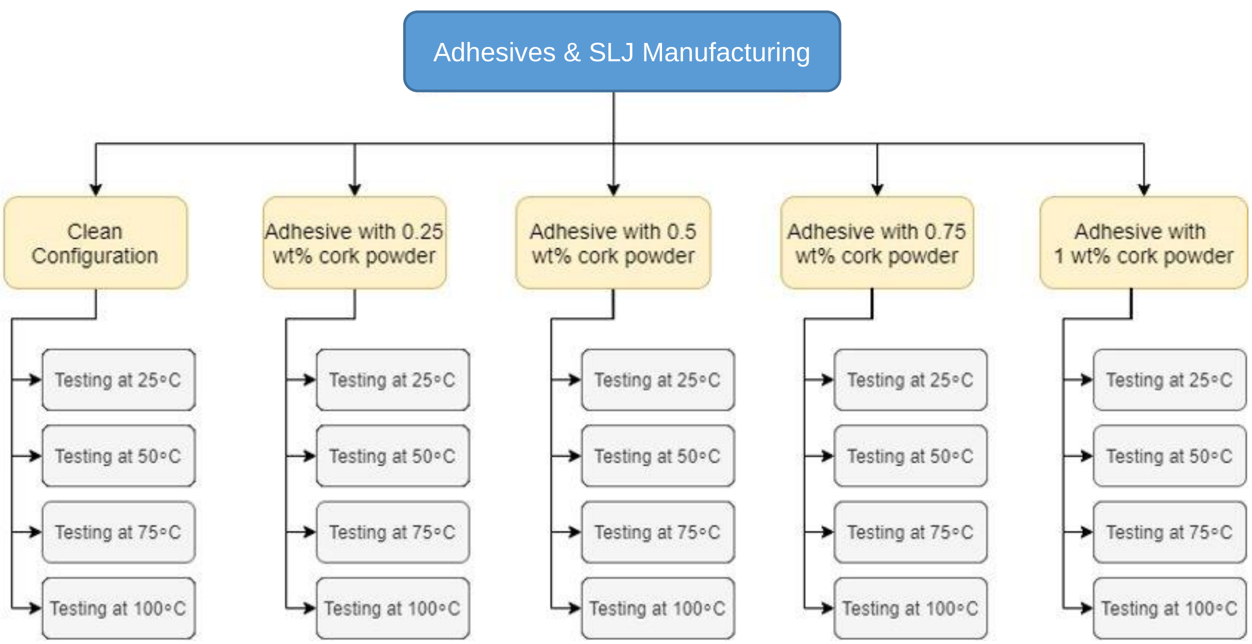



Fig. 1.4 Schematic diagram of cork specimens at different amount and different temperatures

# Chapter 2: Literature Review

This chapter presents a literature review of the changes in the properties of commonly used adhesives & adhesively bonded joints by changing variables such as filler addition, temperature, specimen geometry, surface treatments, joint configuration, adherend geometries and overlap length on adhesive / cohesive properties of both adhesives individually and their response in the adhesive joint configuration.

## 2.1 Introduction

**Adhesives.** Comparing structural adhesives to more conventional methods of joining two components, such welding, structural adhesives are often the best choice. Epoxy resin is one of the most popular structural adhesives. Structural adhesives are innately brittle (low ductility and toughness) and weak against fracture propagation due to the high molecular crosslinking that gives them their exceptional properties. It is possible to toughen and add a second phase to an adhesive to boost its capacity to absorb energy without rupturing. While barely changing the core characteristics of the matrix resin, this increases resistance to impact and fracture.

Because of their superior mechanical, thermal, and chemical characteristics, epoxies are the most often used structural adhesives. They cost more and have more production challenges, but they also have similar greater levels of strength, stiffness, toughness, durability, and chemical resistance. Since epoxies are thermoset materials, they become amorphous and extremely reticulated after polymerizing. Epoxy resins are monomeric or oligomeric compounds having two or more epoxide rings. Epoxy resins can be opened catalytically or stoichiometrically to form a cross-linked network by reacting with multifunctional amines or carboxylic acids. This microstructure produces properties that are extremely advantageous for structural engineering applications, including high elastic and strength modulus, little creep, and great thermal strength.

**Adhesive Joints.** Because they are more cheap and have better strength, durability, and fatigue resistance, adhesive joints are used. Because of the potential uses for them, adhesives have drawn research interest. Today, adhesives are widely used in the aerospace, industrial, and medical industries. In applications involving adhesively bonded joints, it's critical to lower stress concentrations and raise the failure load. The most fundamental kind of adhesive junction, known as a single lap joint (SLJ), makes it simple to attach two adherents together. SLJs are well-liked because of how useful and easy they are. Several methods can be used to predict the strength of adhesive connections. Several numerical and analytical techniques are used to forecast the strength of an adhesive junction, as well as the maximum load. The strength of adhesive joints is influenced by the characteristics of the adhesives, the material of the adherend, temperature, overlap length, and nanoparticle concentration. Many techniques have been tested over a long period of time to increase the strength of single lap joints. The strength of adhesive joints can be predicted using a variety of techniques.

The primary techniques for producing a reinforced material using microparticles are outlined in this study, along with an overview of current advancements in the use of reinforcement particles in adhesive technology. The primary factors that affect the performance of the particles—specifically, their nature and qualities, which depend on their size, interparticle distance, particle/matrix interaction, and volume fraction—and how they affect the final composite's or adhesive's toughness—were discussed. The report concludes with a few suggestions for using cork microparticles as a reinforcement material for adhesive joints. [31].

The behavior of adhesive under the impact of temperature was examined by Nguyen, T.-C., et al., along with time-dependent factors and loading factors. The glass transition temperature Tg for adhesive is 42°C, and the load conditions are 20%, 50%, and 80% of their maximum load. The constant temperature varies from 35 to 50°C, and the cyclic temperature ranges from 20 to 50°C. (UL). Results from various scenarios indicate that when the load is less than its maximum and the temperature is constant, the load carrying capacity diminishes over time. Adhesives exposed to 20 percent UL and a target temperature of 50°C failed after 25 minutes, adhesives exposed to 80 percent UL and a target temperature of 35°C (five 0C below the adhesive's Tg) did not fail after 150 minutes, and after cooling from thermal exposure, the adhesives displayed 100 percent UL in comparison to the unconditioned specimens. In comparison to the unconditioned specimens, the joints loaded at 20 percent of their ultimate load and exposed to cycling temperatures between 20 and 50°C for 400 minutes without failing revealed a 67 to 72 percent UL. Based on these findings, it can be said that adhesives are safe from failure if the adhesive layer is kept at least 7 to 10°C below the glass transition point and that cyclic thermal loading has little impact on the strength reduction of adhesives [32].

Understanding the mechanical and physical behaviour as a function of the cure temperature is crucial since the properties of structural adhesives can vary significantly as a function of the cure temperature.

This study examined how different structural adhesives behave mechanically and physically in relation to cure temperature. Not less significant was the evaluation of how post-cure conditions affected the mechanical and physical behaviour of adhesives. Knowing the Tg and mechanical characteristics of the adhesive after it has been subjected to heat for a period of time is crucial (post-cure conditions). Due to the fact that adhesives are frequently exposed to a wide range of temperatures both during and after manufacture, a complete understanding of the behaviour of adhesives is necessary [33], [34].

Investigated were the mechanical characteristics of epoxy reinforced with silicon carbide nanoparticles at various weight percentages. The experimental results show that strength decreases as reinforcement weight percentage is increased further. The tenuous link between the matrix and the nanoparticles may be to blame for this. The wear data demonstrate that nano particles improved the unreinforced epoxy's wear resistance; this is because of the ceramic The counter face that nano particle slide across has a rough surface due to the nano particles' size. [35].

Researchers have looked into the possibility of altering the behaviour of thermosetting epoxy resin by adding various types of fillers, such as hard ceramics like boron carbide (B4C), silicon carbide (SiC), and alumina (Al2O3), which are discovered to improve the mechanical and wear properties of epoxy. For epoxy to be suited for low friction and low wear conditions, lubricant fillers such as graphite, molybdenum sulphide (MoS2), and polytetrafluoroethylene (PTFE) are sometimes employed as fillers. Fillers are given various pre-treatments in order to improve the interfacial contact between them and the matrix. The outcomes of these studies demonstrated improvement in the mechanical and tribological properties of composites, i.e., the resin's capabilities for friction, lubrication, and wear. [36].

This review paper explored various methods for enhancing the tensile strength of adhesive joints as well as for reducing stress concentration with composite adhesives. Different methodologies are used to compare the geometry design and material arrangement. Different material-based strategies, such as graded adhesive, graded adherends, and transverse adherend toughness, serve to increase the transverse strength of adhesive connections by reducing the stress distribution in the adhesive and adherends and improving surface roughness by employing rivets and bolts. According to the assessment, the shape should be carefully chosen during design to prevent premature failure of the adhesive or adherends. High strength, high delamination resistance, and high surface roughness are all provided through the fabrication process for composites [37].

In this study, the bending effect in adhesively bonded single lap joints is reduced by strengthening the joints by inserting support patches at various distances from the overlap region. Aluminum alloy AA2024-T3 is employed as the adherend, liquid structured epoxy is used as the adhesive, and support patches made of flat or curved aluminum alloy and steel in a range of thicknesses are used. It has been found through testing that support patches between 16 and 94 percent more damage on single lap joints. The load carrying capability of single lap joints will rise as the rigidity in bending increases along with the thickness of support patches. It has been noted that when the overlap portion is equal to the outside section, then the maximum damage load rises. The curved support patches boost the joints' ability to support more weight [38].

The tensile and compressive strengths of green composite single lap adhesive joints were studied in the article. Epoxy, polyurethane, and parent polymers are the bonding materials used to combine the green composites (PLA). The sample overlap length and breadth have an impact on how well adhesive joins operate. It has been shown that single lap joints with greater width and longer overlaps have superior tensile and compressive strength. The bigger bonding surface gives the bonded joints more energy to fail when under strain. As a result of being stiffer and more rigid than the other two bonding materials, it is determined that epoxy adhesive is the best bonding substance. Under tensile and compressive loads, green composites fail owing to cohesive failure, adhesive failure, fiber tear failure and structural failure [39].

This study uses three different adherend thicknesses and two distinct adherend materials with varied mechanical properties, such as yield, tensile strength, and ductility, to explore the strength of bolted, bonded, and hybrid single lap joints. The maximum load and failure displacement both increase with adherend thickness for bonded single lap joints. Maximum load remained relatively constant in both joints when hybrid joints and bonded joints are compared. When aluminum adherends are employed, the energy absorbed in the hybrid joint is equivalent to the total of the joints. Hybrid single lap joints are more robust than the other two single lap joints since they depend on the adherend material [40] .

In this work, molecular dynamic modelling was used to investigate the impact of adhesive thickness on joint strength. The strength between the adhesive and adherend determines the interfacial strength of the joint under tensile, shear, or combination stress, and failure results from a deficiency in the strength at the joint interface. When the joints' interface is stronger under various stress circumstances, cohesive failure develops. Under mixed mode circumstances, it is challenging to detect the bulk shear. The yield strength is influenced by both the thickness and the interface, and it gets stronger as the thickness is reduced. Strength also increases as density and polymer structure increase [41].

The strength of a single lap joint with two adherends that are different is examined in this research using an external bending moment. Based on adherend thickness ratio, adherend length ratio between dissimilar adherend, and young modulus ratio of adherend, the stress distribution at the interface is assessed. It is observed that when the adherend contact gets smaller and thinner, the intensity of stress distribution at the interface gets stronger. Experiments have shown that the young modulus and adherend thickness have an inverse relationship with joint strength, with the length ratio having very little effect on the strength of single lap joints. Finite element analysis is done for single lap joints with different adherends, and the result is that FEA has same results as observed from experiments FEA has same results as observed from experiments [42] .

In this research, the impact of two distinct adhesives—aluminum and carbon fibre reinforcing polymer—on glass sheets with double lap adhesive joints was investigated. In this study, three epoxy and two acrylic adhesives were evaluated under three distinct temperature settings. When compared to epoxy glue, it has been shown that acrylic adhesive exhibits a decline in mechanical performance as the temperature rises. While acrylic glue has the highest joint elongation capability, epoxy adhesive has the highest load bearing capacity. The failure mode in glass-aluminum samples is mainly the adhesive failure while the failure mode in glass- CFRP samples show cohesive failure or light-fiber-tear failure. At high temperatures, the epoxy and acrylic adhesive mostly show the adhesive failure in term of failure modes. Epoxy adhesive  is considered most effective with large elongation capability [43].

In this study, experiments are conducted to determine how overlap length and adherend thickness affect the strength and failure mode of an epoxy composite reinforced with carbon fibre that has a single lap junction. It has been shown that overlap length has a greater impact on shear strength than thickness. The strength and failure mode of the adhesively bonded single lap joints are assessed using multiple linear regression and neutral networks (NNs) that have been trained using algorithms. Ten examples are tested to compare the error rates of the two methodologies, and it is discovered that multiple linear regression has an error rate of 3.12 percent while NNs have an error rate of 2.27 percent. Both methods produce reliable answers that are based on actual data rather than assumptions and both models can accurately improve the strength of adhesively bonded single lap joints. In industries, these two models are utilized for the bond joining procedure [44].

This paper discussed about the effect of reinforcing adhesively bonded single lap joint on the failure of the joints. Three types of adhesive joints (unreinforced adhesive, adhesive with carbon fiber reinforced composites and adhesives with glass fiber reinforced composites) are used at different length and thickness of the joints. Finite element analysis is carried out to evaluate to failure progress and numerical techniques such as Hashin failure criteria and Tresca failure criteria are used to compare the experimental values with numerical results. When both results are compared it is observed that carbon fiber reinforced composites has highest failure load capacity for thin bond-lines as compared to the other two adhesive joints. For thick bond-lines, the glass fiber reinforced composite is most favorable because it gives highest failure load for thick bond-lines. It is concluded that by decreasing the thickness of the adhesive joints and by increasing the overlap length, the strength of adhesive joint will be increased [45].

The paper studied the strength of single lap joint for three different adhesives using critical longitudinal strain criteria (CLS) with rapid point interpolation method (RPIM). When critical longitudinal criteria used with rapid point integration method give accurate results for all adhesives from brittle to highly ductile and predict the strength of single lap joint with the maximum error of 17%. The critical longitudinal criteria are sensitive toward overlap length so it is suitable to choose smallest and largest overlap length with first intersection point to fix this. The strength prediction of single lap joint is accurate when critical longitudinal criteria with rapid point integration method is used instead of finite element method [46].

The paper studied about strength of single lap joints for different material and dimension using critical longitudinal strain technique (CLS). Fracture mechanism is analyzed by using specific distance and longitudinal strain parameters for five different adhesives (epoxy, silicon, polyurethane, bismalemides and acrylic) and for two different substrates that is steel and aluminum alloys. Critical longitudinal strain technique can also predict failure mode for brittle and ductile adhesives. In this technique, 120 different configurations of single lap joints are used and short and long overlap length, thick to thin bond line and different substrate thickness is also taken. The CLS technique predicts the failure load accurately for intermediate and brittle adhesives for different configurations. CLS is linear function of adhesive and stiffness ratio and for intermediate joints, the relation between stiffness ratio and CLS can easily be attained [47].

This paper studied the effect of tensile load on the adhesively bonded CFRP single lap joint. They take 7 different overlap length, 5 adherend width and 3 stacking sequence of joints to evaluate the experimental and numerical investigation. Finite element is considered to be an accurate method for analyzing the failure mode of single lap joint. It is observed that increasing the adherend width is most suitable for improving the load carrying capacity as compared to increasing the overlap length of the joints and by increasing the overlap length will decrease the cohesive failure and increasing the width is proportional in increasing the cohesive failure. Due to increase in stress concentration across the overlap edges, the stress level is higher and leads to premature failure. Both the cohesive and adhesive failure occur in [45/0/-45/90] composite single lap joints which shows less tensile strength while the adhesive failure occurs in [90/-45/45/0] composite single lap joints which shows a small load carrying capacity [48].

The paper compares the tensile shear strength of single lap joint with different adherend. The material used is carbon/epoxy composite, high elastic limit steel and aluminum alloy. Adherend stiffness and high stiffness adherend material largely impacts the shear strength and by using high stiffness adherend material, shear strength will be highest. Also the shear strength is effected by overlap length depending on the different adherend material. From the numerical analysis it is concluded that with increase the rigidity of the adhesive, the stress distribution will be uniform and by increasing in yield stress reduce the stress level and increase the strength of single lap joints. From experimental results, it is concluded that for steel/steel joints the strength is higher as compared to composite/composite joints which has lower strength [49].

This paper investigates the strength of aluminum double lap joint with different adherend material for artificial aging condition and non-aging conditions to check the performance and mechanical properties of adhesive joints. It is concluded that for EXP1 adhesive, high stiffness is observed after 28 curing phase but EXP2 adhesive shoe high stiffness at all phases and EXP3 shoe irregular behavior. The failure mode in EXP1 and EXP3 are adhesive failure and EXP2 adhesives show mixture of both cohesive and adhesive failure. It is observed from experiment that EXP2 proves to be a most favorable adhesive in term of load bearing capacity and mechanical performance is also maintained under artificial aging [50].

Quasi static shear strength of aluminum single lap adhesive joints is studied at different wt.% of the sphere and rod shaped Nano alumina. The maximum shear strength for both nano alumina is observed at 1.5 wt.%. Split Hopkinson pressure bar system is used for the prediction of dynamic shear strength at two different loading rates and at 1.5wt.% of both nano aluminas. Dynamic shear strength shows significant improvement that is 3 to 7 times than the static shear strength. Sphere nano alumina increases the static and dynamic shear strength as compared to the nano rod alumina and neat adhesives. Sphere nano alumina show cohesive failure as compared to the neat adhesive or adhesive with nano rod [51].

This paper studied about the fatigue behavior of the carbon fiber reinforced plastic (CFRP) and aluminum single lap joint at different cyclical loading and under the quasi- static loadings after a transverse pre-impact. It is evaluated from experiments that with increase in the pre-impact energy, fatigue life of CFRP/Al decreased. Based on the fatigue testing, S-N curves are drawn to get the data and it is shown that by increasing the cyclical loading, fatigue life of joints decreases. It is observed that failure mode in cyclical loading under quasi- static load is cohesive failure because cohesive failure is sensitive to the adhesive strength between adherend and adhesive mainly in aluminum substrate and interfacial failure occur before reaching the static failure strength of adhesive. Transverse pre-impact cause damage in adhesive and adhered and produces mechanical interlocking due to indentation in aluminum adherend. By improving the surface texture on aluminum adherend, the bonding capacity will be highly improved thus increasing the fatigue properties of the joints [52]

This paper experimentally studied about the carbon fiber reinforced plastic and aluminum single lap joints at different strain rates. The microscopic and DIC analyses are used to evaluate the fracture mechanism and deformation process under different loadings. It is observed experimentally with the increase strain rate; joint strength demonstrates increasing trend. Also the joint strength and failure elongation are independent of the axial tensile velocity and both decreased with increasing the transverse pre-impact. At high strain rate, brittle failures occur in the carbon fiber reinforced plastic single lap adhesive joints. Also by increasing strain rate, the failure mode of adhesive is transformed from adhesive and cohesive failure to cohesive failure and fiber tear failure mode [53].

