Addis Ababa University

Addis Ababa Institute of Technology

School of Mechanical and Industrial Engineering

"Experimental Evaluation of Mode I and Mode II Fracture Behavior of Glass Fiber Reinforced Polymer Composite by Considering Different Environmental Aging"

A Thesis Submitted to the School of Mechanical and Industrial Engineering in Partial Fulfillment of the Requirements for the Degree of Masters of Science in Mechanical Engineering

(Mechanical Design)

By: Bekalu Nebiy

Advisor: Ermias G. Koricho (PhD)

Co-advisor: Araya Abera (PhD candidate)

Addis Ababa, Ethiopia

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Declaration

I declare that the thesis presented here, entitled; **Experimental Evaluation of Mode I and Mode II Fracture Behavior of Glass Fiber Reinforced Polymer Composite by Considering Different Environmental Aging** contains my own original work except I acknowledged and refereed. In addition, it has not been submitted partially or in full for a degree in any institution.

Name of the student: Bekalu Nebiy Endalew Signature _____

Date of submission: June 6, 2020

Place: Addis Ababa, Ethiopia

This thesis has been submitted for examination with my approval as a university advisor.

Main Advisor: Prof. Ermias G. Koricho

Signature: _____

Co-Advisor: Mr. Araya Abera (PhD Candidate)

Signature: _____

Thesis Approval Sheet

The undersigned have examined the thesis entitled "Experimental Evaluation of Mode I and Mode II Fracture Behavior of Glass Fiber Reinforced Polymer Composite by Considering Different Environmental Aging" set by Bekalu Nebiy, a candidate for the degree of Master of Science has successfully presented the necessary written final thesis and oral presentation and certified for partial fulfillment of the thesis requirements for the Degree of Master of Science in Mechanical Design.

Approved By:

Prof. Ermias G. Koricho	_____	_____
Main Advisor	Signature	Date
Mr. Araya Abera	_____	_____
Co-Advisor	Signature	Date
Mr. Behailu Mamo	_____	_____
Internal Examiner	Signature	Date
Dr. Daniel Tilahun	_____	_____
External Examiner	Signature	Date
Dr. Yilma Tadesse	_____	_____
School Dean	Signature	Date
Dr. Ermias Tesfaye	_____	_____
Assoc. Graduate Program Director	Signature	Date

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Contents

Abstract

The influence of different environmental aging on mode I and mode II interlaminar fracture resistance of glass fiber reinforced polymer composite material is experimentally studied in this paper. Test specimens of unidirectional E-glass fiber with epoxy resin as a matrix material are used in this study. The manufacturing techniques include hand layup process that assisted with vacuum bagging system. Double cantilever beam (DCB) specimens for mode I and end-notched flexure (ENF) specimens for mode II loading conditions are used to realize the test. These specimens were categorized to be immersed in pure water, saline water, engine oil and benzene environments for different period of time to visualize the effects. Through this comparison, the mode I interlaminar resistance $G_I = 50.8\, J/m^2$, which is the maximum one, is resulted in the normal specimen test. The other fracture resistance, which is decreased by 10.27% from the normal condition specimen, is observed in 15^{th} day engine oil immersion. On the other hand, in mode II fracture resistance the maximum value of $G_{II} = 2.49\, J/m^2$ is obtained in specimens immersed for 30^{th} day in benzene media. The other proportionate result $G_{II} = 2.29\, J/m^2$ was seen in specimens tested from 15^{th} day pure water media. While in the normal condition specimens, the fracture resistance obtained is $G_{II} = 1.58\, J/m^2$. The result of the experimental work showed that, the resistance to fracture of environmentally exposed specimens are lower compared to the normal specimens in a DCB test. This can be interpreted as the distribution of such environments on the exposed specimens facilitates the dislocation of the mating matrix and hence increases the opening of the laminate. While, in the mode II loading condition, the exposed specimens are in good performance of resistance to delamination compared to the normal. Again, this is the reason behind the absorbed particles help to increase the flexural strength of the immersed specimens.

***Key Words**: Glass Fiber, Epoxy Resin, Environmental effect, Delamination, Fracture resistance*

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Abbreviations, Nomenclatures and Acronyms

A	Area
ASTM	American Standard for Testing and Materials
CFRPs	Carbon Fiber Reinforced Composites
CMCs	Ceramic Materials Composite
DCB	Double Cantilever Beam
EPFM	Elastic Plastic Fracture Mechanics
Eqn.	Equation
FEM	Finite Element Method
FRP	Fiber Reinforced Polymer
G	Energy release rate
GF	Glass Fiber
GFC	Glass Fiber Composite
GFRP	Glass Fiber Reinforced Polymer
K	Stress intensity factor
L	Litter
l	Length
LEM	Linear Elastic Mechanics
MMCs	Metal Matrix Composites
NASA	National Aeronautics and Space Administration
PMCs	Polymer Composites
PVA	Polyvinyl Acetate
SEM	Scanning Electron Microscopy
UAV	Unmanned Air Vehicle
V_f	Volume of fibre
V_m	Volume of matrix
W_f	Weight of fibre
W_m	Weight of matrix
ρ	Density

Chapter One:

1. Introduction

1.1. Background

Mostly design engineers cope with the dilemma of material selection. The usual case is materials that would work the best for one specific element of the design is lacking in properties required by other elements of the design. This always leads engineers to study further to avoid these problems. Through this, new materials with new better properties can discovered. A material consisting of two or more components with different properties and distinct boundaries between the components can referred to as a composite material. Moreover, the idea of combining several components to produce a material with properties that are not attainable with the individual components used by man for thousands of years. These composite materials can classified into two main groups. The first group comprises composites known as 'filled materials'. The main feature of these materials is the existence of some basic or matrix material whose properties are improved by filling it with some particles. Usually the matrix volume fraction is more than 50% in such materials, and material properties, being naturally modified by the fillers, are governed mainly by the matrix. As a rule, filled materials can be treated as homogeneous and isotropic. The second group of composite materials called 'reinforced materials'. The basic components of these materials sometimes referred to as 'advanced composites' are long and thin fibers possessing high strength and stiffness. The fibers bound with a matrix material whose volume fraction in a composite is usually less than 50%. The main properties of advance composites due to these materials find a wide application in engineering are governed by fibers [1].

Composites make up a very broad and important class of engineering materials. World annual production is over 10 million tones and the market has in recent years been growing at 5- 10% per annum. Composites are used in a wide variety of applications. Furthermore, there is considerable scope for tailoring their structure to suit the service conditions. This concept is well illustrated by biological materials such as wood, bone, teeth and hides; these are all composites with complex internal structures designed to give mechanical properties well suited to the performance

requirements. Adaptation of manufactured composite structures for different engineering purposes requires input from several branches of science [2]. The science verifies how the composite materials cope up to be used for engineering applications.

The application of composite materials have recently increased in the field of aerospace, automobile, nuclear, marine, biomedical and in other engineering areas due to following reasons; high strength or stiffness for lower weight, superior fatigue characteristics, and facility to change fiber orientations etc. As the paper remarks, weight is the most significant variable in the overall design of structures for example for vehicles. A decrease in structural weight of Vehicles will result in reduced fuel load, reduced cost, or increased payload. The weight of the tank can be reduced by the use of new and advanced materials with good strength to weight ratios. Hence, the application of lightweight composites over metals seems to be the best alternative. The use of light-weight composites will also reduce the inertial loads. For example in this study, the X-33 RLV (Reusable Launch Vehicles), which was a joint venture of NASA and Lockheed Martin Aeronautics Company, utilized liquid hydrogen (LH_2) tanks were made of an advanced carbon composites which results decrease in total weight [3].

Now a day composite materials are widely used materials based on their advantages and uses over others. The service conditions of Products that made from these composite materials are exposed to different environmental conditions. Hence, it is found that the effects of the media or environmental condition should be studied. As already known that the uses and advantages of composite materials over metals were found preeminent due to the reasons illustrated as below[4] [5].

Uses of composite materials:

- Space technology and production tails, wings and other compartments.
- Sport goods like racing car bodies and bicycle frames.
- For wind turbine blades.
- For civil infrastructures.
- Fuel-efficient transport vehicles etc.

Advantages of using composites over metals: [4].

- Cost effective
- High strength and low weight
- Complex shape parts can be simply manufactured
- Excellent resistance against acids, chemicals etc.
- Excellent weather/water resistance.
- Excellent isolation of electrical habits
- High thermal isolation, fire retardant habits, and high resisting temperature performance.

At the same time, composite materials are exposed to different problems such as inter ply-cracking, inter laminar delamination, and fiber cracking. Among these three problems, inter laminar delamination is a common failure mode in the composite materials. The delamination mechanism is very complicated and many factors will influence the failure process. Even though numbers of methods of measuring delamination resistance have been developed, the physical origin of the delamination is still not well understood [6]–[8]

As mentioned due to many advantages, recently most researchers gives an attention on the resistance of fracture of composite materials. However, regarding to considering the environmental exposures of the composites found rare. In this paper, there are different environmental conditionings taken into consideration by undertake the service situations of products of these materials may exposed into different environments. One of the reasons for increasing attention to composite materials is the strength to weight ratio for light weight mechanical parts. In the present work, a simple and standardize DCB as well as ENF specimens were prepared for delamination fracture toughness test. The estimation of crack length after the initial crack has been registered by direct vision during test. The test has been developed on the universal testing machines unto the guidelines from ASTM standards and results were obtained accordingly. After the completion of this research, the developed composite will be applicable for semi structural parts of vehicles like compartments, for fuel reservoir, and for seat.

1.2. Problem Statements

Even though fiber reinforced composite polymer materials has several advantages including: low weight, low cost, and friendliness to environment and high specific mechanical performance to weight ratio of metals, delamination is a common problem on the interlaminar features of composites. One factor for this phenomenon to occur and leads to cause failure of composite materials is the medium in which the material purposely used on [4], [9]. The effect of this media should be addressed for the prevention of failures to come. Hence, the influence of environmental effects on composite materials has to be considered which greatly supports the design purpose. In this paper, composite materials of glass fiber-epoxy polymer matrix are supposed to investigate the fracture behaviors under mode I and mode II loading conditions comprising various medium of exposure.

1.3. Objectives

1.3.1. General Objective

The main focus of this work is to determine the effect of different environmental aging on the fracture behavior of glass fiber reinforced epoxy composite material under mode I and mode II loading conditions.

1.3.2. Specific Objectives

The specific objectives of this research includes:

- Investigating the effect of saline water media on fracture toughness value of unidirectional E-glass fiber epoxy composite laminates for mode I mode II loading conditions.
- Investigating the effect of Engine oil media on fracture toughness value of unidirectional E-glass fiber epoxy composite laminates for mode I mode II loading conditions.
- Investigating the effect of Benzene media on fracture toughness value of unidirectional E-glass fiber epoxy composite laminates for mode I mode II loading conditions.

- Investigating the effect of pure water media on fracture toughness value of unidirectional E-glass fiber epoxy composite laminates for mode I mode II loading conditions.

1.4. Scope of the Study

The research presented here focuses on the determination of fracture resistance of glass fiber reinforced epoxy resin composites, under mode I and mode II loading conditions based on ASTM standards and the results are obtained accordingly. The thesis work comprises different environmental effects to know its influence only on the delamination resistance of the specified composite material it does not include the other properties. The evaluation of fracture resistance of DCB and ENF specimens to mode I and mode II loading conditions respectively were studied only by experimental work, comparisons by an analytical and by numerical methods are left for future works.

1.5. Limitations

In order to have values that are more accurate in researches, proper testing machines should be available. One inadequacy is an appropriate specific UTM machines for composite materials that fits for different sized and designed specimens.

1.6. Organization of the Paper

The thesis presented here is organized into five main chapters. Based on the titles under each chapter, the corresponding points were discussed in detail. In the first chapter, the background, statement of the problem, objective of the study are presented. In the second chapter, different literatures related to this work are reviewed. In the third chapter this work; materials used, manufacturing of test specimens, experimental methods and conditions has been stated. In chapter four, the investigated results and discussions are obtained. In the last chapter five, conclusions drawn, recommendations and future developments are presented.

Chapter Two:

2. Literature Review

2.1. Fibers for Advanced Materials

Fibers with the polymer matrix are used as reinforcement in composite materials. Based on the reinforcement, fibers can be either continuous or discontinuous. Continuous fibers are in the form of several patterns based on the orientation. In this study the unidirectional continuous E-glass fiber have been used as reinforcement. Continuous glass fibers which is the first type of fibers used in advanced composites are made by pulling molten glass at a temperature about 1300^{o}C through 0.8-3.0 mm diameter dies and further high-speed stretching to a diameter of 3-19 μm. Usually glass fibers have solid circular cross sections. However there exist fibers with rectangular, triangular, and hexagonal cross sections, as well as hollow circular fibers [1]. Important properties of glass fibers as components of advanced composites for engineering applications are their high strength with lower weight which is maintained in humid environments but degrades under elevated temperatures, relatively low stiffness which is about 40% of the stiffness of steel, high chemical and biological resistance, and low cost.

2.2. Types of Composites

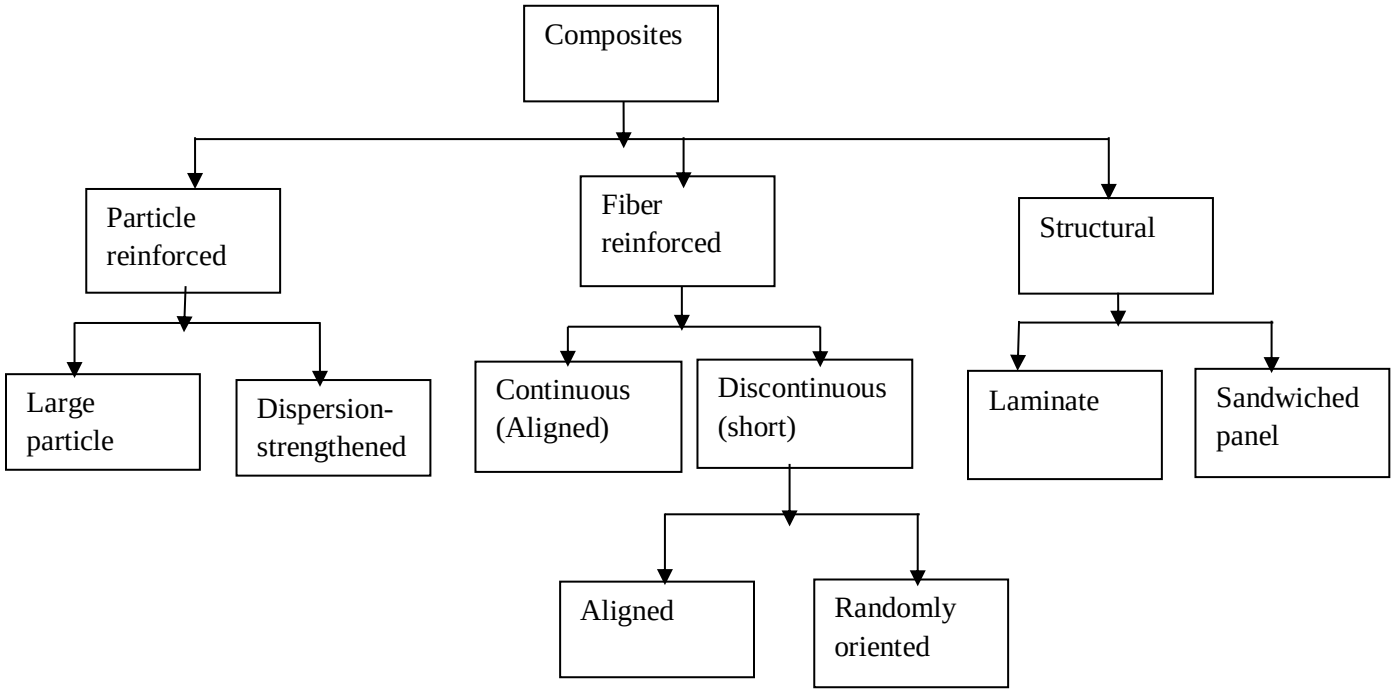
When we see the typical microstructure classifications of composite materials grouped according to the nature of the matrix; are metal matrix composites (MMCs), ceramic materials composite (CMCs) and polymer composites (PMCs). Most composites in industrial use are based on polymeric matrices; thermoset and thermoplastics. These are usually reinforced with aligned ceramic fibers, such as glass or carbon. They commonly exhibit marked anisotropy, since the matrix is much weaker and less stiff than the fibers. More recently, there has been considerable interest in metal matrix composites (MMCs), such as aluminum reinforced with ceramic particles or short fibers, and titanium containing long, large-diameter fibers. The property enhancements being sought by the introduction of reinforcement are often less pronounced than for polymers, with improvements in high-temperature performance or tribological properties often of interest. While various industrial applications have been developed or are being explored for MMCs, their

commercial usage is still quite limited when compared to that of polymer composites (PMCs) [2]. To end with, in this thesis, fracture toughness behavior of composites based on ceramic fibers of glass fiber and polymer composite of epoxy resin has been the research point.

