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A Study of the Interlaminar Fracture Toughness of Unidirectional Flax/Epoxy Composites

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Abstract: Having environmental and economic advantages, flax fibers have been recognized as a potential replacement for glass fibers as reinforcement in epoxy composites for various applications. Its widening applications require employing failure criteria and analysis methods for engineering design, analysis, and optimization of this material. Among different failure modes, delamination is known as one of the earliest ones in laminated composites and needs to be studied in detail. However, the delamination characteristics of unidirectional (UD) flax/epoxy composites in pure Mode I has rarely been addressed, while Mode II and Mixed-mode I/II have never been addressed before. This work studies and evaluates the interlaminar fracture toughness and delamination behavior of UD flax/epoxy composite under Mode I, Mode II, and Mixed-mode I/II loading. The composites were tested following corresponding ASTM standards and fulfilled all the requirements. The interlaminar fracture toughness of the composite were determined and validated based on the specific characteristics of natural fibers. Considering the variation in the composite structure configuration and its effects, the results of interlaminar fracture toughness fit in the range of those reported for similar composites in the literature and provide a basis for the material properties of this composite.

Keywords: flax-epoxy composite; interlaminar fracture energy; fracture toughness; delamination; Mode I; Mode II and Mixed-mode I–II interlaminar fracture; critical energy release rate

1. Introduction

In the recent past decades since the 1990s, the increasing public environmental awareness and commencement of new legislation are demanding more sustainable and ecologically efficient products [1–7]. Synthetic glass and carbon fiber-reinforced polymer composites (FRPCs) have high performance and are extensively applied in different industrial sectors, but they are not environment-friendly [7–9]; thus, environmentally sustainable replacements are required. Numerous research efforts have been dedicated to finding a sustainable substitute for synthetic FRPCs, and have confirmed that plant-based natural fiber-reinforced polymer composites (NFRPCs) are the most promising alternatives (to petroleum-based FRPCs) for many applications [2,3,5,10–17]. Having many advantages over man-made FRPCs such as improved sustainability and competitive properties at a lower cost, NFRPCs have attracted much attention worldwide and have penetrated the composites world with a rapidly extending industrial applications from packaging to structural construction components [1,6,13,16,18–21]. Replacing the engineered FRPCs with fully biodegradable

21]. Replacing the engineered FRPCs with fully biodegradable composites is not yet an economically and mechanically desirable solution in many applications. Nevertheless, the synthetic polymers reinforced with natural fibers to produce economic and partially biodegradable composites can cover the requirements of a wide variety of applications and have been a viable alternative for petroleum-based FRPCs [5,7,22]. For example, flax, hemp, and ramie fibers, placed within the category of plant-based bast fibers, are the most widely used reinforcement in NFRPCs, offering comparable mechanical properties to glass fiber-reinforced polymer composites (GFRPCs) [16,19,23–25]. Among them, flax fibers have excellent performance, and many manufacturers are interested in using them in NFRPCs for various applications [8,12,17,26–36]. Also, epoxy resin, a thermoset polymer matrix, offers high mechanical performance and durability and is the most commonly used matrix to produce flax fiber-reinforced polymer composites (FFRPCs) [29]. Furthermore, the application of a unidirectional (UD) or optimally woven reinforcement structure is a primary factor in maximizing the structural performance of FFRPCs [39,38], therefore, UD flax fiber-reinforced epoxy composites (FFRECs) are of outstanding importance and are used ubiquitously in the industry.

There are, however, some downsides to extending their application field, for instance, workpiece defects in the machining process, required for complex components, and in-service degradation when exposed to a humid (harsh) environment [6,28]. Accordingly, FFRECs have been the subject of several pieces of research, trying to explore their properties and enhance their performance and durability to resolve these drawbacks, aiming at facilitating their applications [2,29,32,39–42].

One of the commonly observed and major damage phenomena in laminated composites is the separation of two adjacent laminae (plies), which is known as interlaminar failure or delamination damage. This failure mode is the primary challenge for the laminated FRPCs that generally affects the in-service functionality of the component by reducing its overall stiffness and load-bearing capacity and subsequently leading to the failure of the entire component [43–45]. It might form through the production process and exist as a defect in the composite, occur under different loadings and environmental conditions, or incur during machining operations, particularly drilling [46–48]. In fracture mechanics, the interlaminar failure, according to the relative displacement of crack surfaces, has been classified into three fracture modes: opening or tensile mode (Mode I), in-plane shear or sliding mode (Mode II), and out-of-plane shear or tearing mode (Mode III) [43,49]. Figure 1 [49] illustrates the crack propagation caused by different modes of failure and the corresponding loadings acting on the cracks. Delamination can initiate and propagate due to any of these fracture modes or any combinations of them (mixed modes).

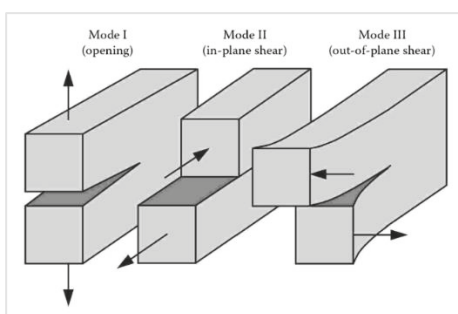


Figure 1. Fracture modes and applied loadings [49].

