



ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

SCHOOL OF MECHANICAL AND INDUSTRIAL ENGINEERING

DESIGN STREAM

INVESTIGATION OF THE INTERLAMINAR FRACTURE TOUGHNESS OF GLASS
HYBRIDIZED WOVEN SISAL REINFORCED PLASTIC COMPOSITE

Submitted by: Kerod Sisay

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Advisor: Mulugeta H. (Ph.D.)

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Approved by the Board of Examiners:

<u>Mulugeta Habtemariam (Ph.D.)</u>	_____	_____
Advisor	Date	Signature
<u>Araya Abera (Ph.D.)</u>	_____	_____
Internal Examiner	Date	Signature
<u>Behailu Mamo</u>	_____	_____
External Examiner	Date	Signature
<u>Abdulkadir Aman (Ph.D.)</u>	_____	_____
School Dean	Date	Signature

Declaration

I hereby declare that the work which is being presented in this thesis entitled “Investigation Of The Interlaminar Fracture Toughness Of Glass Hybridized Woven Sisal Reinforced Plastic Composite” is original work of my own, has not been presented for a degree of any other university and all the resource of materials used for this thesis have been duly acknowledged.

Kerod Sisay

Date

This is to certify that the above declaration made by the candidate is correct to the best of my knowledge.

Dr. Mulugeta Habtemariam (Advisor)

Date

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Abstract

Natural fibers are deemed as a perfect replacement to synthetic materials, because of their advantages over them. However, natural fiber reinforced materials are also subjected to many disadvantages, including lower thermal stability, poor adhesion, and susceptibility to delamination, as compared to synthetic materials. Hybridization with synthetic fibers like glass can compensate for these disadvantages.

This experimental study investigates the interlaminar fracture toughness of sisal/glass fiber hybrid composites, aiming to evaluate their mechanical properties in comparison to pure sisal and glass composites. Using ASTM D5528-13 standards, Mode-I fracture tests were conducted on fifteen specimens manufactured via the hand layup method. The study included evaluating the crack propagation behavior as well as measuring the critical strain energy release rate (GIC) under an opening load condition.

The experiments show that while pure glass fiber composite has the highest toughness and stiffness, the hybrid composites demonstrated an improvement over pure sisal laminate. The finding suggests that hybrid composites are suitable for medium and low mechanical performance and green applications. The study concludes that while glass fiber has the best mechanical strength, making it suitable for applications requiring high stiffness or robustness, hybrid sisal/glass composites offer a balanced alternative in terms of mechanical strength, environmental sustainability, and economic attractiveness for structural or quasi-structural applications.

Key words: Interlaminar Fracture, natural fiber, hybridization, energy release rate, failure mode

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CHAPTER I

1. Introduction

1.1. Background of the study

Composite materials are materials that help achieve different material properties that are not normally achieved by using a monotonic material. Composite material is formed by combining two or more materials with different properties to produce an end material with unique characteristics. These two materials are mixed and bonded on a macroscopic scale. Generally, a composite material is composed of reinforcement (fibers, particles, and flakes) embedded in a matrix (polymers, metals, or ceramics) [1, 2]. The purpose of the matrix is to offer a satisfactory level of performance to the composite structure by holding the fibers jointly and transmit the load to the fibers, which finally halts the formation of the crack. Reinforcement helps in the load-carrying property of the composites. Almost every mechanical property, from stiffness to strength and strength to thermal stability, results from reinforcement type used in the composite materials [3].

Of the reinforcement materials used, recently natural fibers are gaining a lot of interest. This is because natural fibers have high specific strength and modulus, are abundant, very light weight, non-toxic, reliable, easy to make, inexpensive, require less energy to manufacture, and are environmentally friendly [4, 5]. Among the natural reinforcement sisal fiber, are used as potential replacement reinforcement in place of glass and other synthetic fibers due to their low density, low energy consumption, adequate specific properties, low cost, acceptable biodegradability, and efficient stress transfer [6].

Hence, for these reasons natural fiber reinforced composite materials have many applications area. Some of these sectors include transport (automobile, marine and aerospace), construction, sports, and the packaging. Some examples of natural reinforcements include Sisal, Jutes, Banana, Flax, Jute, Hemp, Ramie, and Kenaf [5].

Sisal fiber reinforced composite materials have many applications in current days. For instance, sisal fiber reinforced materials are traditionally has been used for manufacturing rope, twines, carpets, and general cordages, it has recently been introduced in other industries as well[7]. One of these industries is the automotive industry. In recent years' big car manufacturing companies

like Audi and BMW have started using sisal fiber in the interior trim, which resulted in extremely low mass per unit volume, and the products also exhibit high dimensional stability, including high strength and impact resistance. Another industry that has started using sisal fiber reinforced composite is the construction industry. In this industry sisal fiber reinforced cement products such as roof tiles and building blocks are being produced [2]. However, the application of sisal fiber reinforced material is not only limited to these, in addition they are used to make appliances such as computer casings can be manufactured with injection molding techniques. Leisure products such as skis, golf clubs and butt stocks are also being manufactured using sisal fiber reinforced composites [2, 8, 9].

However, using these natural fiber reinforced polymers has some disadvantages. These include sensitivity to climatic conditions, low thermal stability, poor microbial resistance, hydrophobic nature, poor adhesion, and delamination[1]. One method of compensating for these disadvantages is to combine natural and synthetic fibers in a single matrix forming a hybrid composite where the other compensates the disadvantages of one. In this way, sustainability and cost minimization are achieved with acceptable mechanical and physical responses [1, 4, 5].

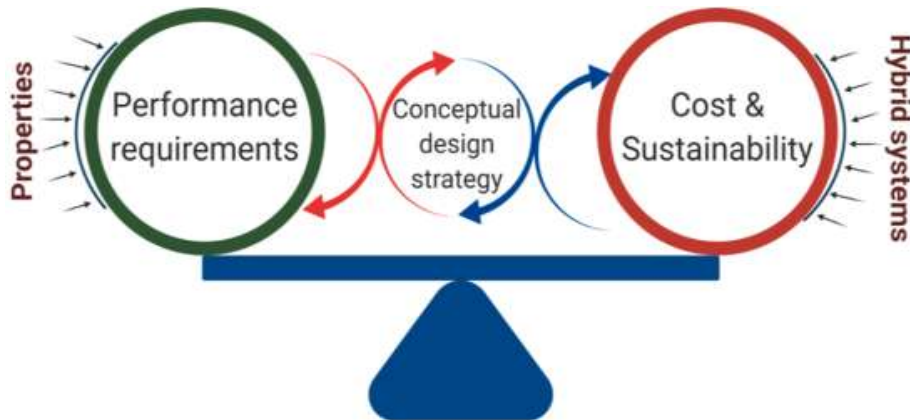


Figure 1-1 Connection between hybridization and required performance through proper design strategy [5]

Fracture of Composites Materials

There are several modes of fracture in composites materials such as interlaminar fracture (delamination), intralaminar fracture (matrix cracking), matrix-fiber debonding (non- adhering), fiber breaking, fiber pullout etc. [10].

Interlaminar Fracture (Delamination) Toughness

Delamination is a mode of failure for composite materials and is characterized by separation of the layers within the material. It is similar to peeling off the layers of an onion. Delamination can occur due to various reasons such as impact, fatigue, or manufacturing defects. Interlaminar fracture or delamination is the major challenge for fiber reinforced composite materials. Delamination greatly reduces the stiffness, degrades the strength of the laminates, eventually leading to failure. Interlaminar fracture differs from fracture toughness of metals, because it also includes energy losses in terms of matrix cracking, fiber breakage, fiber pull-out, fiber slipping and fiber debonding. Delamination occurs from low velocity impact caused by interlaminar shear stress and occurs when the interlaminar stress exceeds the interlaminar strength level. The interlaminar strength of a composite material is characterized by their strength/weakness under tensile stress and/or shear stress. Where strain energy rate and the applied load characterize delamination and its growth. Thus, the objective of studying the interlaminar toughness of a composite material is to measure the critical strain release rate at which the delamination begins to extend vary significantly depending on the mode of loading [1].

Modes of Fracture

There are three modes of loading that can cause fracture, that is, Mode I, opening (tensile) mode, Mode II, sliding (shear) mode, and Mode III, tearing mode. Fracture mechanics concepts are essentially the same for each mode [11].

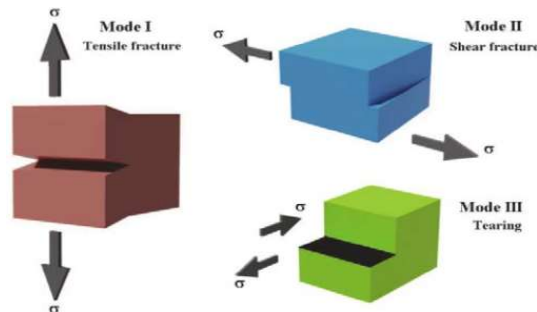


Figure 1-2 Modes of Loading[1]

The interlaminar fracture toughness of glass hybridized sisal fiber needs to be done. The effect of hybridization is also required to be studied by comparing the result with the interlaminar fracture toughness of glass/epoxy, and sisal/epoxy.

1.2.Statement of the Problem

Interlaminar fracture is a predominant phenomenon in a fiber reinforced composite materials. Delamination can occur to any material due to various reasons such as impact, fatigue, or manufacturing defects. Interlaminar fracture or delamination is the major challenge for fiber reinforced composite materials. It greatly reduces the stiffness, degrade the strength of the laminates, eventually leading to failure. Small delamination's are dangerous as they cannot always be detected by nondestructive inspections, and can potentially grow to unstable configurations due to in-service loads. In some cases, catastrophic failure of machines and structures can occur without any external warning signs. Which makes it one of the major reasons for failure of fiber reinforced materials[14].

The high cost of fuel and the demand for lightweight materials in several industries, such as aerospace, automotive, and other transportation, has been driving the global composite material market. It is estimated that by 2027 the global composite material market will grow to US\$ 152.14 billion with a constant annual growth rate of 6.3%. It is also estimated that more than 61.1% this market share is owned by glass fiber and 30% by carbon reinforced polymers [12]. Hence, with rapid growth in synthetic materials also come environmental pollution concerns and high manufacturing costs [13]. Natural fibers are a sustainable alternative, but they suffer from poor thermal stability, low adhesion, and susceptibility to delamination. Hybridization with synthetic fibers like glass can address these issues [4, 5].

This study aims to investigate the interlaminar fracture toughness of sisal/epoxy, glass/epoxy, and sisal/glass/epoxy composites to understand how hybridization affects fracture toughness and to recommend suitable materials for structural and semi-structural applications. Hence, the objective of this study is to find the critical strain energy release rate of woven sisal fiber/epoxy composite material, and find out the effect of hybridization with glass fiber, on the interlaminar fracture toughness under Mode-I loading.

1.3.Objective

1.3.1. General objective

The general objective of the research is to investigate the interlaminar fracture toughness of glass hybridized woven sisal composite under opening mode.

1.3.2. Specific objective

The specific objectives of this research are the following

- Generate the load vs displacement graph of the composites when subjected to opening load.
- Asses the strain energy release rate G_{Ic} during crack propagation of the materials.
- Visually inspect the crack propagation behavior of the specimen's post-test.