This paper studied about the effect of different loading rates on the strength and failure of single lap joints with carbon fiber reinforced plastic and aluminum alloys. Digital image correlation (DIC) technique is used to evaluate the strain rate at four different loading rates from 2mm/min to 12 mm/min. It is observed that with increasing loading rate from 2 to 12 mm/min, the shear strength increases from 19.3 to 29.2 MPa. The cohesive failure and fiber tear failure occurs at the end of bonding areas and the middle of the bonding areas the failure are due to resin matrix failure of CFRP. In quasi static condition, larger failures in adhesives are due to cohesive failure. The plastic deformation in aluminum plate occur due to torque and lead the adhesive to fail early [54].

This paper studied about the failure analysis or strength of composite-aluminum adhesively bonded single lap joint with different overlap length using finite element method coupled with cohesive zone models. It is observed that stresses are peak at the overlap edges and these stresses are higher at adhesive adherend interface. The behavior of joints is numerically modeled, and it is observed numerically that the strength and failure mode of joints highly depends on the adhesives types. It is observed that maximum load in brittle adhesive with different overlap length is negligible and the maximum load in ductile adhesive shows a linear behavior with different overlap lengths. The brittle adhesives show a quicker failure process and ductile adhesives show cohesive failure under global yielding conditions [55].

In this paper, durability and strength of adhesively bonded aluminum joints in wet environment are investigated. Aluminum alloy are conquered to two distinct surface treatment with chromic sulfuric acid (FPL) and sulfuric acid ferric sulfate (P2). Both surface treatments give same results for strength of joints but the adherend treated with sulfuric acid ferric sulfate in humid condition have high durability. The amount of water absorbed by epoxy adhesives has largely effected the glass transition temperature of epoxy adhesives and strength of the joint. The good mechanical properties, high durability, high glass temperature and high lap shear strength is observed experimentally when new epoxy adhesives are treated with siloxanic hardener. When homopolymerized epoxy adhesives used as initiator, then strength of joint will be improved in wet environment but the strength of joints remains constant after aging [56].

The paper showed the effect of two sided adhesive tape and rigid point connection made from two epoxy adhesive on the strength of single lap joint. TESA dual adhesive and Distal epoxy adhesive is used. Aluminum and GFRP are the composite materials that are joined and static tensile testing at room temperature for 25 samples is being analyzed. It is concluded that highest strength is achieved for 4-point connection model. Model 3 is most satisfactory for energy absorption. Two component epoxy adhesive results in delamination of double sided adhesive tape and rigid point connections. Uniaxial tensile test is carried out on 5 type of mixed-adhesive lap joints and they showed high aesthetics with double sided tape and epoxy adhesives [57].

The paper discussed about strength and failure mode of double strap adhesive joint and single lap GFRP joints. It is also concluded the adhesive type, adhesive thickness and overlap length effect the strength of adhesive joints. The joint strength and load- displacement curve are independent of the adhesive type. The joint strength is decreased by increasing the adhesive thickness and strength is increased by increasing the overlap length. It is noted that joint strength is double strap adhesive joint increase with increase in overlap length but the single joint does not show same behavior. It is also observed that double strap adhesive joint show greater load carrying capacity than the single lap joints. The peel stresses are greater in single lap joint than the double lap joints. In double strap joints, peel stresses are more severe near the tip of joints then at the middle of the joints [58].

In this paper, rapid point interpolation meshless method is used to analyze the stress distribution and strength of adhesively bonded composite single lap joints. To predict the strength, brittle adhesive with varying overlap length is being tested. The stress distribution results obtained from meshless method is then compared with finite element method results and both method show similar trend. Similarly, the strength predicted from critical longitudinal strain criteria will matches with strength predicted experimentally. When rapid point interpolation meshless method is used with bi-material come up with a difficulty which is interface region between the material and this difficulty is solved with simplicity that restrict influence domain in that region [59].

The paper discussed about the effect of notching of adherend on the strength of single lap joints. Finite element method is used for different notch parameter to check the strength of single lap joints. The notch parameter includes notch angle, notch width, notch depth and distance from overlap length. To check the failure mode of single lap joint, 90-degree notch angle is selected by numerical results and 3 different depths with two different adhesive curing method is considered. Adherend notching leads to plastic deformation of adherent based on the geometry and properties of adhesive and adherend thereby improving the joint toughness and give advantage of low energy absorption capability. A simple 90-degree notch with 20% notch ratio increases the strength of single lap joints to 55% and increase the load carrying capacity of the joints. Adherend notching technique not dependent on the curing method [60].

In this paper the effect on adherend notching with one ductile and brittle adhesive are investigated to predict the failure load in single lap joints. This paper includes two steps, in one step the finite element analysis is used to evaluate the effect of different notch parameter on the single lap joint strength and in second step, numerical values are taken to perform experiments. It is observed that failure load in single lap joints depend on the mechanical properties of adhesive material and the notch depth. Adherend notching technique is appropriate technique for improving the strength of joints but the notch in adherend makes the adherend weak. The strength of single lap joint for brittle adhesives improved to 100% when the notch depth ratio is 20% while at same notch depth ratio, the strength single lap joint for ductile adhesives improves only 25% [61].

In this paper, single lap joint with brittle and tough adhesives is studied to check the fracture mechanism of the joints. It is observed experimentally that single lap joint bonded with brittle adhesive show cohesive failure mode while single lap joints bonded with tough adhesive show inter-laminar delamination in carbon fiber reinforcement plastics. To determine the damage in material and confirm the fracture mechanism, finite element method with cohesive zone is used. The failure load decreases as the mesh size decreases. The joints show cohesive failure whose surface is treated by acetone or plasma. The sandpaper treatment on adherend cause intralaminar delamination and leads to inaccurate fracture mechanism. The numerical values match with the experimental data when the current experimental parameters were used in term of failure mode. When different parameters are used then numerical results differ from experimental values in term of failure modes [62].

In this paper, the dual and single adhesive bond is used to check the shear and strength of the single lap joints. Between dissimilar adherend such as CFRP and aluminum, the Araldite and brittle adhesives are used separately. The ductile adhesive is used at the end because of their strength and brittle material is used at the middle of the bonded region. To check the relative displacement between dissimilar materials, digital image correlation method is used and for Finite element analysis, ABAQUS software is used. The peel shear and stresses values are calculated numerically and experimentally, and it is found that both values match closely to each other. In single adhesive, the failure occurs at the interface between aluminum adherend and adhesive. In dual adhesive, the failure at bonded material is not easy at interface between adherend and adhesive and hence increases the bond strength. It is concluded that for better performance and for higher strength, dual adhesive should be most favorable option [63].

The strength of epoxy adhesive, carbon fiber reinforced plastics (CFRP), and adhesively bonded aluminum alloy is examined in this research for scarf and butt joints that are subjected to high temperatures and rapid speeds. The strength and mechanical characteristics of joints at high temperatures are investigated using a variety of analytical techniques, including thermogravimetric analysis and

Fourier transform infrared spectroscopy (FTIR). After testing with heat exposure, several surface treatment techniques and scanning electron microscopy are employed to analyze the fracture mechanism and failure strength of CFRP. It has been found that the adhesive Araldite's post-curing behavior increases its tensile strength, thermal stability, and glass transition temperature. Unaged CFRP exhibits uneven and rough epoxy matrix fracture surfaces, whereas deteriorated CFRP exhibits smoother and regular surfaces. The increased normal stress brought on by the bigger area of fiber tear that causes the joints to fail early considerably reduces the failure strength in deteriorated butt joints. It has been shown that the thermal environment causes the failure strength of the adhesively bonded aluminum alloy to decrease more quickly with increasing normal stress and exposure duration [64].

In this study, the temporal behavior of double-strap adhesive connections made of steel and CFRP is examined at constant temperature and various loading rates. The strength function of time and temperature is used to examine the temporal behavior of joints. Shorter time to failure occurs at the same temperature near the glass transition point and increased load. When compared to constant temperature and the same loading levels, the joint's strength under cyclic temperature increased by up to 47%. Keep the temperature between 7 and 10 degrees centigrade or below the glass transition temperature to prevent strength loss caused by the effects of temperature. When joints are subjected to thermal temperatures of 40, 45, or 50 degrees centigrade, their strength gradually declines over time. The time-failure of steel/CFRP double strap joints is projected when adhesive is subjected to tensile tension and exposed to constant temperature near or above the glass transition temperature [65].

This paper examines the impact of high and low temperatures on adhesive joints. Different components are regulated because of the mechanical characteristics of adhesive joints changing with temperature. Improve the performance of the adhesive joint as well as its resistance to temperature changes. Review shows that tensions caused by shrinkage are much lower and insignificant than stresses caused by thermal expansion. Compared to hot cure adhesives, the water expansion cure adhesives virtually never shrink. To prevent adhesive junction failure, great consideration should be given to both material selection and geometry. While the strength and modulus of adhesive joints are strong at temperatures below the glass transition temperature (Tg), their ductility is decreased. Adhesive is malleable and durable above the glass transition temperature, although its strength will decline. Large temperature loads induced in composite substrate and stiffness adhesive bonding have caused them to break prematurely [66].

The paper covered the topic of single lap joints' dynamic strength at high temperatures and under various loading scenarios. Split Hopkinson pressure bar (SHPB) tests the joints while they are subjected to dynamic loading (SHPB). Temperatures range from 25 to 100 degrees, and equilibrium loading conditions are managed by appropriate pulse shaping. Failure is found to be within the adhesive layers, as seen from failure joints. The results of the experiment show that adhesive lap joints have greater strength than quasi-static joints at the same loading rate, and their strength increases with loading rate. While dynamic strength at 100 ºC is 25% less than at 25 ºC, it is still 50% larger than at 25 ºC due to the temperature-dependent decrease in dynamic strength than the static strength at 25 ºC [67].

In this study, hybrid composites and adhesive bonded joint composites are explored and reviewed. To demonstrate the effectiveness of adhesive joints, several factors are mentioned, such as temperature and surface treatment. Hybrid joints are described as having high static strength and a longer fatigue life. It has been shown that failure load increases with bond thickness in thin bond lines whereas failure load decreases with bond thickness in thick bond lines. Compared to other substrates, the SLJ of a carbon-carbon substrate provides a higher level of strength. In comparison to brittle adhesive connections, the ductile adhesive bonded joint produces better outcomes. The failure of the adhesive-adherend contact is inferred from the composite adhesive analysis. Additionally, hybrid joints are examined and shown to be stronger than adhesive-bonded ones [68].

This study used a servo-hydraulic high-rate testing equipment to examine the effects of various temperature ranges and dynamic loads on the strength of steel single lap adhesive joints. The strength and failure of joints are assessed using the digital image correlation approach, and the experimental findings revealed the strength, toughness, and strain distribution for various overlap lengths. It has been found that raising the loading rate increases bond and shear strength. Additionally, it has been found that the average bond strength rises at temperatures between -25 and 50 ºC and decreases between 50 and 100 ºC. At normal temperature, the adhesive/steel contact is what causes failure; however, when the temperature rises rapidly, the failure mode switches to the adhesive/CFRP interface. It has been determined that temperature affects how well adhesives bind [69].

The influence of graphene-oxide nanoplatelets on nanocomposites was investigated in this research at various temperatures, from ambient temperature to the glass transition temperature. The neat and varied weight percentages of graphene-oxide nanoplatelets are tested in a single lap adhesive junction. At temperatures close to room temperature, it has been seen that graphene increases the strength of joints.

Additionally, experimental findings show that the impact of graphene-oxide nanoplatelets reduced with rising temperature. If the temperature is rising at a critical pace, the nanoplatelet reduces the strength of joints. At the critical testing temperature of 60 ºC, 0.1 weight percent of graphene-oxide nanoplatelets were introduced, and for 0.3 weight percent of nanoplatelets, the critical testing temperature was lowered to 40 ºC [70].

The performance of cork and ceramic matrix composite joints (CMC) at high temperatures and during in-situ cork polymerization at the top of CMC was studied in the research. At room temperature and using liquid nitrogen, shear force and strain are measured. It is noted that the shear strength for the adhesive junction between alumina and graphite is closer to 0.53 MPa and increases by 47% for Zr02-Zrsio4. Shear strain is reduced by up to 55% and shear strength is boosted by up to 80% at liquid nitrogen. Shear strength is unaffected by the in-situ polymerization of cork at the top of the CMC, but shear strain is enhanced since the cork is a part of the crack. When compared to alumina and graphite adhesive joints, it is shown that overall Zro2-Zrsio4 adhesive joints exhibit stronger shear strength and less shear strain [71].

In this paper, the strength and roughness of adhesives were examined through the employment of various binder types and cork particles. The thermal conductivity and porosity of mortars, as well as the compressive strength and tightness, are shown to be linearly related. As concrete density dropped, it was found that the thermal conductivity of concrete composites also reduced. The structure and roughness of mortars affect the mechanical characteristics and adhesive joint strength. It has been shown that mechanical qualities like strength and resistance diminish as the amount of cork particles in mortars rises. The quantity of cork and hydrated lime will improve the mortar's absorptivity and reduce the density of the mortar [72].

The fatigue strength of single lap adhesive joints was examined in this work in relation to nanoparticles. The strength of a single lap joint with and without nanoparticles is the subject of experiments. The nanoparticles employed in this research are nano-Al2O3, nano-SiO2, and nano-TiO2. Steel plate adhesive joints are used. It has been noted that adding reinforcement nanoparticles to adhesive joints causes an increase in the average damage load, with the highest damage load being attained at 4 weight percent nano-Al2O3 in epoxy adhesives. As the stress increases, a combination of cohesive and interfacial failure occurs. When nano-Al2O3 and nano-SiO2 are used to reinforce adhesive joints, the strength of the joint rises, but the strength drops when nano-TiO2 is used [73].

Since it is concluding that type of nanoparticles will affect the strength of adhesive joints and strength of joint will increase with the overlap length. At 20mm overlap length and 22.3% sample of nano-$Al_2O_3$, the highest fatigue strength of single lap joint is observed. The strength of adhesive junctions will rise with the duration of the overlap, it is concluded, regardless of the type of nanoparticles present. The greatest recorded single lap joint fatigue strength is at 20mm overlap length and 22.3 percent sample of nano-Al2O3 [74].

The influence of stress rate on the shear strength of an aluminum single lap joint bonded using nano alumina adhesives was examined in this research. There are two types of alumina nanoparticles used: spherical and rod-shaped. To test the static shear strength of aluminum alloy single alp joints under compression stress, various weight percentages of nano alumina particles are utilized. At a weight of 1.5 percent, the dynamic shear strength of neat adhesive and nano alumina adhesive is also studied. It has been demonstrated that a joint's dynamic shear strength is three to five times greater than its static shear strength. Because the spherical shaped nano adhesive has better interfacial characteristics with epoxy, its dynamic shear strength is greater than that of the rod-shaped nano adhesive [75].

This study examined how Nano alumina affected the single lap, double cantilever, and contoured cantilever beam joints in aluminium for strength and toughness. Different percentage weights of alumina nanospheres and nanorods are used to create alumina nanocomposites. When using nanocomposites rather than plain epoxy adhesives, joints' strength and hardness are seen to significantly improve. It has been shown that joints with 1.5 weight percent of nanospheres and 1 weight percent of nanorods adhesives had the highest shear strength and toughness. Additionally, it has been shown that nanospheres' 1.5 weight percent fracture toughness is higher than nanorods' 1 weight percent fracture toughness. It has been determined that adding more Nano alumina would result in a drop in fracture toughness and shear strength. At all weight percentages of Nano alumina, the average toughness measured from the contoured cantilever beam is lower than the average toughness recorded from the double cantilever beam [76].

The strength of an epoxy composite junction held together by two acrylic adhesives was examined in relation to the impact of SiC nanoparticles. Nanoparticles between 25 and 40 nm are employed, and in situ polymerisation is the method used. SiC nanoparticles have greater failure loads than plain adhesives, and the increased failure load for SiC at 1% is studied. Additionally, a single alp joint's shear strength and load bearing capability are 38 percent greater at 0.75 percent nanoparticle content compared to plain adhesives. Conversely, adhesive junctions with SiC nanoparticles exhibit a combination of adhesive and cohesive failure. Adhesive joints without nanoparticles exhibit adhesive failure. The overlap length affects the load bearing capacity, which grows linearly as the overlap length does.  By using finite element analysis, the distribution of various stresses is examined in order to study the impact of nanoparticles in adhesive joints [77].

The influence of two distinct nanoparticles, multi-wall carbon nanotubes (MWCNTS) and silica nanoparticles (SNPs), on the durability of single lap joints was studied in the work. It has been shown that adding the two nanoparticles to the adhesive junctions greatly improves strength and failure modes. By using a scanning electron microscope, it was possible to examine several mechanisms after adding MWCNTS to the joints, including shear yielding, crack growth deviation, and plastic deformation. It is determined that shear strength is significantly increased when MWCNTS is added at low weight, while it is decreased when MWCNTS is used at higher weight. For SNPS, the phenomenon was the opposite, with a significant improvement in shear strength at higher SNP weights. The cohesive failure modes are more pronounced due to the presence of MWCNTS and SNPs because the adhesion between the adhesive and adherend is improved [78].

To increase the adhesive's tensile strength, cork powder was used in this paper's evaluation of epoxy edhesive. It has been found that the size, distance, and volume fraction of a particle all affect its toughness. A benefit of using micro particles with epoxy resin to boost toughness is that it lowers the cost of the component while still giving it the desired properties. When examining the toughness of glassy polymers and metal plastics, the phenomena of shear yielding, and crazing are seen. The adhesion between the filler and matrix affects the ultimate performance of the composite material. Additionally, brittle, and ductile particles are employed to increase the durability of adhesive junctions. Additionally, it is discussed how adding a small amount of ductile material will make adhesives more resilient. The toughness of bulk adhesives increases with particle size. The introduction of cork particles increases the toughness of brittle materials, according to research on their impact [79].

In this paper, a sandwich panel with an optimized composition of cork granule and green epoxy resin to check the mechanical strength and viscoelastic response by perform static bending tests. Kohlrausch-william- watts model is used to collect experimental data and stress relaxation test proves that stress is reducing over time. The KWW model is most favorable for short prediction and it predict the stress relaxation time accurately. To analyze and predict the long term strength, Findley powder law is favorable one. it is concluded that fatigue increases as compared to the strength in sandwich with synthetic foam [80].

The paper discussed about the toughness of structural adhesives in the presence of cork particle. Cork act as crack stopper and particle ranging from 38-250 micrometers and amount is between 1 and 5%. Surface treatment is carried out to show the effect of adhesive-cork with several adhesions. By the pressure plasma treatment, it is shown that increase in surface energy will also increase the adhesion between cork particle and epoxy resins. Cork powder density decrease with plasma treatment. Small amount of cork particle will result in better impact energy absorption that large amount of particles. Low density plasma treatment decrease contact angle and increase wet ability. The behavior of cork/resin composite is effected by the number of cells in the particle [81].

This paper discussed about the effect of cork particle on the strength of structural adhesive. Cork particle ranging from 125-250 micrometers mixed with epoxy adhesive Araldite and amount of cork between 0.5-5 percent is used. Tensile test carried at room temperature and SLJ joint tested on same testing machines. It is concluded that large particle gives better results than the small particles. Tensile test carried with and without pre-heating and it is observed that pre-heating does not influence the behavior of epoxy. The adhesive with 1% cork particle show more ductility behavior as compared to neat epoxy resins. SLJ joint at 1% cork particle show higher strength. 1% cork particle show lower glass transition temperature and resulted in more ductile behavior [82].