These classifications here are usually to impart toughness to the matrix by the introduction of other constituents and the simplicity of manufacturing.

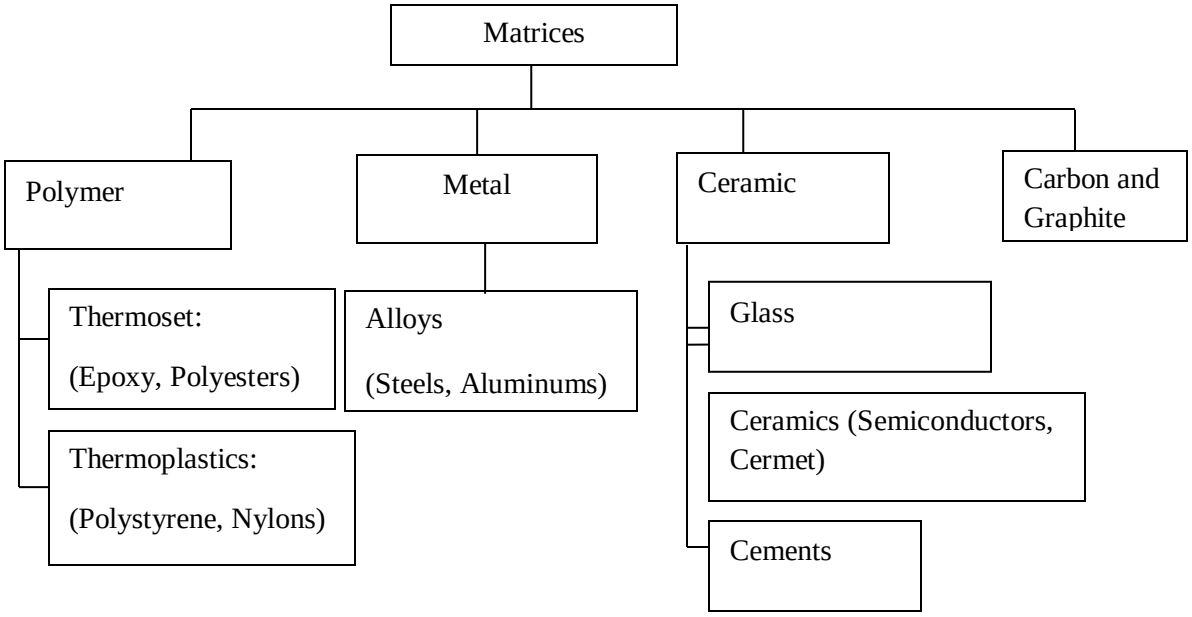
The classification of composites based on the reinforcement is as depicted in the chart below.

Chart 1: Classification of composites



The matrices of composite materials can also classify as follows.

Chart 2: Classifications of matrices



As shown in the above chart, Polymer matrices can be categorized as thermoplastic and thermosetting [10]. These two categories are illustrated as follow.

Thermoplastic: in this type of matrices, processing is faster than thermoset composites since no curing reaction is required and softens upon heating and can be reshaped with heat and pressure. Examples of thermoplastics are; polypropylene, polyvinyl chloride (PVC), nylon, polyurethane, poly-ether-ether ketone (PEEK), Polyphenylene sulfide (PPS), polysulpone.

Thermosetting: this type of matrix material become cross-linked during fabrication and do not soften upon reheating. Examples of thermoset polymers are; polyesters, epoxies, polyimide. From these polymers, epoxy resins are widely used for most advanced composites and is used in this work based on the properties presented on materials and methods section.

2.3. Environmental Effects on Composite Materials

Properties of composite materials, as well as properties of all structural materials are affected by environmental and operational conditions. Moreover, for polymeric composites this influence is more pronounced than for conventional metal alloys because polymers are more sensitive to

temperature, moisture, and time than metals. Moisture content in the material depends on environmental conditions, temperature, pressure, fiber and matrix materials, levels of porosity, and material damage. Because the stable moisture content is rarely reached in real composite structures, its current distribution through the laminate depends on the laminate structure and thickness. Among the polymeric composites, the highest capacity for water absorption under room temperature is demonstrated by aramid composites **(7**±0.25 percentage by weight) in which both polymeric matrix and fibers are sensitive to moisture. In real aramid-epoxy and carbon-epoxy composite structures moisture content is usually about 2% and 1%, respectively. The lowest sensitivity to moisture was shown in boron composites. Metal matrix, ceramic, and carbon-carbon composites are not affected by moisture. While materials exposed to temperature and absorb water it expands and decreases the mechanical strengths and stiffness. For carbon-epoxy composites this reduction is about 12%, for aramid-epoxy composites – about 25%, and glass-epoxy materials – about 35%. After drying up, the effect of moisture usually disappears [1].

Composites are affected by heat and moisture during operation under severe environmental conditions. This paper considers moisture content conditions as a variable, which has great effect on the fracture mechanics of composite materials. Composite materials absorb moisture and expanded gradually with time. These will in turn reduce the failure time. The most moisture is absorbed by the epoxy and expands gradually to the reinforced fiber this incase affects the interfacial bonding strength either negatively or positively based on the behavior of the environmental conditioning. Due to the plasticization effect the epoxy during immersion on water, most studies revealed that the tensile strength and elastic modulus has been decreased [11].

2.4. Fracture of Composite Materials

Fracture can defined as the mechanical separation of a solid material due to the application of stresses. The fractures of engineering materials are classified as ductile or brittle fractures. Ductile fractures of a material absorb more energy before failerity; while brittle materials fracture, absorb little energy. Ductile fracture is with a high-energy dissipation associated with a large plastic deformation before crack instability occurs in which it can be said high-energy process. Whereas brittle fracture is a low-energy process, which leads to catastrophic failure without warning as the

crack velocity is typically high. Hence, little or no plastic deformation may possibly involve before separation of the material [12]. Furthermore, recently, the fracture toughness of a material is related to the amount of energy required to create fracture surfaces. The application of fracture mechanics concepts has identified the primary parameters that affect structural integrity [8]. These parameters include the magnitude and range of the applied stresses, size, shape, orientation of cracks, rate of propagation of the existing cracks and the fracture toughness of the material.

There are two categories of fracture mechanics in which the fracture of a material going to be studied, Linear Elastic Mechanics (LEM) and Elastic Plastic Fracture Mechanics (EPFM). The LEM approach to fracture analysis undertakes the material nominally elastic at regions away from the crack, except for a small region of inelastic deformation at the crack tip. The toughness of the material, which is its resistance to fracture, is determined in terms of the stress intensification factor, K and strain energy release rate G.

One of the underlying principles of fracture mechanics is that the unstable fracture occurs when the stress intensity factor at the crack tip reaches a critical value K_C. The EPFM is used when there is large-scale crack tip plasticity or blunting (flat or large opening). Polymer composite materials often show a mixture of ductile and brittle failure processes. There are several fracture modes in polymer composites such as delamination or interlaminar fracture, matrix cracking or intralaminar fracture, matrix-fiber debonding, fiber breaking, fiber pullout etc. [8].

2.4.1. Interlaminar fracture toughness (Delamination)

The occurrence of interlaminar fracture greatly reduces the stiffness of a structure and leads for the failure of the material during service. The interlaminar performance is characterized by weakness under both tensile and shear stresses, the stress occur hear become significant and affect the overall performance where geometrical and material discontinuities exist. A delamination may be loaded in Mode I (tensile), Mode II (shear), Mode III (tearing shear), or it may be loaded in combination of these Modes [8].

There are two alternative fracture analysis approaches to determine the fracture toughness of a composite material; the stress intensity and the energy criterion approach.

Stress intensity Approach

As mentioned above the stress intensity approach based on LEM uses a constant known as the stress intensity factor K. The stress intensity factor completely characterizes the crack tip conditions in a linear elastic material. The fracture may occur at some critical combination of stress and strain, and hence it follows that failure occurs at some critical stress intensity, K_{IC}. K_{IC} is a measure of the fracture toughness and is a material constant which is independent of the size and geometry of the cracked body [3].

The parameter K_I is the crack driving force and its critical value is a material property known as fracture toughness, which in turn, is the resistance force to crack extension. In fact, K_I equation is applicable to a specimen containing other crack geometry subjected to a remotely applied tensile stress. Experimentally the critical value of K_I, known as fracture toughness can be determined at a fracture stress when the crack length reaches a critical or maximum value prior to rapid crack growth. Thus, K_I can be K_{IC} as the applied stress, σ reaches the yield point σ_f when the crack length a goes to the critical length (a_c), which can be expressed as:

$$K_I = \sigma_f \sqrt{\pi a_c f(g)} \qquad \text{Eqn. 1.1}$$

Where 'σ' is the stress applied on the material, '$f(g$' is the dimensionless factor for the specimen geometry and loading condition and K_{Ic}, the mode I critical stress intensity factor.

Energy Criterion Approach

The energy approach predicates that a crack propagates when the energy available for crack growth is sufficient to overcome the resistance of the material. The material resistance may include plastic work, surface energy or other types of energy dissipation connected with a propagating crack. The energy available for the increment of the crack is called the energy release rate, which is also called the crack extension force or the crack driving force.

The energy release rate G is defined as the rate of change in potential energy with crack area. It is an alternative measure of the fracture toughness. The energy release rate at the moment of crack propagation is called the critical energy release rate G_c which is the material resistance to fracture. The critical energy release rate, G_c for a DCB specimen is given by:

$$G_C = \frac{F^2 a^2}{BEI} \qquad \text{Eqn. 1.2}$$

Where; F is the critical force at the crack propagates, a is the initial crack length and B is the width of the specimen and EI is the flexural rigidity of the specimen. To conduct the mode I fracture toughness the paper follows the following methods. The specimen is first loaded and the crack is allowed to propagate to a specified length (Δa). The specimen is then unloaded. If the DCB specimen is loaded linearly to P_1 where the crack of size a_1 begins to grow and the load drops P_2 while the crack extends from a_1 to a_2, then the loss in strain energy due to crack extension is given by the area ΔA between the loading and unloading curves. The fracture toughness is measured as the energy release rate 'G', which is calculated from the load-displacement diagram for the specimen from the DCB test. The area method is used to calculate the fracture energy, which is given by [3]:

$$G = \frac{\Delta A}{B \Delta a} \qquad \text{Eqn. 1.3}$$

Where; ΔA the area between the loading and unloading curves, Δa is the crack extension and B is the width of the specimen.

2.5. Modes of Fracture

In Fracture mechanics there are three modes of loading as shown in the figure below. In mode I the forces act perpendicular to the crack it also called the opening mode. In Mode II the forces act parallel to the crack plane. One force is pushing the top half of the crack back and the other force is pulling the bottom half forward. This creates a shear crack. Since the forces do not cause the material to move out of its original plane this is also called in-plane shear. In mode III the forces are perpendicular to the crack and pull the top half left and the bottom half right or vice versa. This causes the material to separate out of its plane. It is also called out-of-plane shear.

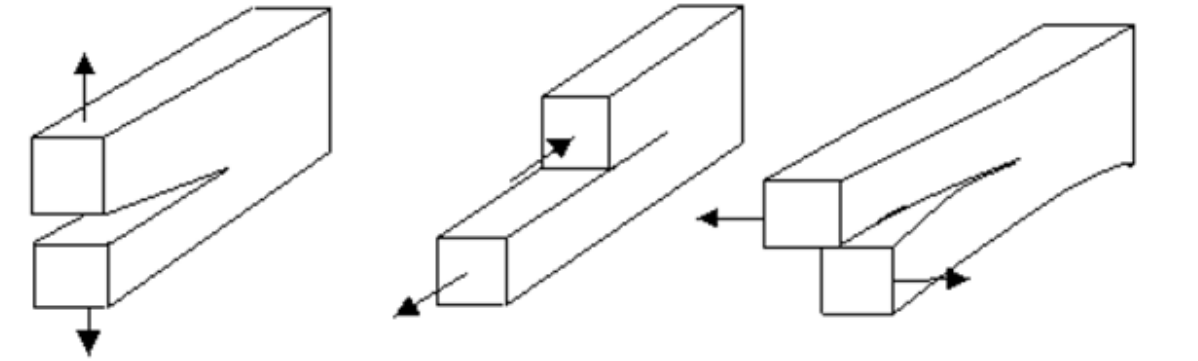


Figure 2-1 Basic fracture mode types

2.6. Overview of Related Previous Works

The fracture toughness of different composite materials based on several parameters is studied on different literatures. Some are deal on the enhancement mechanisms of fracture toughness by introducing different interleaves. Other studies try to investigate the effects of different environmental conditions on fracture toughness behavior of composite materials. The thesis presented here deals on the fracture toughness of a composite material composed of E-glass fiber reinforced epoxy matrices by introducing effects of different environmental exposures such as saline water, pure water, benzene and engine oil for different durations of time. There are different researchers gave attention and devote their time on the fracture behaviors of composite materials. These researchers have used their own parameters for the prediction of fracture toughness and the mechanical properties of different composite materials. Among the several literatures that are related to the fracture toughness of composite materials, some are listed below.

Mode I fracture toughness test as per ASTMD 5528 standards has been carried out on fracture toughness testing machine by varying the composition of the composite constituents. The samples with the ratio of 30:70, resin and fiber have more fracture toughness compared to the samples with the ratio of 40:60. The samples with the ratio of 40:60 have more fracture toughness compared to the samples with the ratio of 50:50. From these we observe, the fracture toughness increases as the ratio of fiber increases [6].

Another paper which goes through dealing the effects of bucky paper interleaves on the fracture toughness of composite materials made from carbon nanofibers on interlaminar shear properties of carbon fiber reinforced composites (CFRPs) are studied. The study includes fabrication of bucky papers, resin impregnation by different techniques, i.e., soaking, hot-compression and vacuum filtration, followed by b-stage curing and the integration with carbon fiber prepregs to produce CFRP composites with bucky paper interleaves. The vacuum infiltration technique results in the best quality of polymer impregnation through bucky papers. Remarkable 31% and 104% improvements in interlaminar shear strength and mode-II shear interlaminar fracture toughness of the multiscale composites, respectively, are achieved with the incorporation of interleaves at failure-prone locations [13].

According to Saghafi [14], the experimental results showed that composites containing 5 wt.% whiskers (SiC) exhibited 67% increase in the crack initiation interlaminar fracture toughness G_{IC}, whereas it exhibited 55% increase in the maximum G_{IC}.

Ali [15] studies about the effect of environmental exposures on the fracture properties of glass fiber polymer composite materials. From the findings as the pollutant from outdoor environment, UV radiation and sunlight increases, the mechanical properties of the glass fiber decreases. Even though, the study has not tried salty water and oily media to relate the mechanical property results. Having this, the points mentioned above are to be dealt in this research.

Zhao, Liu, Seah and Chai [16] conducted that the fracture toughness decreases monotonically with seawater exposure and increasing immersion temperature except for pure Mode II results, where it surged up nearly 20% compared with the results of dry specimen. However, most specimens with seawater absorption exhibited increasing resistance with the delamination growth. In the study, there is no description about the standards. In addition, there is nothing about time intervals taken or immersion duration of cycle and the other point is what else if oil used.

Yu, He, Jiang and Yang [17] shows how the surface treatment increases the interlaminar fracture toughness of Fiber Metal Laminates (FMLs) by immersing for 60 days on seawater, which enhances 25.5 times greater than from the normal. Even though, it has seen a gap about what will

be the effect for the composite of glass fiber with matrix of epoxy resin. Having this study, we can have something useful for enhancing the fracture toughness of glass fiber/epoxy composite.