The resistance of the material to crack propagation, or to delamination damages in composite laminates, is known as interlaminar fracture toughness (IFE) [49]. The Dineen Elastic Fracture Mechanics (ELEM) approach, initially developed by isotropic materials, has been successfully adopted and applied as a predictive approach to the IFE of the laminated FRPCs [70,51]. A fracture FRPCs [50,51] ELEM assumes an elastic material behavior for the whole body, except for a certain damage zone around the crack tip (which is a provision for the validity of the results). The ELEM has a fundamental material property and can be determined based on either the stress intensity factor (K_{IC}) or the Critical Strain Energy Release Rate or the Critical Strain Energy Release Rate (G_c) of the material; the latter is commonly named Interlaminar Fracture Energy (IFE). They are, respectively, a measure of the stress intensity level and

Rate (G_C) of the material; the latter is commonly named Interlaminar Fracture Energy (IFE). They are, respectively, a measure of the stress intensity level and energy required for an increment of the crack growth [49]. For the isotropic, homogeneous, and relatively brittle metallic materials, IFT depends on the energy dissipation at the crack tip and can be characterized by K_C . However, for FRPCs, the stress field in the vicinity of the crack tip is more complicated, and IFT is affected by the result of energy losses of different failure mechanisms such as matrix cracking, fiber fracture, fiber pullout, and fiber-matrix debonding [50,52]. Therefore, G_C is commonly used for the determination of IFT and the study of delamination in FRPC laminates for different failure modes [44,52–55]. By comparing the energy release rate (G) for a loading case to the G_C of the material, the capability of the body to resist crack extension can be assessed.

Ever-growing applications of laminated NFRPCs and their penetration into structural applications, as well as the vital role of delamination in these composites, necessitate the investigation of the interlaminar fracture behavior and determination of IFE of these materials. IFT, as a primary property of a material, is required for engineering design purposes as well as numerical analysis methods such as the Finite Element Method (FEM). The interlaminar fracture of the synthetic FRPCs under different modes of failure has been extensively investigated, and its state of the art has been comprehensively reviewed by many authors [43,44,50,52,56]. However, in the literature, limited work has been documented addressing the interlaminar fracture behavior of the laminated NFRPCs, particularly FFRECs that show better toughness [57]. Wong et al. [58] studied the influence of polymer modification on IFT of a randomly oriented-flax/poly(L-lactic acid) composite and noted that by adding hyperbranched polymers (HBPs) to the matrix, G_{IC} of the composite significantly increased: from 38.9 J/m² for the original composite to 115 J/m² for the composite with the highest volume of HBP. Investigating the properties of a hybrid flax/glass UD-fabric (twisted flax yarns) reinforced phenolic resin composite, Zhang et al. [59] measured $G_{IC} = 550$ J/m² for flax composite that increased by hybridization to 560 J/m² for [(flax/ glass)_n]_s composite. Their study shows that adding off-center plies of different fibers to the laminate will change the IFT, although the delamination is happening between the same type of composite plies. Li et al. [60] investigated the effect of fiber surface treatment on the fracture properties of sisal-fabric/vinyl-ester composites with a fiber volume fraction (V_f) of around 32% ($V_f \approx 0.32$) and observed significant improvements in fracture energies of Mode I (G_{IC}) and small increases in that of Mode II (G_{IIC}) in comparison to the untreated system. While G_{IC} of the permanganate-treated fiber experienced the most significant improvement. The authors followed ASTM D5528 and measured the G_{IC} values of 320.5/1158.7 [888.0/1682.2] J/m², and G_{IIC} values of 2141.0 [2384.7] J/m² for the crack initiation/propagation of untreated and [permanganate-treated] sisal composites. In an experimental study, Chen et al. [45] investigated the potential of carbon nanotube buckypaper interleaf for improving the IFT of flax-fabric/phenolic resin composites with $V_f \approx 0.73$. They followed ASTM D5528 and tested some partially and fully interleaved laminates at the mid-plane as well as laminates without interleaf for IFT. They observed a 26% [22%] and 29% [51%] increase in initiation G_{IC} , and [propagation G_{IC}], respectively, for partially and fully interleaved composite in comparison to those of the original one (≈ 4.4 and [≈ 5.6] kJ/m²) and related them to the extensive favorable fibrillations observed in the interleaved laminates. Trying to enhance the IFT of flax-fabric/epoxy composite of $V_f = 0.60$, Li et al. [61] interleaved them at the mid-plane with chopped flax yarns of different lengths and content and followed ASTM D5528 to evaluate their G_{IC} . The authors observed a 4% to 31% improvement in the propagation G_{IC} , with the maximum increase for the moderate length and content of the chopped yarns, compared to that of the original composite, $G_{IC} = 1.40$ kJ/m². It should be noted that they have used Modified Compliance Calibration method but have stated that used Modified Beam Theory (MBT). Ravandi et al. [62–64] conducted experimental investigations, according to ASTM D5528, and numerical analysis, using FEM, to study and predict the effects of through-the-thickness stitching and reinforcement architecture on the G_{IC} of the UD ($V_f \approx 0.31$) and woven fabric ($V_f \approx 0.40$) FFRECs. The authors tested double cantilever beams (DCBs), according to ASTM D5528, to determine the G_{IC} of the FFRECs as well as UD-glass/epoxy