The paper discussed on the effect of cork particle on the toughness of brittle epoxy adhesives. Brittle resin is used with cork and without cork particles to analyze the kinetics of specimens. In order to relate the mechanical properties with thermal and chemical properties tensile test is being performed. It is observed that with increase of temperature of cure, the degree of conversion is also increases. The mechanical property of composite cork/resin not depend on curing process. Specimen with cork particle show lower transition temperature than the specimen without cork particles. According to the DiBenedetto equation it is concluded that cork particle applies plasticizing effect on resin and difference in mechanical properties observed. Brittle resin with 1% of cork particles with structure composite by limited number of cells show more ductility as compared to the other percentage of cork [83].

## 2.2 Conclusion

To strengthen or raise the strength of adhesives & adhesive based single lap joints, various sorts of experiments are being conducted. It has been found that a variety of parameters, including temperature, nanoparticles, surface treatment, adhesive thickness, kind of adhesive, etc., affect the strength and toughness of adhesives and in turn adhesive joints. The review's conclusion is that employing different nanoparticles for various materials or lengthening overlaps generally increases strength. Additionally strengthening adhesive junctions is cork powder, and adhesives with 1% cork particles exhibit greater ductility than the adhesives with lower concentrations of cork powder. Additionally, the impact of temperature is being researched on the durability of both epoxy adhesives & single lap joints. The effect of temperatures ranging from 25 to 100 degrees at various concentrations of cork powder is being experimented in the current work for initially pure adhesives, cork powder added adhesives and then single lap joints (with and without cork) having two similar adherends at various temperature conditions.

# Chapter 3: Experimentation

The experimentation setup, fabrication processes, techniques, tools, and testing processes employed in this study are covered in this chapter. There were two main components to the specimen production and experimentation.

- **Phase – I** The initial step involved manufacturing of adhesive samples through a mould designed to house specified samples as per designed experiments.
- **Phase – II** Adhesive joints (SLJs) were manufactured using same adhesive and then cured in oven to develop specified set of joints. Adherends were chemically cleaned to get the optimal surface treatment on the adherent (Aluminum) for adherence.
- **Phase – III** The third part consisted of testing structural adhesive samples and adhesively bonded single lap joints to determine the effects of cork powder concentration and temperature under tensile loading.

## 3.1 Materials Used

### 3.1.1 Adhesive

#### *3.1.1.1 Epoxy resin (LY-556)*

Table 3.1 Physical Properties of Epoxy Resin

| S No | Parameter | Details |
|---|---|---|
| **1** | Aspect (visual) | clear liquid |
| **2** | Viscosity at 25ºC (ISO 12058-1) | 10000 – 12000 [mPa s] |
| **3** | Density at 25 °C (ISO 1675) | 1.15 - 1.2 [g/cm3] |
| **4** | Epoxies' index (ISO 3001) | 5.30 – 5.45 ** [Eq/kg] |

#### *3.1.1.2 Hardener (AD-22962)*

Table 3.2 Physical Properties of Hardener

| S No | Parameter | Details |
|---|---|---|
| **1** | Aspect (visual) | Colorless-little yellow liquid |
| **2** | Viscosity at 25°C (ISO 12058-1) | 5 - 20 [mPa s] |
| **3** | Density at 25°C (ISO 1675) | 0.89 - 0.90 [g/cm3] |

- **Storage**

  Both the resin and the hardener are kept in a container that is properly closed and kept dry. Containers that have been partially used should be closed right away.

- **Mixing Ratio**

Table 3.3 Components mixing ratio

| Components | Parts by weight | Parts by volume |
|---|---|---|
| **Araldite LY-556** | 100 | 100 |
| **AD-22962** | 23 | 30 |

To avoid mixture errors that could affect the matrix physical properties, it is advised that each component be weighed using a proper balance with calibration. To ensure homogeneity in the mixture, the components must be well combined through hand mixing and then magnetic stirring. It's essential to incorporate the vessel's side and bottom into the blending process. Exothermic reaction might cause the pot life to shorten when processing large amounts of mixture. It is preferable to divide large mixtures into several smaller containers.

- **Curing Time of the epoxy and Hardener:**

  Cure at 100ºC for 2 hours.

### 3.1.2 Adherends

#### *3.1.2.1 Aluminum Coupons:*

- **Material Specifications:** Aluminum 5052

Table 3.4 Mechanical Properties of Aluminum

| S No | Parameter | Details |
|---|---|---|
| 1 | Young’s modulus | 70.3 GPa |
| 2 | Yield Strength | 193 MPa |
| 3 | Ultimate Strength | 228 MPa |
| 4 | Shear Strength | 138 MPa |
| 5 | Poisson Ratio | 0.33 |

- **Dimensions:**

$$Width\ of\ aluminum\ coupons\ =\ 25.4\ \pm\ 0.2mm$$

$$Length\ of\ Aluminum\ coupons =\ 101.6\ \pm\ 0.2mm$$

$$Thickness\ of\ Aluminum\ coupons =\ 3.0\ \pm\ 0.1mm$$

- **Aluminum Coupons Characteristics:**

  This alloy has high corrosion resistance, improved weldability, high fatigue strength and light weight in composition. This makes Al 5052 very useful for application in fuel tanks, pressure vessels, oil lines, transportation, heat exchangers, chemical storage etc.

### 3.1.3 Filler

As filler, cork powder has been used in these experiments. Benefits of cork are:

- Because of its nearly impermeable nature, cork's flexibility makes it a particularly good material for crack stoppers.
- Since cork has a nearly zero Poisson's ratio, pulling or compressing it does not greatly alter its radius.
- A homogenous tissue with thin-walled cells that are aligned uniformly and without intercellular gap can be used to describe cork. Cork exhibits an alveolar structure resembling a honeycomb with closed units and no vacant areas between continuous cells.

Compared to brittle resin without particles, this cell arrangement gives the composite the ability to absorb more impact.

#### *3.1.3.1 Filler concentrations*

Following cork powder concentrations have been used:

- 0.25 wt.%
- 0.5 wt.%
- 0.75 wt.%
- 1wt.%

## 3.2 Equipment

Following equipment has been used for experimentation purpose:

1) Electronic balance
2) Magnetic stirrer with heating plate
3) Curing oven
4) Ultimate tensile machine

### 3.2.1 Electronic balance

The tool used for precise adhesive & cork powder measurement is electronic balance. It is utilized in the experiment to accurately quantify the resin and hardener. This device could measure amounts up to 0.001 grams.


Fig. 3.1 Electronic Balance

### 3.2.2 Magnetic stirrer with heating plate

Magnetic stirrer is the device used to create a spinning field in order to perform mixing of epoxy adhesive. The spinning field produced by the magnetic stirrer is supported by a rotating magnet bar or a rotating magnet-containing plate. The magnet is often covered with plastic, and the plate has a spinning magnet. With the aid of a revolving magnet, it is possible to create a rotating field. Magnetic stirring can be performed at

- **Room temperature.** To mix epoxy and hardener
- **Higher temperature.** To mix epoxy and hardener at various cork powder concentrations and to heat sodium hydroxide for surface treatment.


Fig. 3.2 Magnetic stirrer with heating plate

### 3.2.3 Curing Oven

A curing oven is a thermal processing machinery created to increase a material's tensile strength and durability by quickening a desired chemical reaction through higher but controlled temperature. In its most basic form, a curing oven accomplishes this by raising a sample material's temperature to within or over a predetermined limit. This might be sufficient to enhance the product's mechanical properties.

Fig. 3.3 Curing Oven

### 3.2.4 Universal Testing Machine

A universal testing machine (UTM) can be referred as a testing setup to study mechanics of various materials under various loading conditions such as flexural strength tests, tensile strength and compressive strength of materials etc.Below stated are the specifications of UTM used throughout the experimental setup:

Table 3.5 Specifications of UTM

| Description | Details |
|---|---|
| **Specification** | HD-B607-S HAIDA INTERNATIONAL EQUIPMENT CO., LTD |
| **Capacity** | UTM of 100KN load cells |
| **Load accuracy** | Less than equal to ± 0.5% |
| **Test Speed** | 0.5 mm/min. |
| **Operation Mode** | Computer tensile testing machine with PC control software “TESTER”. |
| **Display** | It will show the maximum failure load, duration, time, and position after testing. In an excel sheet, data can be manually stored. The user can adjust the product's length, width, and thickness in accordance with the dimensions of the sample. |

## 3.3 Manufacturing of Adhesive Samples

### 3.3.1 Dimensions of Adhesive Samples

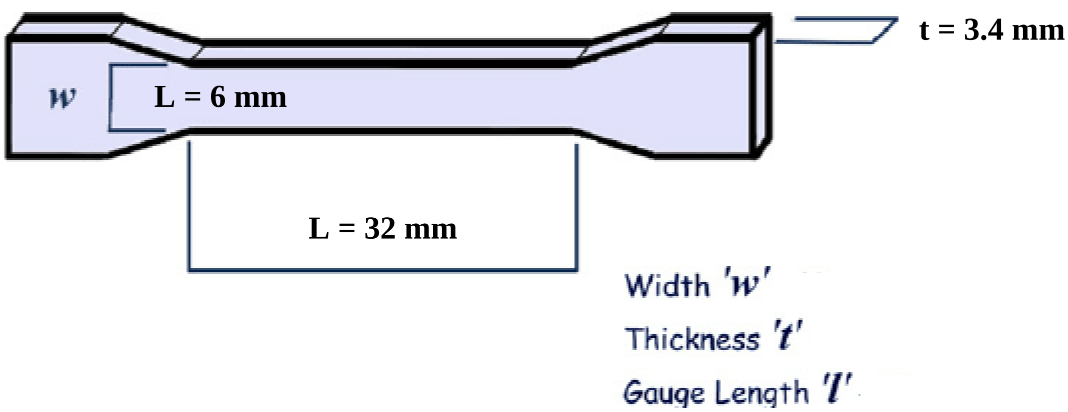



Fig. 3.4 Adhesive Dimensions

$$\boldsymbol{Dimensions\ of\ each\ sample\ =\ 32\ x\ 3.4\ x\ 6\ (gauge\ length\ area)}$$

### 3.3.2 Mould for Adhesive Preparation

A customized mould was designed using Al-5086 alloy for adhesive samples preparation. Detailed dimensions of mould are stated in the figure below:

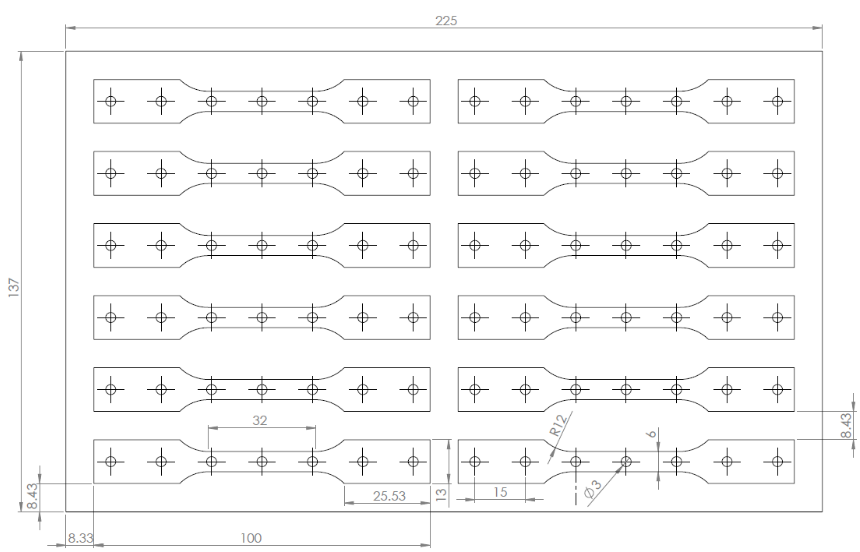



Fig. 3.5 Mould CAD

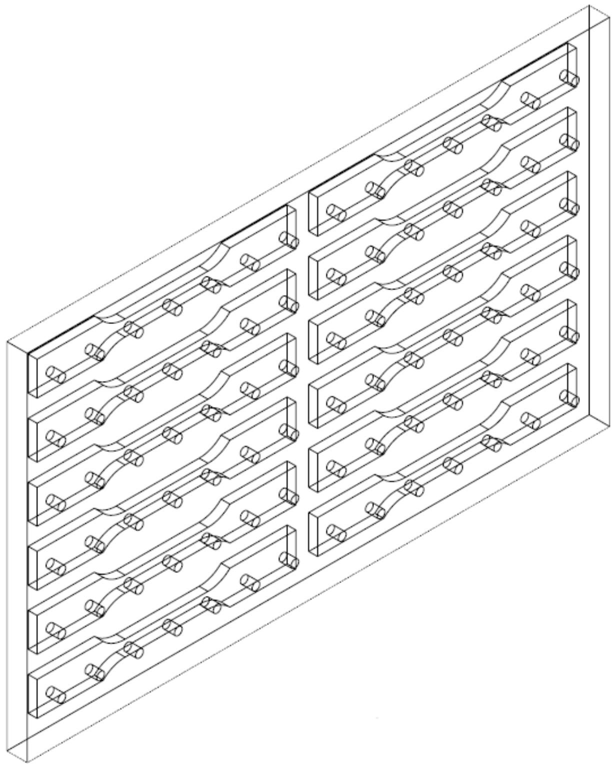


Fig. 3.6 Isometric view of mould

The actual mould developed based on CAD design above is shown below: -

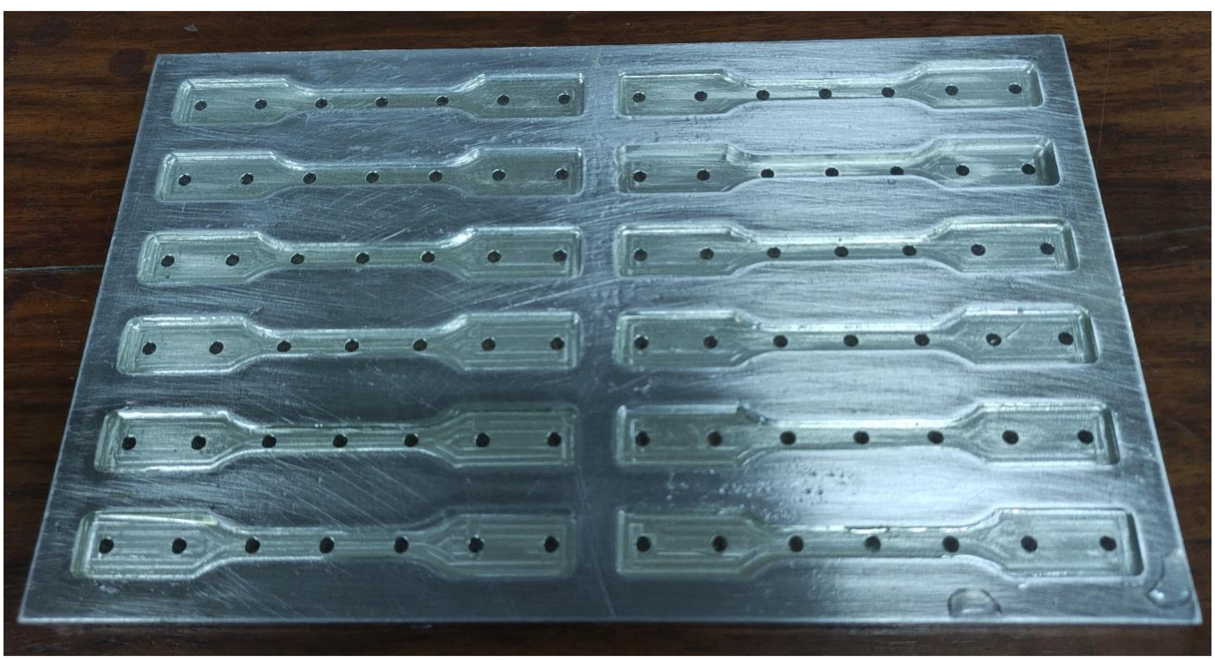


Fig. 3.7 Actual mould made for specimen preparation

### 3.3.3 Design of Experiments

Dog-bone shaped specimen of adhesives were made in following configuration:

- Without addition of cork powder (neat configuration)
- With addition of various concentrations of cork powder

As per ASTM standard, 03 samples were manufactured for each case and following experiments were designed to determine the behavior of adhesives with and without cork powder

$$\boldsymbol{Temperature\ conditions\ =\ 25, 50, 75, 100\ ^\circ C\ (04\ cases)}$$

$$\boldsymbol{Cork\ powder\ concentrations\ =\ 0.25, 0.5, 0.75, 1\ wt\%\ (05\ cases)}$$

$$Samples\ per\ case\ (repeats)\ =\ 03$$

$$Total\ samples\ =\ 04\ x\ 05\ x\ 03$$

$$\boldsymbol{Total\ samples\ =\ 60}$$

**Full factorial design of experiment for adhesives sample testing is tabulated below:**

Table 3.6 Design of Experiments for Adhesive

| **Temp / Conc** | **25$^o$C** | **50$^o$C** | **75$^o$C** | **100$^o$C** |
|---|---|---|---|---|
| **Neat adhesive** | X | X | X | X |
| **0.25 wt%** | X | X | X | X |
| **0.5 wt%** | X | X | X | X |
| **0.75 wt%** | X | X | X | X |
| **1.0 wt%** | X | X | X | X |

### 3.3.4 Preparation of epoxy samples

Following steps are involved in the manufacturing process of adhesive specimen

- Mould preparation

- Adhesive preparation
- Curing of specimens
- Testing of specimens

### 3.3.5 Mould preparation

The mould needs to be prepared before epoxy adhesive is poured inside it and for this purpose, it needs to be ensured that epoxy does not stick to sides of the mould. For this purpose:

(i) Initially, the mould is degreased with detergent solution to remove the excess impurities from the surface

(ii) Clean any remaining impurities / dirt through an acetone solution

(iii) Carefully apply the adhesive releasing agent on each slot of mould (total 12 slots per mould for the specimens). This releasing agent must be applied to cover the adhesive cut out fully, in case of failure to apply this, epoxy will stick to the sides of the mould.

(iv) By using a cutter / blade, carefully remove the excess releasing agent

(v) Apply grease on sides of mould to ensure easily removal of specimens after curing process

Figure below shows the prepared mould for adhesive preparation.