Veazie, Robinson and Shiva Kumar [18] conducted their study by exposing specimens prior to testing to three forms of marine environments for 500 hrs. at wet, hot/wet at a temperature of 80^{o}C and hot/dry at 80^{o}C environments. The desire of this study beyond the presented paper is just to add and see the effects of oil on the fracture toughness of glass fiber /epoxy matrix. Results showed that exposure to elevated temperatures, along with inducing cracks between the face sheet and a PVC core degraded by elevated temperature exposure appear to be the most detrimental to interfacial fracture toughness.

Another study showed results when moisture increase from high humidity the fracture toughness decrease under mode I and mode II loading conditions. Moreover, it has seen significant increase in mixed mode fracture toughness under low humidity conditions. Although the mixed-mode toughness reduced with increasing levels of humidity, the values are not differing from the joints fabricated using materials without environmental effects [19]. The gap visualized from this paper is nothing about the effects of other media such as salty water and oil.

Maud and Zaman [20] investigate a significant degradation on the fracture toughness of glass fiber-polyester resin under different environmental exposure such as; water, saline water, engine oil, and normal temperature condition. Only on the saline water environment exposure there about 0.3% reduction in fracture toughness from the normal which indicates the possibility to use the composite material under this condition. On the other hand, the paper does not state clearly about the type of fracture mode in detail and it is difficult to conclude the environmental effects on the composite of glass fiber-epoxy resin composite materials of the aimed loading conditions.

Another study takes three different temperature condition (0oC, 25^{o}C, and 50^{o}C) for three months and the result shows, the mode I fracture was highly decreased at 50oC and moderately for the other two. Whereas for mode II fracture it has affected by only the temperature not the duration affects [21]. Having this results what will be if other environmental exposures applied is the research point of this paper.

The other study by Tang [22] and his colleagues takes different immersion duration in seawater (7days, 15 days and 30 days). Not significant changes have seen for 3 days and regarding to the fracture toughness of the epoxy, but for the 7 days it shows 18.5% reduction in the fracture toughness and no further decrease for 30 days immersion. On the other hand, the composite used is Nano-silica/epoxy and the results found are regarding this composite. Having these, the paper wants to study the effects on glass fiber-epoxy resin composites.

Influences of temperature and moisture content on the fracture toughness of carbon/epoxy composite were investigated. The critical strain energy release rate was unaffected by changes in moisture content and increase slightly at elevated temperature. In pure mode II the critical Strain energy release rate drops with moisture content and increase in temperature [23]. On the other hand, it has seen difficulty to conclude the weathering condition effect on the fracture toughness of the composite due to lack of clear standard and justifications.

Different literatures go through determining the effect of environmental conditions and aging only on the polymer matrix. For example, El and his colleagues [24] deals about composites that has been introduced in primary components such as the fuselage. Under service conditions, such components are subjected to varied environmental conditions that can lead to material degradation. Exposure to water vapor simultaneously with cyclic temperature changes is the predominant cause of degradation related to the outdoor environment. Although the phenomenology aging of the laminated composites can appear to be very complex, a mandatory starting point is to understand the evolution of the properties of the constitutive resin. Many studies considered temperatures above 25^{o}C, but few addressed aging under representative cold (40^{o}C) and warm (70^{o}C) conditions. An experimental analysis of the aging of composite specimens is therefore required to identify its effect on the fracture toughness. In this paper, the research point is to know the general effect of different environmental conditions to the normal one.

Fiber reinforced polymer composites are exposed to the moist environment and high temperatures in many applications. According to Sharma and his colleagues, [25] the aging of GFRC specimens results in the property of flexure strength. The durability of fiber reinforced epoxy composites during hygrothermal ageing is dependent on the damage resistance of matrix and integrity of fiber-

matrix interface. The flexural modulus of GFRC immersed in seawater at 25 °C for 120 days increased by 83%, whereas flexural strength increased by 43%. A similar increase in GFRC specimens was also observed after ageing in seawater at 55 °C, with flexural modulus increasing by 83% after an ageing for 60 days only and stabilizing thereafter. The flexural strength had increased by 19% after an ageing of 30 days and showed no significant change after 30 days. The C15GFRC specimen also showed an increase in both flexural modulus and flexural strength after hygrothermal ageing. The increase in flexural properties can be attributed to the additional secondary cross-linking of epoxy and increase in the modulus of elasticity of composite after seawater ageing [25]. It is generally accepted that the absorption of water in polymer composites/nanocomposites results in deterioration of mechanical properties in some literatures, but contrasting results have also been reported.

Sugiman [11] reported that, for the steady condition, in distilled water, the increase of the fracture toughness had been observed since 7 days of immersion, and then the fracture toughness tended to decrease after an immersion time of up to 30 days. In salt water, the fracture toughness increased after 15 days of immersion and then tended to decrease after 30 days of immersion. However, because the data scatter is high, the trend is likely to be the same as that in the distilled water. For the fluctuating condition, in both ageing media, there is a similar trend for the fracture toughness with the immersion time; however, after 30 days of immersion, the fracture toughness of the specimen immersed in distilled water was lower than that in salt water. After 7 days, because the immersed specimens were removed and then left in room temperature for a day before they were tested, the absorbed water had been desorbed. In the crack tip, where the fracture occurred, water desorbed more quickly, and it was possible that the region had dried out; therefore, the fracture toughness was close to that of the dry specimen. It is likely that the desorption, particularly at the crack tip, is faster than that in salt water, as there are no obstacles from salt ions for water molecules to come out during desorption. This may result in a much lower fracture toughness of specimens aged in distilled water than in salt water, such as the result observed after 30 days of immersion.

Liao, Schultheisz and Huston [26] conducted an experimental work on the flexural properties (strength and modulus) were measured to 0^o specimens as received and after aging in fluids. The

fluids included water and two salt solutions, one containing a mass fraction of 5% NaCl and the other containing a mass fraction of 10% NaCl. According to this study, the aging conditions are:

a) De-ionized water for up to 3900h at room temperature,

b) 5 % salt solution for up to 3980h at room temperature,

c) 10 % salt solution for up to 6570h at room temperature,

The paper showed results of decrease in tensile and flexural strength as the aging condition time increases.

The study investigates as the temperature increased; the stiffness of the material was significantly reduced. Similarly, as the moisture content increases the stiffness and strength of the composite material decreases. Due to humid in the composite, there is reduction in toughness compared to that of the dry composite [27]. The point here to be raised is what will happen if saline environment and oil environment conditions used.

Having these different literatures, because of delamination is a major problem in Glass Fiber Reinforced Composites, the focus will be on determining the fracture toughness of glass fiber reinforced epoxy matrix under the loading conditions of mode I and mode II by using standard work specimens of double cantilever beam (DCB) and ENF test pieces. So analyzing and justifying the effect of the different environmental conditions on the fracture behavior of the composite on the specified loading conditions has been the main task of the research as clearly stated on the section of result and discussion.

Chapter Three:

3. Materials, Methods and Conditions

3.1. Materials

The DCB and Flexure Test specimens of composite laminate were fabricated using a hand layup process which is assisted by a vacuum bagging system. For the fabrication of these specimens, the basic materials used are reinforcement fibers of unidirectional (UD) glass fiber and epoxy resin matrix. Depending on its nature, an effective polymer used in hand lay-up method assisted by vacuum bagging manufacturing system is an epoxy resin that grouped under thermoset resins. The mixture of the matrix to its hardener used is a 2:1 ratio.

Specifically materials has been used in this research are mentioned as follows:

Glass Fiber (GF) it is one fundamental type of synthetic fiber mostly has laminate structure with different design of fiber orientations. Among the different types of glass fibers, E-glass fiber that dominantly exist in three principal arrangement types including; unidirectional glass fiber, chopped glass fiber and continuous glass fiber. Unidirectional (UD) Glass fiber of grade E-Class of Surfacing mats composed of continuous glass filaments as shown in figure 3.1 below has been used as reinforcing agent.

The reason behind using E-glass fiber is [2], [10], [28]:

- Its low cost
- High bending strength
- Its high ultimate tensile strength
- Its high flexibility
- Its high stiffness
- High resistance to chemical harm

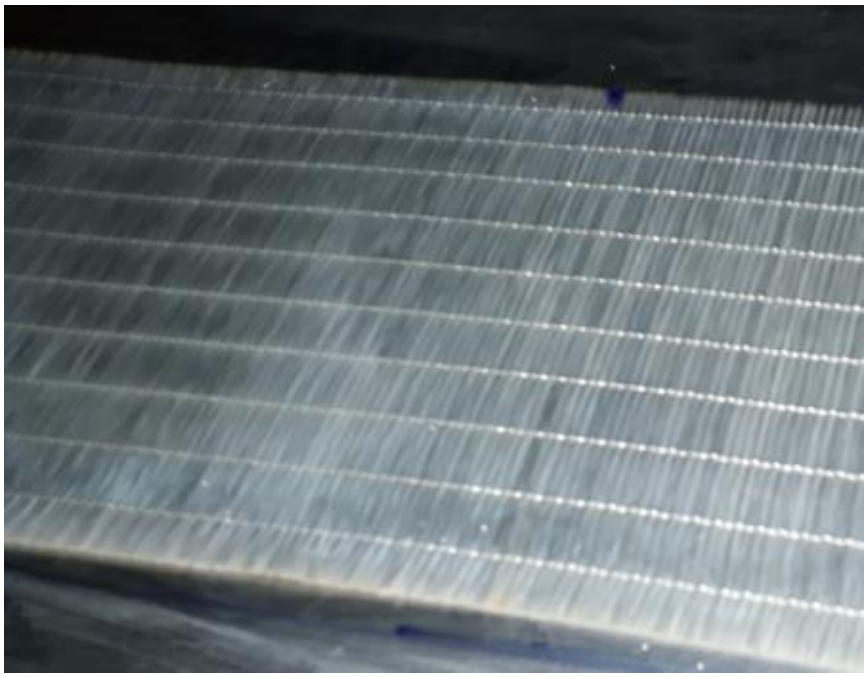


Figure 3-1 Unidirectional E- glass fiber

Epoxy Resin: this is used as a matrix material and is widely used in industrial application because of their high strength and mechanical adhesiveness characteristic. It is also good solvent and have good chemical resistant over a wide range of temperature [2] [10].

The reason behind using Epoxy resin is:

- Its superior mechanical properties
- Excellent adhesion
- Its good possibility of utilizing addition type reaction
- Its low cure shrinkage and low cost
- high fatigue resistance and superior flexural strength

Additives: for the case of treating the specimen fabrication process, wax, jells and hardeners are used.

Thinner: thinner is a chemical which is used for washing and cleaning purposes.

Teflon Insert: PolyTetraFluoroEthylene is a fluorocarbon-based polymer and is commonly abbreviated PTFE with brand name **Teflon®** purposely to be used here as an insert for the fabrication of composite materials to create initial cracks.

Size: the sizes needed for each specimen fabrication are as follows:

Thickness = 0.013mm (13µm), length = 63mm and width = 500mm

Useful Properties of Teflon Insert for the research:

- Non adhesive
- Resistance to water
- Chemical resistance to corrosive reagents
- Non solubility
- Long term weatherability
- Stability at high temperatures
- Low coefficient of friction

Vacuum Bag System: this system is used to avoid voids and porosity problems. The detail is explained below.

3.2. Experimental Methods

To achieve the objectives of the study, there have been followed and adopted different methods from organizing necessary materials and equipment up to experimental testing. The DCB test and ENF test are the most common test methods from the different types for determining the fracture toughness of composite materials. These two methods were chosen for the simplicity to prepare the work specimens and for the ease of the test. The experimental work handled were opening case of mode I and flexure test of end notched flexure case for mode II fracture tests.

To understand the environmental effects on the fracture toughness of reinforced composite materials, performing fracture toughness test by introducing different environmental exposures is essential. Except the normal condition specimens, the remaining were immersed on different environmental media such as engine oil, saline water, benzene and pure water (cyclic).

The test for both DCB and ENF specimens were performed on universal testing machine of model WAW-600D with the requirements of ASTM standard's cross head displacements. The G_I and G_{II} values were obtained from the result of load Vs displacement graph using the load, deflection, crack length, specimen width and length.

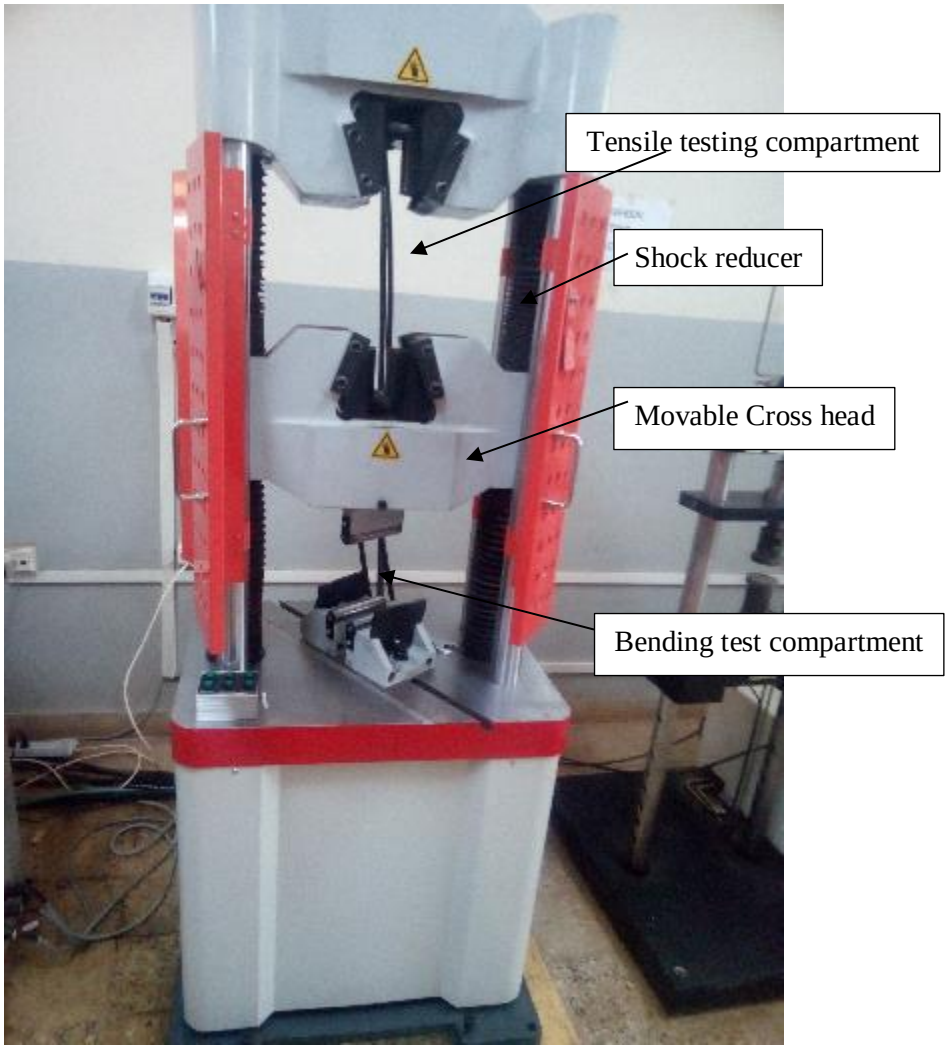


Figure 3-2 WAW-600D UTM

The different methods and conditions followed in this paper are as illustrated under the following subtitles listed below.

3.2.1. Test specimens

All specimens were prepared using hand layup method, which is assisted by vacuum bagging system at Dejen Aviation unmanned air vehicle (UAV) department. The test specimens are brought to an experimental test according to the recommended ASTM standards. The table below gives short summary for test conditions.

Table 1Test specimen experimental condition:

Test type	Rate of applied load	Layup	Initial crack length	Specimens tested
DCB	2 mm/min	[0]8	50 mm	3-5
ENF	0.5 mm/min	[0]8	30 mm	3-5

3.2.1.1. Volume fraction contents

The volume fraction is necessary to know the amount of fiber and matrix contents. It can be evaluated based on initial known values, the density of the fiber and resin and areal weight of glass fiber. As the composite ply consists of two constituents such as fibers and matrix whose quantities in the materials are specified by volume, v, and weight, w, fractions.

$$Fiber\ volume\ fraction = \frac{volume\ of\ fiber}{total\ volume}$$

$$v_f = \frac{V_f}{V_c}, \qquad \text{Eqn. 3.1}$$

$$Matrix\ volume\ fraction = \frac{volume\ of\ matrix}{total\ volume}$$

$$v_m = \frac{V_f}{V_c}, \qquad \text{Eqn. 3.2}$$

$$Fiber\ weight\ fraction = \frac{weight\ of\ fiber}{total\ weight}$$

$$w_f = \frac{W_f}{W_c}, \qquad \text{Eqn. 3.3}$$

$$Matrix\ weight\ fraction = \frac{weight\ of\ matrix}{total\ weight}$$

$$w_m = \frac{W_m}{W_c}, \qquad \text{Eqn. 3.4}$$

Here, 'V' and 'W' are volume and weight, while subscripts 'f', 'm', and 'c' correspond to fibers, matrix, and composite material, respectively.