($V_f = 0.60$) composites, and observed that average G_{IC} of the UD-glass/epoxy was less than half of that of unstitched UD-FFRECs ($G_{IC} \approx 1.3 \text{ kJ/m}^2$). Also, their findings showed that the average G_{IC} value of the UD-FFREC was three times lower than that of woven-FFRECs ($G_{IC} \approx 3.2 \text{ kJ/m}^2$), and generally, stitching with flax yarns induced continuous improvement in IFT of the laminates, increasing with stitch areal fraction to a maximum of 21% for the highest investigated level (of aerial fraction). Besides, they reported that their FEM results agreed with the experimental ones giving the G_{IC} values of 0.771 and 1.25 kJ/m^2 , respectively, for the crack initiation and propagation of UD-FFRECs as well as 0.671 kJ/m^2 for the G_{IC} value of glass/epoxy composite. Bensadoun et al. [57] tested some FFRECs reinforced with seven different fiber configurations; i.e., one plain weave, two twill weaves (low/high twist), a twisted-yarn-fabric cross-ply and an untwisted-fiber cross-ply architecture (cross-ply laminates were tested in both $0^\circ/90^\circ$ directions), to investigate the effect of the reinforcement architectures on the G_{IC} and G_{IIC} of the composites with $V_f = 0.40$. In terms of delamination fracture, their composites with untwisted-fiber cross-ply reinforcement denoted as UD [90,0], has a $0^\circ/0^\circ$ ply interface at the mid-plane of the laminate and is the closest one to the UD-FFRECs assessed in the current study. They reported initiation G_{IC} values between 457 J/m^2 for plain woven and 777 J/m^2 for twisted-yarn-fabric cross-ply composite when tested in [90,0] direction, as well as $G_{IC} = 496 \text{ J/m}^2$ for UD [90,0] tests. While the propagation values of G_{IC} vary from 663 J/m^2 for UD [90,0] to 1597 J/m^2 for the low-twist twill-woven composites. Besides, they obtained G_{IIC} values between 728 J/m^2 for UD [90,0] and 1872 J/m^2 for plain weave laminates. The authors named the cross-ply twisted-yarn-fabric (with 90% of yarns in longitudinal direction and 10% along the transverse direction) and the UD-untwisted-fiber architecture respectively “quasi-UD” and “UD” architecture, whereas, their interlaminar fracture behavior could be different with those of UD laminates, where all plies lay in one direction. Therefore, the results may not coincide exactly with the results of UD laminates. In two different works, Almansour et al. reported their findings of the effect of water absorption on the G_{IC} [47] and G_{IIC} [48] of the woven flax ($V_f = 0.31$) and woven flax/basalt (basalt plies in the skin of the laminates and overall $V_f = 0.33$) reinforced vinyl-ester composites in $[\pm 45^\circ]$ arrangement. Their DCB test results showed that upon immersion in water, the initiation and propagation G_{IC} of the flax/vinyl-ester, as well as the initiation G_{IC} of flax/basalt hybrid composites, decreased by 27%, 10%, and 23%, respectively. In contrast, the propagation G_{IC} of flax/basalt hybrid composites increased by 15%. They have documented the average initiation/propagation G_{IC} values of 3870/12093 J/m^2 , and 4431/9738 J/m^2 , respectively, for flax and flax-basalt hybrid composites in dry condition. The concerning issue in this study is that the crack did not propagate in the mid-plane of the laminate and deviated from that by crossing the layers; it thus cannot be considered a pure interlaminar failure. Along with the high G_{IC} value of vinyl-ester (410 J/m^2 compared with 69–150 J/m^2 for epoxy [57]), this might be another reason for higher measured values of IFT. Also, in spite of the increase in initiation G_{IC} , it seems that for the flax/basalt hybrid composites, the shorter final deviated-crack growth combined with the ever-increasing R-curve resulted in a smaller propagation G_{IC} . Whereas, comparing the propagation G_{IC} associated with the last crack length exhibited by the hybrid composites with that of flax composite, an increase in propagation G_{IC} can be observed. In terms of G_{IIC} values, testing end-notched-flexure (ENF) specimens, the authors observed that hybridization by basalt fibers (adding basalt composite plies to the skin of the flax laminates) improved the initiation G_{IIC} value of flax composite by 58%; also, moisture absorption improved the initiation G_{IIC} of flax composite by 29% and that of hybrid composite by 20%, due to improved matrix ductility. Their published average initiation G_{IIC} values for the dry flax and flax/basalt hybrid composites are 253 J/m^2 , and 400 J/m^2 , respectively. Nevertheless, considering that the basalt plies are located at the surface of the laminate, while the IFT is being evaluated between flax plies at the mid-plane, the basalt layers have the same function that the stiffeners (bonded as tabs to the skin of DCB and ENF test specimens to avoid large deformations and specimens arm failure) used by some researchers measuring the IFT of flax composites have [57,62–65]. Accordingly, the findings of these studies show that the stiffeners affect the values of both Mode I and Mode II IFT, which contradicts the assumption of these researchers. As a result, stiffening the arms of the DCB and ENF specimens