1.4 Significance of the study

With a rapidly growing composite material market, the need for environmentally friendly material has never been higher. Reinforcements extracted from natural plants are the most environmentally friendly amongst others. In this paper natural fiber, and synthetic fibers are hybridized to produce environmentally friendly, and less costly composite with a better mechanical, and physical property. For the proposed material to be recommended for structural and/or semi-structural component, the fracture characteristics of a material is a vital property. It is important to know how the material behaves during crack initiation and crack propagation in order to avoid catastrophic failure during failure. The physical and mechanical behaviors of sisal fiber depend on their source, age, and location, but also on their fiber diameter, experimental temperature.

1.5 Scope of the study

This research assesses the interlaminar fracture toughness of sisal/epoxy, glass/epoxy, and sisal/glass/epoxy composites. The strain energy release rate is only being assessed when these materials are subjected to opening load. A total of fifteen specimens (five specimens for each group of composites) has been be manufactured as per ASTM D5528-13. The staking sequence, weight fraction, and weaving type are selected so that they have better mechanical, physical, and fracture property as per previously done literatures

1.6 Limitations of the study

This work investigates the interlaminar fracture toughness of sisal/epoxy, glass/epoxy and sisal/glass/epoxy composites under Mode-I loading as per ASTM D5528-13. However few limitations should be noted. First, this study is limited to Mode-I loading only, hence the effects of Mode-II or mixed-mode conditions which are also important in real world applications are excluded. The study uses a specific stacking sequence, fibre weight fraction and weaving type which may limit the generalizability of the results to other composite configurations. The specimens were fabricated using hand layup process, a cost effective method which inherently introduces variability in fibre distribution, resin content and pre-crack initiation. The fracture toughness of the composites may be affected by this fabrication technique. Furthermore, visual inspection of the fracture surfaces was conducted without the aid of an electron microscope, which may have limited the ability to capture detailed microstructural features and accurately characterize the failure mechanisms at a microscopic level.

1.7 Organization of the thesis

The thesis document is organized into five chapters, each contributing to the overall structure of the study. The first chapter, Introduction, provides a comprehensive background, outlining the research problem, objectives, significance, and scope, while introducing the key questions or hypotheses that guide the investigation. The second chapter, Literature Review, offers a well-organized and exhaustive review of relevant research, identifying key themes, trends, and gaps in the current knowledge, forming the theoretical framework for the study. The third chapter, Materials and Methods, details the methodology, including research design, data collection methods, and analytical techniques, along with the materials and procedures used, justifying the choice of methods in relation to the study's objectives. The fourth chapter, Results and Discussion, presents the findings systematically, interprets the results in the context of the research objectives. Finally, the fifth chapter, Conclusion and Recommendations, summarizes key findings, draws conclusions, provides practical and theoretical recommendations, and suggests directions for future research.

CHAPTER II

2. Literature Review

The study of composite materials has recently been increasingly focusing on achieving improved mechanical properties, environmentally friendliness, and cost-effectiveness. Composite materials contain two or more different materials and thus allow the final product to benefit from the properties of each included material. Reinforcing composite materials with natural fibers is a relatively new trend, but one that is gaining considerable interest, especially given their sustainability, renewability, lightweight and low cost. Sisal has been emerging as a promising candidate among these natural fibers due to its good mechanical properties, biodegradation potential, and economic benefits.

Nevertheless, despite these advantages, natural fibers, such as sisal, also have some drawbacks, including poor interfacial adhesion, high moisture absorption, and low thermal stability. To address these issues, hybridization with synthetic fibers like glass has been suggested. The use of both bio-based and synthetic fibers may address the shortcomings of the individual reinforcements, yielding a well-balanced property combination.

Despite these advances, a critical gap exists in our understanding of how hybridization, specifically, the combination of woven sisal with glass fibers affects the interlaminar fracture toughness under Mode-I loading. While many studies have explored tensile, flexural, and impact properties, very few have systematically investigated the critical strain energy release rate of hybrid systems. It is crucial to addressing this gap to improve composite design for structural applications.

This literature review discusses the existing research on sisal fiber, glass fiber, and their hybrid composites. It also explores the effect that hybridization has on the interlaminar fracture toughness of these composites and addresses the key factors that are influencing their mechanical properties, these factors include fiber treatment, volume fraction, and fiber orientation. Furthermore, the research gap concerning the interlaminar fracture toughness of sisal-glass-epoxy composites has been highlighted, which motivates the present study.

2.1.Sisal Fiber

The sisal fiber is extracted from the leaves of the sisal plant (a.k.a. *Agave sisalana*), which consists of a sword like shaped leaves that reach up to a height of 100 – 150 cm tall and 13 – 15 cm wide. The sisal plant produces about 200 – 250 leaves over its life time, while each leaf contains a thousand fiber bundles, hence a sisal plant can produce about 200,000 – 250,000 fiber bundles over its life time [15]. Some of the properties that makes Sisal fiber stand out include renewability, low density, and biodegradability, coupled with a high specific modulus and a moderate mechanical strength[2]. The content of Sisal fiber's cellulose varies between 67% to 78% as reported by literatures, while the remaining composition is made up of lignin, ash, wax, and hemicelluloses. Factors such as geographical location, growing condition, and age has a significant influence on the chemical composition of the plant[2, 16]. These variations of chemical composition could affect the mechanical properties of the fiber, which in turn could affects its suitability for various applications.



Figure 2-1 Sisal Plant [17]

2.1.1. Types of Sisal Fibers

Sisal fibers can be classified into three main types: mechanical, xylem, and ribbon fibers, which are extracted from different parts of the leaf. The mechanical fibers have the highest modulus out of the three due to their rigid cell structure and are also the most commercially. On the other hand, ribbon fibers have a good mechanical property and are longer, however, during processing they tend to split. Finally, the xylem fibers are known to be easily lost during the process of extraction, and are also less robust[2].

2.1.2. Extraction of Sisal Fiber

The extraction of sisal fiber can be done by retting, scraping, or mechanical decortication. Among these mechanical decortications is the most widely used method. It involves crushing and/or beating the leave until only fiber remains, and moisture and the fleshy pulp are removed from the fiber. In this process, water is used to clean debris present in the leaves. Finally, the obtained sisal fiber is dried under the sun. The extracted fibers are generally 90 cm long [6, 15].



Figure 2-2 Extracted Sisal Fiber [18]

2.1.3. Mechanical and Physical Properties of Sisal fibers

Due to its strength, stretchability, resistance to saltwater deterioration, and durability, Sisal fiber has traditionally been used to manufacture ropes, cordages, and twines. With recent developments, it is being used in various structures such as the automotive industry, as an environmentally friendly reinforcement replacement of fiber glass and asbestos[2].

The modulus, density and specific strength of as Sisal fiber is comparable with that of Glass fibers [2, 19]. Sisal fibers have a Young's modulus of approximately 9-24 GPa and a tensile strength of 610-720 MPa. This makes Sisal fiber relatively better tensile property when compared to other natural fibers, but falls short when compared to synthetic fibers such as carbon fiber and glass fibers[3]. However, due to its renewability and light weight properties, makes it a promising candidate for environmentally friendly composite[3, 20]. The ability of sisal fiber to withstand a moderate amount of tensile forces makes it well-suited for uses in the construction and automotive applications[3, 20].

The high specific modulus and moderate tensile strength of Sisal fiber coupled with physical properties, such as low density of approximately 1.33 g/cm^3 , makes Sisal fibers a good alternative replacement to synthetic fibers in non-load bearing applications[2, 16].

2.1.4 Properties of Sisal fiber reinforced composite

Different literatures have developed Sisal fiber reinforced composites (SFRC) by using different types of matrices. These include thermosets such as polyester and epoxy, and thermoplastics such as polypropylene. These composites have shown a notable improvement s in properties such as tensile strength and stiffness, which is due to the reinforcing effect of the fibers[2, 21-24].

The characteristics of sisal fiber–reinforced composites are mainly due to the properties of matrix and the fibers-matrix interface. The tensile strength, flexural strength and impact resistance of the composites are extremely improved when reinforced with sisal fiber. An example is the work by Murali Mohan Rao and colleagues have proven that sisal-based composites with optimum fiber content has a high tensile strength and modulus. Tensile strength increases with the increase of fiber volume fraction, reaching an optimal value before declining due to increased brittleness[21].

Sanjay et al. evaluated a sisal-glass fiber reinforced hybrid composite. The resultant hybrid composites showed improved tensile strength, stiffness, and impact resistance as compared to composites with sisal or glass fibers separately[3]. Hybrid combination of natural fibers and synthetic fibers provides the best advantages with less cost but also to more eco-friendly materials.

2.1.5. Factors affecting Sisal fiber reinforced composites

The main problem faced to develop sisal fiber reinforced composites is that the hydrophilic nature of sisal fibers has difficulty in bonding with the completely incompatible surface properties of poor water-resistance and wettability[1]. This in turn leads to low fiber-matrix adhesion and, hence poor mechanical properties. Some of the surface treatment methods that have been used to improve complete fiber-matrix interaction are alkali-treatment, silane coupling agents and acetylation[25]. Torres et al. found that stearic acid treated sisal fibers could increase the interfacial shear strength by 23 %, which indicated the necessity of surface treatments[26].

Several other key factors that influence the performance of sisal fiber-reinforced composites, includes fiber treatment, moisture absorption, fiber orientation, fiber volume fraction, and weight

fraction, in case natural fibers content due to its porous surface must be regarded as volume phase and not related with aspect ratio [10].

The chemical treatments are widely employed to enhance the fiber-matrix adhesion. For example, alkaline treatment erodes the hemicellulose and lignin of fiber surface thereby increasing roughness and adhesion at the interface. Senthilkumar et al. and Wambua et al. highlighted the effectiveness of chemical treatments in enhancing fiber-matrix bonding, resulting in improved tensile, flexural, and impact properties of sisal composites [19, 20].

However, the disadvantage of sisal fibers was poor moisture absorption. The high moisture uptake derived from the hydrophilic nature of sisal fibers can cause composite swelling inducing degradation and loss in mechanical properties with time. In general, both chemical treatments and hybridization had a positive effect on moisture absorption, improving the long-term durability of sisal composites[15].

The fiber content and their orientation also affect the performance of sisal composites. Wambua et al. has shown that composites with ideal fiber volume fractions (generally 30–40%) have superior mechanical properties including tensile strength and stiffness. As the fiber content increases, however; it may go beyond a limit to create poorly distributed fibers into weaker composite[20]. Embedding fibers in the desired orientation is also crucial to increase mechanical performance of the composite.

While sisal fibers offers a considerable environmental and economical benefits, their inherent mechanical drawbacks, such as poor interfacial adhesion and low stiffness, highlights the necessity of hybridizing with synthetic fibers, a strategy that this study investigates to enhance interlaminar fracture toughness.

2.2 Glass fiber

Glass fibers are among the most widely used reinforcing materials in polymer composites due to their favorable balance of mechanical properties, lower cost, and versatility. The main types of glass fibers are E-glass, S-glass, C-glass, and R-glass, each offering different properties suited for various applications. E-glass is the most commonly used type due to its high strength, moderate cost, and good chemical resistance. S-glass, which has higher tensile strength and modulus, is

typically used for aerospace and military applications where performance is prioritized over cost [27, 28].

Glass fibers are produced by melting glass at high temperatures and drawing it through fine orifices to create filaments. The properties of these fibers depend significantly on their composition and the manufacturing process. For instance, E-glass fibers are made from alumino-borosilicate glass, which provides good insulation properties and mechanical strength, while S-glass is composed mainly of silicon and magnesium oxides, offering superior tensile properties[29]. The fibers can be produced as continuous rovings, chopped strands, or woven mats, depending on the intended application[29].