And because of:

$$\left.\begin{aligned} V_c &= V_f + V_m \,, \\ W_c &= W_f + W_m \end{aligned}\right\} \qquad \text{Eqn. 3.5}$$

We have:

$$v_f + v_m = 1, \text{and } w_f + w_m = 1 \qquad \text{Eqn. 3.6}$$

From the above relations, we can have the following relationships between volume and mass fractions by taking into considerations the relation, $W_f = \rho_f V_f$ and $W_m = \rho_m V_m$.

$$v_f = \frac{\rho_m W_f}{\rho_f W_m + \rho_m W_f} \qquad \text{Eqn. 3.7}$$

Density of composite: the density of the prepared composite can be evaluated from the following relations:

$$\rho_c = \frac{W_c}{V_c} \qquad \text{Eqn. 3.8}$$

Where:

$v_f = volume\ fraction\ of\ fiber(E - glass\ fiber)$

$W_f = weight\ of\ fiber(E - glass\ fiber)$

$W_m = weight\ of\ matrix(Epoxy\ resin)$

$\rho_f = density\ of\ fiber(E - glass\ fiber)$

$W_c = weight\ of\ the\ composite$

$V_c = volume\ of\ the\ composite$

$$\rho_c = density\ of\ the\ composite$$

$$\rho_m = density\ of\ matrix(Epoxy\ resin)$$

Based on the above formulas taken, the results are obtained as given below in the table.

Table 2 Total required values of the reinforcement and the matrix

Primary and Predetermined values		Calculated Results	
Parameters	Values	Parameters	Values
Density of E-glass fiber	2.6 g/cm^3	Total weight of fibers	1,263.6 g
Density of Epoxy resin	1.2 g/cm^3	Total weight of matrix	712.8 g
Total volume of laminate (plate) of thickness 3mm	1,080 cm^3	Ratio of weight of fiber to matrix	1.77:1

After estimating the amount of fractions of the composite needed, the manufacturing technique has been used hand layup assisted by vacuum bagging system.

3.2.1.2. Specimen Preparation

Even though there are plenty of composite manufacturing methods, based on accessibility and the behavior of the matrix, hand layup method which is assisted by vacuum bagging system is found to be preferred for this research.

Hand layup method:

Hand layup is the common method for making composite products. It is easy and requires no technical skill and no machinery. A typical structure of hand lay-up method is shown in Figure 3.3 below.

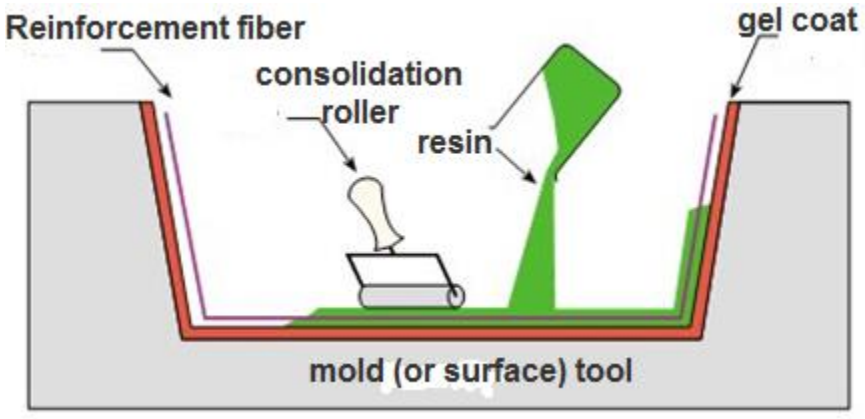


Figure 3-3 Hand layup methods [29]

Hand lay-up technique is the simplest method of composite processing. The processing steps are quite simple. First of all, a release gel is sprayed on the mold surface to avoid the sticking of polymer to the surface. Thin plastic sheets are used at the top and bottom of the mold plate to get good surface finish of the product. The Reinforcement is then applied in between. The polymer is uniformly spread with the help of brush and a roller is moved with an easygoing pressure on the fiber-polymer layer to remove any air trapped as well as the excess polymer present. The process is repeated for each layer of polymer and fiber, until the required layers are stacked.

Tools for the Lay-up Process:

1. Weighing balance - to weigh the chemicals.
2. Mugs and small bowls - for taking the resin mix for lay-up.
3. Brushes - to apply resin for both gel coat application and for lamination.
4. Rollers - to remove the air bubbles and also for applying resin.

A flat composite plate was manufactured with a pre-implanted non-adhesive film insert. And specimens are to be cut from these plates. The film insert is to be implanted at the mid plane of the laminate during layup to create an initiation site for the delamination. The ASTM D7905of mode II loading recommended film insert for epoxy resin polymer matrix cured at or below 177^{o}C is PolyTetraFluoroEthylene (PTFE) with thickness not more than 13μm. The ASTM D5528 as well recommends similar Teflon with insert length of 63mm with thickness of not more than 13 μm and curing temperature of 93 to 121^{o}C [30], [31].

Specimens has been fabricated after collecting all necessary components such as glass fiber, the polymer composite, and other chemicals. The specimen preparation uses unidirectional glass fiber and epoxy resin with complementary materials. The epoxy resin used was mixed with its hardener with the ratio of 2:1 by weight ratio as recommended by the manufacturer. The procedures followed are as described below.

The first task in the manufacturing process is preparing the surface or molds. Then apply the wax with a dry cloth/towel on the surface of the mold and wait for 30 min for the wax to dry. Next, apply vacuum tape around the mold edges without leaving any gap between vacuum tape and mold surface and put on scotch tape to protect the vacuum tape from epoxy until all the fabrics, peel ply,

perforated nylon and breather fabrics are applied. Having this the next task was preparing all the fabrics, peel ply, perforated nylon and breather fabric with a dimension of 90cm X 180cm and then prepare the vacuum hose, connectors, vacuum pump, spiral hose and resin trap with gauge. After this, the main tasks were laying the fabric on to the thin layer of epoxy and wet out the applied fabric with the brush gently. By repeating this until all the fabrics and sandwich are wetted gently, the composite plate were produced. Here, the pre-crack is created in the laminate by placing a non-porous Teflon® sheet in the mid of plies. Once the hand layup process completed, the next task was applying a vacuum bagging method. That is, lay the peel ply, perforated nylon, and breather fabric respectively over the surface of wetted fabric and apply vacuum by connecting the vacuum pump to UPS (Power backup). Then, leave the vacuum pump on as per the epoxy curing time (8hrs) required. After this leaving the part cure for three complete day (72 hrs.). Finally, the part produced were cut into required size as per requirement of ASTM standards.

Vacuum Bagging System

Vacuum Bagging Process is a closed-mold process that is capable of manufacturing high performance and large scale glass fiber reinforced polymer parts with low tooling cost. It utilizes the pressure to achieve the unique requirements. Some basic advantages of vacuum bagging system are [32]:

- It eliminates random bonding, avoids unwanted porosity, and can make free void content on the product.
- It provides uniform pressure and eliminates putty pooling or putties starved areas because of unequal clamping pressure.
- It is with easier manufacturing process.
- Vacuum bagging is easier on tools in that it produces very little stress on the tool itself.

Vacuum bagging requires some basic materials in order to successfully pull vacuum and apply even pressure. These are mentioned as follows:

The bleeder is placed to absorb the excess resin from the lay-up, thus controlling the amount content during the curing process. Pressure plates are introduced to supply additional compression to the lay-up. Barrier film is used to control the resin flow in the bleeder.

Peel Ply (Release fabric) is a smooth woven fabric that will not bond to epoxy. It is used to separate the breather and the laminate.

Breather film is given beneath the vacuum bag to allow the uniform application of vacuum all over the area of the laminate and removal of excess air or volatiles developed during the cure.
Mastic sealant is used to provide a continuous airtight seal between the bag and the mold around the perimeter of the mold.

Mold Release is preventing the epoxy from sticking to the mold when laminating a part.

Vacuum bag is used to contain the vacuum generated by the pump and applied to the lay-up. The vacuum maintained till the resin gels [32].

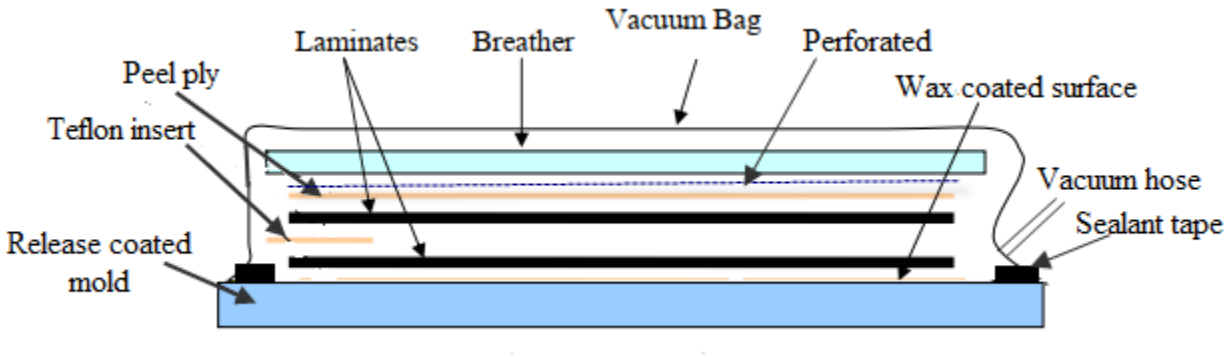
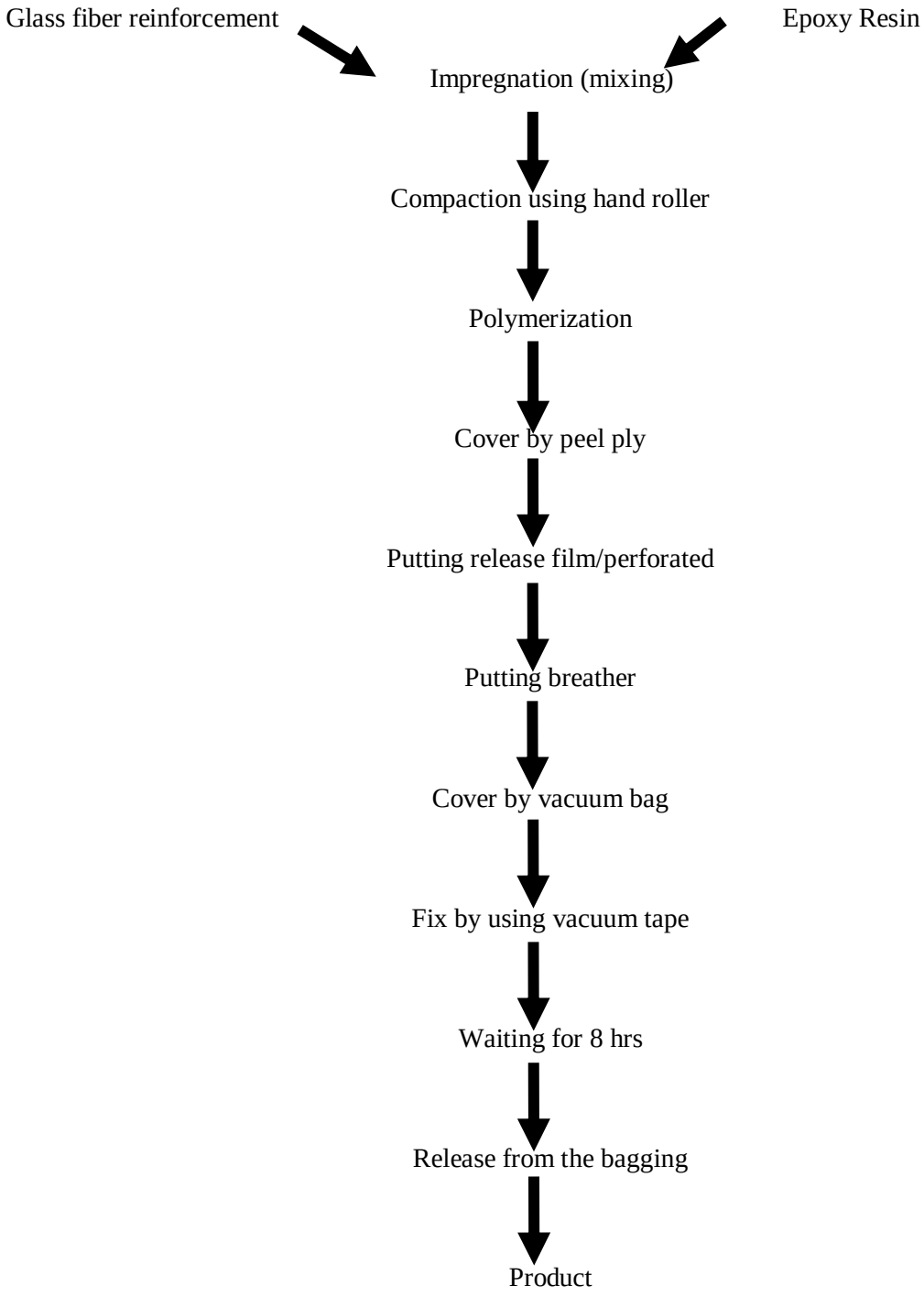


Figure 3-4 Vacuum bagging system

Chart 3: The composite manufacturing steps



The stacking sequence:

The stacking sequence for mode I and mode II fracture toughness test specimen preparation containing an initial delamination is **0º unidirectional fiber orientation layup**. The numbers of layers are required to be 8 for each test parameters.

- A unidirectional E-glass fiber with density 2.6g/cm3 mixed with epoxy resin is used for this work. The orientations or an arrangement of the fiber follows 0° considering high delamination of composites happens to this arrangement.

Unidirectional E-glass fiber

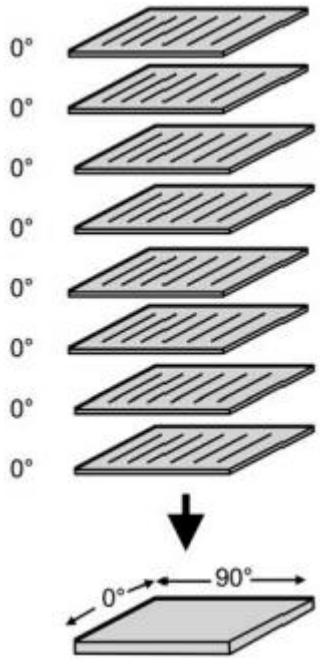


Figure 3-5 Unidirectional orientation lay-up (Lamina) [33]

The samples were produced by hand layup method that is assisted by vacuum bagging system. The specimens were made of 8-ply unidirectional E-glass fiber reinforced epoxy composite. The test samples with a length of 125±0.5 mm and a width 25 ±0.5 mm for mode I delamination were cut

from 200x900 plates. To create a pre- crack in the DCB specimen, Teflon film with a length of 60 mm was placed in the composite specimens during hand lay-up process.

Similarly, specimens with length of 160 ±0.5 mm and a width 25 ±0.5 mm for mode II delamination were cut from 200x900 plate.