will affect the IFT values and is not a proper solution, instead, increasing the thickness or changing the initial crack length to improve the specimen stiffness seems desirable, as recommended by the corresponding ASTM standards [53,54]. Trying to improve the IFT of UD-flax/gliadin composites, Vo Hong et al. [65] investigated the influences of some processing conditions, fiber surface treatment, and matrix plasticization on IFT by fabricating and testing flax/gliadin composite laminates with $V_f = 0.40$. Testing the original composite DCB specimens according to ISO 15024 standard, the authors reported $G_{IC} = 50$ to 100 J/m^2 and $G_{IIC} = 450$ to 550 J/m^2 , respectively, for crack initiation and propagation measurements. Their findings show that the optimum value of IFT, $G_{IC} \approx 1000 \text{ J/m}^2$, was obtained with a combination of all of the investigated processing and materials parameters, i.e., fiber treatment, adding glycerol to the matrix, and medium cooling rate. Rajendran et al. [66] investigated the behavior of twill-weave flax/epoxy composites in Mode I and Mode II delamination (no V_f reported). The authors tested DCB and ENF specimens and, following the experimental calibration method (ECM), calculated a $G_{IC} = 485 \text{ J/m}^2$ and $G_{IIC} = 962 \text{ J/m}^2$ for the composite. In another report [67], they published their findings of the Mode I, Mode II, and Mixed-mode I/II interlaminar fracture toughness of the same composite with $V_f \approx 0.44$. In this work, they followed ASTM D5528 and D7905 standards and tested standardized DCB and ENF specimens to determine the IFT in Mode I and Mode II, respectively, while, they tested single-leg-bending (SLB) specimens, which is not standardized, to measure the Mixed-mode I/II fracture toughness ($G_{(III)C}$). Their obtained values in this recent report are $G_{IC} = 363 \text{ J/m}^2$, $G_{IIC} = 962 \text{ J/m}^2$ and $G_{(III)C} = 649 \text{ J/m}^2$ (for a $G_{II} / (G_I + G_{II}) = 0.43$), which shows a discrepancy in G_{IC} value for the same material from two different reports. In a recent study, Saidane et al. [68] used the acoustic emission (AE) method to investigate the Mode I fracture toughness of flax, glass, and hybrid flax/glass woven-fiber/epoxy composites, flax laminates had a $V_f = 0.40$. They conducted DCB tests according to ASTM D5528 standard but used AE to detect the onset of delamination and the corresponding critical load to calculate G_{IC} . The initiation G_{IC} values of 1079 J/m^2 , 945 J/m^2 and 923 J/m^2 were determined respectively for flax, hybrid flax/glass and glass composites. Nevertheless, the method used in this work to detect the delamination initiation and the corresponding critical load used in calculation of G_{IC} influences the obtained values, thus, this fact should be considered when using the results for comparison purposes. The interesting point in this study is that the propagation G_{IC} value of flax composites (R-curve) continuously increased with delamination length which contradicts the findings of previously cited studies, including those of Zhang et al. [59] for the similar materials which show a stabilized plateau value after a certain crack length. Also, their findings for hybrid flax/glass composites regarding the behavior of R-curves and the decrease in G_{IC} is in contrast with those of Zhang et al. [59] for the similar materials.

As summarized above, the literature review shows that the previous works addressing the IFT of flax composites have mostly studied flax-fabric reinforced composites, while few researchers investigated UD-FFRECs and only in Mode I [62]. Also, to the knowledge of the authors, no study has, in particular, investigated their delamination behavior in Mode II and Mixed-mode I/II. Therefore, the damage tolerance and behavior knowledge of UD-FFRECs, as the basic building block of the FFRECs, particularly in Mode II and Mixed-mode I/II are missing. This is of paramount importance in using flax/epoxy composites in recently demanded structural applications as well as employing widely-used and reliability-proven FEM numerical analysis method to study this environmentally and economically-advantageous and ubiquitously used material. Consequently, an in-depth understanding of their delamination behavior in different modes is essential both for improving their material properties and for generating demanded knowledge and mechanical properties in the engineering design field and for future studies employing analytic and numerical resolutions. Because of the variations in the fabrication processes, origins and properties of raw materials, and the mechanical properties evaluation methods of these composites, the use of well-defined and precisely controlled uniform composites as well as standard test methods is a pre-requisite to achieve reliable results.

The aim here is to investigate the interlaminar fracture behavior of the UD-FFRECs in three different modes of failure, namely, Mode I, Mode II, and Mixed-mode I/II and determine the corresponding

interlaminar fracture toughness. For this purpose, UD-FFRECs are fabricated using identical raw materials and processing techniques with a precisely controlled fiber volume fraction. The commonly used ASTM standard test procedures are followed to evaluate the composites under different modes of loading to determine their IFT and produce valid and reliable data by respecting the defined validity criteria.