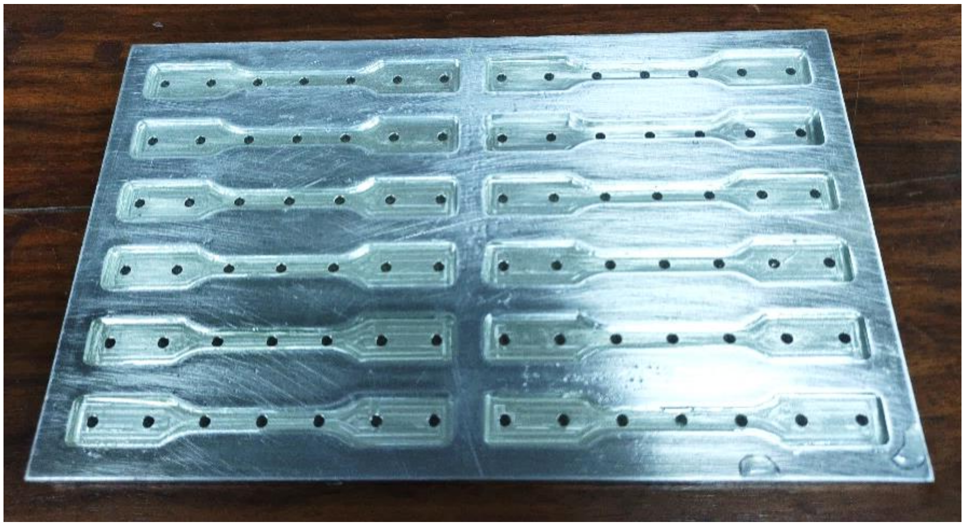


Fig. 3.8 Mould prepared for adhesive specimen

### 3.3.6 Neat adhesive preparation

#### *3.3.6.1 Mixing Ratio of Epoxy and Hardener*

In this case, two component epoxy resin is used (resin and hardener). Once both parts of adhesive are mixed, a chemical reaction takes place and adhesive is formed which cures to form a solid. It is to be ensured to have proper mixing of both parts and precise measurement is necessary to achieve desired properties of epoxy resin. For this step, initially quantities of epoxy and hardener are to be measured as per the mixing ratio:

- Mixing Ratio = E:H = 100: 23 (100 parts of epoxy we take 23 parts of hardener).

Total five sets of experiments will be conducted for neat, 0.25, 0.5, 0.75 & 1 wt%. We have prepared 55 grams of solution for every set of experiment. As per mixing ratio

$$\boldsymbol{Amount\ of\ epoxy\ in\ 55g\ solution\ =\ 55g\ x(100/123)\ =\ 44.71g}$$

$$\boldsymbol{Amount\ of\ hardener\ in\ 55g\ solution =\ 8g\ x\ (23/123)\ =\ 10.29g}$$

OR

$$\boldsymbol{Amount\ of\ hardener\ in\ 55g\ solution =\ 55g - 44.71g\ =\ 10.29g}$$

**(ii) Measurement & mixing of epoxy and hardener**

- Start with a 100 ml beaker. We use a weighted scale or an electronic compact scale to measure an exact amount. The scale is now first set to grammes. Set the beaker down on the scale.
- To make the beaker's weight zero, first click the tare button on the electronic compact scale. Once 44.71g of epoxy has been added to the beaker, slowly pour the epoxy into the container. With the use of a spatula, we may remove any excess epoxy that has been added to the beaker.
- Add 10.29g to the beaker as the next step. To start, click the tare button to reset all values to 0. Then, pour the hardener slowly and carefully because it is difficult to control if a little excess is poured.


Fig. 3.9 Weight measurement for epoxy and hardener

- After combining the two components in the proper proportion, thoroughly stir them with a mixing stick for a full 2–3 minutes. When working with bigger quantities, stir for a longer amount of time.
- Several times while mixing, scrape the bottom, sides, and corners of the container. This makes sure that every last bit of the hardener is mixed into the epoxy, which should stop the resin from curing wrongly.
- Scrape the mixing cup's sides as well. Continue blending the mixture if the mixture does not reach a uniform consistency (streaks still exist).
- After combining with a spatula, mix the epoxy and hardener for around 10 minutes on a magnetic stirrer to guarantee good mixing and a bit higher rpm. After 10 minutes, stir the epoxy hardener one more for two to three minutes.


Fig. 3.10 Epoxy stirring on magnetic stirrer

#### *3.3.6.2 Adhesive pouring in mould*

- The prepared epoxy resin will now be poured into mould. For precise injection of adhesive in mould slots, fill the epoxy resin in a 60 ml injection and carefully pour inside the mould.
- Once all the epoxy is poured inside the mould, burst any bubbles on the surface of poured adhesive to ensure smooth epoxy resin.

Once epoxy resin is poured, the mould will look like this

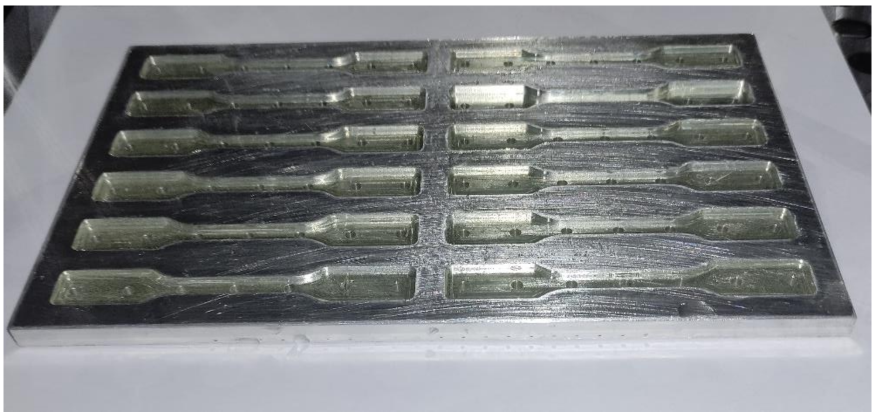


Fig. 3.11 Adhesive poured inside the mould

### 3.3.7 Cork powder adhesive preparation

#### *3.3.7.1 Mixing Ratio of Epoxy and Hardener*

In this case, same process will be followed as in case of neat adhesives.

$$\boldsymbol{Amount\ of\ epoxy\ in\ 55g\ solution\ =\ 55g\ x(100/123)\ =\ 44.71g}$$

$$\boldsymbol{Amount\ of\ hardener\ in\ 55g\ solution =\ 8g\ x\ (23/123)\ =\ 10.29g}$$

OR

$$\boldsymbol{Amount\ of\ hardener\ in\ 55g\ solution =\ 55g - 44.71g\ =\ 10.29g}$$

Along with this, various concentrations of cork powder will be added w.r.t to 55 g epoxy resin solution in following amounts:

Fig. 3.12 Percentage of cork powder in adhesives

| S No | Concentration (%) | Amount (g) |
|---|---|---|
| **1** | 0.25wt% | 0.1375 |
| **2** | 0.5wt% | 0.275 |
| **3** | 0.75wt% | 0.4125 |
| **4** | 1wt% | 0.55 |

#### *3.3.7.2 Measurement & mixing of epoxy and hardener*

- Start with a 100 ml beaker. We use a weighted scale or an electronic compact scale to measure an exact amount. The scale is now first set to grammes. Set the beaker down on the scale.
- To make the beaker's weight zero, first click the tare button on the electronic compact scale. Once 44.71g of epoxy has been added to the beaker, slowly pour the epoxy into the container. With the use of a spatula, we may remove any excess epoxy that has been added to the beaker.
- Add cork powder in given amount in table stated above as per % (0.25, 0.5, 0.75, 1).
- A magnetic stirrer is now utilized to mix and heat the epoxy and filler properly.
- As indicated in Figure, the filler and epoxy resin are magnetically stirred for 30 minutes at a temperature of 50 degrees.


Fig. 3.13 High temperature mixing of epoxy and cork powder

- It is challenging to keep the magnetic stirrer at a temperature of 50 degrees; thus, when the temperature hits 32 degrees, turn off the heat and stir the solution. The magnetic stirrer's plate is already hot, so the temperature continues to rise and reaches a maximum of 50 degrees. A temperature gauge dipped in the epoxy and filler solution is used to gauge the temperature. To achieve appropriate mixing, the rpm should be little higher than the earlier. Turn on the heat button for a time while the magnetic stirrer plate begins to cool down and the temperature drops below 50 degrees. Therefore, it was necessary to continuously evaluate the epoxy and filler mixture for 30 minutes.
- Switch off the magnetic stirrer after 30 minutes. The beaker should be covered with aluminum foil and allowed to cool to room temperature. 8,9 minutes are needed for the temperature to drop.
- Add 10.29g to the beaker as the next step after the epoxy and filler mixture has cooled. To start, click the tare button to reset all values to 0. Then, pour the hardener slowly and carefully because it is difficult to control if a little excess is poured.

- After combining the two components in the proper proportion, thoroughly stir them with a mixing stick for a full 2–3 minutes. When working with bigger quantities, stir for a longer amount of time.
- Several times while mixing, scrape the bottom, sides, and corners of the container. This makes sure that every last bit of the hardener is mixed into the epoxy, which should stop the resin from curing wrongly.
- Scrape the mixing cup's sides as well. Continue blending the mixture if the mixture does not reach a uniform consistency (streaks still exist).
- After combining with a spatula, mix the epoxy and hardener for around 10 minutes on a magnetic stirrer to guarantee good mixing and a bit higher rpm. After 10 minutes, stir the epoxy hardener one more for two to three minutes.


Fig. 3.14 Mixing of hardener

#### *3.3.7.3 Adhesive pouring in mould*

- The prepared epoxy resin will now be poured into mould. For precise injection of adhesive in mould slots, fill the epoxy resin in a 60 ml injection and carefully pour inside the mould.
- Once all the epoxy is poured inside the mould, burst any bubbles on the surface of poured adhesive to ensure smooth epoxy resin.

Once epoxy resin is poured, the mould will look like this

Fig. 3.15 Cork powder samples poured inside the mould

### 3.3.8 Curing of epoxy samples

The prepared epoxy batch as shown in figure above will be now cured at 100°C for two hours in curing, for this purpose mould is carefully placed inside oven and cured for the specified duration.

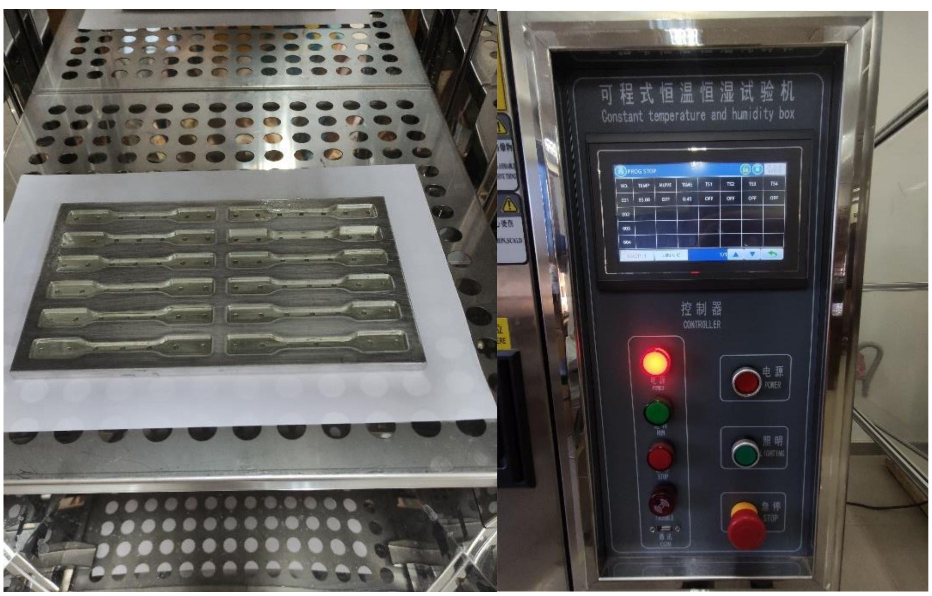



Fig. 3.16 Mould is placed inside the curing oven

### 3.3.9 Adhesive Samples Prepared

Once the whole cycle of specimen preparation is complete, the adhesives will be gently removed from the mould by lightly tapping the bottom of the mould and the epoxy sample will look like this.

Fig. 3.17 0.25 wt% samples

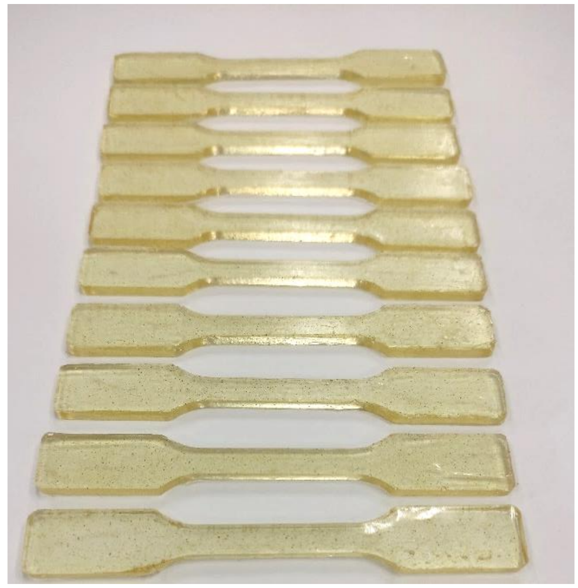


Fig. 3.18 0.5 wt% samples

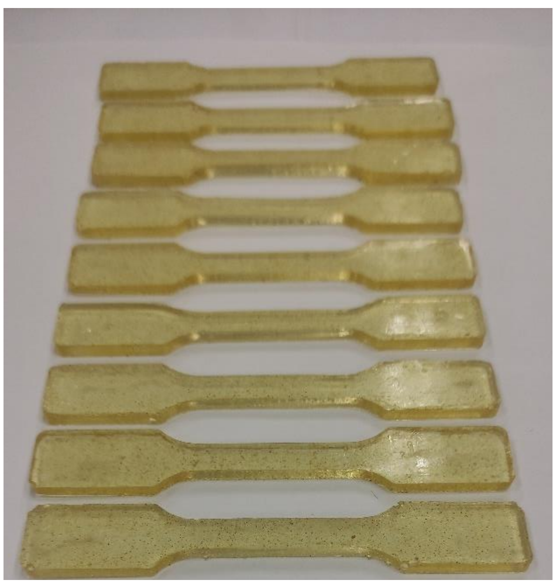


Fig. 3.20 0.75 wt% samples

Fig. 3.19 1 wt% samples

### 3.3.10 Testing of epoxy adhesive samples at 25 degrees

A material that has a tensile load applied to it resists the load by creating an internal resisting force. A material's stress value has a maximum value in addition to increasing as the applied tensile load increases. The stress at which a material fails is known as its ultimate tensile strength. The elastic limit ends at the yield point (load). The original cross-section area continues to shrink when loading surpasses the elastic limit until it hits its minimal value, which is when the specimen breaks, as will be further discussed in the procedure.

For room temperature testing case, UTM is utilized, and samples are put through a 25°C tensile testing process through following steps:

- Turn on the power switch for the UTM and check the PC it is connected to it.
- Start the "TESTER" software. Then, after choosing the new file and entering the width, length, and thickness of your specimen, click "OK."
- After that, mount the specimen using the proper edges and grips. The specimen length or grip should be considered when selecting a load cell (we are employing a 0–7mm grip load cell here).
- It is advised to install the specimen extremely carefully because specimen breakage may occur when the grips are tightened.
- The machine can be operated without a computer connection by moving the grip jaws with the aid of an LCD display screen.
- The specimen needs to be mounted straight to prevent bending during tensile testing from leading to its failure. Check to see if the specimen is mounted straight after mounting it.
- Place the extensometer on sample mounted in UTM to ensure accurate calculation can be obtained for strain measurement.
- Check the load and length while mounting the specimen on the computer screen. Prior to testing, we apply a preload of 100 -130 N while maintaining a 0.5 mm/min speed.
- A graph is displayed on the screen continually during testing up until the specimen breaks. The maximum failure load is displayed on the screen when the specimen breaks.
- First, save the data by selecting it from the menu, then save the file on your computer. It is suggested to save the data first because occasionally software will become stuck and you won't be able to access the data.
- Now Go to Calculate Data, select Tensile Test Data, and then select any value. For sample, copy the data of testing into an excel file.
- After the data is saved, release the failed specimen from the grips.
- Now same procedure is repeated for the all specimens for 0.25%, 0.5%, 0.75%, 1% filler concentration at 25 degrees.

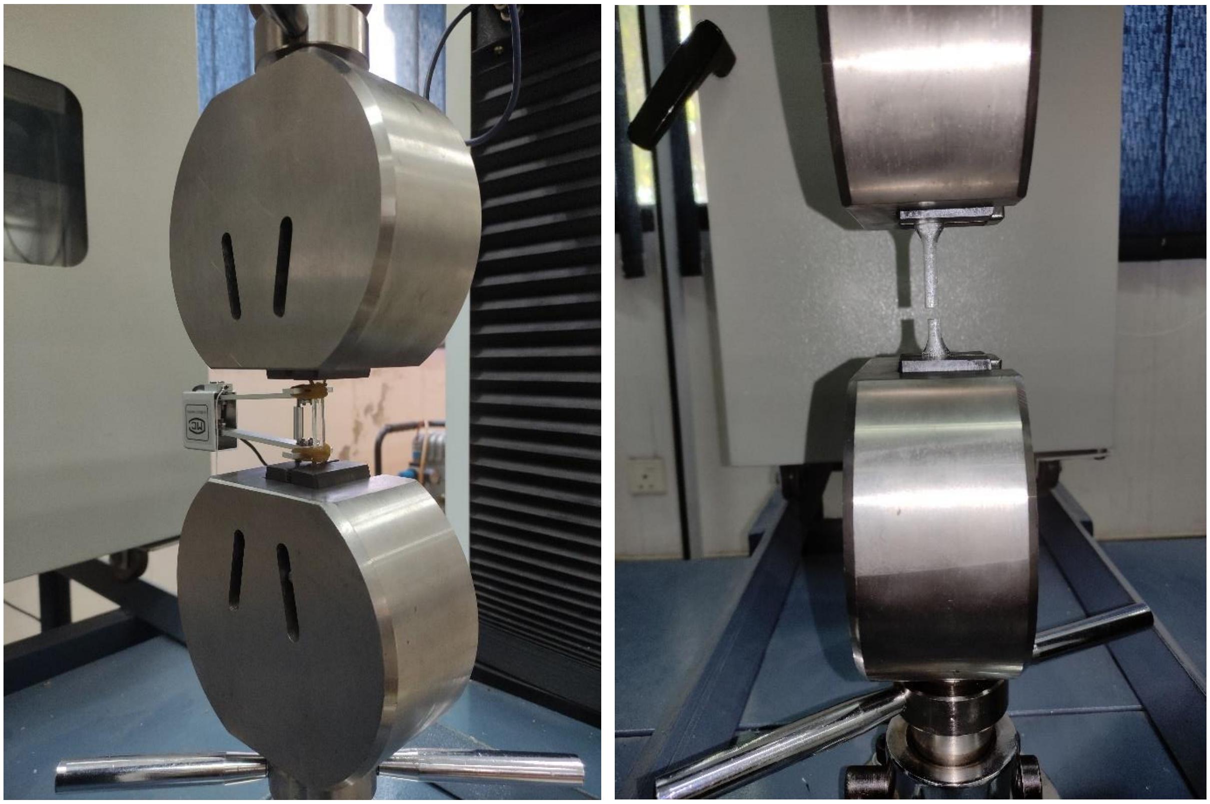


Fig. 3.21 UTM testing of adhesives at room temperature

### 3.3.11 Testing of epoxy adhesive samples at 50, 75 & 100 degrees

The Temperature Chamber (oven), which is connected to the ultimate testing equipment during testing, is used to test the specimen at temperatures of 50, 75 & 100 degrees. A stand with a track for connecting to a universal testing device was attached. Added a thermal discharge extension shaft to the tester to lessen the impact of temperature. The oven's maximum temperature is 300 degrees, and the temperature can be manually adjusted. Following steps will be followed:

- First, adjust the ultimate testing machine's configuration so that it runs tests at a temperature of 50 / 75 / 100 degrees in the oven. The UTM's load cells should be removed. The oven should be moved forward and then insert the new load cell into the oven.
- On the TESTER software first, install the specimen as previously described. The specimen must remain upright.