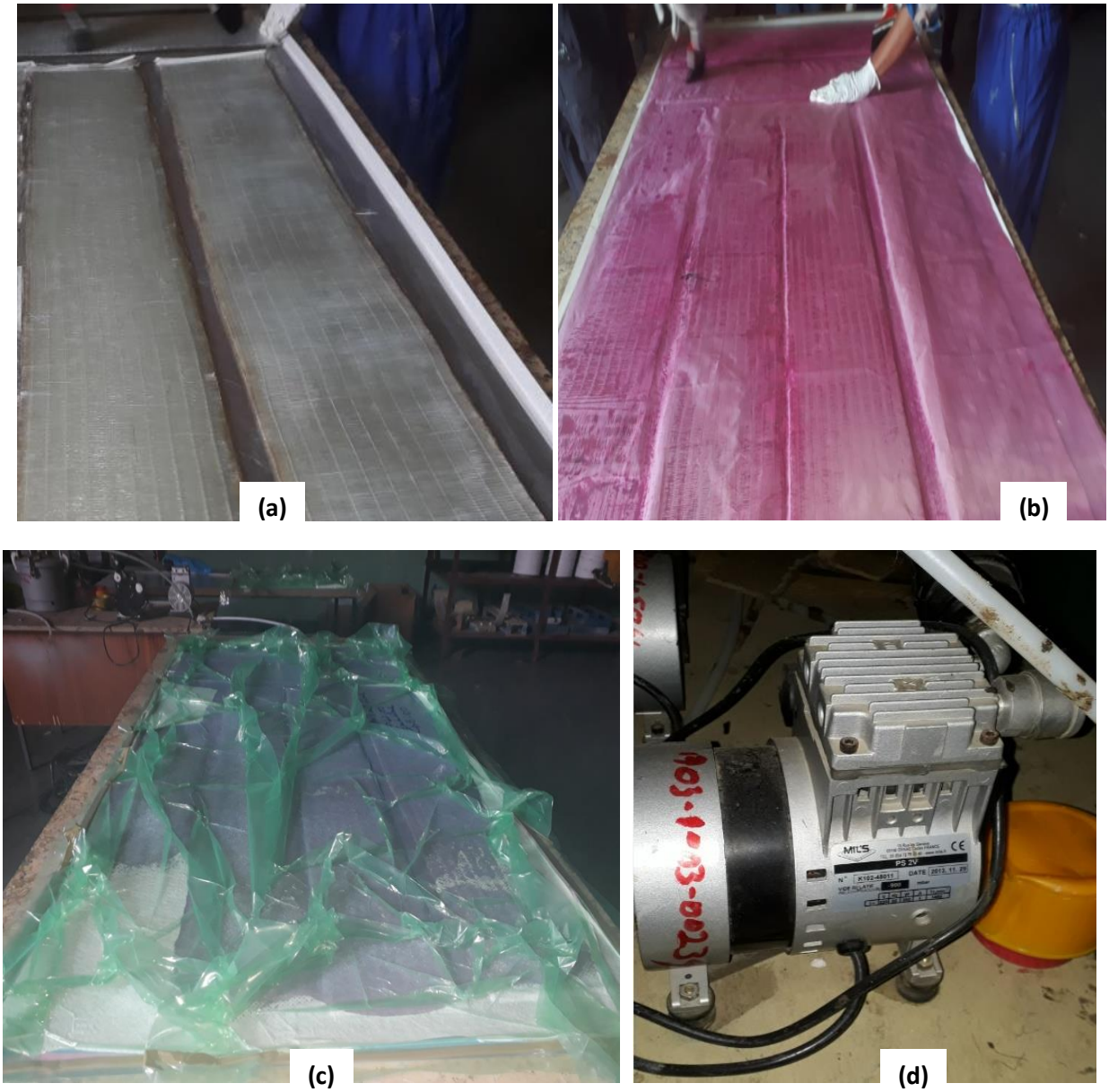


Figure 3-6 Specimen Preparation by hand lay-up using vacuum bagging

(a) Impregnation stage (b) Introducing peel ply (c) Vacuum bagging system (d) Vacuum pump

3.2.2. Experimental conditions:

3.2.2.1. Environmental conditioning

In addition to the normal condition, the experimental work comprises different variables of environmental conditions in which the specimens were inserted to;

I. Pure water (cyclic) exposure
II. Saline water
III. Engine oil
IV. Benzene

Except the cyclic case the other three conditions are taken to be exposed for three different time durations of 7days, 15 days and 30 days. For the cyclic case, from the 24 hours per day, half (12hrs) on immersion and the other half on normal condition at room temperature are applied for 15 days and 30 days on pure water.

As depicted in figure 3.7 below after the work specimens cut into size, the different environmental exposures like Engine oil, benzene, saline water and pure water (cyclic) are used for the effects on fracture toughness to be analyzed in this paper.

Figure 3-7 (a) Sized specimens before exposure, (b) Engine oil, (c) benzene

For studying the long-term effect of water, benzene, engine oil and marine environments on composite materials, the prepared specimens were immersed in the aforementioned environment conditions. The specimens were soaked in small plastic containers at room temperature that contains the aimed different environmental conditions. The containers were kept closed to maintain the conditioning from other environmental effects. The saline water condition was obtained by dissolving 5 % sodium chloride (NaCl) in distilled water by using proportionate weight ratio. After the specified period of immersion specimens weight were measured to obtain the weight gain ratio based on initially known weights. The amount of moisture absorption is expressed as the percentage change in weight of the specimen after immersion (see equation 3.9). The different environmental conditioning as shown on figure 3.8 below were used for visualizing the effects on the fracture toughness of the composite.

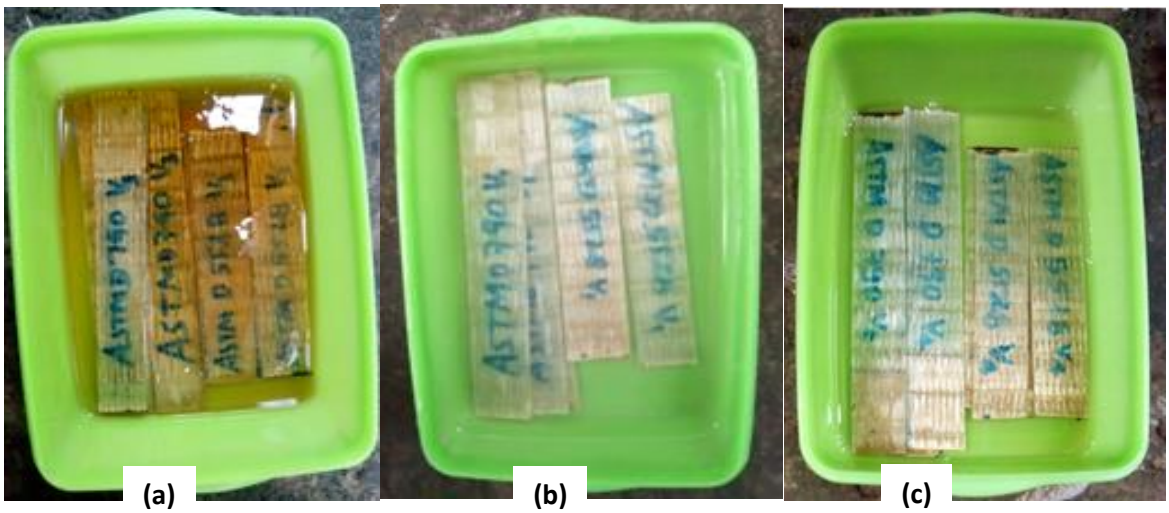


Figure 3-8 Conditioning (a) In engine oil, (b) In saline water, (c) In benzene

Composite materials absorb moisture and expanded gradually with time. These will in turn reduce the failure time. Since the composites contain epoxy matrix and fibrous material, most of moisture is absorbed by epoxy as depicted by figure 3.9 below. Therefore, the volumetric expansion takes place along the matrix content that localizes stress and strains inside it. Thus, the most moisture absorbed by the epoxy will expanded gradually with time. This moisture absorption tends to change the mechanical properties of the composite. Based on the behavior of the environmental

conditioning, the interlaminar strength of the composite may increase or decrease. This effect are presented in detail on the result and discussion section.

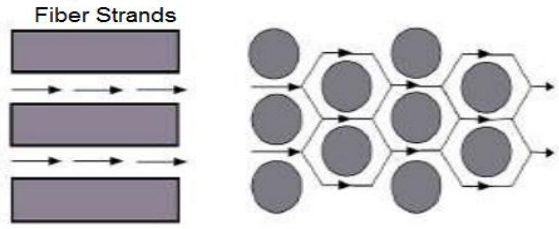


Figure 3-9 Diffusion path of moisture into composite laminate in the thickness direction [4]

The amount of moisture content over the span of the reference time period in a material is taken as the ratio of the mass of the moisture in the material to the mass of the normal material and expressed as a percentage, as follows: [34], [4]

$$M = \frac{W_i - W_o}{W_o} \times 100 \qquad \text{Eqn. 3.9}$$

Where;

$$M = average\ moisture\ content(\%)$$
$$W_i = current\ specimen\ mass(g)$$
$$W_o = normal\ condition\ specimen\ mass(g)$$

3.2.2.2. Test specimen configuration

To understand the nature of fracture toughness of glass/epoxy composite under the parameters taken in this research experiments are to perform fracture toughness tests on the laminated composite. There are different techniques by which one can measure the fracture toughness of a material. The most common tests are the drum peel test, double cantilever beam test (DCB), three point bending test and four point bending tests. DCB and Three Point Bending (Flexure Test) specimens are selected for this research. ASTM D-5528 describes the standards for performing the DCB test on unidirectional composite laminates. Similarly, ASTM D7905 states about the determination of mode II interlaminar fracture toughness tests.

The fracture toughness of glass fiber polymer composite matrix under loading conditions of mode I and mode II are configured based on standard test procedures. Based on these requirements the

fiber weight and volume of epoxy are to be determined using volume fraction formulas by taking the densities and mass of each constituent as an input.

The test method consists of a rectangular, uniform thickness, unidirectional laminated composite specimen containing a non-adhesive insert (Teflon) at the mid plane that serves as a delamination initiator. And the forces are applied to the specimen through the DCB for the Mode I and an end notch flexure (ENF) fixture for Mode II under displacement controlled loading.

The experimental detail describes the determination of mode I and mode II interlaminar fracture toughness of G_{IC} and G_{IIC} of unidirectional fiber reinforced epoxy resin matrix composite laminates using DCB and three point bending test methods respectively.

Specimen test and Test configuration: The test laminates are required to be an even number of plies and unidirectional glass fiber with delamination occurring in the 0^o (zero degree) direction. And the specimen dimensions are required to confirm the ASTM standard which is presented as below.

Dimensions:

The dimensions for the composite material to be prepared based on the known ASTM standard are as depicted on the figure 3.10 and figure 3.11 below.

DCB for Mode I with loading blocks: According to ASTM D5528; Specimens shall be at least 125 mm long and nominally, from 20 to 25 mm and in this paper a 25 mm width is taken. Panels shall be manufactured, and specimens cut from the panels, such that the insert length is approximately 63 mm. This distance corresponds to an initial delamination length of approximately 50 mm plus the extra length required to bonding the hinges or loading blocks. As the ASTM D5528 recommends, the laminate thickness shall normally be between 3 and 5 mm and the prepared specimen thickness reads 3mm.

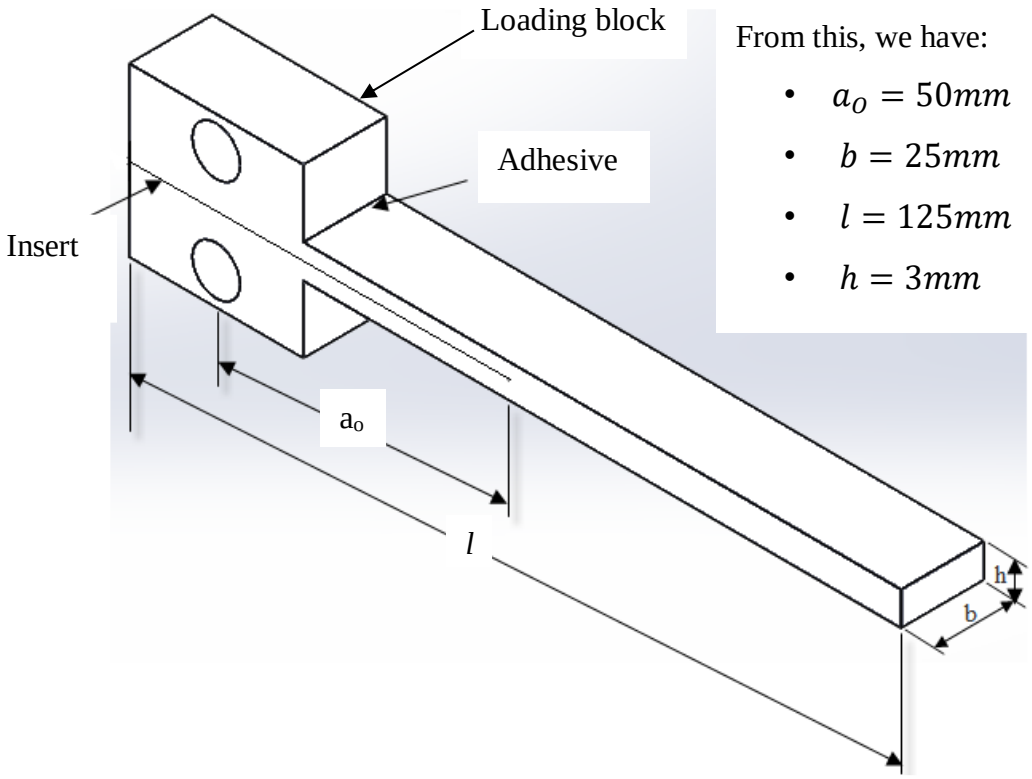


Figure 3-10 Mode I DCB with loading blocks

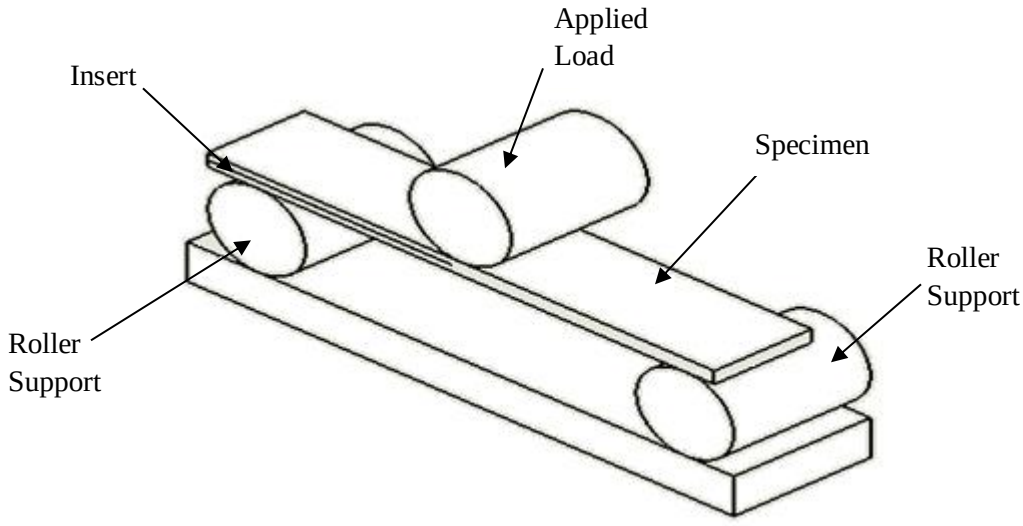


Figure 3-11 End notch flexure (ENF) test loading condition

❖ Specimen Dimension for ENF test (Mode II):

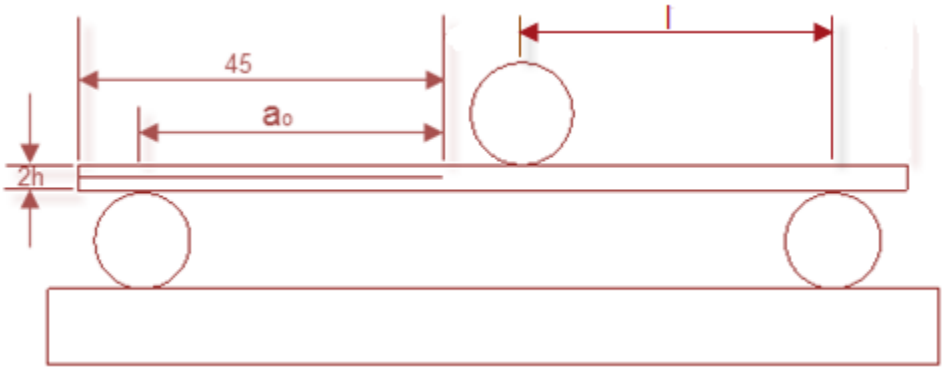


Figure 3-12 ENF test configuration and specimen dimensions

Where;

$$a_o = 30mm$$

$$l = 65mm$$
$$b = 25mm$$

$$2h = 3mm$$

Sampling: As per ASTM standards, the number of five specimens is required per test condition unless valid results can be gained with fewer specimens. Having this, eighty total specimens is needed for the total test variables such as the pure water environment, the saline water environment and the engine oil environment.