2. Materials and Methods

2.1. Material System and Test Specimen Preparation

Unidirectional tapes of flax fibers (Eco-Technik, AXA TAP 200 100 (from ECHO, Liqville, France; <https://eco-technik.fr>)) with a areal density of 200 g/200² were cut in 300×300×300 dimensions and stacked to form the UD UD reinforcement preform in Figure 2a. The thermosetting Marine 820 Epoxy System, mixed with 18 Wt.% Marine 824 hardener (from ADITECH® Plastic Systems, Madison Heights, MI, USA, www.axson-technologies.com), was used to impregnate the fibers employing the resin transfer molding (RTM) process. The number of layers is [0]₁₂ layer and the thickness of laminates were regularly checked according ASTM D3017 procedure and controlled to a result of V_f of 41% for all composite plates and quarter thickness of the plates of the ASTM test plates regarding the regular thickness of test specimens. In order to produce a pre-crack in the initiation site for delamination in a film of thick polytetrafluorethylene (PTFE) film was attached to the mid-plane of the laminate in the longitudinal direction of the preform. The preform was placed in the mold and the resin was injected into the mold. The resin was cured in the mold. The resulting pre-cracked laminate is shown in Figure 2b.

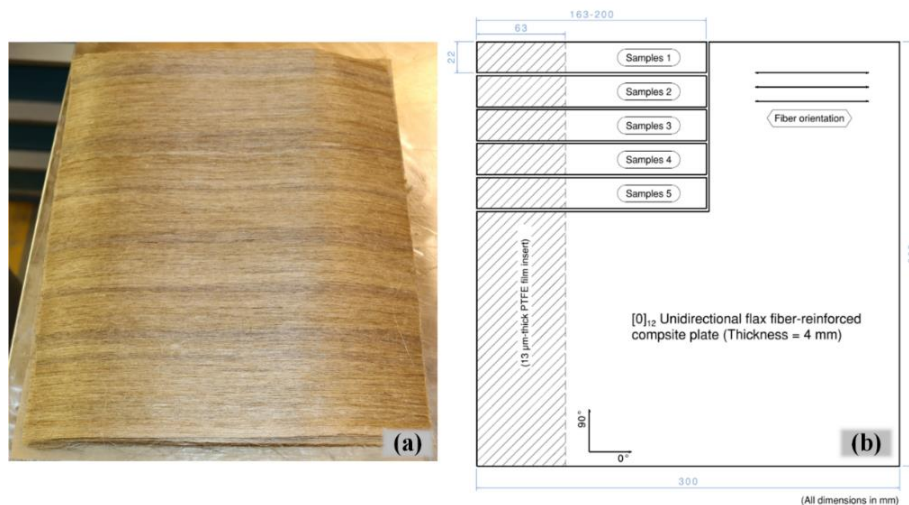


Figure 2. (a) Unidirectional flax fiber preform and (b) configuration of flax/epoxy pre-cracked laminate.

After fabrication and post-curing of the composite laminate, the PTFE film was located via X-ray imaging and the laminate was trimmed to reach the dimensions of the insert that is recommended by the ASTM standard as shown in Figure 1b. Next, the rectangular UD laminated composite specimens with uniform thickness and thickness containing a PTFE film and PTFE film attached to the mid-plane were cut to their desired dimensions by a diamond/90° tool in DM/90/LO/015, DINGAO, loading with a high density with high density parallel to the plates, as illustrated in Figure 2b.

2.2. Mechanical Testing

Three different types of fracture tests were conducted to evaluate the interlaminar fracture toughness of the composite in pure Mode I and Mode II as well as in Mixed-mode I/II loading conditions. Numerous test methods and specimen configurations have been developed over the years to evaluate the IFT of the laminated FRPCs; however, only some of them have been standardized [43,69]. In this study, the most commonly used standardized test methods by ASTM will be employed. All of the tests were carried out at ambient conditions, and the specimens were exposed to the test conditions at least 48 hours before the experiments. The details of the corresponding specimens, test procedures, and data reduction methods are described as follows.

2.2.1. Mode I Test: Delamination Fracture Toughness

In this study, the most commonly used standardized test methods by ASTM will be employed. All of the tests were carried out at ambient conditions, and the specimens were exposed to the test conditions at least 48 hours before the experiments. The details of the corresponding specimens, test procedures, and data reduction methods are described as follows.

2.2.1. Mode I Interlaminar Fracture Toughness

Following the ASTM D5528 standard test method, DCB specimens were prepared and tested to determine the Mode I interlaminar fracture toughness of the composite. The thickness, h , and initial delamination length, a_0 , of the composite laminate were designed to fulfill the requirements of the standard. After cutting the test coupons to their desired dimensions, two piano hinges were bonded to the delaminated arms of the DCB specimen to introduce the tensile opening load and induce a Mode I delamination fracture; the configuration and nominal dimensions of the DCB specimen are illustrated in Figure 3a. To facilitate the detection of the onset and propagation of the crack, the side edge of the specimens was coated with white correction fluid, and the approximate location of the pre-crack tip and required final crack length were marked, as shown in Figure 3b. The DCB tests were conducted on an MTS Landmark 370 hydraulic machine equipped with a 1 kN-capacity load cell, and MTS 647/10 hydraulic grips. The specimens were loaded and unloaded, respectively, at a constant crosshead speed of 3 and 25 mm/min in two loading/unloading cycles. The initial loading was stopped after a crack growth of 3 to 5 mm, and the specimens were unloaded; this was a procedure to create a natural Mode I pre-crack in the DCB specimens. They were then reloaded until the delamination crack (a) propagated past 50 mm from the tip of pre-crack, and then they were unloaded.