2.2.1. Glass Fiber Reinforced Composites (GFRC)

Glass fiber reinforced composite (GFRC) is a complex in which the reinforcing fibers are embedded within a polymer matrix. The matrix may be a thermoset (such as epoxy, polyester) or thermoplastic material such as polypropylene. These composites are extensively used in various industries such as automotive, aerospace, construction and marine due to their excellent specific strength, stiffness corrosion resistance and design flexibility [27, 28].

The production methods of GFRC differ based on the kind of matrix used and how fiber is positioned. Hand lay-up, compression molding and hot press molding are common techniques. Every method has its advantages and drawbacks which in turn influence the quality of that composite. The hand lay-up method for instance, is low cost and good approach to large composite parts (Gostaert et al., 2020), however it may not provide uniform fiber distribution like the automated processes like filament winding[28, 29].

2.2.2. Applications of GFRC

GFRCs are used in a variety of applications, including automotive parts (e.g., body panels, bumpers), aerospace components (such as rudders and landing gear doors), and construction materials like roofing sheets or panels. In automotive and aerospace, composites like these bring about a meaningful reduction in weight versus their metal counterparts to bolster fuel efficiency while lowering emissions[28]. In addition to the panels, GFRCs are also used in consumer goods, sporting equipment and even dental applications as they have excellent mechanical properties and fabrication ease[30].

2.2.3. Properties of Glass Fiber

The tensile strength, modulus of elasticity and elongation at break are fundamental mechanical properties that affect these artefacts when glass fibres (GFs) were used as reinforcements. E-glass is one of the most common glasses used for fibers in composites with a tensile strength up to 3.5 GPa and a modulus of approximately 73 GPa, making them suitable for most structural applications[19, 31]. Tensile properties of GFRC fibers are determined by the tensile strength and modulus of glass fiber, as well as its content, orientation, and the type of matrix used.

Glass fibers are great thermal stability fibres and can be able to take higher processing temperatures making them ideal for applications that require heat resistance. Having even better thermal properties than E-glass, S-glass was expected to be significant in structures that operate at high temperature[27]. These glass fibres also have high chemical inertness, which enhances the durability of composites in corrosive environments such as marine and, food handling or storage equipment, and other chemical processing applications[29].

Glass fibers are additionally recognized for their high impact resistance and fatigue strengths. On the other hand, GFRC has lower fatigue strength compared to carbon fiber-reinforced composites. GFRC performance during fatigue loading is affected by factors like fiber volume fraction, fiber angle and type of bonding between the fibers with matrix [27, 31].

2.2.4. Factors Affecting the Properties of GFRC

The quality of adhesion of the glass fibers to polymer matrix is one of key factors directly influencing on mechanical properties GFRC. Adhesion is the starting point in composite structures and poor adhesion can lead to fiber pull-out, matrix cracking, which means delamination between these materials when loading, decreases mechanical properties significantly. However, improvement can be found in better adhesion between these polymers media by treating the glass fibers with coupling agents such as silanes which is reportedly to improve chemical bonding between glass surface and polymer matrix[27, 29].

In the composite, the volume fraction of glass plays an important role in determining its mechanical properties It is widely found that tensile strength, flexural strength and modulus of the composite increase with higher fiber volume fractions. However, beyond a certain limit, defect formation may occur related to poor fiber wetting by resin and reduce the fibers may not be adequately wetted

by the resin, leading to defects and reduced mechanical properties[32]. As such, the fiber content has to be tuned just right for a balance between strength and ease of manufacture.

Also, the direction and duration of glass fibers is another major factor that specifically affects GFRC properties. Long, unbroken fibers aligned with the load direction Maximum strength and stiffness. Consequently, short and randomly-oriented are usually employed because they make the mechanical processing easier to perform and reduce costs (although providing lower mechanical properties compared with continuous fibers) [32]. Especially in aerospace components, sourcing directionally strong fiber orientation can be crucial to end-use applications for things like aircraft and defense systems.

Environmental conditions such as moisture, temperature and UV radiation exposure influences properties of GFRC. Although glass fibers alone are stable in humid conditions, the polymer matrix is capable of absorbing water, causing swelling and potentially disrupting fiber-matrix adhesion, thereby diminishing strength [27, 33]. These may include the matrix material and fiber surface treatments that can have a significant impact on composite performance in an adverse environment.

The processing conditions during composite manufacturing, such as temperature, pressure, and curing time, also affect the properties of GFRC. Improper processing can lead to defects such as voids, uneven fiber distribution, and poor fiber wetting, which compromise the mechanical performance of the composite. The use of controlled processes like injection molding can help achieve better consistency and quality compared to manual methods like hand lay-up[31].

The superior mechanical properties of glass fibers including high tensile strength, and stiffness has positioned them as the ideal reinforcements. However, due to their environmental impact and cost the exploration of hybrid systems prompt, which is central to addressing the research gap in this study.

2.3. Hybridization

Hybridization refers to combination of different reinforcements within a composite system [4]. Depending on the distribution of reinforcement, hybridization of natural fiber reinforced polymers is categorized into three types. These are interply, intraply, and super hybridization. Interply hybridization is obtained when individual constituents are stacked in layers at laminate level,

intraply hybridization is formed from parallel combination of constituents within plies, whereas super hybridization involves layers containing metal/composite and matrix arranged in a specific order [5].

2.3.1. Effect of hybridization

Many researches done on hybrid natural fiber composite, and specifically hybrid sisal fiber reinforced composites suggested that hybridization have enhanced the properties of the composite [17]. For instance, a paper by John and Naidu reported that by hybridizing sisal fiber with glass fiber, it was possible to increase the impact strength by up to 301%, and an increase of up to 19% in compressive strength [34]. While, in another paper by Venkateshwaran and his colleagues, concluded that hybridization of banana with sisal fiber resulted in an increase of the tensile strength, flexural strength and impact strength around 16%, 4% and 35% respectively [25]. According to a paper by John and Naidu, states that the flexural strength of pure sisal was increased by up to 115% by hybridizing with glass fiber [35], while, a 20% increase in tensile strength and 21% increase in elongation was also achieved by hybridization of sisal fiber with glass fiber [36]. In addition to this, it was proven that the addition of glass fiber to sisal fiber reinforced composite had improved the thermal property and the water absorption resistance [37]. Hence, the mechanical, and physical properties increased due to hybridization with glass [3].

2.3.2. Factors affecting Hybridization

According to Shrivastava and Singh[11], the fiber volume fraction of the composite does not influence the fracture toughness during crack initiation, which is due to the similarity of damage zone. However, the fiber volume fraction had positive impact on the fracture toughness during crack propagation. This increase in fracture toughness was due to an increase in fiber bridging. On a paper by Gill and his colleagues, it was found that high fiber volume fraction composites had 72.02% times more mode-I fracture toughness values compared to low fiber volume fraction samples. These increase in fracture toughness was due to energy absorption mechanisms [38].

The number of layers has a huge impact on the interlaminar fracture toughness of fiber reinforced composites. As the number of layers increase, so does the stiffness of the fibers, hence, increase in the interlaminar fracture toughness. According to a paper by Zulkifli R et al., an increase in the number of layers of lamina results in a slower crack growth and more stability [39]. Hence, a higher number of layers of fiber is directly related to higher interlaminar fracture toughness, and

an increase of stiffness of the fiber. The increase in interlaminar fracture toughness results in a slow and stable delamination growth [1].

According to Suppakul and his colleagues, it was observed that the weaving architecture of the woven fiber composite had a great effect on the interlaminar fracture initiation and propagation values. This was because of the high number of fractured fibers due to fiber bridging, hence, having higher value of strain energy release rate during initiation and propagation of crack [40]. As reported by M. Pinto, due to the presence of fiber bridging in woven fiber reinforced composites, they generally have shown 28% higher fracture initiation toughness as compared to unidirectional fiber reinforced composites [41]. Woven reinforcements also experience high degree of nesting. Hence, the paper reports that the strain energy release of woven flax is three times higher than unidirectional one [1]. In conclusion, it has been found that twill weave resulted in the highest critical strain energy release rate value [1, 40].

From the previous studies reviewed, it suggests that hybridization can synergistically combine the advantages of both natural and synthetic fibers, directly addressing the critical research gap regarding the interlaminar fracture toughness of sisal/glass composites and setting the stage for the present investigation.

However, during the literature review survey no research on the interlaminar fracture toughness of sisal fiber reinforced materials as well as sisal/glass/epoxy composites was found. Hence, there is a research gap that needs to be filled, in order to recommend sisal fiber reinforced composites for structural application.

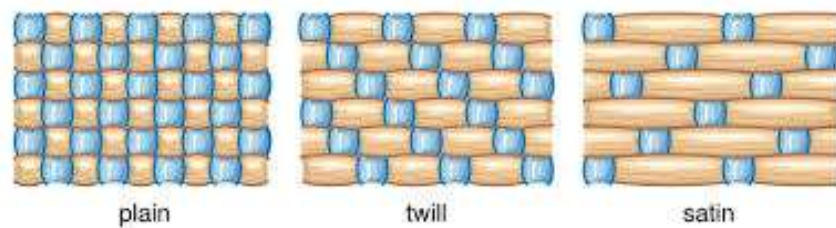


Figure 2-3 Basic weaves [42]

2.4. Interlaminar Fracture Toughness Test

ASTM is working on standardizing tests that is used to measure the critical strain release rate under different loading conditions, but it is still a work in progress. Hence, the ASTM D 5528 is the

recommended test to measure the Mode I fracture toughness G_{IC} of fiber reinforced polymer composites by using Double Cantilever Beam (DCB) specimen. While the ASTM D 7905 recommends using the End Notch Flexure (ENF) test for testing pure Mode II. And ASTM D6671 recommends the use of Mixed-Mode bending (MMB) test that can measure fracture toughness over a wide range of combinations of Mode I and Mode II loading. And finally, even though it is not standardized yet, the Edge Crack Torsion Test (ECT) is used to measure the fracture toughness of pure Mode III [10, 11].

2.4.1. Mode I Interlaminar fracture toughness testing

The Mode-I testing follows the standardized test of ASTM D5528 - 13 to measure the Mode-I fracture toughness, fiber reinforced composite material. The specimens used in this testing are the double cantilever beam (DCB) specimen. This specimen consists of a rectangular cross section with uniform thickness specimen, that is at least 125 mm long, and 20 - 25 mm wide. The specimen contains a non-adhesive insert during fabrication, of thickness of not more than 13 μm , on the midplane which serves as a crack initiator. Piano hinges or loading blocks, that are bonded to the top and bottom of one end of the specimen serves as means of applying the opening force. These ends are loaded either by controlling the opening displacement or the crosshead movement. The load, crosshead movement (or crack opening) displacement and delamination length are recorded continuously during the test. The delamination lengths are determined visually or by using a traveling microscope for more accuracy. Finally, the initiation and propagation value of G_{IC} can be calculated based on these recorded data using beam theory and data reduction methods [43].