The number of test specimens prepared for the experiment is based on each experiment variables. For each experiment variables, at least three to five specimens are needed. The dimensions of the prepared test specimens are based on the standards allowed regarding of this study.

- The prepared specimens have been cut into desired sizes by using three mm grinding cutter.
- Then it has to been exposed into the different environmental conditions.
- Then after based on the ASTM standards five specimens for each test has taken into the UTM testing machine.

Total specimen required:

Based on test variables by considered, the total required specimens are listed below. Five specimens are allowed for each parameter of tests.

- For saline water;

Time durations to be considered are; 168hrs, 360hrs, and 720hrs.

5×3=15 specimen

- For pure water;
- Cyclic

Taking 15 and 30 days for the cyclic condition, which is estimated to 360 and 720 hours respectively. Then specimens were provided to immersed on the water for half of these days to observe just the effects of this cyclic condition on the interlaminar fracture toughens for the two mode of delamination.

5×2 =10 specimens are required.

- For engine oil;

Taking five specimens into engine oil environment for 168hrs, 360hrs, and 720hrs is used to justify the effects of engine oil on composite material structures.

5×3 =15 specimens are required.

- Total

$15 + 10 + 15 = 40$ Specimens for mode I loading type.

Similarly, for mode II fracture toughness test numbers of 40 specimens are required. Therefore, the total specimens required are **80** in number.

Table 3 Summary of standard dimensions, number of layers and curing temperature.

Loading Type	No of layers	Stacking Oreintation	Thickness, 2h (mm)	Length after insert, L (mm)	Width, B (mm)	Insert Length (mm)	Curing Temperature (hr.)
Mode I	8	0^o	3	62	25	63	72
Mode II	8	0^o	3	65	25	45	72

3.3. Apparatus, Experimental Setup and Testing

The design of the experiment is the main thing in which useful for identifying the input parameters that have a significant effect on the response of the system. It is a technique that can be used in finding the optional solution, selecting significant parameters and construction of theoretical models. The design of experiment process involves three steps: [35]

- Designing the experiment,
- Conducting experiments, and
- Analyzing the experimental data

In the first step while designing an experiment the major input parameters selected for the study are normal environmental condition, saline water condition, Engine Oil environment and benzene environment each with several range of time duration.

3.3.1. Mode I Fracture Testing

For the mode I loading condition, specimens should be connected with loading bocks such that the jaws of the UTM can handle it and makes it firm during the operation.

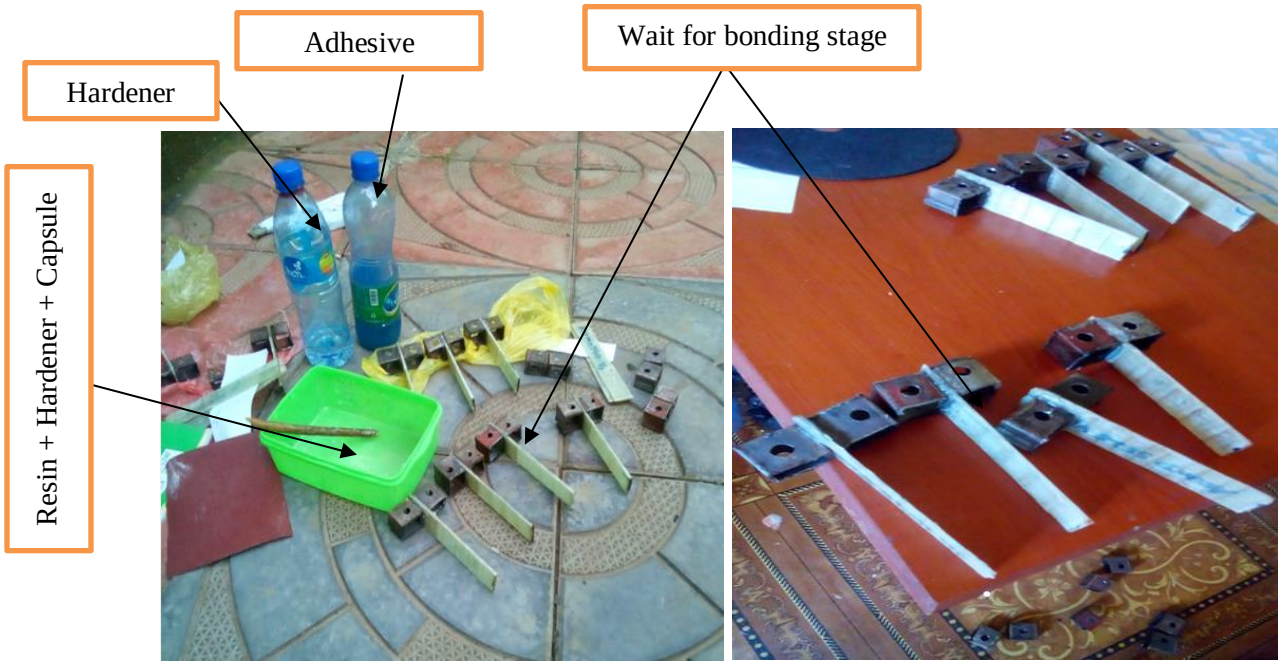


Figure 3-13 Bonding the holding blocks

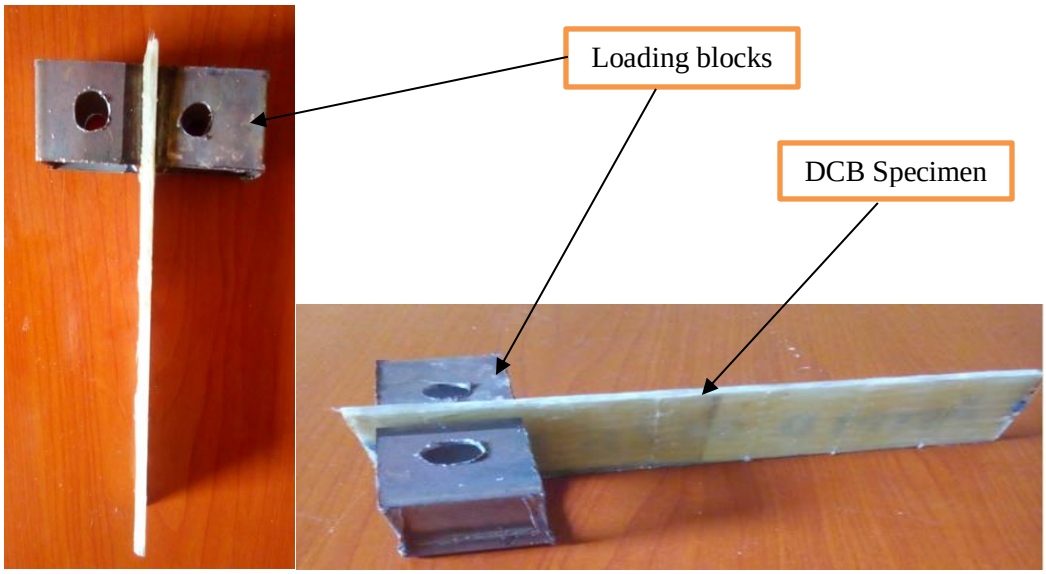


Figure 3-14 Prepared samples of DCB test specimen

The bonding elements used are polyester resin with its hardener and capsule as a strengthening guidance. The time required for full adhesion were about one and half day.

These DCB test specimens were used to measure the mode I fracture toughness G_{Ic} of composite of E-glass reinforced epoxy resin polymer matrix. As shown in the figure 3.14. DCB specimens are with a rectangular uniform thickness contains a non-adhesive Teflon film inserted in the mid-plane of the laminate during fabrication that allows as a delamination initiator. The calculations of the fracture toughness G_{Ic} is based on modified beam theory with experimental compliance calibration as recommended by ASTM D5528 [31].

The resistance to fracture or strain energy release rate based on modified beam theory (MBT) for mode I fracture is expressed as:

$$G_I = \frac{3P\delta}{2b(a+|\Delta|)} \qquad \text{Eqn. 3.10}$$

Where,

$$P = Load\ (N)$$

$$\delta = Load\ point\ displacement\ (mm)$$

$$b = specimen\ width\ (mm)$$

$a = Delamination\ length\ (mm)$, and

$$\Delta = C^{1/3},\ C = \frac{\delta}{P},\ P = P_{cr}$$

$$C = Compliance\ calibration$$

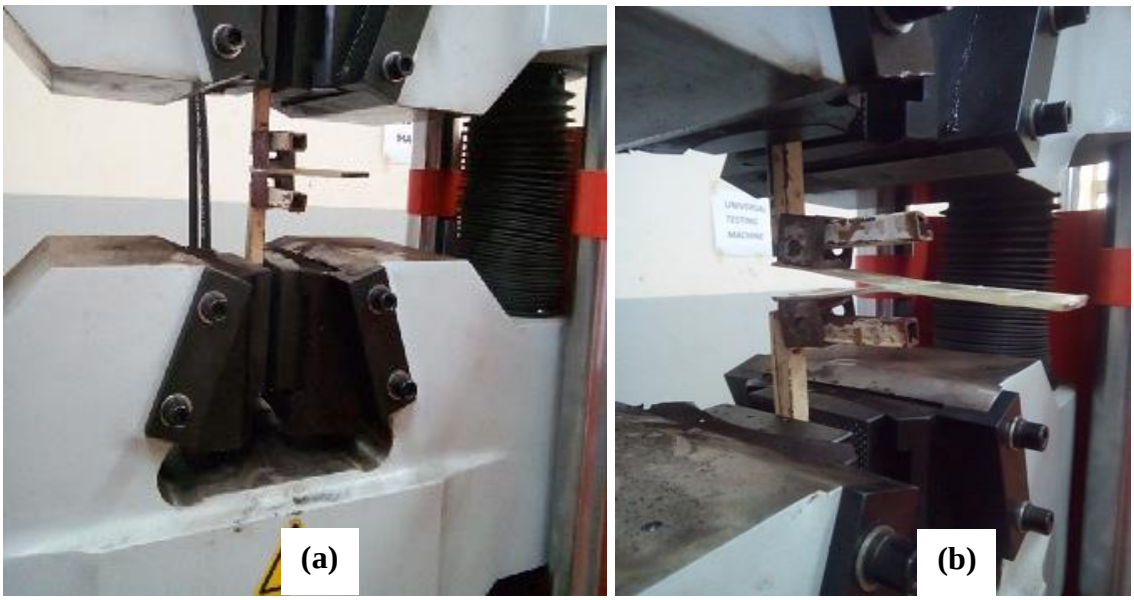


Figure 3-15 (a) DCB test setup on WAW 600D UTM, (b) Delamination proceeds

3.3.2. Mode II Fracture Testing

An End-notched flexure (ENF) test commonly known as - Mode II is one of the methods intended to measure the interlaminar delamination [8] [30]. This method able to determine the delamination resulted due to a combination of compressive and bending stresses caused by the initially cracked plies as they buckle out of plane.

Figure 3-16 ENF Test setup on WAW 600D UTM, (a) before operation takes place (b) in loading

Based on the experimental data, the mode II fracture resistance can be evaluated by using the following equation:

$$G_{II} = \frac{9Pa^2\delta}{2b(2l^3+3a^3)} \qquad \text{Eqn. 3.11}$$

Where;

$$P = Load\ (kN)$$

$$\delta = Load\ point\ displacement\ (mm)$$

$$b = specimen\ width\ (mm)$$

$$a = Delamination\ length\ (mm),$$

$$l = the\ effective\ length\ of\ the\ specimen$$

Chapter Four:

4. Results and Discussion

The results of the investigation on the study of influence of environmental conditioning on the fracture behavior of DCB and ENF specimens is presented in this chapter. As well, the weight gain ratio of exposed specimens and their behavior during the immersion period is obtained. The experimental work was handled on Model WAW-600D universal testing machine at Bahirdar University Institute of Technology, Ethiopia. The experimental test results obtained from all DCB and ENF specimens under several environmental exposures is summarized below in table 4. As it is supported by different literatures [7][36] one point to be mentioned here is, the higher maximum load value obtained during the experiment indicates that the relative greater resistance of crack opening achieved in tested specimens.

4.1. Moisture Absorption from different environmental exposures:

The weight gain of exposed specimens over each specified times is obtained in figure 4.1 below. During conditioning, specimens were immersed constantly for the prolonged time period in saline water, engine oil and in benzene environment. While in case of pure water conditioning, periodically removed from the media.

As shown, the cyclic case is fully in Fickian process which defined by the linear relationship of diffusion with time. While the others can be categorized into three stages.

Stage I: the diffusion process is Fickian.

Stage II: the moisture absorption relationship deviates from linearity with the conditioning time.

Stage III: the diffusion process is non-Fickian

Most likely, all are non-Fickian after about 15-$hr^{0.5}$ immersion time. This means there is a development of cracks, which leads the diffusion process to be increase. The moisture content is increased rapidly in the first stage following the Fickian diffusion behavior. The moisture absorption performance is determined by a combined effect of the concentration gradient

controlled diffusion and polymer relaxation. The first stage of absorption is dominated by the Fickian diffusion, as it is a more rapid process than the polymer relaxation. The non-Fickian diffusion behavior shows the plasticization of the matrix and the possible fiber-matrix debonding [16].

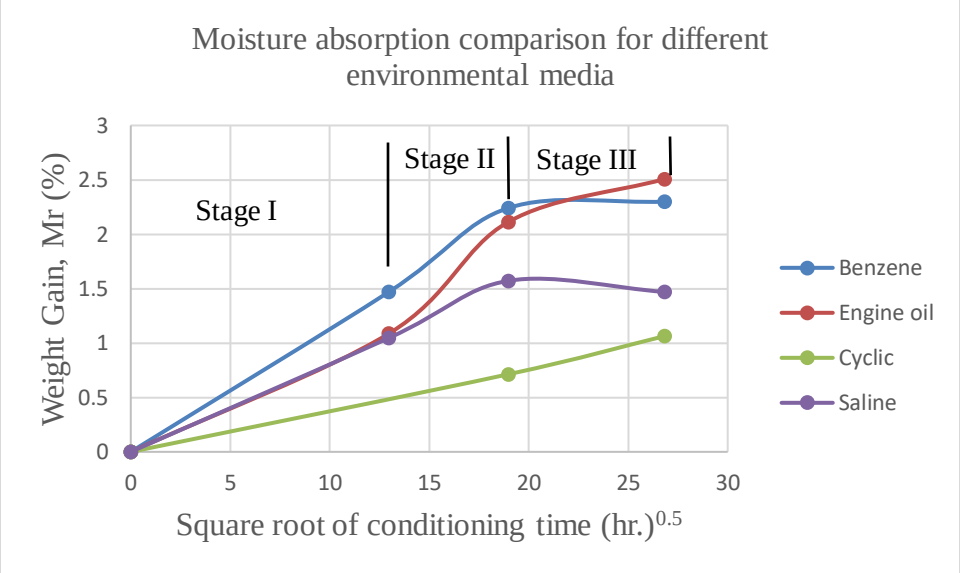


Figure 4-1 Weight gain of E-glass/epoxy composite specimens as a function of square root of time (hr.) $^{1/2}$

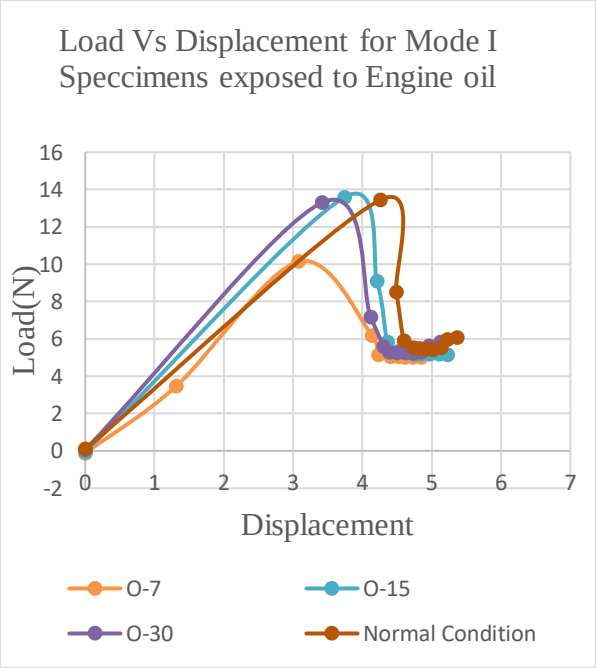
4.2. Mode I Test:

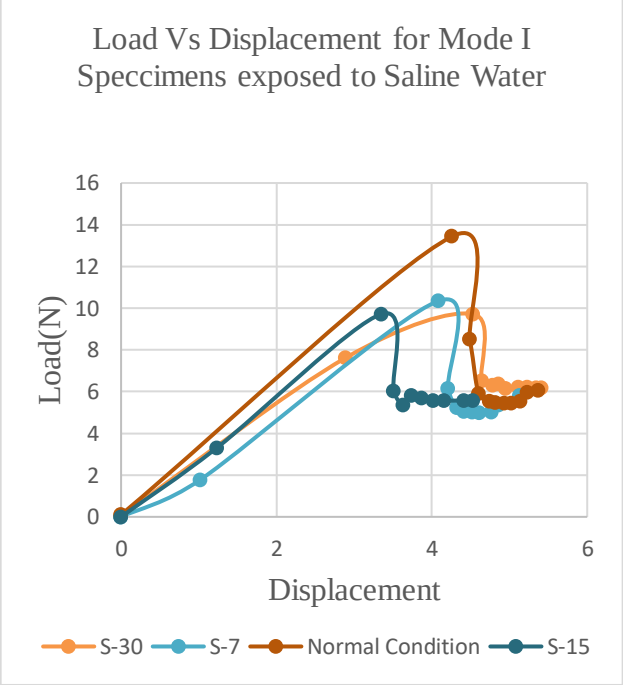
As depicted in figure 4.2, the load Vs displacement for the different environmental conditions and normal condition were performed using a cross head displacement of 2 mm/min. Based on the experimental data, the mode I fracture resistance were evaluated by using equation 3.10 and results are obtained accordingly.