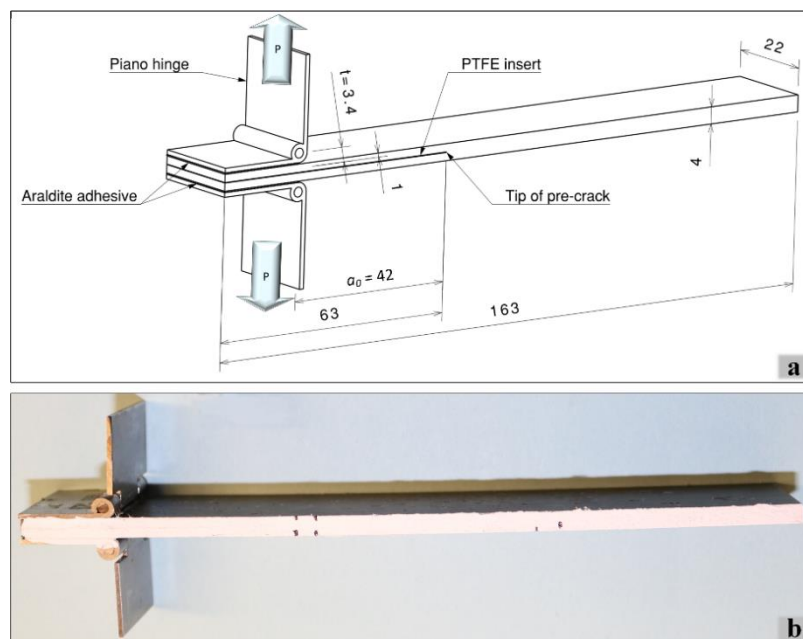


Figure 3. Double Cantilever Beam (DCB) specimen: (a) configuration and dimensions, (b) final specimen.

A Digital Image Correlation (DIC) system was employed to observe and track the delamination front; the test setup is shown in Figure 4. DIC is an optical non-contact deformation measurement technique [70,71] with adequate performance, low cost, and handy experimental setup that has been used in many research fields for strain analysis studies, including crack initiation [72,73]. The principle of DIC and theoretical basis for the calculation of strains using consecutive images has been reported by many researchers and is available in the literature [74–76]. The recorded images were synchronized with the load and displacement signals of the MTS machine; the crack opening displacement (COPD) was estimated as the crosshead displacement, and the data was recorded for the entire loading/unloading cycles. Then, the registered information was processed to associate the load and displacement values at the intervals of delamination growth defined by the standard. These values were used to calculate G_c for different delamination lengths and to form the delamination resistance curves (R-curves). Eight DCB specimens were tested to obtain at least five valid tests to determine G_c of the composite. The piano hinges were extended with a bracket to avoid the interference that occurred

values, has been recommended. In this study, MBT was implemented to calculate G_{IC} values based on the load versus displacement curve and the data was recorded for the entire loading/unloading cycles. Then, the registered information was processed to associate the load and displacement values at the intervals of delamination growth defined by the standard. These values were used to calculate G_{IC} for different delamination lengths and to form the delamination resistance curves (R-curves). Eight DCB specimens were tested to obtain at least five valid tests to determine G_{IC} of the composite, where P is the applied load, δ is the COD on the load line, b is the width of the specimen, h represents the delamination crack length, and Δ is the crack length correction factor. Δ can be determined experimentally as the x-intercept of linear least squares plot of C^2 versus delamination length, where C is the compliance of the specimen ($C = \delta/P$). G_{IC} is the IFT of the composite, associated with critical load (P_C) determined as follows.

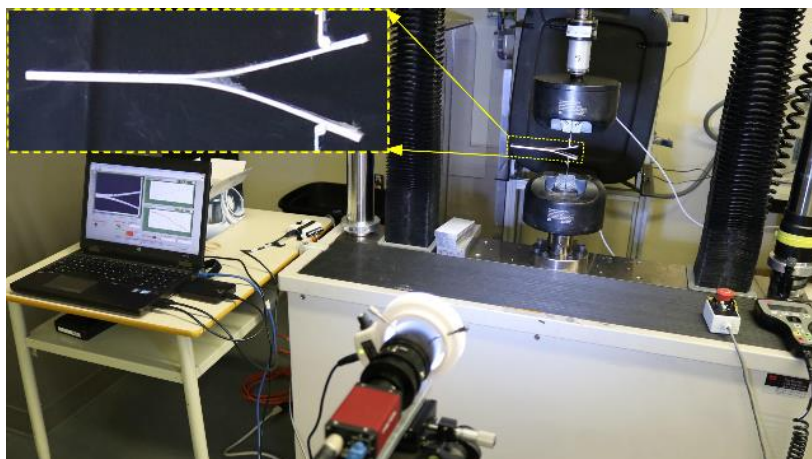


Figure 4. Mode I delamination test setup with a DIC system to follow crack propagation.