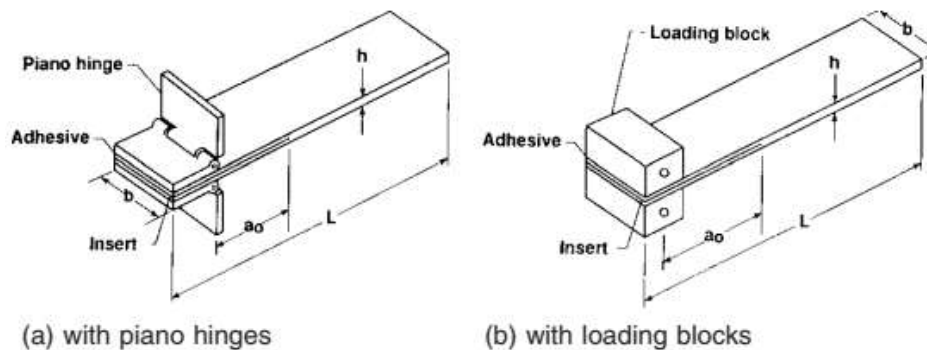


Figure 2-4 DCB test Specimen geometry[43]

The three data reduction methods recommended in (ASTM D5528-13 2013) are: Modified Beam Theory (MBT) method, (B) Compliance Calibration method, and Modified Compliance Calibration (MCC) method.

A. Modified Beam Theory (MBT) - This method considers the delamination front to be clamped perfectly for the strain energy release rate. The formula for this method is: $G_I = \frac{3P\delta}{2ba}$

P = load,

δ = load point displacement,

In the above method, the specimen is considered to be fixed and not subjected to rotation at the delamination front. But this is not the case in the real world. Rather, the beam is not perfectly built in and is subjected to rotation on the delamination front. Hence to correct this we consider the delamination to be slightly longer, $a + |\Delta|$, rather than just 'a.' where, Δ is calculated experimentally by generating a least squares plot of the cube root of compliance, $C^{1/3}$, as a function of delamination length. Where $C = \delta/P$

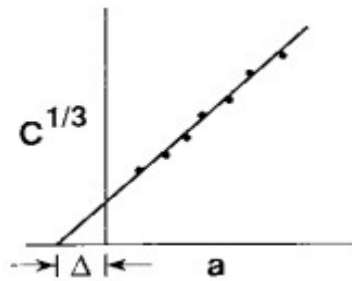


Figure 2-5 Modified Beam Theory graph[43]

Hence, the formula becomes: $G_I = \frac{3P\delta}{2b(a+|\Delta|)}$

B. Compliance Calibration (CC) Method - In this method the strain energy release rate is calculated as follows: $G_I = \frac{nP\delta}{2ba}$

Where, n is calculated as the slope from the graph generated from a least squares plot of $\log(\delta/P)$ versus $\log(a)$ using the visually observed delamination onset values and all the propagation values. Draw a straight line through the data that results in the best least-squares fit. $n = \frac{\Delta_y}{\Delta_x}$

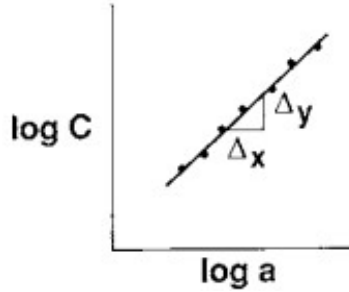


Figure 2-6 Compliance Calibration Method graph [43]

C. Modified Compliance Calibration (MCC) Method - Generate a least squares plot of the delamination length normalized by specimen thickness, a/h , as a function of the cube root of compliance, $C^{1/3}$, as shown in Fig. 6, using the visually observed delamination onset values and all the propagation values. Calculate the Mode I interlaminar fracture toughness

as follows:
$$G_I = \frac{3P\delta p^2 C^{2/3}}{2A_1 b h}$$

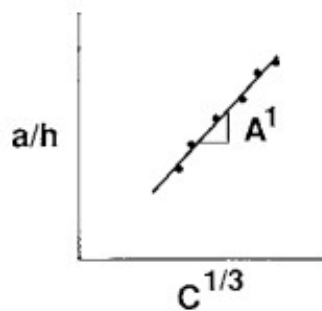


Figure 2-7 Modified Compliance Calibration Method graph [43]

Fiber Treatment

Sisal fiber is mostly made up of 50 -80% cellulose. Cellulose is a hydrophobic polymer, making the sisal fiber have hydrophobic property as well. Due to this, sisal fibers are well known for their weak adhesion to hydrophobic matrixes [16]. This leads to natural fibers such as sisal fiber, having lower strength, lower adhesion, higher moisture absorption, and lower specific strength.

There are different methods of treatment. Alkaline treatment is the most widely used treatment for cleaning oil and debris from the surface of the fiber, and by creating rough surface on the fiber[44].

Crack Initiation

According to Brunner [45], the type of defects (pre-crack) have a significant effect on the experimental results. In his paper, he used three methods to introduce initial defect to the specimens

Method (A) involves inserting a thin film between two laminas during the layup process of a laminated material without pre cracking prior to testing.

This method has proven to be the easiest method out of the three methods. Using an insert film as a crack initiator allows to form a crack tip at a defined point, and also a well-defined starter crack tip informed which is observable macroscopically.

However, the thickness of the insert film needs to be less than or equal to 13 micro meter in order to not yield a result that is not affected by the crack initiator film [46]. If thicker insert film is used, it will result in a region which yields a different critical fracture toughness that is different from the rest of the composite. This is because the region ahead of the tip of the insert film is a resin rich region[45]. Another disadvantage of using insert film as starter crack is that crack propagation from the starter film was unstable in brittle epoxy unidirectional laminates without mechanically-induced pre cracks.

Method (B) involves inserting a thin film between two laminas during layup, and the wedge is used to grow the delamination beyond the tip of the starter film (Mode 1 load).

Method (C) involves inserting a thin film between two laminas during layup process, and the specimen is pre cracked by Mode 2 or Mode 1 load to grow the delamination point beyond the starter film's tip.

Under this method, delamination growth under Mode 1 loading yields a thumb-nail shape curve, which is symmetrical with respect to the median. While, delamination under Mode 2 usually yields an extended damage zone with a fuzzy crack tip. However, it was observed that under both loading methods, the “insert films yields a straight starter crack tip across the starter crack tip.”[45].

Pre cracking in Mode I has been shown to give lower values of fracture toughness compared with insert, especially for Mode II testing. Mode I or wedge crack initiation has been shown to produce starter tip shapes similar to those observed during Mode I testing. However, pre cracking may also

create a damage zone ahead of the crack tip and irregular crack front shape. Another issue is that fibers tend to bridge between interfaces with crack extension, which results in a higher initiation value than an insert film defect type.

Method of loading and unloading

Method of loading/unloading for a typical DCB test, are of two types. The first method, one or a maximum of two loading and unloading cycles. In this method, the first loading unloading creates a crack length of typically 3-5 mm, while second loading and unloading creates a 50-60 mm. In the second method, several loading/unloading cycles take place. A typical load against opening displacement curve of loading and unloading procedures is shown in the figure below. The purpose of the loading/reloading process is to obtain a more precise compliance value for the crack lengths at which the specimen is unloaded[47].

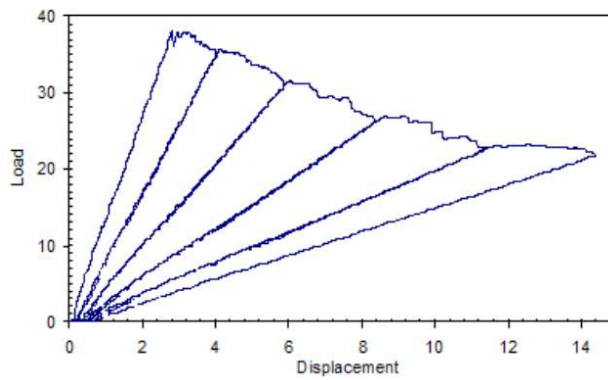


Figure 2-8 Load vs Displacement curve of a DCB specimen with multiple loading/unloading cycles[47]

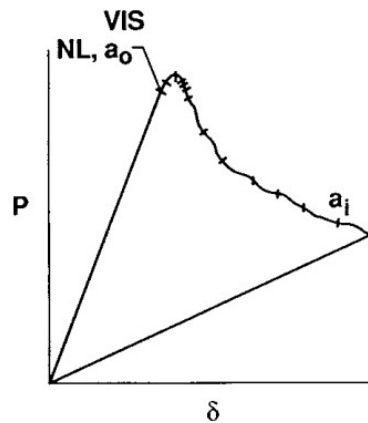


Figure 2-9 Load vs Displacement curve of DCB specimen under two loading/unloading cycles[43]

Interlaminar Fracture toughness of Glass Fiber

Blake et al. had conducted a detailed study of the interlaminar fracture of unidirectional E-glass fiber reinforced epoxy composite, by using the double cantilever beam (DCB) tests to evaluate the G_{IC} . The study had employed the modified beam theory (MBT) as data reduction method to determine the fracture toughness, and the results highlighted significant change in delamination behavior based on the fiber orientation and stacking sequence[49]. While another experiment by Hodzic et al. investigated the effect of water aging on the interlaminar fracture toughness for glass polyester and glass phenolic composites. From the experiment, the G_{IC} was found to decrease significantly after prolonged exposure to moisture. This was due to the moisture weakening the fiber-matrix interface. This reduction in fracture toughness was caused by interfacial debonding, which was observed through fractographic analysis using scanning electron microscopy (SEM)[50].

A study by Bhandakkar et al. on the interlaminar fracture toughness of epoxy glass fiber reinforced composite, it was found that the addition of fly ash fillers had improved the Mode I fracture toughness by approximately 50%. This emphasizes the potential of fillers in the enhancement of delamination resistance of glass fiber composites[51]. In addition, a study by Saravanakumar et al. studied the influence of milled glass fiber fillers on the interlaminar fracture toughness of glass fiber. It was found that incorporating this milled filler increases the G_{IC} through a bridging effect, which stabilizes the crack propagation[52].

Interlaminar Fracture Toughness of Natural Fiber for Mode I Loading

The interlaminar fracture toughness under Mode I of natural fibers such as flax and Bamboo has been studied. Bensadoun et al. had conducted an investigation on the interlaminar fracture toughness of flax epoxy composite. The study assesses various types of fiber architectures including unidirectional and woven architectures. The result show that the interlaminar fracture toughness in Mode I of the woven flax fiber is better than the unidirectional, which is due to the enhancement of fiber bridging and the increase of energy dissipation mechanisms[53]. In a study by Chen et al. the Mode I fracture toughness of bamboo fiber reinforced composite had been studied. This study focused on the intrinsic and extrinsic toughening mechanisms such as crack kinking and fiber bridging. They concluded that the highest fracture toughness value was found in

the middle region of bamboo which indicates that optimizing the fiber content could enhance the resistance to crack[54].

A study by Ravandi et al. investigates the Mode I interlaminar fracture toughness of stitched unidirectional (UD) flax fiber/epoxy laminates. The composite specimens were fabricated using vacuum-assisted resin infusion (VARI) with a layup of [0]16 and stitched with twistless flax yarns in a modified lock stitch pattern. The stitch row spacing was 4 mm, and the stitch length was 12 mm. The fiber content achieved was not explicitly stated, but the study emphasized the role of flax fibers in reinforcing the epoxy matrix. The Double Cantilever Beam (DCB) test, performed following ASTM D5528. The results revealed that introducing flax yarn stitching increased the delamination resistance by twofold compared to unstitched specimens [55].