The experimental result showed that the interlaminar fracture toughness G_{Ic}, is maximum for the normal condition which is $50.8. J/m^2$. Moderately the other visible fracture toughness next to the normal DCB specimens is seen on the 15th day Engine Oil environment i.e. $45.58\, J/m^2$. While the minimum fracture resistance G_{Ic} value is observed on the 7th day benzene environment DCB specimen which is $28.52\, J/m^2$. Through this comparison, the delamination resistance in 15th day engine-oil immersion duration it reduced by 10.27% from the normal condition specimens. When we see the specimens immersed in pure water the fracture resistance comparably lowest for all 7

days and 15 days aging periods. In general, the accomplished experimental work showed that, the resistance to fracture of environmentally exposed specimens are lower compared to the normal specimens. This can be interpreted as the distribution of moistures absorbed on the exposed specimens facilitates the dislocation of the mating matrix and tends an increase of laminate opening. The details of all fracture resistance values for the different environmental conditioned specimens is illustrated below in table 4 and figure 4.4.

The load Vs Displacement graphs of a DCB specimens for different exposure media and duration:





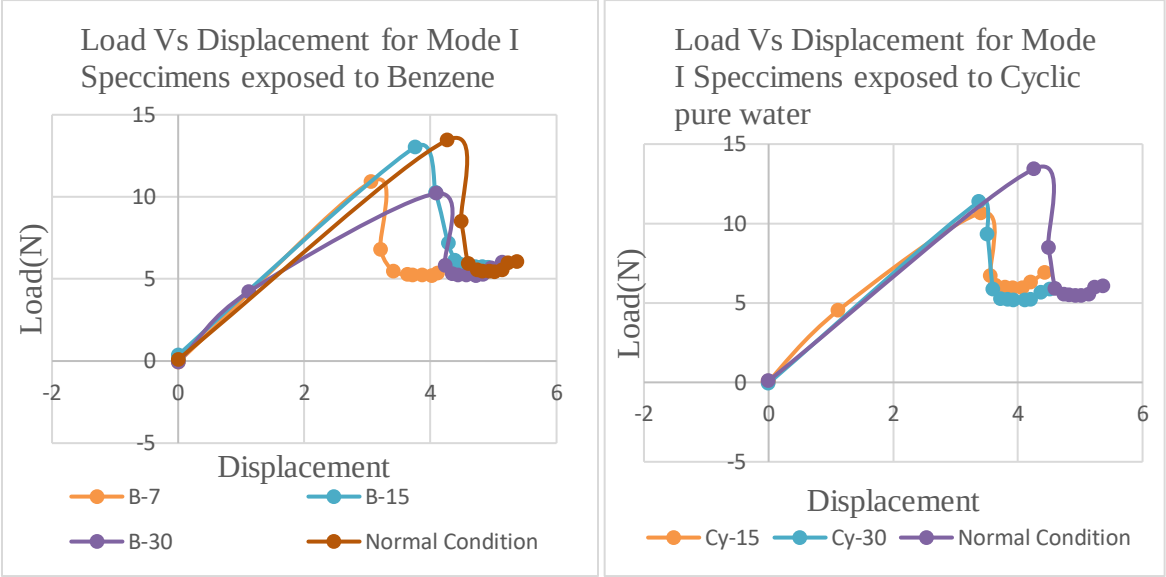


Figure 4-2 Load Vs Displacement graph of tested DCB specimens

4.3. Mode II Test

The mode II interlaminar fracture toughness G_{II} is held to represent the strain energy release rate that is the resistance to crack propagation from the insertion film. In this test, the load is led by flexural force on the specimen supported at the two ends to produce a crack from the insert. Then, the crack has been extended due to the shear effects of these forces at the crack tip. As recommended by ASTM standards, the mode II test uses the maximum load and crack length $a_o = 30mm$ which measures the crack initiation value. Having all necessary data, the mode II fracture resistance were obtained by using equation 3.11 and the results are obtained accordingly.

As can be seen from figure 4.5, the maximum fracture resistance, $G_{II} = 2.496\, J/m^2$ is obtained in an ENF tested specimen exposed to 30 day of benzene environment condition. Similarly, the other moderate strain energy value $G_{II} = 2.297\, J/m^2$ is observed on the 15th day cyclic pure water environment condition. While in the normal condition specimens, the fracture resistance obtained is $G_{II} = 1.58\, J/m^2$. When we see the saline water environment aging condition, specimens immersed for 30 days found in better resistance to delamination, which is $G_{II} = 2.031\, J/m^2$. In the same way, specimens immersed in an engine-oil of 30 day aging showed $G_{II} = 1.687\, J/m^2$ which increased by 6.3% than the normal condition specimens. This shows, in the mode II loading condition, the exposed specimens are in good performance of resistance to delamination compared

to the normal. The reason behind this is absorbed particles help to increase the flexural strength of the immersed specimens. The detail of the ENF experimental test results for the five different environmental conditions are illustrated in table 4 and comparisons are presented in figure 4.5.

The load Vs Displacement graphs of ENF specimens for different exposure media and duration:

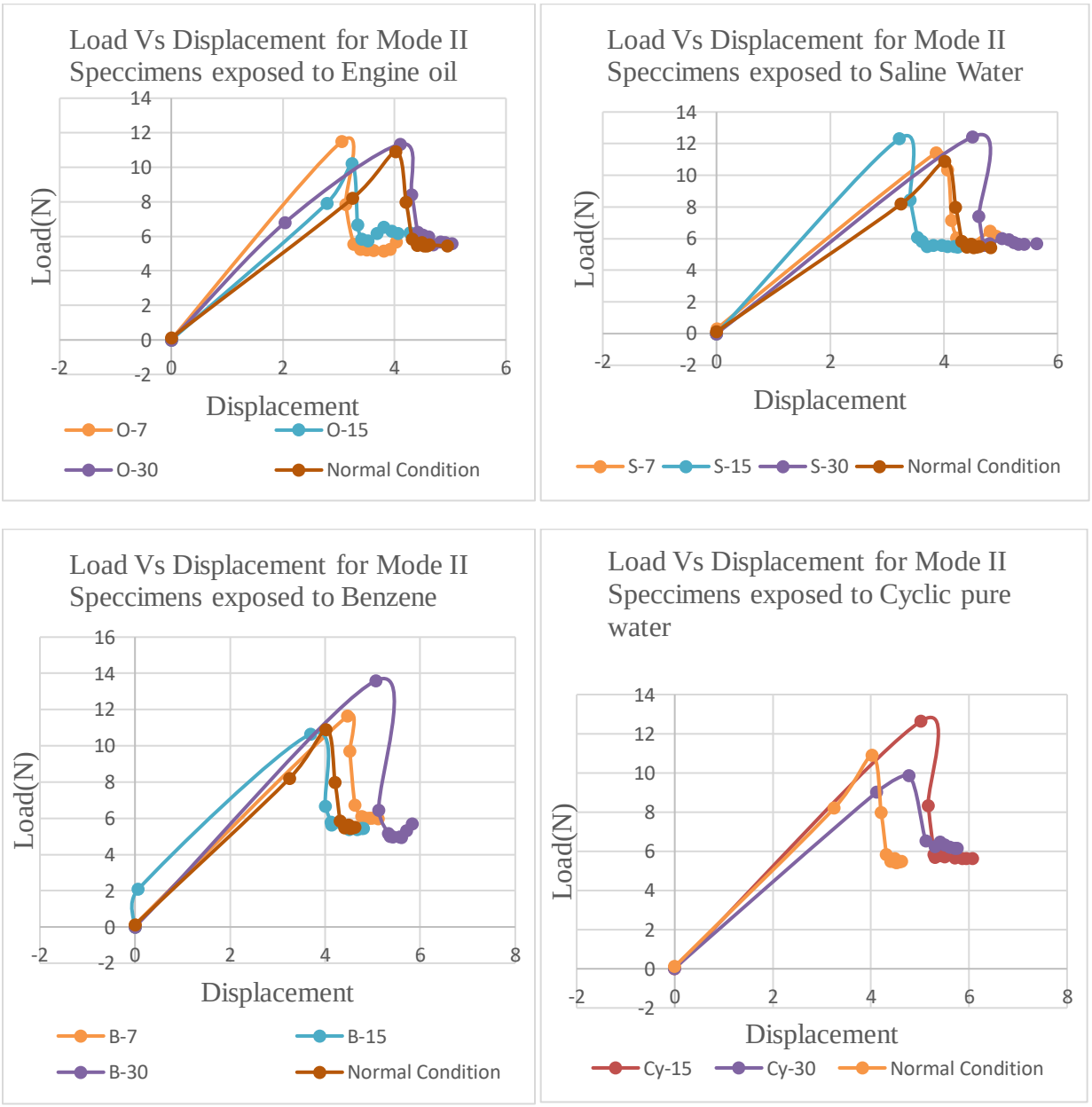


Figure 4-3 Load Vs Displacement graph of tested ENF specimens

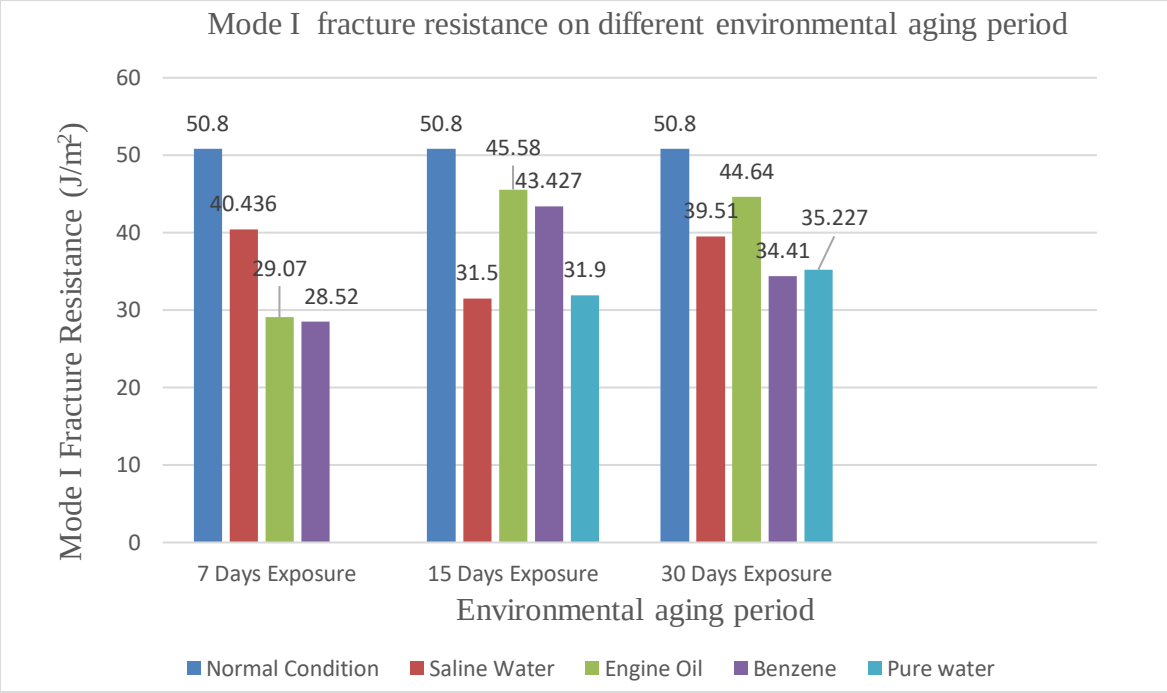


Figure 4-4 Comparison of mode I fracture toughness results

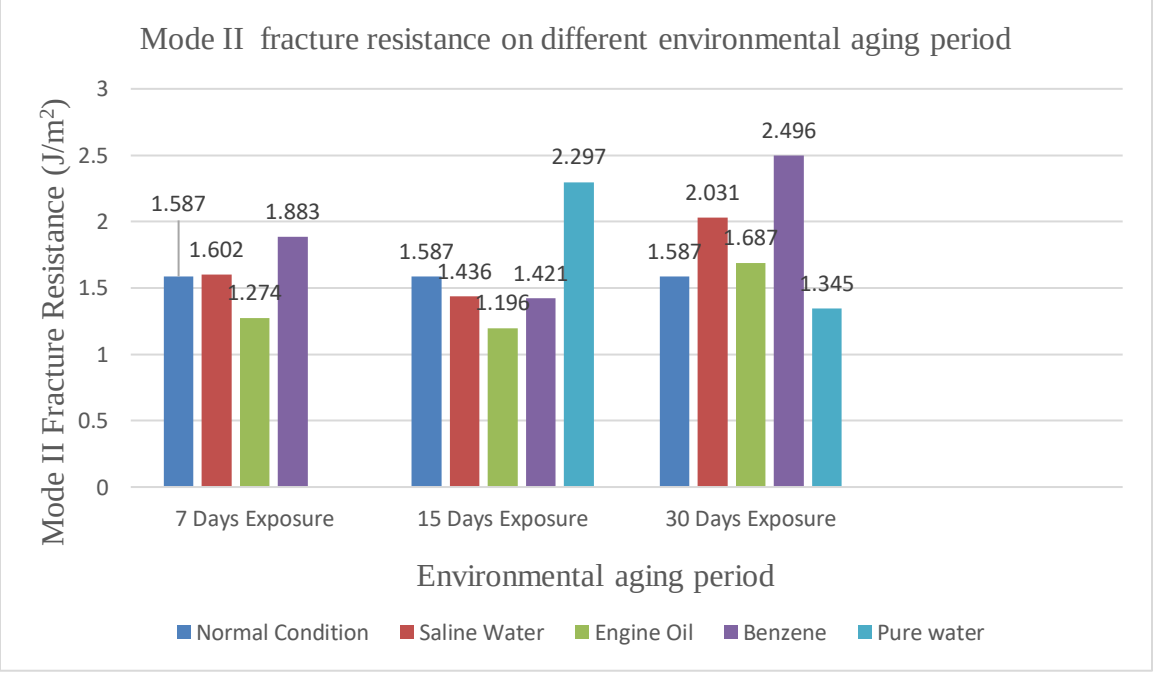


Figure 4-5 Comparison of mode I fracture toughness results

Table 4 Summarized results obtained from the experimental work

Environmental conditioning	Exposed Duration (days)	Test temperature	Mode I interlaminar fracture resistance (J/m^2)	Mode II interlaminar fracture resistance (J/m^2)
Normal condition	_	Room temperature	50.8	1.587
Saline	7	Room temperature	40.436	1.602
	15	Room temperature	31.5	1.436
	30	Room temperature	39.51	2.031
Benzene	7	Room temperature	28.52	1.883
	15	Room temperature	43.427	1.421
	30	Room temperature	34.41	2.496
Engine Oil	7	Room temperature	29.07	1.274
	15	Room temperature	45.58	1.196
	30	Room temperature	44.64	1.687
Pure water(cyclic)	15	Room temperature	31.9	2.297
	30	Room temperature	35.227	1.345

4.4. Failure Mode

From the tested specimens, it was seen some unique failure kinds especially on mode I loading conditions. Except some specimens that delaminates totally from the initially created crack on the mid plane, others were failed by different pattern like as shown on figure 4.6. From these the one observed is branching, which goes out of the initially cracked plane. This was observed mainly on specimens immersed in saline water. The reason behind these failure modes differences is not

identified but it seems due to the cause of the absorbed moisture sensitivity. While in mode II tests, the specimens were commonly fail at the mid bottom surface and the breakage is transferred throughout around the load application point. During the bending force application at the center of the specimen, the failure starts by damaging the bottom layers of the laminate tensional and compressively upper layers of the laminate. The flexural test specimens' mode of failure is presented in figure 4.7.