The crack initiation data recorded on the load-displacement curve are defined as follows: G_{IC}^{NL} is the value of G_{IC} at the deviation from linearity point in the load-displacement curve (NL), when delamination is visually observed (VIS) and at 5%-increased-compliance point or the maximum load, as illustrated in Section 3.1. The NL G_{IC} is the lowest and recommended for damage tolerance analysis, and the first propagation value of G_{IC} is known as Mode I pre-crack G_{IC} [53]. Concerning deflections of the specimen arms due to relatively low flexural modulus of the FFREC, the values of h and a_0 were calculated according to the recommendations outlined in the ASTM standard and based on the elastic properties of the composite, leading to a shorter $a_0 = 42$ mm compared to $a_0 = 50$ mm, recommended for synthetic FRPCs. Nevertheless, for some delamination lengths, the ratio of the load-line displacement to delamination length was higher than 0.4 ($\delta/a_0 > 0.4$). Consequently, according to the standard, a large-displacement correction factor (F) was calculated using Equation (2) and incorporated in the data reduction method by multiplying F by G_{IC} values calculated using Equation (1):

The crack initiation value of G_{IC} (G_{IC}^{ini}) was determined using three definitions presented in the standard, namely, at the deviation from linearity point in the load-displacement curve (NL), when delamination is visually observed (VIS) and at 5%-increased-compliance point or the maximum load, as illustrated in Section 3.1. The NL G_{IC} is the lowest and recommended for damage tolerance analysis, and the first propagation value of G_{IC} is known as Mode I pre-crack G_{IC} [53].

2.2.2. Mode II Interlaminar Fracture Toughness

Concerning deflections of the specimen arms due to relatively low flexural modulus of the FFREC, the values of h and a_0 were calculated according to the recommendations outlined in the ASTM standard and based on the elastic properties of the composite, leading to the shorter $a_0 = 42$ mm compared to $a_0 = 50$ mm (recommended for synthetic FRPCs). The only exception for some delamination lengths, for the ratio of the load-line displacement to delamination length was higher than 0.4 ($\delta/a_0 > 0.4$), mentioned previously according to the standard, a large-displacement correction factor (F) was calculated using Equation (2) and incorporated in the data reduction method by multiplying F by G_{IC} values calculated using Equation (1) for the range of delamination lengths and widths specified in the standard. Both sides of the specimens were painted with a correction liquid to help the detection of crack initiation, and the end of pre-crack, as well as the required loading points, was marked. Figure 5a illustrates the configuration and dimensions, and Figure 5b shows a picture of

and incorporated in the data reduction method by multiplying F by G_{IC} values calculated using Equation (1):

$$F = 1 - \frac{3}{10} \left(\frac{\delta}{a}\right)^2 - \frac{3}{2} \left(\frac{\delta t}{a^2}\right) \tag{2}$$

where t was measured as defined in the standard and is shown in Figure 3.

2.2.2. Mode II Interlaminar Fracture Toughness

End-notched flexure (ENF) tests were conducted following the procedure outlined in ASTM D7905 to determine the Mode II interlaminar fracture toughness, G_{IIC} , of the UD-FFRECs using the compliance calibration (CC) method, which is presented as the only acceptable data reduction method for this test [54]. The specimens were cut from the same laminates used for DCB specimens mentioned above (Figure 1) and with the same tool to the dimensions that fall within the range of allowable lengths and widths specified in the standard. Both sides of the specimens were painted with a correction liquid to help the detection of crack initiation, and the end of pre-crack, as well as the required loading points, was marked. Figure 5a illustrates the configuration and dimensions, and Figure 5b shows a picture of the ENF specimen with markings. The ENF specimens were mounted in a three-point-bending (TPB) fixture with a 100-mm-span and loaded on the same machine fitted with the 1kN-loadcell that was used for DCB specimens, as shown in Figure 6. The coupons were loaded and unloaded at 0.5 mm/min crosshead speed, and the data acquisition was performed for the entire fracture tests and the loading phase of the CC tests.

According to the standard, the non-pre-cracked (NPC) and pre-cracked (PC) CC tests, at $a_i = 20$ and 40 mm, and fracture tests, at $a_0 = 30$ mm, were carried out on the same specimen to obtain the compliance plots as well as the maximum loads ($P_{M_{max}}$), to be used for determination of G_{IIC} . The initial the ENF specimen with markings. The ENF specimens were mounted in a three-point-bending (TPB) CC forces (P_i) were approximated using the flexural modulus (E_f) of the same material [77] and G_{IIC} of a similar material [66], then, the result of ENF tests with accepted G_{IIC} was used to update the P_C and P_i . The calculated crack length after NPC tests (a_{calc}) was calculated using unloading data of the NPC fracture test, as mentioned in the test procedure.