- Finally, shut the oven close. Set temperature to 50 / 75 / 100 degrees and press the heat and power buttons. Wait until the mercury reaches desired degrees. Upon reaching 50 / 75 / 100 degrees, which takes some time, hit the UP button to get the 100 – 130 N preload.

✓ As force reaches 130N, click the stop button and zero all the values.

✓ Click on start and the testing start at 50 / 75 / 100 degrees.

✓ As the specimen breaks, maximum failure load is obtained.

✓ First save the data and then go to calculate data and copy the whole data and paste it into excel file. Then click on exist and open the new file for next specimen.

✓ The turn off the power and heat button of the oven. Open the oven and the failed specimen is removed. Wear heat resistant gloves before removing the specimen.

✓ Repeat the process for testing the other specimen at 50 / 75 / 100 degree temperature.


Fig. 3.22 UTM testing of adhesives at higher temperatures

## 3.4 Adhesive Joints Manufacturing

Different types of methods are explained in literature review for testing the strength of single lap joints. The methodology part was done by tensile testing for dissimilar adherend at different temperatures. The methodology part consists of three main steps.

### 3.4.1 Dimensions of Single Lap Joint Samples

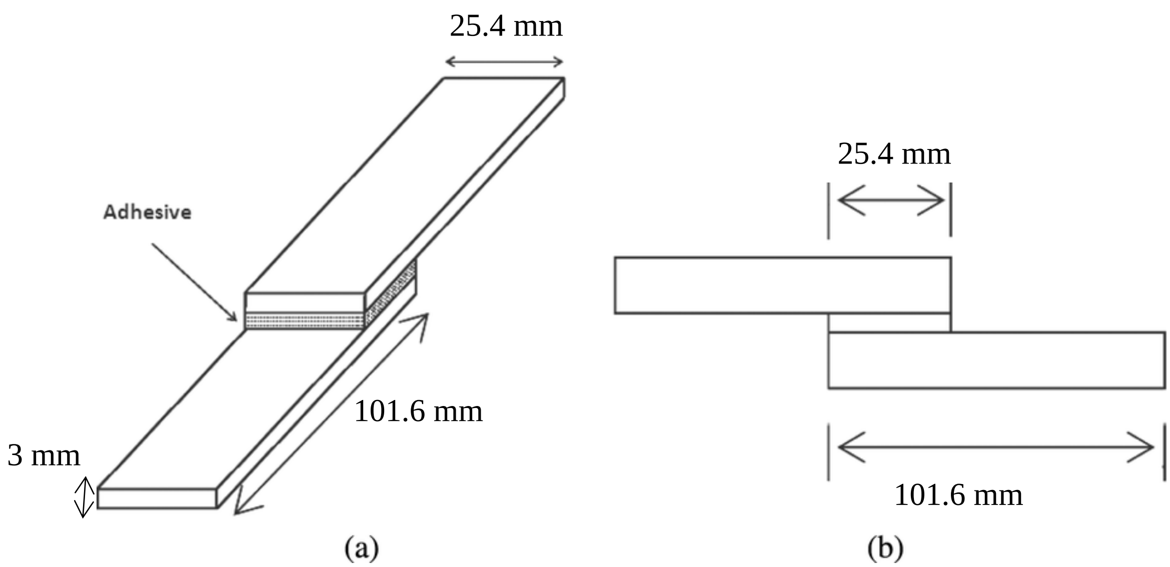



Fig. 3.23 Dimensions of SLJ

$$Width\ of\ aluminum\ coupons\ =\ 25.4\ \pm\ 0.2mm$$
$$Length\ of\ Aluminum\ coupons =\ 101.6\ \pm\ 0.2mm$$
$$Thickness\ of\ Aluminum\ coupons =\ 3.0\ \pm\ 0.1mm$$

### 3.4.2 Design of Experiments

Single lap joints aluminum plates were made in following configuration:

- Without addition of cork powder (neat configuration)
- With addition of various concentrations of cork powder

As per ASTM standard, 03 samples were manufactured for each case and following experiments were designed to determine the behavior of SLJs with and without cork powder

$$\boldsymbol{Temperature\ conditions\ =\ 25, 50, 75, 100\ °C}\ (\boldsymbol{04\ cases})$$

$$\boldsymbol{Cork\ powder\ concentrations\ =\ 0.25, 0.5, 0.75, 1\ wt}\%\ (\boldsymbol{05\ cases})$$

$$Samples\ per\ case\ (repeats)\ =\ 03$$

$$Total\ samples\ =\ 04\ x\ 05\ x\ 03$$

$$\boldsymbol{Total\ samples\ =\ 60}$$

**Full factorial design of experiment for SLJ sample testing is tabulated below:**

Table 3.7 Design of experiments for SLJ

| **Temp / Conc** | **25°C** | **50°C** | **75°C** | **100°C** |
|---|---|---|---|---|
| **Neat adhesive** | X | X | X | X |
| **0.25 wt%** | X | X | X | X |
| **0.5 wt%** | X | X | X | X |
| **0.75 wt%** | X | X | X | X |
| **1.0 wt%** | X | X | X | X |

### 3.4.3 Preparation of single lap joints

Following steps are involved in the manufacturing process of adhesive specimen

- Surface treatment of coupons
- Adhesive preparation
- Joint preparation
- Testing of joints

#### *3.4.3.1 Surface treatment of coupons*

A popular technique for bonding two materials together with an overlapping surface is single-lap joints. They are relatively robust and straightforward. Increasingly different materials have to be joined as a result of the growing use of composite materials in modern design processes. Therefore, it is crucial to comprehend how single-lap joints behave when their adherends are different. In order to prevent other factors from affecting the strength of single lap joints, it is vital to clean the adherends of all forms of particles before constructing the joints. Following steps are following for cleaning of coupons

**Degreasing of Aluminum Coupons**

Degreasing is sometimes known as oil removal or grease removal. The goal is to get rid of oil-adhering filth, hand sweat, rust-resistant oil, naturally occurring oxide coating, and process oil. to make sure the surface of alkali erosion is uniformly corroded.

- **Washing of Aluminum coupons with Detergent.** The first step in degreasing is to wash all of the aluminum coupons in detergent. The main job of a detergent is to break down the surface tension between water and grease. They are referred to as "surfactants" or "surface active agents." The removal of dirt from the surface of aluminum coupons has another benefit. The washing powder is used to first wash all of the aluminum coupons (that is used as detergents). It is advised to wear gloves when washing with detergents since aluminum coupon's sharp edges might cause hand injuries.
- **Filling of Aluminum Coupons.** The removal of sharp edges is important before preparing the joints since they may lead to hand injuries. In this procedure, a file is used to remove the 45o edge. I basically take a file, hold it at a 45-degree angle with the coupons, and go forward while crossing it. Repeat the procedure until the edges are smooth, then do the same for each additional corner.
- **Identify the Surface of Aluminum coupon.** With the aid of the nail, we can locate the degreased surface. Choose a surface that is frictionless and needs to be cleaned first. Then, while holding the nail at the corner of the coupon, strike it with a hammer so that the surface is pointed. As shown in figure, a tiny dot is placed on the corner of the coupons to indicate the aluminum coupons' degreased surface.
- **Clean the surface with Toluene.** Toluene, also referred to as methylbenzene or phenyl methane, is a colourless, water-insoluble liquid with the distinct odour of paint thinner. A thinner called toluene is employed in the production of specialised paints and coatings. It works wonders as a general cleanser and degreaser. Compared to acetone, it evaporates more slowly, but more swiftly than Xylene. Put on the gloves first, then grab a towel. To clean the surface of the coupon that will be used for preparing joints, wrap the towel over your finger, dunk it in toluene, and then wipe it down. The towel can be used to wipe two or three coupons before being dipped in Toluene once more and the coupons.

- **Clean the surface with Acetone.** Acetone can be used to clean, degrease, finish, and remove paint, among other things. It can effectively remove a lot of grease and other unwanted materials from surfaces. Acetone overcomes a lot of the issues with heavy-duty equipment or products and techniques for eliminating surface impurities when used for degreasing. After using toluene to wipe the surface, we rapidly close the acetone container after dipping one corner of the towel in it. The coupon's side that will be used for attaching the joints should then be wiped. Repeat for the remaining coupons after wiping the first two or three with acetone. The acetone-degreased aluminum single lap shear (SLS) test coupons were degreased with acetone prior to immersion in the NaOH solution.
- **Immersion of Aluminum coupons in NaOH Solution.** The degreased Aluminum coupons were dipped into a 6 weight percent NaOH solution for the chemical etching operation. Begin with a 1000 ml beaker. The NaOH solution is then diluted to 400 ml in a beaker. The NaOH solution should next be heated using a magnetic stirrer until the temperature reaches 50 degrees in Fig (ii). By dipping the temperature gauge into the NaOH solution, you may measure the temperature. When the solution reaches 50 degrees, switch off the magnetic stirrer, dip no more than 10 coupons into it, and put it outside to cool. This is because aluminum and NaOH combine to form flammable and explosive hydrogen gas, which can irritate the skin, eyes, and respiratory system and produce smells. Set the timer for 6 minutes after the aluminum coupon has been soaked in the NaOH solution. Compared to the black dots that were visible on the surface after it had been wiped with acetone, the surface after six minutes of treatment looks to be much cleaner and may even be free of organic impurities.
- **Immersion in water.** The aluminum coupons will be taken out of the NaOH solution and placed in the water as the timer expires. For that, take a 1000 ml beaker and pour 600 ml of pure water into it. Ultra-pure water was used to rinse the etched samples. In order to clean the coupon's surface and remove any leftover particles from the NaOH solution, the coupons are submerged in water for five minutes.
- **Immersion of aluminum coupons in Acid cleaning solution.** We take 9 wt.% of $HNO_3$. Take 1000ml beaker and pour $HNO_3$ up to 400ml. The aluminum coupon is then soaked in to $HNO_3$ solution for 3 minutes at ambient temperature in Fig3.1 (v). Acidic cleaning has been shown to effectively remove corrosion products formed on the aluminum

surface as well as intermetallic particles following alkaline etching. After 3 minutes, the aluminum coupons are immersed into water as explained in step 6.

- **Wash the coupons with distilled water.** We use 9 weight percent HNO3. Pour 400 ml of HNO3 into a 1000 ml beaker. In Fig.3.1, the aluminium coupon is then submerged in an HNO3 solution for three minutes at room temperature (v). It has been demonstrated that after alkaline etching, intermetallic particles and corrosion products produced on the aluminium surface may be successfully removed using acidic washing. The aluminium coupons are submerged in water as described in step 6 after three minutes.

Fig. 3.26 Degreasing with detergent


Fig. 3.27 Cleaning with Toulene solution


Fig. 3.24 Acetone solution cleaning


Fig. 3.25 Treatment with NaOH


Fig. 3.28 Post treatment rinsing with HNO3 and water

Fig. 3.29 Drying of coupons

#### *3.4.3.2 Degreasing of end tabs of single lap joints*

The single lap joints are aligned using the end tabs, which are composed of Aluminum 5052. Before inserting it into the testing machine, some joints would automatically misalign, thus bond tabs are placed at the end joints to help with alignment. In order to prevent them from affecting the strength of adhesive joints, the end tabs are also degreased.

- **Wash the end tabs with detergent.** The end tab coupons must first be washed with detergent before the alignment tabs can be degreased. Detergents are used to wash end tabs in order to clean them of dirt and soil particles. When cleaning the end tabs with detergents, it is advised to use gloves because the end taps' sharp edges can cause hand injuries.

- **Filling of end tabs.** Filling the joints on the aluminum end tabs is the second stage in degreasing the end tabs. Take a file, hold it at a 45-degree angle to the coupon, and move over it moving forward. Repeat this process until the edges are smooth. For each additional edge, repeat the procedure.
- **Mark sides to be joined.** Identifying the surface that will be employed as end tabs is the third stage in degreasing the end tabs. Take the side that is smooth, free of curves, and without resistance.
- **Clean with Toluene.** Cleaning the designated surface with toluene is the fourth step in the degreasing of the end tabs. Put on gloves before taking a tiny hand towel corner and dipping it in Toluene before using it to clean the coupons.
- **Clean with Acetone.** The designated surface is cleaned with acetone in the fifth and final phase of degreasing the metal end tabs. Because acetone evaporates quickly, we first take a corner of a hand towel, dip it in the solvent, and then seal the container.

Now take end tabs coupon wipe the coupons with that dip corner. Now place the degreased coupons on the tissue paper and cover with clean tissue paper.

#### *3.4.3.3 Adhesive Preparation for single lap joints*

After the surface treatment of the coupon is completed, two types of epoxy adhesive are prepared that is for adhesive with and without cork powder. We prepare 12 neat joints, 03 samples for every temperature. The temperatures are 25, 50, 75, and 100 degrees. So we take three samples for each temperature. Similarly, 12 joints for each filler concentration shall be made (design of experiment). In this case, two component epoxy resin is used (resin and hardener). Once both parts of adhesive are mixed, a chemical reaction takes place and adhesive is formed which cures to form a solid. It is to be ensured to have proper mixing of both parts and precise measurement is necessary to achieve desired properties of epoxy resin. For this step, initially quantities of epoxy and hardener are to be measured as per the mixing ratio:

- Mixing Ratio = E:H = 100: 23 (100 parts of epoxy we take 23 parts of hardener).

Total five sets of experiments will be conducted for neat, 0.25, 0.5, 0.75 & 1 wt% SLJs. We have prepared 10 grams of adhesive for every set of experiment and application in the joint. As per mixing ratio,

$$\boldsymbol{Amount\ of\ epoxy\ in\ 10g\ solution\ =\ 10g\ x\left(\frac{100}{123}\right)=\ 8.13\ g}$$

$$\boldsymbol{Amount\ of\ hardener\ in\ 10g\ solution=\ 10g\ x\ \left(\frac{23}{123}\right)=\ 1.87g}$$

OR

$$\boldsymbol{Amount\ of\ hardener\ in\ 10g\ solution=\ 10g - 8.13g\ =\ 1.87g}$$

Same process will be repeated as discussed in adhesive preparation technique stated in section 3.3.6 & 3.3.7.

#### *3.4.3.4 Joining the aluminum coupons*

- After thoroughly combining the epoxy and hardener, cover the work surface with a sheet to prevent epoxy from adhering to it. The mixture was then used to create the SLJs.
- Apply the epoxy on the degreased side of the aluminum coupons at a distance of about one inch for the initial adhesive application.
- Join the aluminum coupons where epoxy has been applied.
- Now press while maintaining the two coupons in alignment.
- Pick a binder clip, bind one side of the coupon, check the alignment, and then use the second binder clip to bind the other side of the joints.
- Recheck the alignment to make sure the coupons are joining straight.
- To join the coupons, go back and repeat the process.

#### *3.4.3.5 Joining the end tabs on joint*

- After joining the composite and aluminum coupons, mix again the epoxy with the help of spatula, then apply the epoxy on the degreased side of 1-inch end tabs with the help of spatula.
- Now apply the mixture on all the remaining end tabs.

- Now take one end tabs on which epoxy is applied and joint the nailed side of aluminum coupons and align the end tabs on aluminum coupons and the press with help of finger and bind it with binder clip.
- Due to epoxy, the end tapes slips or misalign during its binding. Bind the end tabs carefully so that they may not misalign.
- Repeat these steps for each additional coupon now.
- Two end tabs were needed for one joint. Hence, we require 24 end tabs for 12 joints.

### 3.4.4 Curing the joints in the oven

The prepared SLJs batch as shown in figure above will be now cured at 100°C for two hours in curing, for this purpose joints is carefully placed inside oven and cured for the specified duration. After two hours turn off the oven and heat button. Wear the heat resistant gloves, then open the oven and take out the joint. Let the oven to cool down to room temperature and then close the oven. The single lap joints with & without any cork powder concentration were prepared through same method.

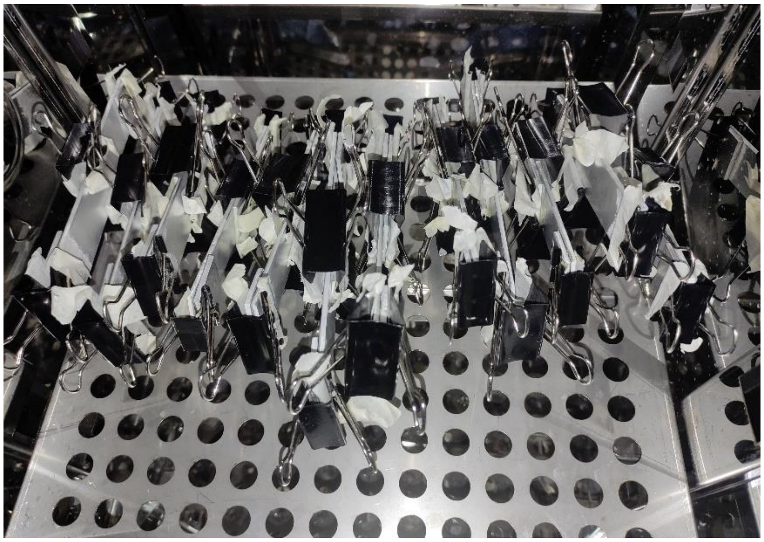


Fig. 3.30 SLJs placed inside curing oven

### 3.4.5 SLJs samples prepared

Once the whole cycle of SLJ preparation is complete, the binder clips were gently removed from the joints and the SLJ sample will look like this.


Fig. 3.31 SLJs prepared after curing process

### 3.4.6 Testing of Single Lap Joints on 25 degrees

Testing of SLJs was then conducted for neat, 0.25, 0.5, 0.75, 1wt% cases at 25°C by following the same steps and setting used in section 3.3.10 with a difference in UTM grip (in this case 0-14 mm grip jaws were utilized).

### 3.4.7 Testing of single lap joint on 50, 75 & 100 degrees

Testing of SLJs was then conducted for neat, 0.25, 0.5, 0.75, 1wt% cases at 50, 75, 100°C by following the same steps and setting used in section 3.3.11 with a difference in UTM grip (in this case 0-14 mm grip jaws were utilized).


Fig. 3.32 UTM testing of SLJs ant room and higher temperatures

# Chapter 4: Results and Discussion

This chapter presents the findings from the experiments conducted for this study. Chapter 3 covered the experimental parameters and their methods. The research findings are reviewed in depth together with the experimental results of tensile tests performed on epoxy adhesives and adhesively bonded single lap joints under various working temperatures and filler concentrations. The failure loads and strength of adhesives and adhesive junctions have been measured and the effects of temperature and filler content evaluated.