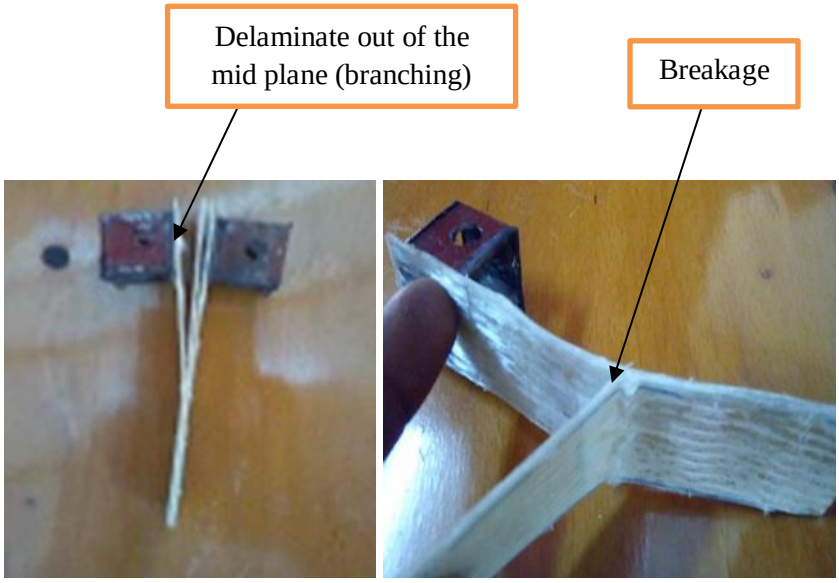


Figure 4-6 Typical failure modes in DCB tested specimens

Figure 4-7 Failure mode of ENF specimens

Chapter Five:

5. Conclusions, Recommendations and Future Development

5.1. Conclusions

The accomplished experimental work describes the influence of environmental aging on the fracture toughness of DCB and ENF specimens made from glass fiber reinforced epoxy resin composite under mode I and mode II loading conditions respectively. The procedures followed ASTM standards and results were obtained accordingly.

Studying the delamination resistance of glass-fiber polymer matrix composite on different environmental exposure by comparing it with the normal condition specimens is useful for designing and setting preconditions for high technology applications. As different environmental conditions on the application of composite materials have great effect on the mechanical properties, it is found that studying these effects can help designers to made preconditions for prevention. The study presents the effect of the environmental exposures on the fracture toughness based on ASTM standards by introducing different environmental exposures such as pure water, benzene, saline water and engine oil.

In general, the accomplished experimental work presented here showed quantitatively the effect of environmental variables on fracture toughness G of E-glass fiber reinforced epoxy resin composite material. As observed from the experimental work amongst the performed media, the mode I DCB specimens immersed in an engine oil shows remarkable fracture resistance relative to the other exposed specimens in mode I loading condition. While in mode II loading condition, specimens immersed in benzene environment showed moderate result of delamination resistance to the normal specimens compared to the other conditioned specimens. These influences are due to the property of moisture absorbed during the immersion. In case of mode I loading, the absorbed moisture increases the dislocation of the mating matrix and makes the laminate easy to delaminate. While in case of mode II loading condition, the absorbed particles help to increase the flexural strength.

5.2. Recommendations

Even though the ASTM standards used in tests, some discrepancies were observed during the test work. Specifically in Mode I loading, branching and deviations of the delamination from the mid plane were visible problems. At the same time in mode II loading, specimen position deviations were observed. This may lead variations in results and is an ongoing research. To resolve the stability problems in three point bending (flexure test), it has to be an improvement on the end supports by introducing fixture mechanisms such as grooves. In addition, further tests of a DCB and ENF tests needs to exercise to provide enough information on an effect of several environments on reinforced composite materials.

5.3. Future Development

In this study as a beginner on the science of fracture of composite materials, it has been attempted to present the Mode I and Mode II fracture resistance of E-glass fiber epoxy resin composite material. Although the science is very vast and complicated, it has to be deal in detail as the future technology requires the use of composite materials.

The listed ideas below are points to be studied extensively for strengthening this paper's primary understanding.

- Studying the different mechanical properties of glass fiber epoxy resin composite material by extending the duration of time of different environmental conditions.
- Investigating the fracture toughness of glass fiber polymer matrix composite materials by integrating analytical and numerical methods with experimental work.
- Design and development of local fiber extracting machine for the production of natural fibers.
- Performing different natural fibers and fiber orientations by exposing into different environmental conditions.

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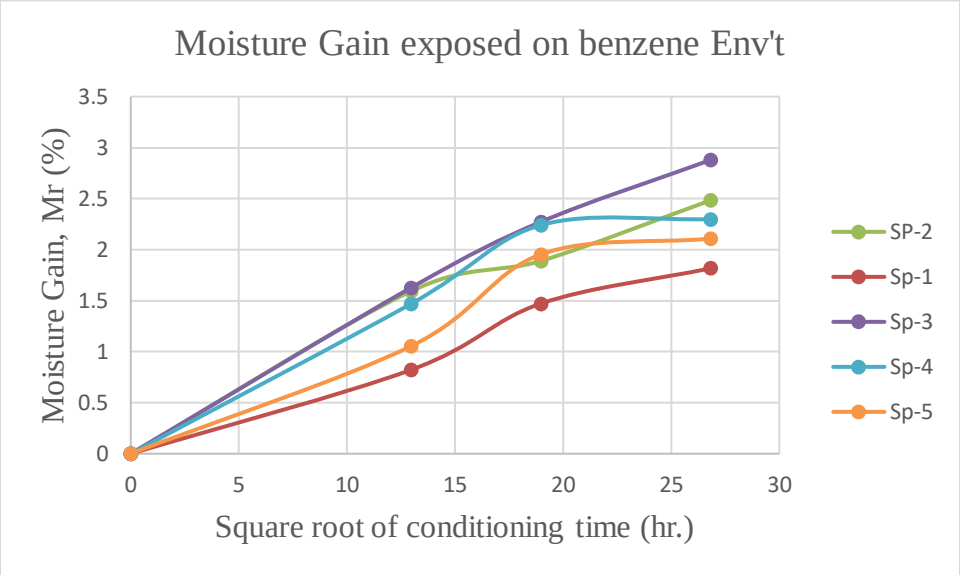
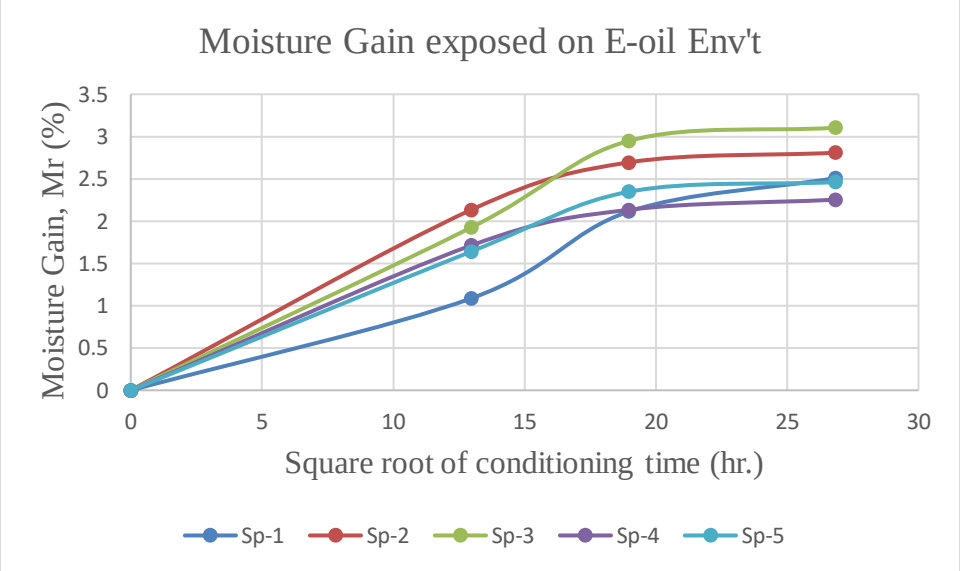
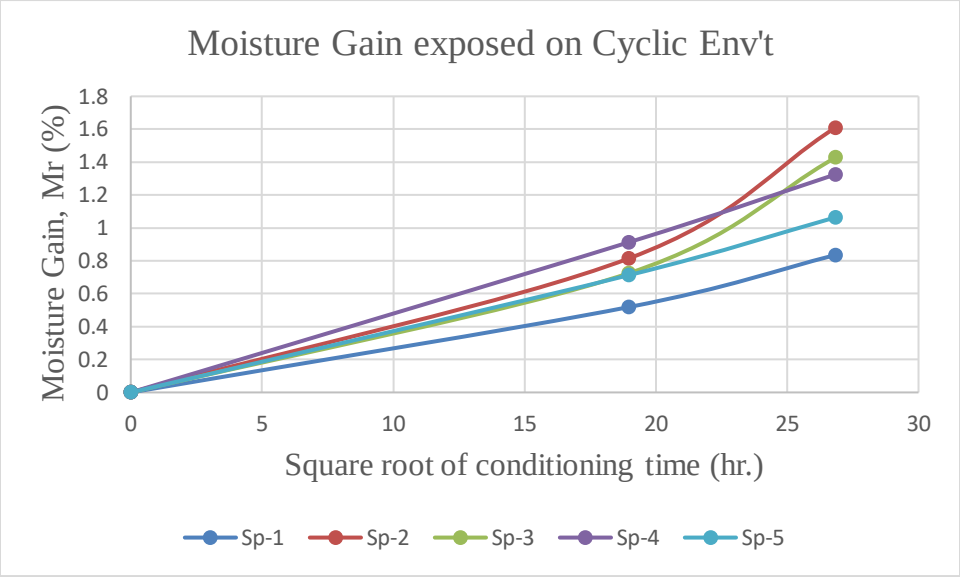
7. Appendices

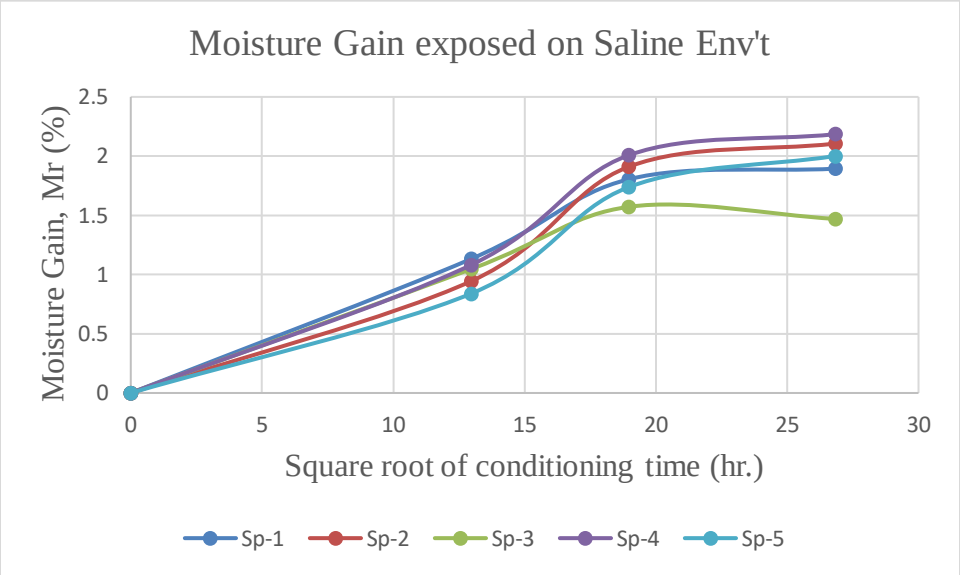
Appendix A: Graphs

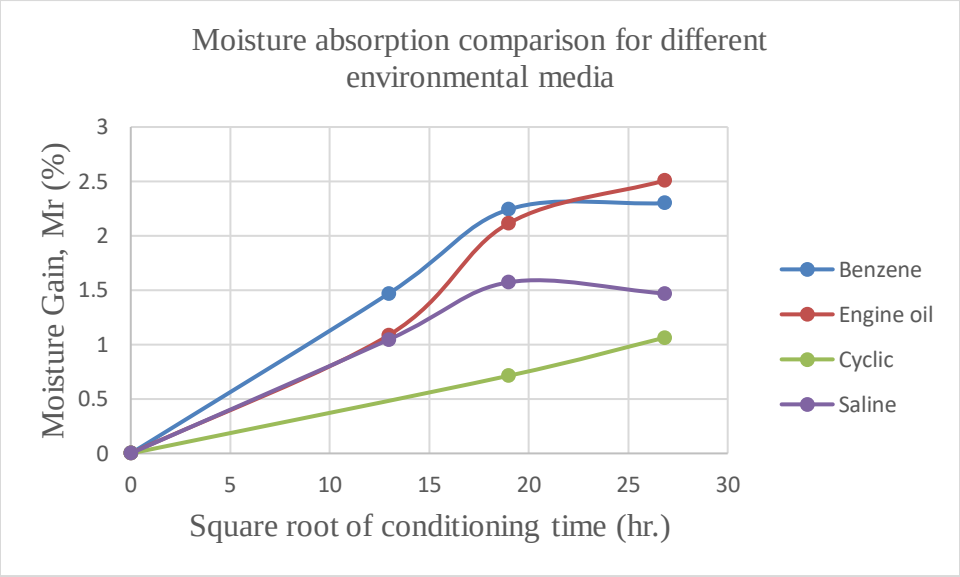
Moisture Absorption from different environmental exposures:






Moisture Gain exposed on Cyclic Env't
Moisture Gain, Mr (%)
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
0
5
10
15
20
25
30
Square root of conditioning time (hr.)
Sp-1
Sp-2
Sp-3
Sp-4
Sp-5


Moisture Gain exposed on Saline Env't
Moisture Gain, Mr (%)
2.5
2
1.5
1
0.5
0
0
5
10
15
20
25
30
Square root of conditioning time (hr.)
Sp-1
Sp-2
Sp-3
Sp-4
Sp-5


Moisture absorption comparison for different environmental media
Moisture Gain, Mr (%)
3
2.5
2
1.5
1
0.5
0
0
5
10
15
20
25
30
Square root of conditioning time (hr.)
Benzene
Engine oil
Cyclic
Saline

Appendix B: Material Properties

Table 5 Properties of epoxy resin [Kadisco Paint and Adhesive Industry]

No.	Property	Value	Unit
1	Density	1.17	g/cm^2
2	Young's modulus	3.4	GPa
3	Tensile strength	0.08	GPa
4	Shear modulus	1.26	GPa
5	Poisons ratio	0.36	
6	Compressive strength	0.104	GPa
7	Glass transition temperature	152	^oC

Table 6 Mechanical properties of E-glass fiber [Dejen Aviation Industry, Bishoftu, Ethiopia]

No.	Property	Value	Test method
1	Density (g/cm^3)	2.6	
2	Young's Modulus (GPa)	72.3	ASTM
3	Tensile Strength (GPa)	1.73	ASTM
4	Elongation (%)	4.8	

Table 7 Nature of E-glass Fiber

Color	White
Areal Weight	1.63 g/cm^2
Thickness	0.35 mm
Weave Type	Plain