Another literature by Li et al. which was aimed to enhance the interlaminar fracture toughness of unidirectional flax fiber/epoxy composites using randomly oriented chopped flax yarn interleaves. The chopped yarns were varied in length (5, 10, and 15 mm) and content (up to 30 g/m²). Fabrication involved impregnating flax fabrics with epoxy resin and using a hot-compaction process, resulting in a fiber volume fraction of approximately 60%. The Mode I fracture toughness was tested using DCB tests. Results demonstrated that an optimal yarn length and content combination could increase the fracture toughness by up to 31%. Scanning Electron Microscopy (SEM) analysis showed that chopped yarn interleaves created tortuous crack paths and trans-layer bridging, contributing to energy dissipation during delamination[56].

A study by Shindo et al. focused on the cryogenic Mode I interlaminar fracture toughness of woven glass-cloth/epoxy laminates. Using a double cantilever beam (DCB) test, the laminates were tested at room temperature, 77 K (liquid nitrogen), and 4 K (liquid helium). The specimens consisted of 20-ply woven glass cloth and epoxy resin cured under pressure at 470 K and 4.9 MPa, yielding a fiber volume fraction of 56%. The critical strain energy release rate (GIC) was significantly lower at cryogenic temperatures compared to room temperature[57].

A paper by Samborski et al. presents the Mode I interlaminar fracture toughness studies on glass/epoxy unidirectional laminates. Specimens were prepared following the ASTM D5528, with 16 plies of unidirectional glass fiber roving tape and a PTFE insert as a pre-crack starter. Fabrication involved autoclaving at 100°C under pressure, and the fiber volume fraction was approximately 35%. The DCB tests were performed using compliance calibration methods, and

the fracture toughness (GIC) was determined. Acoustic emission (AE) analysis was used to monitor damage evolution, identifying distinct frequency bands associated with matrix cracking and fiber bridging. The results highlighted the critical influence of interlaminar delamination and the need for careful damage monitoring[58]

The study of the effect of hybridization on the interlaminar fracture toughness of natural fiber reinforced composites has been studied by Periera et al. In their study it was found that the G_{IC} value of the hybrid composite of sisal and jute was higher when compared to pure sisal or pure jute. This was due to the hybridization limiting crack propagation via improvement of fiber interaction[59].

Summary of literature review

Despite natural fiber composites and hybrid materials having been widely explored, there remains a significant gap in the understanding of the interlaminar fracture toughness of sisal-glass-epoxy composites. A typical failure mechanism for fiber-reinforced composites is interlaminar fracture, which is the failure of material layers within the composite. Since this kind of fracture is responsible for a large loss of toughness in composites, it is important to understand and improve their interlaminar toughness. However, the interlaminar fracture behavior of sisal-glass hybrid composites have not been thoroughly explored, especially in terms of how hybridization can enhance resistance to such failure modes.

The hybridization of natural fiber and glass fiber is effective in overcoming the shortcomings of natural fibers without compromising on the green and economical aspects. The significance of this study is that it has bridged this gap and provided detailed information about the mechanical properties of sisal-glass-epoxy composites, particularly interlaminar fracture toughness. Knowledge of the fracture performance of such hybrid composites is relevant in their usage for structural and semi-structural components, where mechanical performance and environmental sustainability are of relevance. The outcomes anticipated from this research are that hybrid composites will show a better overall interlaminar fracture toughness compared to pure natural fiber composites and will offer a compromise solution suitable for those applications where high toughness performance and sustainability are desired.

CHAPTER III

3. Materials and Methods

3.1. Materials

3.1.1 Fiber

For this experiment, sisal fiber and glass fiber are used as reinforcement material for the composite material. Sisal fiber is a Lignocellulosic fiber that is extracted from *Agave sisalana* plant's leaves [60]. Sisal fiber is mostly cultivated in Brazil, Haiti, East Africa, Indonesia, and India. It has potential to grow in hot climate and arid regions, which are not suitable for other plants [16]. The cultivation of the plant is easy as it grows in all types of soil except clay, and is resilient to diseases[4]

Sisal fiber is a good potential reinforcement for polymer composites due to its flexibility, impact resistance, specific strength, stiffness, durability, ease of availability, and economic feasibility[4].



Figure 3-1 Woven Sisal Fabric

Glass fiber has a versatile application in domestic and industrial materials, as it is durable under varying environmental conditions[61]. Glass fiber reinforced polymer is the most commonly used composite for manufacturing composite materials. It has a wide range of application due to their better tolerance to damage from impact, high specific strength, flexibility, good resistance to environment and to chemical harm, stiffness, and are less expensive to other similar alternatives [28, 62, 63].

3.1.2 Metrics

Epoxy resin is a thermosetting resin, that is one of the most commonly used type of polymer matrix material due to its low viscosity, resistance to chemical, high strength, low volatility, good adhesion, and low shrinkage[64, 65].

Curing agents such as a hardener, play an important role in the hardening process of epoxy resins. They are essential because they define the properties of the epoxy once cured, such as curing kinetics, gel time, viscosity, the extent of curing, reaction rate, and curing cycle[66]. Hardeners essentially activates and controls the chemical reactions that transform liquid epoxy into a solid, durable material, making them integral to the usability and effectiveness of epoxy in various applications[67].

For this experiment, Araldite LY 556 epoxy resin with hardener ARADUR 906 was used. The property of the epoxy resin is presented below.

Table 1 - Epoxy Specification

Specifications	Value
Epoxy to Hardener ratio	1:1
Viscosity at 25 ⁰ C	10,000 – 12,000 mPa
Density at 25 ⁰ C	1.15 – 1.2 g/cm ³
Epoxy index	3.2 – 5.45

3.1.3. Fiber Treatment

Treating natural fibers such as Sisal fiber by using treatment chemicals such as Sodium Hydroxide (NaOH), significantly improves their adhesion with resin polymers. They are crucial for manufacturing natural fiber reinforced composites[68]. The inherent hydrophobic nature of natural fibers is often the cause of poor adhesion between the fiber and the hydrophobic polymer, which in turn causes loss of strength and degradation overtime[68, 69]. Substances that contribute to water absorption of the Sisal fiber such as lignin and hemicellulose, can be removed by applying a 5% NaOH solution to the fibers. This promotes fibrillation, and cellulose content, which enhances the fiber's mechanical properties and its adhesion with the polymer matrix[70].

Distilled water is mainly used to prepare the Sodium Hydroxide solution. It maintains consistency and purity in the process of the treatment, this ensures that unwanted impurities do not infer with the chemical reactions. This do not only extend the lifespan of the composites by reducing the risk of premature aging, but also enhances their structural integrity[68].

For the purpose of surface modification, sodium Hydroxide (NaOH) has been bought with the following properties.

- Grain size – Pellet
- Country of origin – Turkey
- Molecular Weight – 40 gm/mol
- Assay – 99.8%



Figure 3-2 NaOH

3.2. Experimental Methods

3.2.1. Specimen Preparation

- **Sisal Fiber Extraction**

The preparation of the Sisal fiber starts from the collection of the Sisal plant. For this experiment, the sisal plant leaves were collected from Werabe, Silte Zone, Southern Nations, Nationalities and Peoples Region (SNNPR), Ethiopia. After the leaves were collected from the plant by cutting them from the base, they were exposed to direct sunlight for three days. The extraction of sisal fiber from the sisal plant was done by mechanical decortication method. The fiber is extracted with the use of a knife, by securing one end of the sisal plant and slowly scrubbed through until only the

fiber remains. In this process, the fleshy pulp is removed from the fiber as well. Finally, the extracted fiber is left in the sun to dry.



Figure 3-3 Extracted Sisal Fiber

- **Sisal Fiber Treatment**

After the Sisal fiber is extracted, it was treated with a solution of 5% NaOH solution in a 1 liter distilled water. The fiber was emersed in the solution for 3 hours at in a plastic container to protect it from foreign substances, at a room temperature. This process was done twice to ensure better results. After which, the fibers were rinsed by using distilled water to remove any impurities and dried with direct sunlight.



Figure 3-4 Fiber treatment process

- **Sisal Fiber weaving**

For the purpose of weaving the Sisal fiber, a wooden frame with dimension of $30\text{cm} \times 40\text{cm}$ was made. To aid the hand weaving process, the frame was marked and nailed with 5mm gap between their successor for both weft and warp direction to create a $5\text{mm} \times 5\text{mm}$ grid.



(a)



(b)



(c)

Figure 3-5 Sisal Fabric preparation (a) Weaving frame (b) Weaving Process (c) Woven Fabric

For this research, the fibers were woven in twill weaving following the 2x2 form. The weaving was done by hand.

3.2.1 Determination of mass of fiber and matrix

Calculation of the mass of epoxy resin and sisal and glass fibers

A comprehensive analysis of current research revealed that 40wt% had the best overall performance in a number of experiments [71-75] on hybrid composites. As a result, 40% fibre by weight was selected for all composite laminates in this investigation, including hybrid composites, pure glass, and pure sisal.

For all the tests total fiber volume fraction of 40% and number of layers pf six were kept constant. The table below shows the relative percentage of constituents in the composite laminate panels. Detail calculation can be found in the Appendix.

Table 2 - Constitute of the laminates

Laminate Constitiute	Layer designation	Weight Fraction	
		Glass	Sisal
Pure Sisal	S/S/S/S/S/S	0%	40%
Hybrid	S/G/S/G/G/S	20%	20%
Pure Glass	G/G/G/G/G/G	40%	0%

3.2.3 Specimen Preparation

For this research, three composite laminates were manufactured by using the hand layup method, as it is easy to apply and cheaper. The dimensions for these laminates are 125mm × 25mm × 5mm and the number of layers is six, with the arrangement of SGSGGS for the hybrid laminates.

A mold was made for the process of manufacturing the laminates as shown in the image below. In the manufacturing process, first the bottom of the mold was wetted using resin. Then after each layer was places in the mold, the required amount of resin was added. To ensure uniform distribution of the resin, a brush and steel rollers were used. This not only ensures uniform distribution of the resin, but also removes trapped air.

As per the ASTM D5528-13 standard, a Teflon film was used to initiate crack on the specimens. After the third layer was placed in the mold and the required amount of resin was applied, a Teflon

film of 50cm length and 125cm width was put on one edge of the specimen. After this the fourth and other layers were placed in the mold and the required amount of resin was applied.

After the impregnation process was the curing process, where a 5Mpa compression pressure was applied on the mold to remove any excess resin and air gaps.

The hand layup process was used to manufacture the specimens due to its simplicity and cost-effectiveness. Layers of woven sisal and/or glass fibers were manually placed into a mold and impregnated with epoxy resin in this method. Despite careful processing, the manual nature of the technique may introduce certain limitations. Specifically, non-uniform fiber distribution may occur which leads to some variation in local fiber volume fraction and resin content. In addition, the process is prone to the formation of air voids, which act as a site for stress concentrators and could negatively impact mechanical properties. The use of a Teflon film as a pre-crack initiator may also result in slight variations in pre-crack length and quality across specimens, even though it is effectiveness. These limitations are acknowledged as potential contributors to variability in the experimental results.



Figure 3-6 Mold manufacturing



Figure 3-7 Laminate manufacturing



Figure 3-8 Manufactured Laminates

Specimen cutting

For this research, the specimens were cut from the fabricated laminate by following the standard ASTM D5528-13. To aid in the cutting, hacksaw was used, and was done with great care to ensure precision. The specimens were cut into a specimen of $25\text{mm} \times 125\text{mm}$.

After the specimens were cut, piano hinges were glued to each side of the specimen. The piano hinges are $20\text{mm} \times 20\text{mm}$. And the glue needs to be strong enough to support the load that will be introduced by the machine. To make sure that the glue will not break in test, an extra wire was introduced to enhance the strength. These are important for the experiment, because the Universal Testing Machine is going to be grabbing these hinges to introduce the load.