## 4.1 Behavior of epoxy Adhesives

### 4.1.1 Effect of temperature and cork powder on failure loads of epoxy adhesives

Epoxy adhesive (LY-556 / AD-22962) at four different temperatures that is 25, 50, 75 and 100 degrees is experimented to evaluate the average failure load with and without cork powder concentration. The cork powder concentration ranges from 0.25 wt.%, 0.50 wt.%, 0.75 wt.% and 1 wt.%. Three samples for each designed experiment were tested under tensile testing at displacement rate of 0.5 mm/min. After the tensile testing, the average failure load values at each temperature and at each cork powder concentration are recorded. Table below describes the behavior for all cases by plotting average value against each case of the experiment:

Table 4.1 Failure loads of adhesives at all temperatures & concentrations

| Temperature (degC) | Concentration (%) | Failure Load (kN) |
|---|---|---|
| 25 °C | ***Neat*** | 1.474 |
| | ***0.25*** | 1.125 |
| | ***0.5*** | 1.102 |
| | ***0.75*** | 0.909 |
| | ***1*** | 0.684 |
| 50 °C | ***Neat*** | 1.247 |
| | ***0.25*** | 1.198 |
| | ***0.5*** | 1.222 |

| | | |
|---|---|---|
| | ***0.75*** | 1.065 |
| | ***1*** | 1.097 |
| 75 °C | ***Neat*** | 0.837 |
| | ***0.25*** | 0.836 |
| | ***0.5*** | 0.898 |
| | ***0.75*** | 0.779 |
| | ***1*** | 0.464 |
| 100 °C | ***Neat*** | 0.598 |
| | ***0.25*** | 0.535 |
| | ***0.5*** | 0.528 |
| | ***0.75*** | 0.532 |
| | ***1*** | 0.662 |

The behavior of epoxy adhesive has been described in the graph below in terms of the failure loads.

- **Failure Loads at 25 °C.** Fig. 4.1 demonstrates the graph of failure load and displacement with and without cork powder concentration (0.25wt.%,0.50wt.%,0.75wt.% and 1wt.%) at 25-degree temperature. It is shown that as the cork powder concentration increases the failure load decreases and lowest value is exhibited at 1wt% concentration of cork, however the values are relatively higher for 0.25-0.75%. The neat single lap joint shows a failure load of 1.54kN at a displacement of 2.77mm. The failure load at 0.25wt.% of cork powder is 1.12kN at a displacement of 1.5mm, while at 0.5wt.%, the failure load is 1.12kN at 2.22mm, at 0.75wt.% the failure load is 1.01kN at 1.96mm and at 1wt.%, it shows 0.637kN at 1.31mm. The load bearing capacity of adhesive reduces as cork powder increases from 0 - 0.25wt.% but increases between 0.5 - 0.75wt.% and then decreases at 1wt.% at 25-degree temperature. The highest failure strength is at 0% cork powder at 25-degree.
- **Failure Loads at 50 °C.** Fig4.2 demonstrates the graph of failure load and displacement with and without cork powder concentration at 50-degree temperature. It is shown that neat adhesive show a failure load of 1.281kN at a displacement of 3.68mm. The

0.25wt.% show a failure load of 1.195kN at a displacement of 3.7mm, while 0.5wt.% show a failure load of 1.27kN at a 3.62mm, 0.75wt.% show a failure of 0.98kN at 2.6mm and 1wt.% show a failure load of 1.074kN at a displacement of 2.36mm. It is shown that failure strength increases from 0.25wt.% to 0.5wt.% and then start decreasing towards 0.75-1 wt%. It is also observed that displacement also increases at concentration of 0.25-0.5 wt% and decreases at higher percentages. The highest failure strength is at 0.25wt.% cork powder at 50-degree.

- **Failure Loads at 75 °C.** Fig4.3 demonstrates the graph of failure load and displacement with and without cork powder concentration (neat, 0.25wt.%,0.50wt.%,0.75wt.%,1wt.%) at 75-degree temperature. Failure load of 0.86kN at a displacement of 2.96mm is observed for neat adhesive. The failure load for 0.25wt.% is 0.84kN at 2.91mm, while at 0.5wt.% concentration the load carrying capacity is 0.93kN at a displacement of 3.83mm, at 0.75wt.% cork powder concentration the failure strength shows a value of 0.73kN at an elongation of 2.56mm and 1wt.% the failure load is 0.87kN at 2.65mm. The elongation increases from neat to 0.25wt.%. 0.5wt.% and then start decreases at 0.75 - 1wt.%. The failure strength shows same trend as it shows for 50-degree temperature that is the strength is higher at 0.25-0.5wt.% and then decreases form 0.5wt.% to 0.75wt.%. At both temperatures, the higher failure load and displacements are exhibited at 0.25-0.5wt. %. The highest failure strength is at 0.5wt.% cork powder at 75-degree.
- **Failure Loads at 100 °C.** Fig 4.4 demonstrates the graph of failure load and displacement with and without cork powder concentration at a temperature of 100-degree. It shows that at a temperature of 100-degree, neat adhesive shows a failure strength of 0.56kN at 4.12mm, while at 0.25wt.% cork powder concentration the failure load is 0.525kN at 3.2mm, at 0.5wt.% the load carrying capacity is 0.567kN at 2.75mm, at 0.75wt.% the failure load is 0.52kN at an elongation of 2.67mm and at 1wt.% cork powder concentration the failure load shows a value of 0.67kN at a displacement of 2.43mm. The failure strength shows same trend as it shows for 25-degree temperature that is the strength is highest for neat adhesive and then it reduces at 0.25-0.5wt.% but it is still higher than 0.75-1wt%. The overall displacement however are much higher than in case of 25 degrees. The highest failure strength is at 0wt.% at 100-degrees.

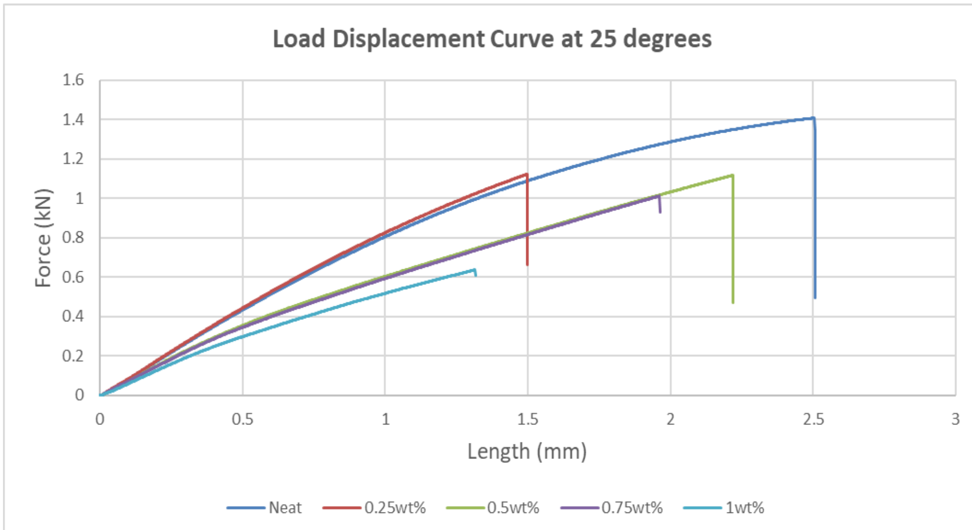



Fig. 4.1 Load-Disp Curves at 25 ºC

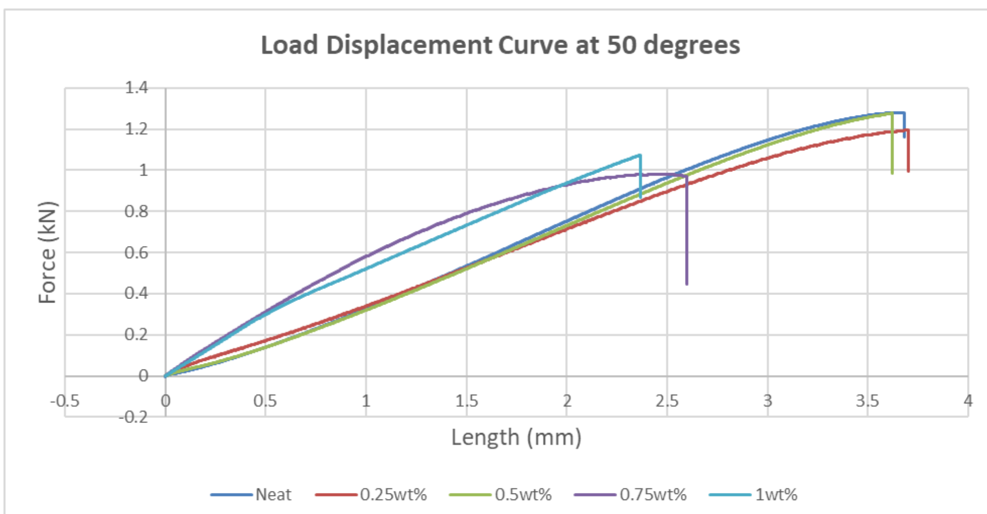



Fig. 4.2 Load-Disp Curves at 50 ºC

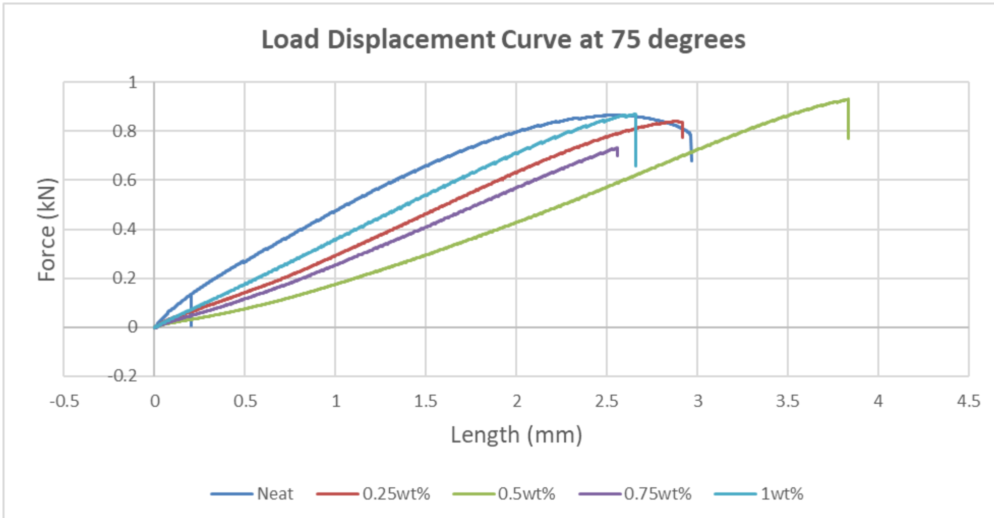



Fig. 4.3 Load-Disp Curves at 75 ºC

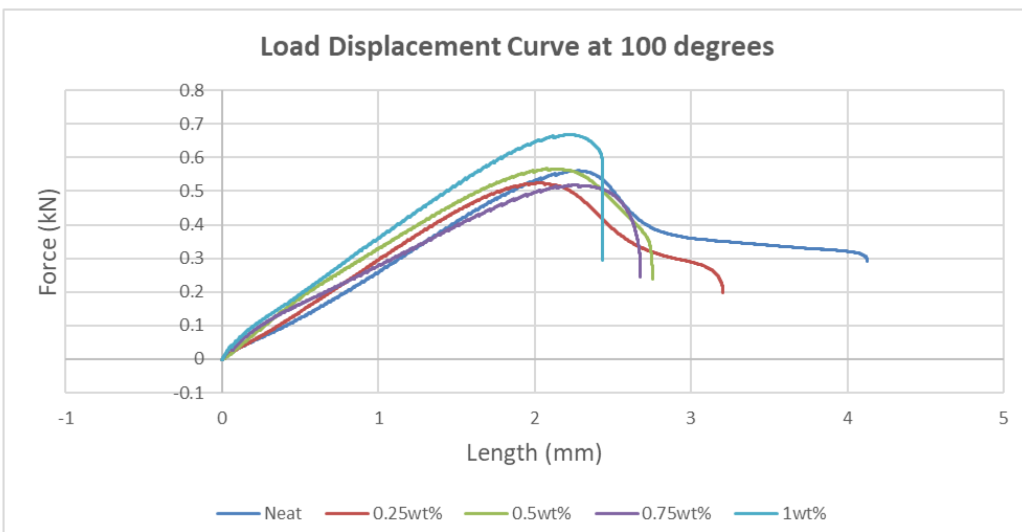



Fig. 4.4 Load-Disp Curves at 100 ºC

#### *4.1.1.1 Observations on failure loads*

A comparative chart has been made to observe the failure load phenomenon in epoxy adhesives.

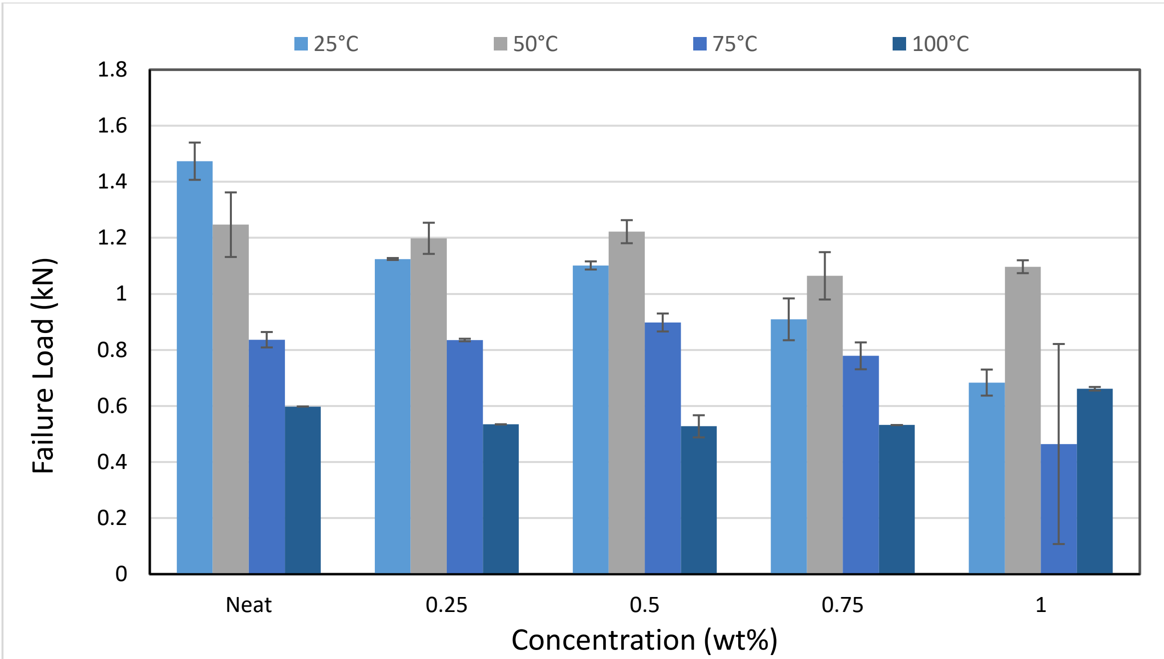



Fig. 4.5 Bar chart depicting failure loads at all temperatures & concentrations

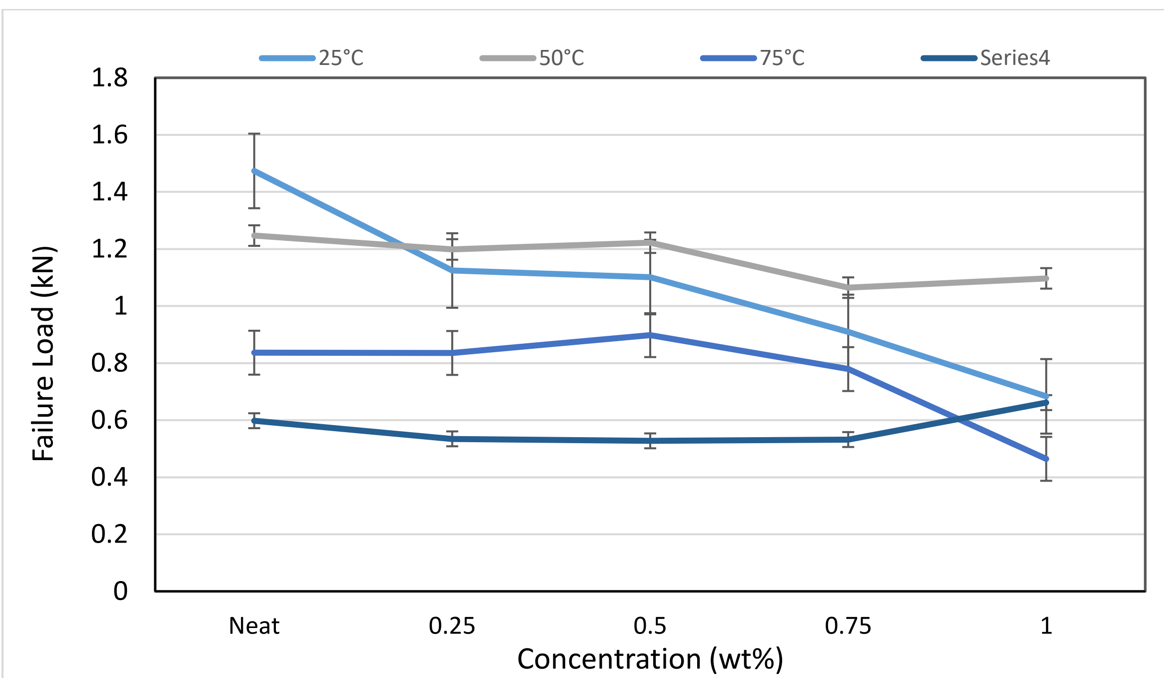



Fig. 4.6 Graphical depiction of failure loads at all temperatures & concentrations

Following observations have been made on failure loads of epoxy adhesives:

- The failure load is larger at neat adhesives as compared to 0.25wt.%. then failure load start increases till 0.5wt.% and show a decreasing value at the concentration in 0.75 to 1 wt.%
- At all temperatures (25,50,75 and 100-degree), the value of failure strength decreases. It means that at same concentration the failure strength decreases by increasing the temperature.
- The highest failure strength shows at room temperature as compared to other temperatures for same concentration.
- The displacement increases with increase in temperature as well as behavior of adhesive becomes more brittle from ductile with increasing temperature

### 4.1.2 Effect of temperature and cork powder on tensile strength of epoxy adhesives

Three samples for each combination were tested under tensile testing with the displacement rate of 0.5 mm/min. After the tensile testing, the average tensile strength values at each temperature and at each cork powder concentration are being calculated and plotted in graphs. Table below describes the behavior for all cases by plotting average value against each case of the experiment:

Table 4.2 Tensile strength of epoxy adhesives at all temperatures & concentrations

| Temperature (degC) | Concentration (%) | Tensile Strength (MPa) |
|---|---|---|
| 25 $^{o}$C | ***Neat*** | 59.870 |
| | ***0.25*** | 55.860 |
| | ***0.5*** | 55.038 |
| | ***0.75*** | 45.998 |
| | ***1*** | 36.347 |
| 50 $^{o}$C | ***Neat*** | 59.881 |
| | ***0.25*** | 60.012 |
| | ***0.5*** | 62.402 |
| | ***0.75*** | 54.546 |

| | | |
|---|---|---|
| | ***1*** | 50.239 |
| 75 °C | ***Neat*** | 42.063 |
| | ***0.25*** | 43.127 |
| | ***0.5*** | 47.711 |
| | ***0.75*** | 42.563 |
| | ***1*** | 39.875 |
| 100 °C | ***Neat*** | 26.417 |
| | ***0.25*** | 26.393 |
| | ***0.5*** | 26.426 |
| | ***0.75*** | 26.255 |
| | ***1*** | 25.407 |

#### *4.1.2.1 Observations on tensile strength*

A comparative chart has been made to observe the tensile strength phenomenon in epoxy adhesives.