Figure 3-9 Test specimens after being cut



Figure 3-10 Glue for piano hinge

3.3. Test Procedure and Experimental Setup

The purpose of this research is to experimentally evaluate the interlaminar fracture toughness of the three composite laminates (i.e., pure sisal, pure glass, and sisal and glass hybrid) in a mode-I tensile test. To achieve this the specimens were subjected to a 1 mm/min crosshead speed by using Universal Testing Machine. The piano hinges were secured to the crosshead, and the propagation of the crack was recorded by using a camera. The load vs displacement graph and data was collected from the machine using a computer.

Table 3 - Specifications of a universal testing machine used for the test

Model	WDW-100S
Max. test load	100kN
Accuracy	±0.5%
Effective measuring range	0.2%-100% of Full capacity
Speed range	0.001-1000mm/min
Resolution	0.006um



Figure 3-11 Test Specimen Setup

Chapter IV

4. Result and Discussion

This chapter presents the findings from the Mode-I interlaminar fracture toughness tests conducted on pure sisal, pure glass, and hybrid sisal-glass composites. The results include the load-displacement curves, strain energy release rates (GIC), and fracture characterization. The results are discussed in detail to highlight the differences in performance between the composites and to understand the impact of hybridization on improving fracture toughness.

4.1. Load-Displacement Curves

The load vs displacement graph of the conducted interlaminar fracture test had been obtained from the machine. The graph for each specimen of the three laminates has been presented below.

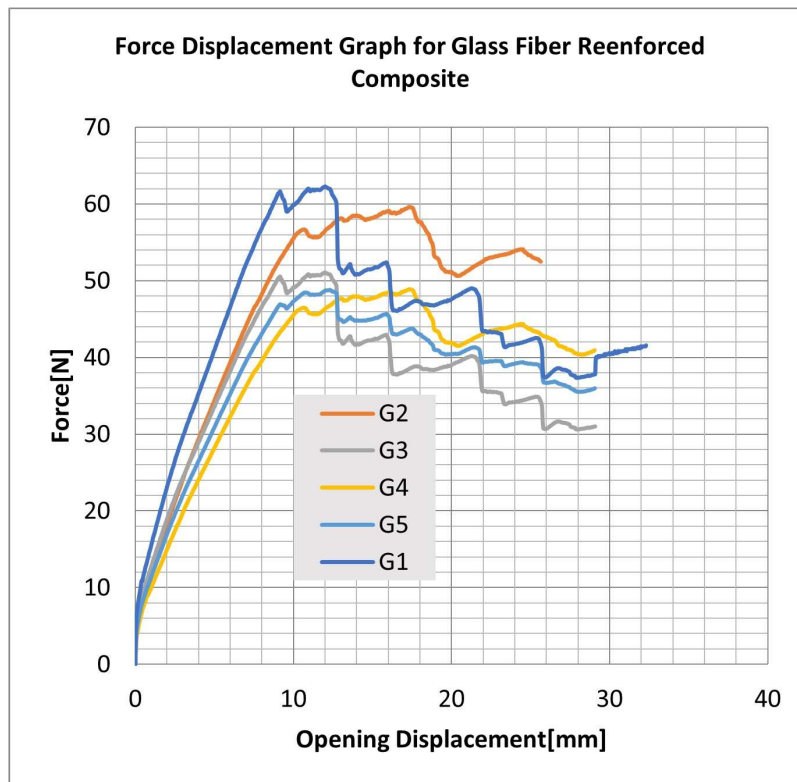


Figure 4-1 Load Vs Displacement graph for Pure Glass

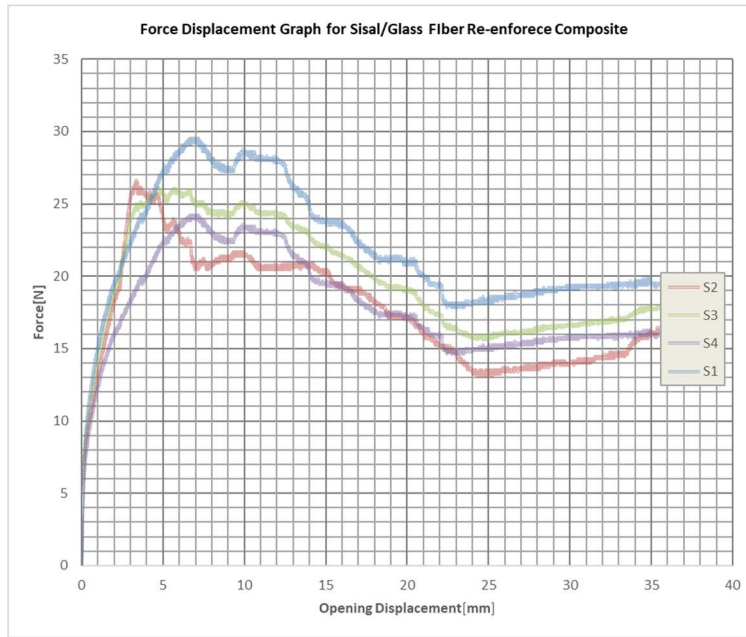


Figure 4-2 Load Vs Displacement Graph for Hybrid Laminates

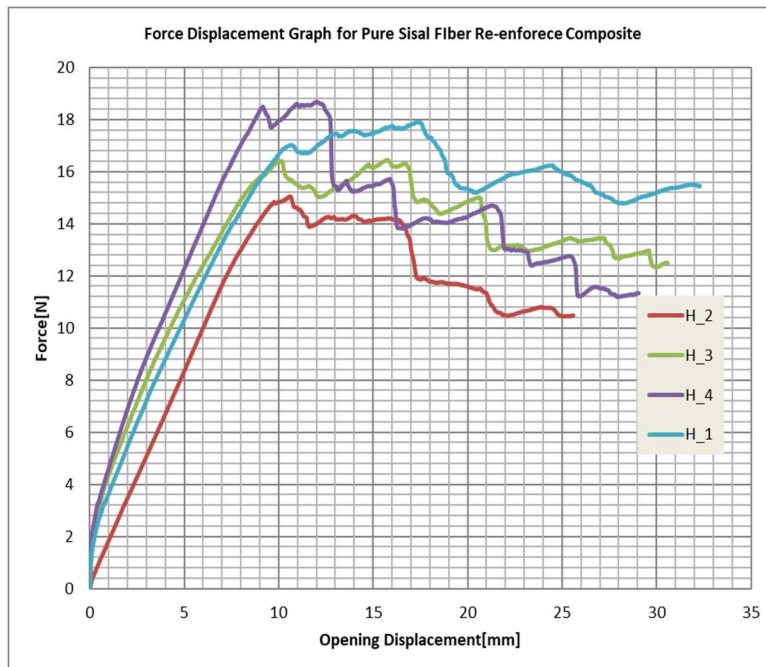


Figure 4-3 Load Vs Displacement Graph for Pure Sisal Laminates

Three types of composites were tested: pure sisal, hybrid sisal-glass, and pure glass. By analyzing the load-displacement curves, these insights of how each material behaves under tensile load are obtained:

Elastic region:

In this region, the deformation is elastic and the laminate is not damaged permanently, which is depicted by a linear relationship between load and displacement.

The pure glass composite has the steepest slope, which suggests that it is the highest stiffness among the three, followed by the hybrid and then the pure sisal composite.

Ultimate load and cracking behavior:

In this context, the ultimate load is the maximum load each specimen can bear before significant cracking occurs (the maximum load before leaving the elastic region). From the curves, the pure glass composite demonstrates the highest ultimate load, which aligns with its expected mechanical strength.

The hybrid composite on the other hand, shows intermediate behavior, achieving an ultimate load which is higher than pure sisal but lower than pure glass. This shows that the hybridization effectively boosts the performance of the sisal fibers.

Once the crack initiates, an uncontrolled propagation phase is entered, particularly in the sisal and hybrid composites. This is due to the matrix's inability to arrest the crack growth. However, for the hybrid composite some delay of this phase when compared to pure sisal, implying improved toughness.

The results of the experiments have shown some variability in the measured load vs displacement values. Which can be attributed to the limitations of the hand layup fabrication process. The Variations in the fiber alignment and their distribution, as well as the presence of voids, could contribute to the local inconsistencies in mechanical performances. In addition to that, some minor discrepancies in the pre-crack initiation that arise from the manual insertion of Teflon films may have influenced the initiation and propagation behavior of cracks during testing. These manufacturing variabilities were evident in the occurrence of inconsistent data points. As a result of this one specimen from the pure sisal and hybrid groups was excluded from the final analysis to ensure a consistent dataset. Overall, while the methodology produced repeatable results, these factors underscore the importance of improved fabrication techniques for more consistent performance.

4.2. Strain Energy Release Rate (G_{IC})

To compute the G_{IC} value, the Modified Beam Theory (MBT) was used as a data reduction method, as it is the most common and recommended method for data reduction[43]. To calculate G_{IC}, the Compliance value was calculated by using the ASTM D5528-13 standard.

$$G_I = \frac{3P}{2b(a+|\Delta|)} \text{ (eq. 4.1)}$$

$$C = \frac{\delta}{P} \dots \dots \text{ (eq. 4.2)}$$

After which the Δ was calculated from the delamination value (‘a’) recorded from visual observation during the test, Vs C^{1/3} graph.

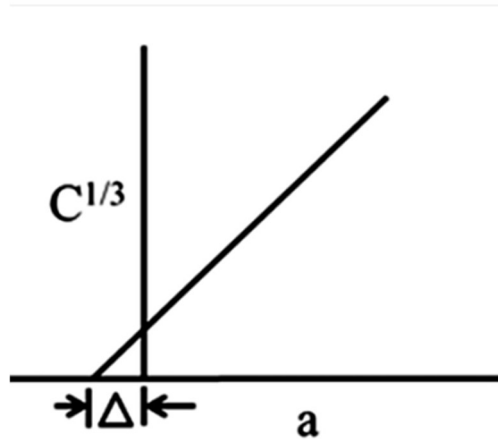


Figure 4-4 Determination of Δ

After Δ was calculated for each laminate, the G_{IC} value was calculated for four points.

Table 4 - Energy release rate value summary

	G _{IC} (VIC)	G _{IC} Propagation	SD	Margin of Error (95% confidence level)
Sisal	166.25	204.71	7.32	±3.14%
Hybrid	176.37	214.03	6.78	±2.97%
Glass	301.1	343.82	6.08	±2.32%

The strain energy release rate (G_{IC}) data provides a more quantitative measure of the toughness:

G_{IC} Initiation:

As expected, the pure glass composite has the highest G_{IC} initiation value of 301.1 J/m², indicating a higher resistance to crack initiation. Next to that, it is observed that the hybrid composite again performs better than pure sisal, with a G_{IC} initiation value of 176.37 J/m², compared to 166.25 J/m² for pure sisal, reflecting the benefits of combining synthetic and natural fibers.

G_{IC} Propagation:

The propagation values for G_{IC} are significantly higher for pure glass (343.82 J/m²), followed by the hybrid composite (214.03 J/m²), and finally, pure sisal (204.71 J/m²). This implies that once a crack starts, the glass fiber's high stiffness and energy-absorbing capacity slow down the propagation compared to the other two.

To ensure the consistency of the data, one outlier specimen was excluded from the pure sisal and the hybrid composites each from the analysis because of the inconsistency in the hand layup manufacturing. After these adjustments, the margin of errors was calculated to be approximately 3.14% for pure sisal, 2.97% for the hybrid composite, and 2.32% for pure glass, underscoring the robustness of the dataset.