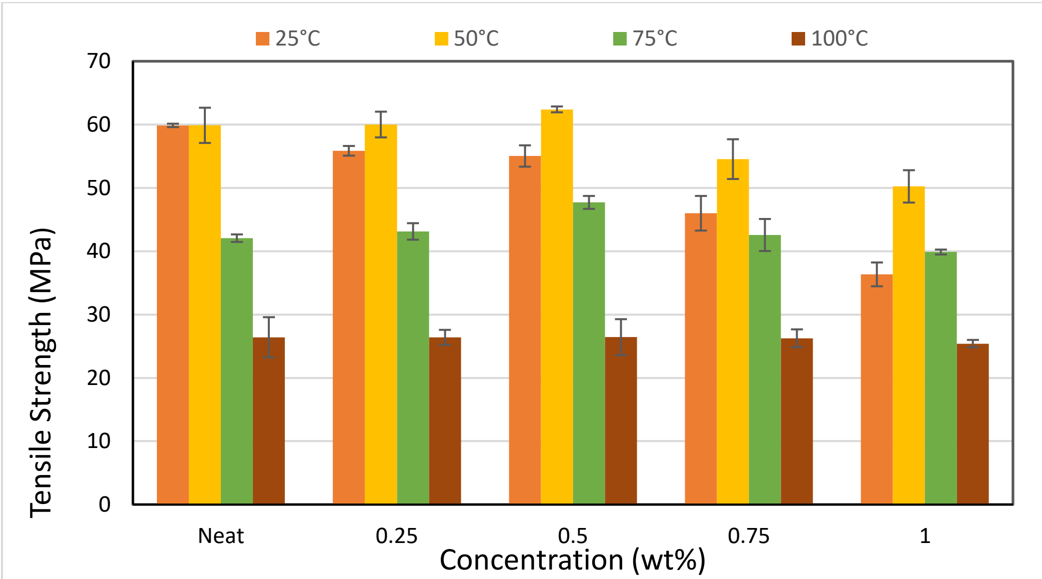



Fig. 4.7 Bar chart depicting tensile strength at all temperatures & concentrations

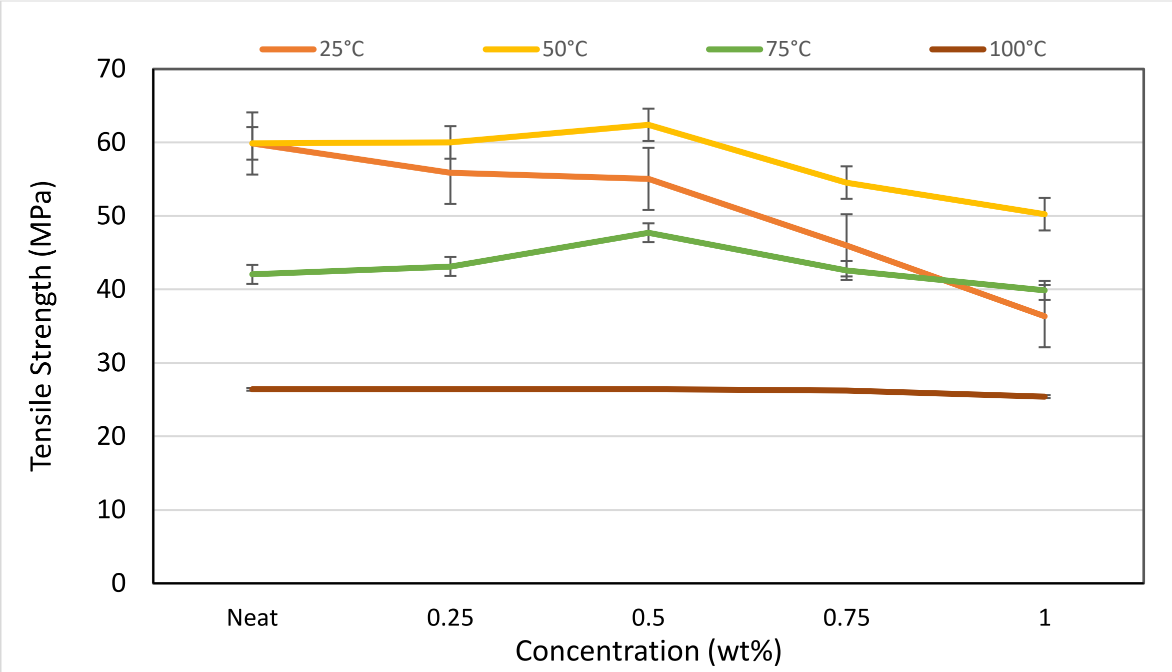



Fig. 4.8 Graphical depiction of tensile strength at all temperatures & concentrations

Following observations have been made on tensile strength of epoxy adhesives:

- The tensile strength increases from neat adhesives till 0.5wt% after addition of cork powder at all the temperatures, however after 0.75wt%, the strength of epoxy adhesives continues to decrease.
- At all temperatures (25,50,75 and 100-degree), the value of tensile strength decreases. It means that at same concentration the failure strength decreases by increasing the temperature.
- The highest tensile strength shows at 50 degrees temperature with 0.5wt% cork powder concentration as compared to other temperatures for same concentration.
- The lowest tensile strength is observed at 1wt% at 100 degrees as compared to other temperatures for same concentration.
- At 100 degrees, the behavior of adhesive does not respond to addition of any amount of cork powder, the adhesive exhibits very low strength and no improvement is seen with cork powder addition since adhesive is very close to the glass transition temperature (Tg).

### 4.1.3 Effect of temperature and cork powder on tensile modulus of epoxy adhesives

Three samples for each combination were tested under tensile testing with the displacement rate of 0.5 mm/min. After the tensile testing, the average tensile modulus values at each temperature and at each cork powder concentration are being calculated and plotted in graphs. Table below describes the behavior for all cases by plotting average value against each case of the experiment:

Table 4.3 Tensile modulus of epoxy adhesives at all temperatures & concentrations

| Temperature (degC) | Concentration (%) | Tensile Modulus (GPa) |
|---|---|---|
| 25 $^{o}$C | ***Neat*** | 2.156 |
| | ***0.25*** | 2.036 |
| | ***0.5*** | 1.457 |
| | ***0.75*** | 1.141 |
| | ***1*** | 1.505 |
| 50 $^{o}$C | ***Neat*** | 0.994 |
| | ***0.25*** | 0.950 |
| | ***0.5*** | 0.952 |
| | ***0.75*** | 0.823 |
| | ***1*** | 0.781 |
| 75 $^{o}$C | ***Neat*** | 0.693 |
| | ***0.25*** | 0.816 |
| | ***0.5*** | 0.789 |
| | ***0.75*** | 0.821 |
| | ***1*** | 0.854 |
| 100 $^{o}$C | ***Neat*** | 0.474 |
| | ***0.25*** | 0.466 |
| | ***0.5*** | 0.513 |
| | ***0.75*** | 0.521 |
| | ***1*** | 0.603 |

#### *4.1.3.1 Observations on tensile modulus*

A comparative chart has been made to observe the tensile modulus phenomenon in epoxy adhesives.

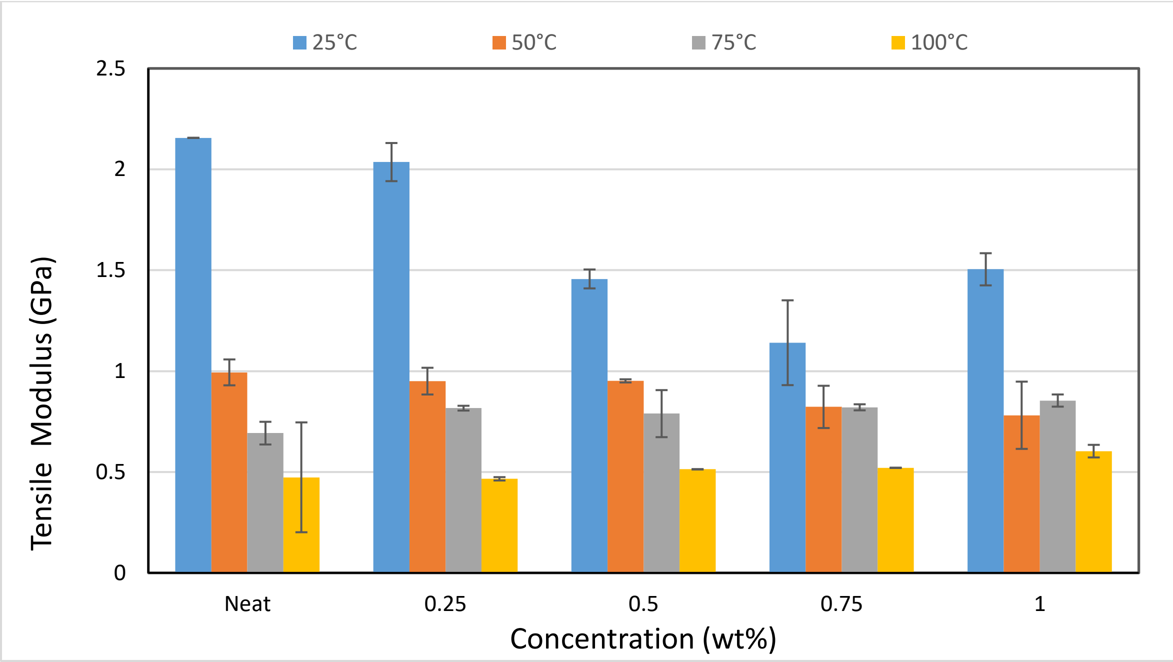



Fig. 4.9 Bar chart depicting tensile modulus at all temperatures & concentrations

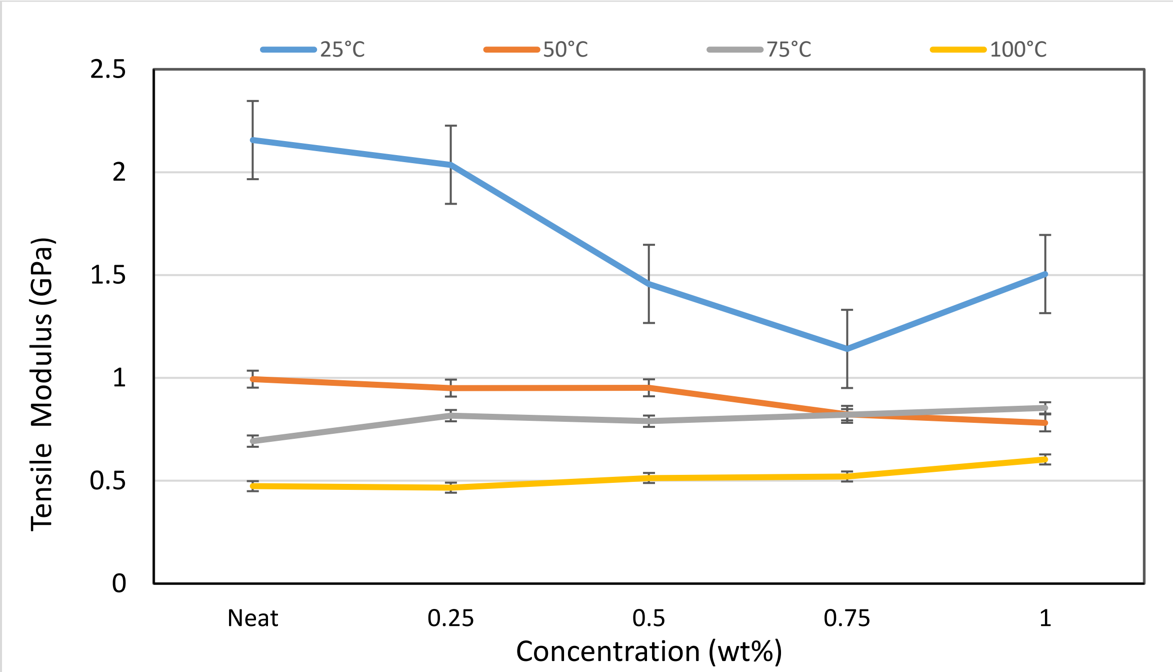



Fig. 4.10 Graphical depiction of tensile modulus at all temperatures & concentrations

Following observations have been made on tensile modulus of epoxy adhesives:

- The tensile strength decreases from neat adhesives till 1 wt% after addition of cork powder at all the temperatures, an overall decrease in tensile modulus since the toughness of adhesive is increasing but overall tensile strength is compromised.
- At all temperatures (25,50,75 and 100-degree), the value of tensile modulus decreases. It means that at same concentration the failure strength decreases by increasing the temperature. This decrease in strength is drastic as temperature increases from 25 – 50 degrees and then uniform decrease is witnessed.
- The highest tensile strength shows at 25 degrees temperature with no cork powder and the lowest tensile strength is observed at 1wt% at 100 degrees as compared to other temperatures for same concentration.
- At 100 degrees, the behavior of adhesive does not respond to addition of any amount of cork powder, the adhesive exhibits very low modulus and no improvement is seen with cork powder addition since adhesive is very close to the glass transition temperature (Tg).

## 4.2 Behavior of SLJs

### 4.2.1 Effect of cork powder on failure loads of SLJs

Three samples of single lap joints for each combination were tested under tensile testing with the displacement rate of 0.5 mm/min. After the tensile testing, the average failure strength values at each temperature and at each cork powder concentration are being calculated and plotted in graphs. Table below describes the behavior for all cases by plotting average value against each case of the experiment:

Table 4.4 Failure loads of SLJs at all temperatures & concentrations

| Temperature (degC) | Concentration (%) | Failure Load (kN) |
|---|---|---|
| 25 °C | ***Neat*** | 10.21 |
| | ***0.25*** | 9.664 |
| | ***0.5*** | 12.941 |
| | ***0.75*** | 11.994 |

| | | |
|---|---|---|
| | ***1*** | 8.313 |
| 50 °C | ***Neat*** | 9.247 |
| | ***0.25*** | 9.066 |
| | ***0.5*** | 11.677 |
| | ***0.75*** | 11.522 |
| | ***1*** | 7.366 |
| 75 °C | ***Neat*** | 8.407 |
| | ***0.25*** | 8.125 |
| | ***0.5*** | 9.247 |
| | ***0.75*** | 9.099 |
| | ***1*** | 7.611 |
| 100 °C | ***Neat*** | 8.069 |
| | ***0.25*** | 7.447 |
| | ***0.5*** | 7.79 |
| | ***0.75*** | 7.484 |
| | ***1*** | 6.971 |

#### *4.2.1.1 Observations on failure loads of SLJs*

An interaction chart has been made to observe the failure load phenomenon in epoxy adhesives to study if the combined effect of temperature and concentration can be witnessed in SLJs. The interaction effect has been studied on Design Expert ® software by plotting all responses (failure loads) against changing temperatures and concentrations. Following data has been used:

$$Inputs = 02\ (temperature, concentration)$$

$$Levels = 04\ for\ temperature, 05\ for\ concentration$$

$$Repeats = 03$$

$$Response = Failure\ Load\ (kN)$$

$$Total\ runs = 04\ x\ 05\ x\ 03$$

$$\boldsymbol{Total\ runs = 60}$$

In this case, the response (failure load) has been plotted on Y-axis and concentration is plotted on X-axis while temperature is plotted on Z-axis to see if there is any interaction between the two factors. The graph shows independent behavior of SLJ under effects of temperature and changing concentrations. The results obtained from Design Expert ® software are shown in the diagram below:

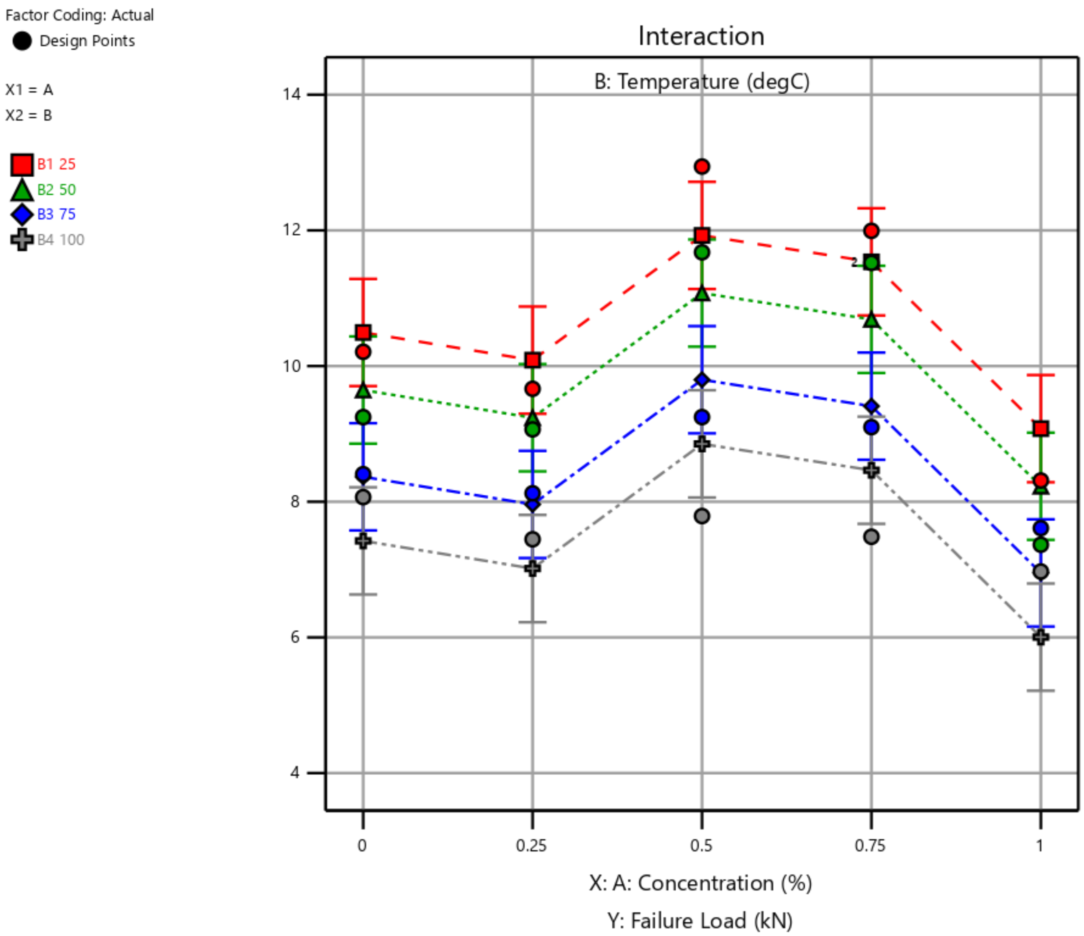



Fig. 4.11 Effect of cork powder on failure loads of SLJs

Following observations have been made on failure loads of epoxy adhesives:

- Overall failure loads of SLJs improve with addition of cork powder in such a manner that initially a small decrease in strength is observed, however at 0.5wt% cork powder concentration, the failure loads become maximum and then minimum loading capacity is observed at 1wt%.

- As seen from figure above, when the temperature increases a small decrease in failure strength is observed between 25-50 °C (upto 50% of Tg value) while a drastic decrease in failure strength is observed between 50°C to 75°C and to °C as the temperature becomes closer to Tg value
- At all temperatures (25,50,75 and 100-degree), the value of failure strength decreases. It means that at same concentration the failure strength decreases by increasing the temperature.
- The highest failure strength shows at room temperature as compared to other temperatures for same concentration.