The observed significant difference in the results between pure glass and the other composites can be attributed to the superior mechanical properties of glass fibers which includes higher stiffness, strength, and energy absorption capacity. Glass fibers provide excellent reinforcement, leading to higher interlaminar fracture toughness. In contrast, sisal fibers have lower stiffness and strength, resulting in lower fracture toughness. The hybrid composite shows intermediate values, benefiting from the combination of glass fiber's high strength and sisal fiber's flexibility.

Comparison of Results to Previous Literatures

The table below shows the interlaminar fracture toughness values reported in previous works on hybrid natural synthetic fiber composites. The results discussed in this research agrees well with these literatures. Moreover, the processing methods and material proportions employed in the

experiment are validated by their agreement with previous findings, reinforcing that our composite system is a viable solution for sustainable, cost-effective applications.

Table 5 - Review of interlaminar fracture toughness values

Material	Condition	G _{IC} value	Reference
Glass Fiber/Epoxy Composites	Room temperature	0.2 - 0.3 kJ/m ²	[58]
Glass Fiber/Epoxy Composites	Cryogenic temperatures (77 K & 4 K)	Decreased G _{IC} values	[57]
Flax Fiber/Epoxy Composites	Interleaved with flax yarns	Up to 0.5 kJ/m ²	[56]
Glass Fiber/Epoxy Composites	0 wt% milled glass fiber fillers	0.4 kJ/m ²	[52]
Flax Fiber/Epoxy Composites	Stitched with flax yarns	1 kJ/m ²	[55]

R-Curve

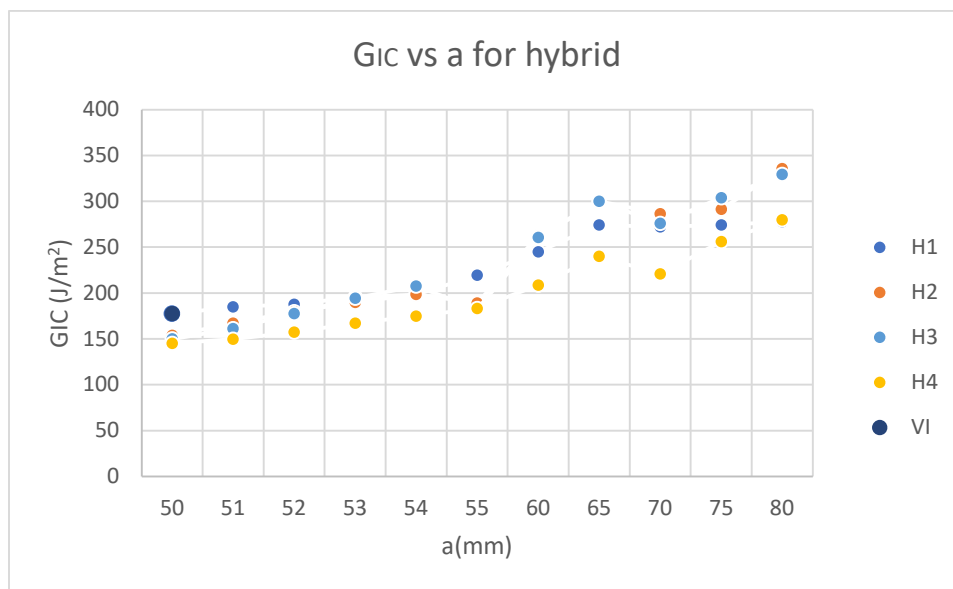


Figure 4-5 R Curve for Hybrid

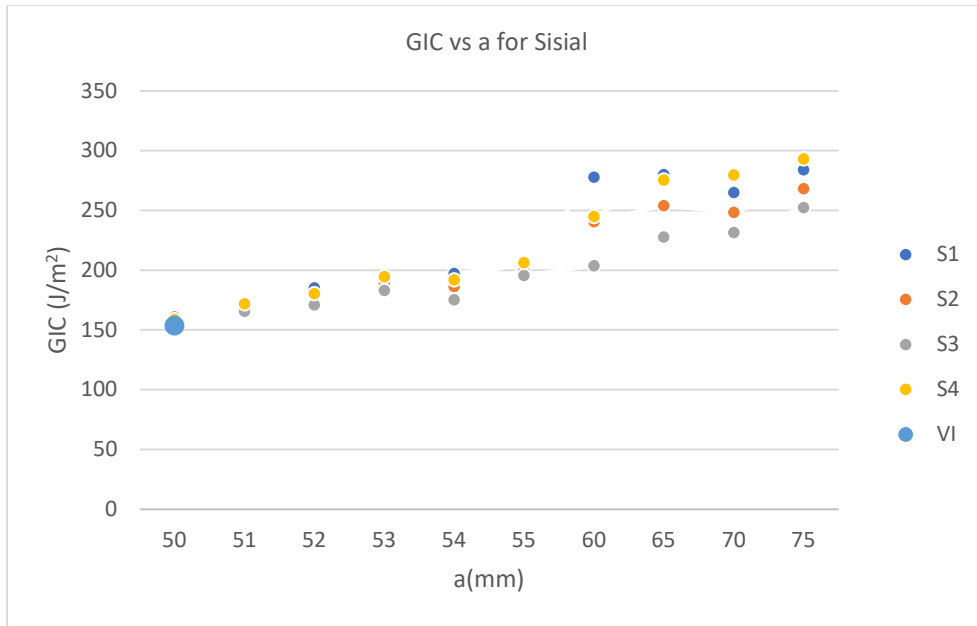


Figure 4-6 R-Curve for Pure Sisal Laminate

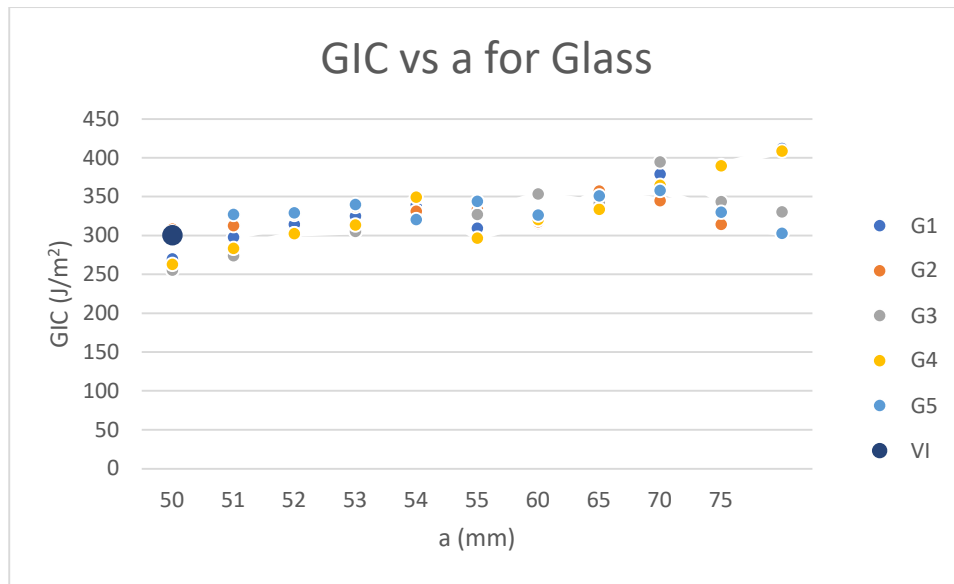


Figure 4-7 R-Curve for Glass Laminate

The interlaminar fracture toughness for the glass composite displayed an increasing trend with increasing crack length. This behavior indicates that the glass-reinforced laminate exhibits a high capacity to absorb energy during crack propagation, which contributes to enhanced toughness. The consistent and upward trend of G_{IC} values suggests that the glass fibers provide excellent reinforcement, allowing the composite to resist delamination effectively. The stable rise in fracture

toughness implies a strong fiber-matrix interaction, which is crucial for applications that demand high structural integrity and resistance to fracture.

The hybrid composite, consisting of both glass and sisal fibers, demonstrated moderate values of fracture toughness. The trend showed a gradual increase in G_{IC} with crack length, highlighting the balance between the properties of synthetic glass fibers and natural sisal fibers. The glass fibers contribute to higher fracture toughness, while the sisal fibers provide improved flexibility and a degree of toughness that lies between pure glass and pure sisal laminates. This balance makes the hybrid composite suitable for applications that require a compromise between stiffness, toughness, and cost-effectiveness.

The sisal composite exhibited relatively low interlaminar fracture toughness compared to the glass and hybrid composites. The trend of G_{IC} versus crack length was more irregular, indicating variability inherent in natural fibers like sisal.

4.3.Fracture characterization

After the experiment was done, the specimens were opened all the way through in order to study the crack propagation behavior of the specimens. Representative specimens from each of the three laminates has been displayed in the figure below.



Figure 4-8 Pure Sisal reinforced composite after complete fracture

Pure Sisal Fiber Reinforced Composite

From the above image of the pure sisal fiber composite, several key fractographic features can be observed. This includes a substantial fiber fracture, which is an indication of a weaker fiber-matrix bonding, that is likely due to the low interfacial strength between the sisal fibers and the epoxy matrix. The fracture surface also reveals exposed fiber ends which suggests that the energy absorption during crack propagation primarily occurred through matrices cracking rather than fiber fracture. In addition to this, the presence of multiple crack paths within the matrix shows brittle failure, which is a characteristic arising from the inherent brittleness of the matrix and also the insufficient stress transfer to the reinforcement fibers. Also, the visible voids and the signs of debonding suggest that there might be an incomplete bonding or a poor wettability between the fibers and the matrix, which in turn contributes to the reduction in mechanical strength and fracture toughness. In a nut shell, the fracture behavior of pure sisal fiber composite shows that the energy dissipation mechanism relies more on matrix cracking rather than cohesive fiber fracture, resulting in lower interlaminar fracture toughness value when compared to glass fiber composites.



Figure 4-9 Pure glass fiber reinforced composite after complete fracture

Pure Glass Fiber Reinforced Composite

From the above figure, it is evidently shows that there are less number of fiber breakage seen in pure glass fiber reinforced composite (Figure 29) than sisal fiber composites (Figure 28), attributing to stronger bonding of glass fibers with the epoxy matrix leading to the phenomenon of cohesive fiber breakage rather than interfacial failure. Fractured surface also shows evidence of fiber fracture, which is a characteristic of good fiber-matrix adhesion, which shows that the stress is effectively transferred by the glass fibers to the matrix thus improving the fracture resistance. Besides, the low void content also means a higher compatibility of the glass fibers with epoxies, which can lead to homogeneous stress transfer and better mechanical performance under fracture loading. Overall, the glass fiber composite is characterized by higher interlaminar fracture toughness due to the strong bonding between the glass fibers and epoxy, enabling energy dissipation through fiber breakage rather than debonding or pull-out.