Figure below shows the comparison of average load for different temperature and different cork powder concentrations. It is observed that the maximum failure load is observed at 0.75wt.% cork powder concentration and at 25- degree. The failure load is maximum at 0.75wt.% for all temperatures but the value of failure load at 0.75wt.% and at 25 degrees is 9.314kN that is large as compared to value of o.75wt.% joints at other temperatures. It is also observed that at 0.75wt.% the values of average failure load start decrease as temperature increases from 25-degree to 100-degree. Hence from the experiments we can observed that composite and aluminum adherend with 0.75wt.% at 25-degree temperature show highest strength and the strength is decreases as we increase the temperature for 0.75wt.%. It is also observed that neat adhesive show slightly higher strength as compared to 0.25wt.% and the strength is increases from 0.25wt.% to 0.75wt.% and decreases at 1wt.%. The maximum strength is observed at 0.75wt.% at 25-degree that is 9.314KN and minimum strength is observed at 1wt.% at 100-degree temperature that is 4.606KN.

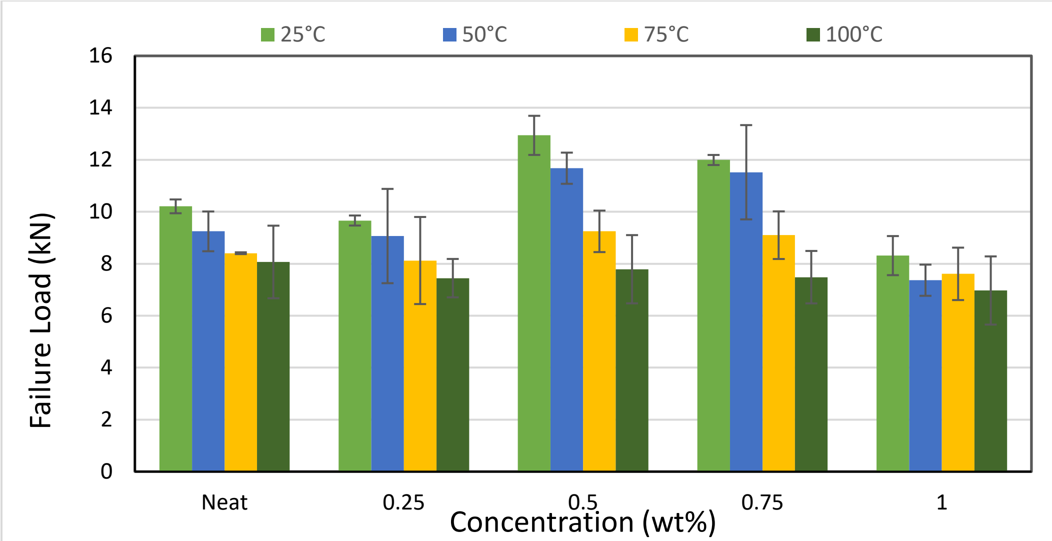



Fig. 4.12 Bar chart depicting failure loads at all temperatures & concentrations

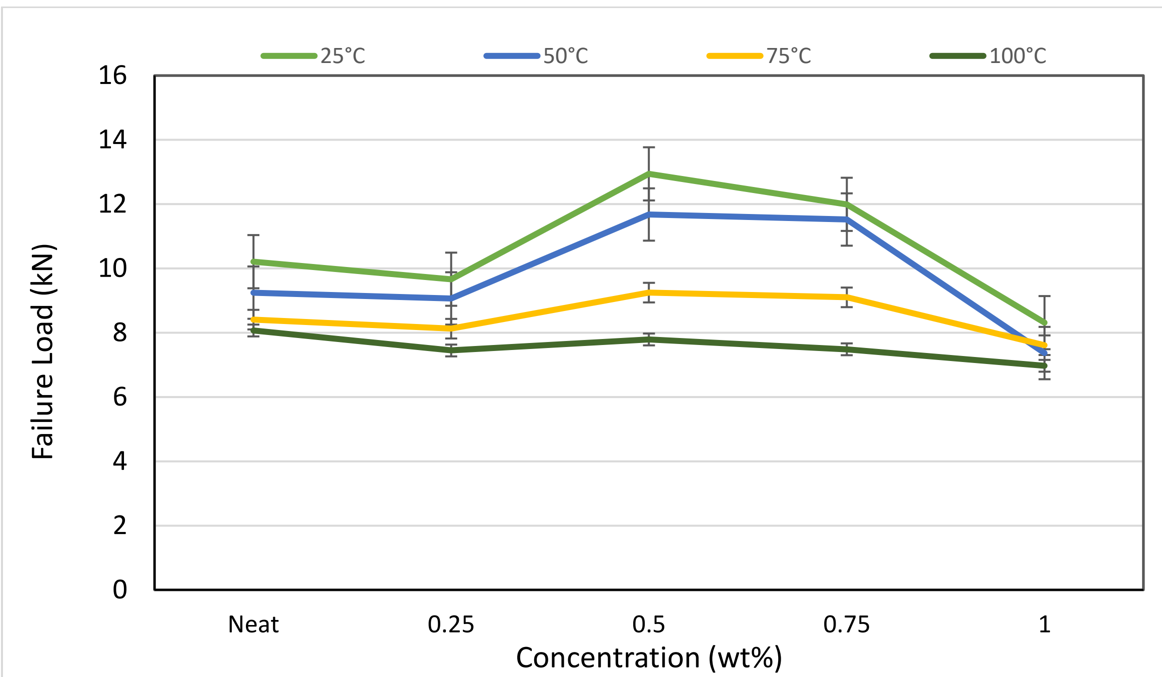



Fig. 4.13 Graphical depiction of failure loads at all temperatures & concentrations

### 4.2.2 Effect of cork powder on failure loads of SLJs

An interaction chart has been made to observe the failure load phenomenon in epoxy adhesives to study if the combined effect of temperature and concentration can be witnessed in SLJs. The interaction effect has been studied on Design Expert ® software by plotting all responses (failure loads) against changing temperatures and concentrations. In this case, the response (failure load) has been plotted on Y-axis and temperature is plotted on X-axis while concentration is plotted on Z-axis to see if there is any interaction between the two factors. The graph shows independent behavior of SLJ under effects of temperature and changing concentrations.

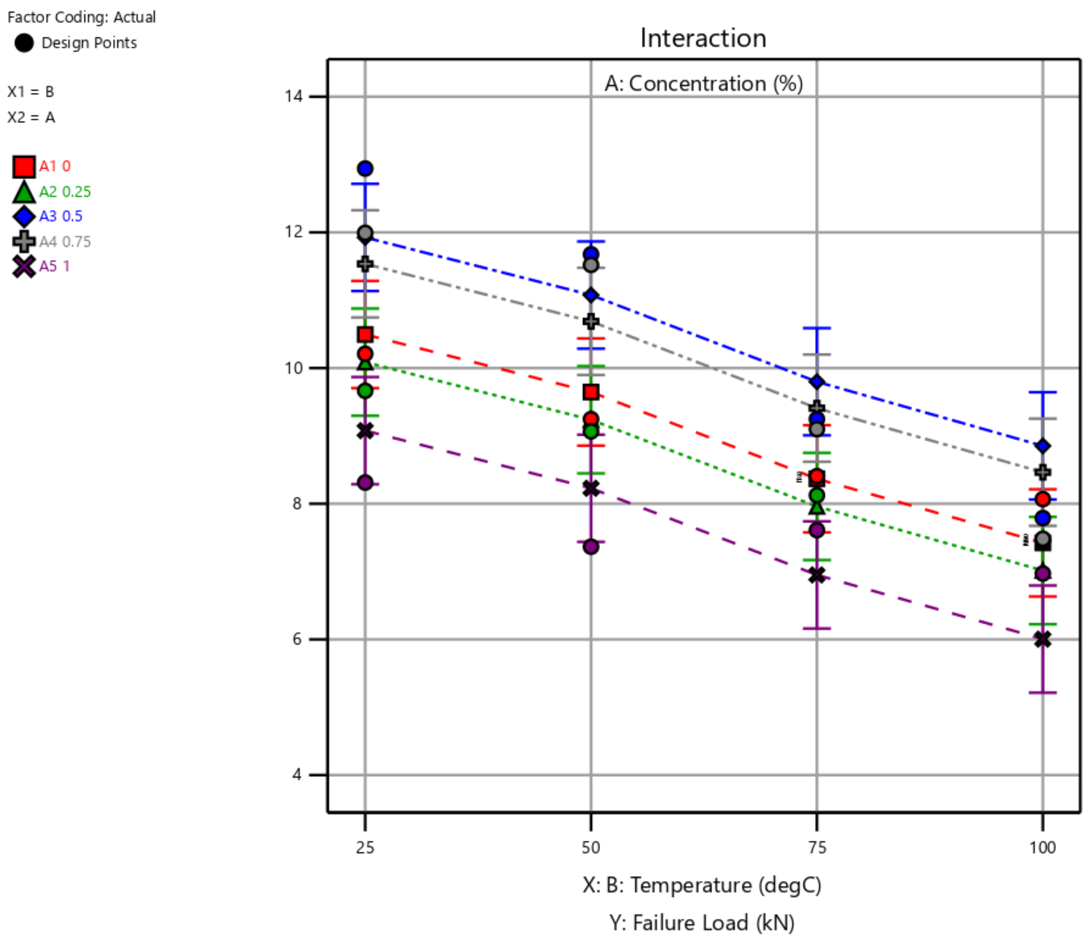



Fig. 4.14 Effect of temperature on failure loads of SLJs

### 4.2.3 Effect of temperature and cork powder on load displacement curves of SLJs

Single lap joints at four different temperatures that is 25, 50, 75 and 100 degrees are experimented to evaluate the average failure load with and without cork powder concentration. The cork powder concentration ranges from 0.25 wt.%, 0.50 wt.%, 0.75 wt.% and 1 wt.%. Three samples for each designed experiment were tested under tensile testing at displacement rate of 0.5 mm/min. After the tensile testing, the average failure load values at each temperature and at each cork powder concentration are recorded.

The behavior of epoxy adhesive has been described in the graph below in terms of the failure loads.

- Figures below shows the average failure load at different cork powder concentration and at different temperature.
- It is noticed that average failure load decreases from neat adhesive to 0.25wt.% and then average failure start increases from0.25wt.% to 0.5wt.%, stay almost the same at 0.75wt% and then decreases at 1wt.%.
- We can say that by increasing cork powder concentration from 0.25wt.% to 0.75wt.% average failure load at different temperatures increases and the average failure load decreases at 1wt.%.
- It is also noticed that average failure at 25-degree temperature is has higher values as compared to the other failure load at other temperatures for 0.5wt.%, 0.75wt.% and 1wt.% concentration.
- As we increase the temperature the average failure load start decreases because the strength of adhesive decreases as the temperature rises, owing to the relative weakness of bonding large objects with a small bonding surface area and the increased difficulty of separating objects during testing.
- For neat and cork powder adhesives, it is shown that average failure load has highest vales at 25 and and has decreasing values at 50 - 100 degrees.
- At 100- degree, the failure load has lowest vales because mechanical properties changes at high temperature and make it weaker and more likely to creep. Hence single lap joints strength decreases with increasing temperature.
- Load displacement curves below shows the failure load comparison of adhesive joints at different temperature. The room temperature is taken as reference temperature and the other temperature value of average failure is compared with that room temperature in term of load displacement curves.
- More ductile behavior is observed in adhesives with increase in the temperature and brittle failure is observed at lower temperatures.
- The failure load is maximum at 0.5wt.% for room temperatures but the value of failure load start decrease as temperature increases from 25-degree to 100-degree. It is also observed

that neat adhesive show slightly higher strength as compared to 0.25wt.% and the strength is increases from 0.25wt.% to 0.5 - 0.75wt.% and decreases at 1wt.%.

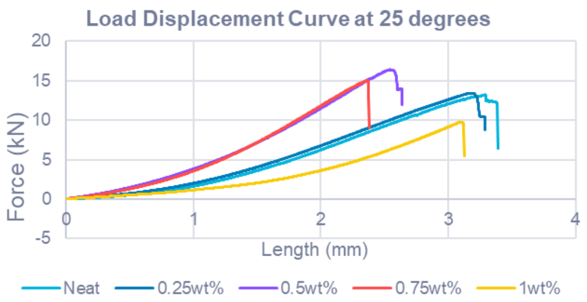



Fig. 4.15 Load disp curves at 25 degrees

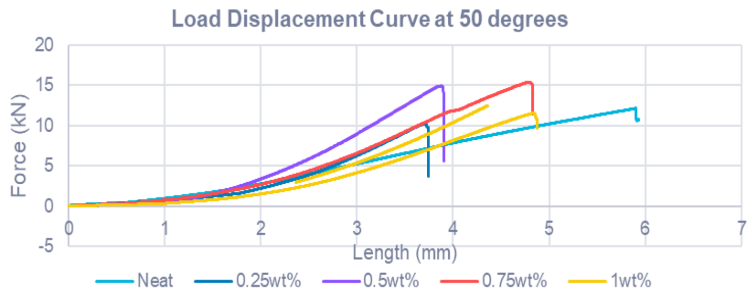



Fig. 4.16 Load disp curves at 50 degrees

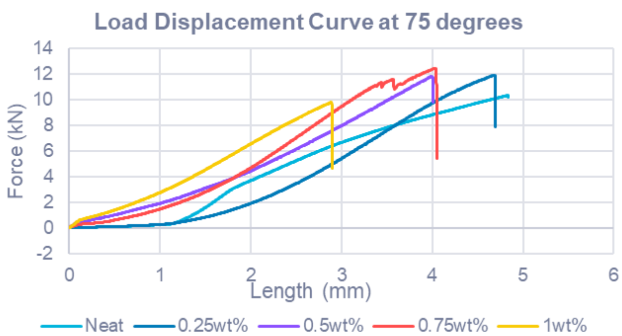



Fig. 4.17 Load disp curves at 75 degrees

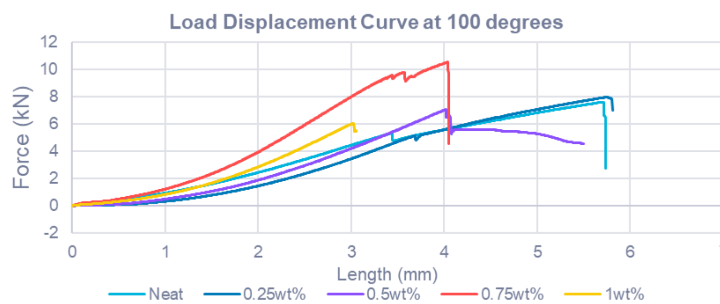



Fig. 4.18 Load disp curves at 100 degrees

### 4.2.4 Percentage failure at different temperatures and different concentrations

Following formula is used here:

**%age difference= ((F.L value at high tem- F.L value at room temp)/value at room temp) *100**

The negative value shows that percentage decrease for every temperature difference from the room temperature/ reference temperature. Similarly, same formula is used for the failure load comparison for neat and with cork filler concentration to see the percentage improvement by taking neat adhesives as a reference at that same temperature as shown in table below.

Table 4.5 Changes in failure loads with concentration change

| Concentration | Temperature | %age Change in Failure Load |
|---|---|---|
| **Neat** | 25ºC | |
| | 50ºC | -5.113 |
| | 75ºC | -27.153 |
| | 100ºC | -19.645 |
| **0.25wt.%** | 25ºC | |
| | 50ºC | -6.777 |
| | 75ºC | -19.054 |
| | 100ºC | -22.318 |
| **0.5wt.%** | 25ºC | |
| | 50ºC | -5.625 |
| | 75ºC | -20.828 |
| | 100ºC | -13.314 |
| **0.75wt.%** | 25ºC | |
| | 50ºC | -5.394 |
| | 75ºC | -21.088 |
| | 100ºC | -14.513 |
| **1wt.%** | 25ºC | |
| | 50ºC | -2.337 |

| | |
|---|---|
| 75ºC | -14.271 |
| 100ºC | -27.804 |

### 4.2.5 Type of failure in SLJs

The table below shows the type of failure as the temperature and concentration changes. From the table it is observed that as temperature and filler concentration changes, the type of failure from mix mode failure are shifted toward the cohesive failure. At higher temperature and filler concentration, cohesive failure is experimented. It is observed that SLJs based on aluminum adherend with brittle adhesives show cohesive failure at high temperatures.

Table 4.6 Change in failure type with temperature

| **Type of Failure in SLJs** | | | | | |
|---|---|---|---|---|---|
| **Temperature** | **Concentration** | | | | |
| | **Neat** | **0.25%** | **0.5%** | **0.75%** | **1%** |
| **25 degrees** | Mix mode failure | Mix mode Failure | Mix mode Failure | Mix mode Failure | Mix mode Failure |
| **50 degrees** | Mix mode Failure | Mix mode Failure | Mix mode Failure | Mix mode Failure | Mix mode Failure |
| **75 degrees** | Mix mode Failure | Cohesive Failure | Cohesive Failure | Cohesive Failure | Cohesive Failure |
| **100 degrees** | Cohesive Failure | Cohesive Failure | Mix mode Failure | Cohesive Failure | Cohesive Failure |

# Chapter 5: Conclusion

The strength and failure mode of epoxy adhesives & single lap joints having aluminum adherends with brittle epoxy is evaluated through tensile test on ultimate tensile machine. The test was performed for neat samples and with the cork powder concentration ranges from 0.25wt.%- 1wt.% and at the temperature ranges from 25 degrees, 50 degrees, 75 degrees and 100 degrees. Following conclusions have been drawn in both cases:

## 5.1 Behavior of adhesive epoxy

- There seems to be an optimum amount of cork particles for obtaining the best adhesive ductility (more than 0.25wt% and less than 0.75wt%). Epoxy with greater strength has less ductility and more likely to be brittle.
- The amount is insignificant at 1wt% the cork particles start to act as defects since the adhesive becomes more brittle.
- As the temperature increases from 50ºC onwards, the adhesive becomes more ductile as it gets closers to glass transition temperature, resulting in higher strain to failure and smaller tensile strength.
- With increase in temperature, overall tensile modulus of epoxy adhesive decreases, which means as the temperature increases, the stress-to-strain bearing ratio reduces in epoxy adhesives.
- As seen from the plots, the strain in epoxy adhesives is increasing with temperature, also the overall response of curves at 25ºC is clearly brittle and becomes more ductile at high temperatures
- Load displacement values of epoxy adhesives continues to slightly increase till 0.5wt% and then decrease indicating that specimen are reducing loading capacity

## 5.2 Behavior of SLJs

- The loading capacity of adhesives has been improved with addition of certain amount of cork powder.
- Cork may be described as a homogeneous tissue of thin-walled cells, regularly arranged without intercellular spaces similar to a honeycomb. This cell configuration improves

epoxy adhesive ability to withstand more damage, compared to the brittle resin without particles.

- However, only up to 0.5-0.75wt% addition may be suitable to these adhesives and any further addition will reduce strength of SLAJs.
- Load displacement values of epoxy adhesives continues to slightly increase till 0.5wt% and then decrease indicating that specimen are reducing loading capacity

## 5.3 Deductions from the research

- Cork powder when added in a certain % acts as a crap stopper and increases the overall capacity of adhesives & SLJs to withstand higher loading
- 0.5wt% of cork particles incorporated in brittle resin give more ductility than other amounts of cork. Specimens above 1% of cork present a worse behavior than specimens without cork probably because from that point cork particles act like a defect.
- Higher temperatures increase the ductility of adhesive joint but tensile strength & modulus of adhesive is compromised
- Strength is drastically decreased in adhesives & joints at temperatures closer to Tg
- As temperature increases, the failure mode of the joint shifts from adhesive failure to cohesive failure

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