Figure 4-10 Hybrid composite after complete fracture

Sisal-Glass Hybrid Reinforced Composite

For the sisal-glass hybrid reinforced composite, the figure shows a combination of sisal fiber pull-out and glass fiber fracture implying that the bonding in the composite is variable as damage is seen to take place predominantly with the sisal fiber pulling out whereas the glass fibers break. This particular failure mode is mixed, which is due to the bond strength of both fiber types with the epoxy matrix. This same hybrid architecture uses the brittle fracture of the glass fibers together with the additional toughening via ductile pull-out of the sisal fibers to potentially achieve significant overall improvements in toughness. Furthermore, some regions exhibit interfacial delamination which is also a result of the difference in the bonding nature of fibers with the epoxy which acts as an energy dissipation mechanism thereby increasing fracture toughness further compared to pure sisal composites. In general, the sisal-glass hybrid composite showcases the synergistic fracture mechanisms of the two fibers in overcoming the weakness inherent in both fiber types, hence increasing fracture toughness, thus a balanced composite and efficient option that offers better toughness than pure sisal composites while maintaining some cost and weight advantages over pure glass composites.

For each specimen, the fractographic analysis consistently revealed that failure occurs predominantly through fiber breakage with minimal evidence of pull-out, thereby confirming strong fiber–matrix adhesion and validating the quantitative G_{IC} measurements as a true reflection of the energy dissipation mechanisms at work during crack propagation.

Chapter V

5. Conclusion and Recommendation

5.1. Conclusion

In this experimental research, the effect of glass hybridization on the interlaminar fracture toughness has been investigated using Double Cantilever Beam Specimens has been carried out. All process starting from the specimen manufacturing up to result analysis and discussion has been performed as per ASTM D 5528 – 01 standards. For this experiment three different types of laminates have been evaluated (i.e., Pure Sisal, Pure Glass, and Sisal Glass Hybrid laminates). And for each type of laminate, five specimens were manufactured and tested as per the standard. The Modified Beam Theory is the selected data reduction techniques, as it is both recommended by the standard and literature. A 50 mm long initial delamination has been introduced to the specimens during the manufacturing process by the use of Teflon film. As a mechanism for load introduction, piano hinges were used, and were glued to the specimens by using high task glue. For the test, a Universal Testing Machine has been utilized. Once the specimens were fixed on the machine, the crossheads were moving at 1mm/s rate.

From the test, the load vs displacement data was gathered as well as crack propagation data was collected from visual inspection during the test. After the test was done, the data was used to calculate the critical energy release rate, resistance curve, and the load-displacement graphs were plotted.

From the result, a typical load-displacement graph was found over the tests. This is desirable as it shows its repeatability and reliability. In addition, the shape and value of the load and displacement were close to the literatures reviewed. The maximum load before uncontrolled fracture of the glass laminates was the highest of all, followed by the hybrid, and finally the pure sisal. This outcome is expected as Glass fiber has superior mechanical properties than sisal.

The critical energy release rate (G_{IC}) values of pure glass, hybrid, and pure sisal fiber reinforced epoxy composites were 343.82, 214.03, and 204.71 J/m², respectively.

Despite the lower toughness values, the sisal laminate offers benefits in terms of biodegradability, lightweight properties, and cost-effectiveness. The behavior observed in the G_{IC} values suggests that while sisal fibers provide some resistance to crack propagation, their performance is limited

by the lower stiffness and strength of natural fibers compared to synthetic ones. This composite is ideal for applications where sustainability and economic considerations are prioritized over high fracture toughness. The hybrid laminate result shows an improvement in the toughness value, proving to be a good balance between a higher fracture toughness, less costly, and environmentally friendly composite.

In conclusion, the experimental investigation has demonstrated that pure glass composites exhibit the highest interlaminar fracture toughness, while the hybrid sisal/glass composites offer a significant improvement over pure sisal laminates. These results directly address the initial research gap by confirming that the integration of glass fibers with sisal fibers not only enhances fracture resistance but also provides a sustainable, cost-effective alternative for structural applications. This study thereby validates hybridization as a viable strategy to overcome the inherent limitations of natural fibers, reinforcing its potential to achieve a balance between superior mechanical performance and environmental sustainability.

5.2. Recommendation

As evident in the comparison between the three laminates, the glass composite clearly has the highest fracture toughness indicating its high mechanical strength and good resistance to crack propagation under demanding applications. On the opposite side the hybrid composite offers a balanced combination of properties so it can be used in applications requiring mechanical performance at a lower price point. Finally, the sisal composite provides a sustainable and pocket-friendly alternative, although at the cost of low toughness that can be utilized in non-structural applications or for less-environmentally friendly impact applications.

Trends in the fracture toughness data highlight the need to select a composite material based on the application requirements. Each composite reveal particular asset in particular use such as Glass composite with constant strength, hybrid composite with balance property and sisal composite with eco-friendly property.

5.3. Future Works

In this research, the effect of hybridization on the Mode I interlaminar fracture toughness of 2x2 twill woven composites has been experimentally tested and discussed. For future work that are related to this topic, one could consider the following suggestions:

- Effect of Fiber Volume Fraction on Interlaminar Fracture Toughness
- Impact of Weaving Architecture and Fiber Orientation on the fracture toughness of the composite
- A full characterization of interlaminar fracture of sisal fiber reinforced laminate, under Mode2, and Mode3 loading.

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Appendix

A. Fiber and matrix volume fraction

The density of a material is defined as the mass of the material per unit volume.

$$\rho = \frac{w}{v} \quad \text{Equation 1}$$

Hence, we can rewrite it as:

$$w = \rho * v \quad \text{Equation 2}$$

The total mass of a composite (w_c) is the sum of the mass of the reinforcement (w_f) and the mass of the matrix (w_m):

$$w_c = w_f + w_m \quad \text{Equation 3}$$

The mass fraction is characterised by the ratio of the weight of the reinforcement/matrix to the composite. And the sum is always 1.

$$W_f + W_m = 1 \quad \text{Equation 4}$$

The mass fraction of the reinforcement:

$$W_f = \frac{w_f}{w_c} \quad \text{Equation 5}$$

The mass fraction of the matrix:

$$W_m = \frac{w_m}{w_c} \quad \text{Equation 6}$$

For a given volume of the fiber (v_f) and matrix (v_m) the total mass of the composite (v_c) is given by

$$v_c = v_f + v_m \quad \text{Equation 4}$$

The volume fraction of the fiber:

$$V_f = \frac{v_f}{v_c} \quad \text{Equation 5}$$

The mass fraction of the matrix:

$$V_m = \frac{v_m}{v_c} \quad \text{Equation 6}$$

The sum of the mass fractions is

$$V_f + V_m = 1 \quad \text{Equation 7}$$

Equation (3.6) and (3.7) can be rewritten in terms of mass and volume fraction as

$$W_f = \frac{\rho_f}{\rho_c} V_f \text{ and } W_m = \frac{\rho_m}{\rho_c} V_m \quad \text{Equation 8}$$

Or in terms of individual constituent

$$W_f = \frac{\frac{\rho_f}{\rho_m} V_f}{\frac{\rho_f}{\rho_m} V_f + V_m} \text{ and } W_m = \frac{1}{\frac{\rho_f}{\rho_m} (1 - V_m) + V_m} V_m \quad \text{Equation 9}$$

Density

From Equation (3.8) the density of a composite can be calculated as:

$$\rho_c v_c = \rho_f v_f + \rho_m v_m \quad \text{Equation 10}$$

The density of the composite is:

$$\rho_c = \rho_f V_f + \rho_m V_m \quad \text{Equation 11}$$

In terms of weight fractions of the fiber and the matrix:

$$\frac{1}{\rho_c} = \frac{W_f}{\rho_f} + \frac{W_m}{\rho_m} \quad \text{Equation 12}$$

The volume of each laminate is $125\text{mm} \times 125\text{mm} \times 5\text{mm}$.

By using ASTM D3800 standard testing the average density of glass and sisal fibers found to be 2.57 g/cm^3 and 1.45 g/cm^3 respectively. The densities of the sisal, and glass fibers found through test were acceptable in reference to the prior literature.

The density of sisal is taken to be 1.45 g/cm^3 [16]

The density of glass fiber is taken to be 2.57 g/cm^3

And the density of epoxy is taken to be 1.17 g/cm^3

Pure Glass

$$\rho_f = 2.57 \text{ g/cm}^3$$

$$\rho_m = 1.17 \text{ g/cm}^3$$

$$v_c = 15.625 \text{ cm}^3$$

$$w_f = 0.4w_c$$

$$w_m = 0.6w_c$$

From Equation 2 we have:

$$w_f = \rho_f * v_f$$

$$w_f = 2.57 * v_f$$

Similarly,

$$w_m = 1.17 * v_m$$

But from Equation 7 we have

$$w_m = 1.17 * (15.625 - v_f)$$

From Equation 15 we can find the density of the composite

$$\frac{1}{\rho_c} = \frac{0.4}{2.57} + \frac{0.6}{1.17}$$

$$\rho_c = 1.496 \text{ g/cm}^3$$

$$\text{Hence, } w_c = 23.375g$$

Finally, from Equation 3:

$$w_c = 2.57v_f + 1.17(15.625 - v_f)$$

$$23.375 = 2.57v_f + 18.28 - 1.17v_f$$

$$v_f = 3.64 \text{ cm}^3 \text{ and } v_m = 11.985 \text{ cm}^3$$

$$w_f = 9.35g \text{ and } w_m = 14.02g$$

Pure Sisal

$$v_f = 5.62 \text{ cm}^3 \text{ and } v_m = 10.005 \text{ cm}^3$$

$$w_f = 7.86g \text{ and } w_m = 11.74g$$

Hybrid

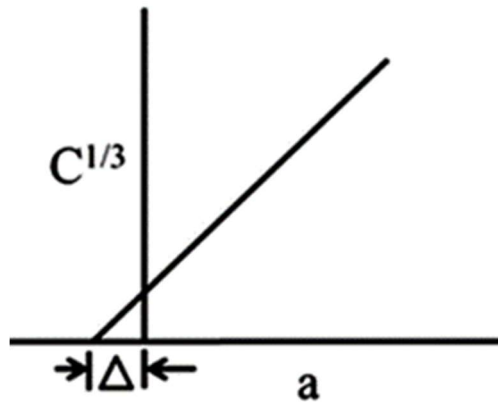
$$v_g = 1.77\text{cm}^3 \quad v_s = 2.98\text{cm}^3 \text{ and } v_m = 10.85\text{cm}^3$$

$$w_g = 4.26\text{g} = w_s \text{ and } w_m = 12.81\text{g}$$

B. Calculation of Δ

Δ is found by finding the x - intercept of the line a vs. $C^{1/3}$. Where a is the delamination length observed, and C can be found by using:

$$C = \frac{\delta}{P}$$



Here is a summary table for finding Δ for 1 sample of Glass reinforced specimen:

Points from initial ($a=a_0 + \delta a$) (mm)		δ (mm)	P(N)	δ/a	C	$C^{1/3}$
a_0	50	9	61	0.18	0.147541	0.52841
$a_0 + \delta a$	51	10.2	60.5	0.2	0.168595	0.552436
$a_0 + \delta a$	52	10.9	60.9	0.209615385	0.178982	0.563555
$a_0 + \delta a$	53	11.5	60.75	0.216981132	0.1893	0.574183
$a_0 + \delta a$	54	12.3	60.4	0.227777778	0.203642	0.588332
$a_0 + \delta a$	55	13	53	0.236363636	0.245283	0.625973
$a_0 + \delta a$	60	15	51	0.25	0.294118	0.665029

$a_0 + \delta a$	65	18.3	48	0.281538462	0.38125	0.725109
$a_0 + \delta a$	70	21.3	50	0.304285714	0.426	0.752437
$a_0 + \delta a$	75	22.998	51	0.30664	0.450941	0.766843
$a_0 + \delta a$	80	25.361	52	0.3170125	0.487712	0.787144