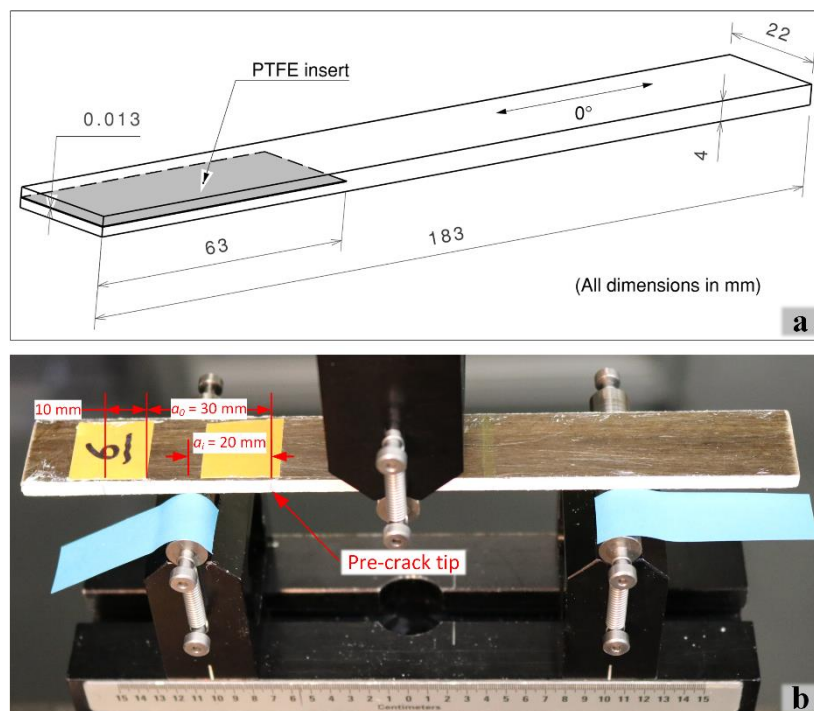


Figure 5. ENF specimen for Mode II delamination: (a) configuration and dimensions; (b) specimen and loading fixture.





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 Figure 5. ENF specimen for Mode II delamination: (a) configuration and dimensions, (b) specimen and loading fixture.

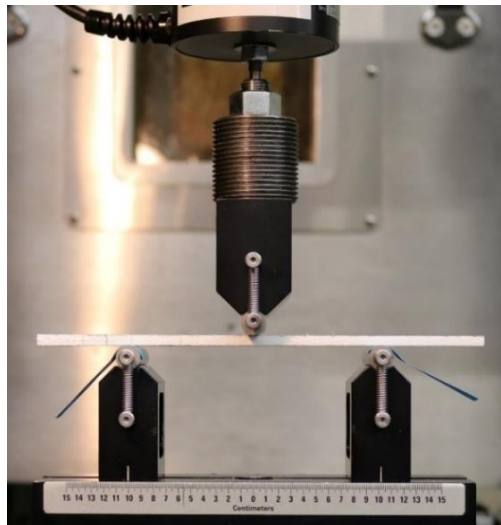


Figure 6. Mode II delamination test setup.

According to the standard NPC and PC stacked (NPC) and PC-wrapped (PC) CC tests of the and 40 mm displacement tests had a 20 mm by 20 mm linear regression (LSRA). The compliance plot as well as the maximum load (P_{Max}) and the CC coefficient (m) of Equation (3), G_{IIC} values were approximated using the flexural modulus (E_f) of the same material [77] and G_{IIC} of a similar material [66], then, the result of ENF tests with a accepted G_{IIC} was used to update the P_c and B . The calculated crack length after NPC tests (a_{calc}) was calculated using unloading data of the NPC fracture test, as mentioned in the test procedure. G_{IIC} values were calculated using Equation (4) and validated based on the criteria of the ASTM standard:

$$G_{IIC} = \frac{3mP_{Max}^2a_0^2}{2B} \quad (4)$$

where m is the CC coefficient, a_0 and P_{Max} are respectively the crack length used and the maximum force measured in the fracture test, and B is the specimen width. Six ENF specimens were tested to obtain adequate results for the calculation of G_{IIC} . The value of G_{IIC} is considered as IFT, only if it satisfies the criteria defined in the standard.

2.2.3. Mixed-Mode I/II Interlaminar Fracture Toughness

The Mixed-Mode Bending (MMB) tests were carried out according to ASTM D6671 standard test method [55] to obtain the Mixed-mode I/II fracture toughness ($G_{(I/II)C}$) of the composite. Following the guidelines of the standard, piano hinges, with drilled holes matching to the hinge clamp of the MMB test fixture, were adhesively bonded to the pre-cracked specimens cut from the same plate as for DCB and ENF specimens, to form an initial crack length of $a_0 = 28$ mm. The length of the initial crack was calculated based on the criteria defined in the standard and using the thickness, mechanical properties, and an estimated value of fracture energy, $G_{(I/II)C}^{est}$, to result in an ultimate allowable deflection. Also, the side edges of the specimens were painted white and marked. Figure 7a presents the configuration, dimensions, and load application points of the MMB specimen. Figure 7b shows the MMB apparatus with a mounted specimen and the finalized MMB specimen. Mixed-mode delamination under a mode mixture ratio of $G_{II} / (G_I + G_{II}) = 0.55$ was applied by setting the length of the lever as $c = 47.4$ mm (Figure 7b). The tests were performed at a constant crosshead speed of 0.5 mm/min on an MTS-322.31 machine equipped with a 5 kN loadcell, and the force versus displacement data was recorded. A moveable microscope was synchronized with the load-displacement data and used to observe the crack tip to detect the onset and follow the propagation of the delamination; the test setup is shown in Figure 8. The loading of the specimen was continued until the delamination extended past 25 mm. The required elastic and shear properties were used from our previous work [77], and the out-of-plane

shear modulus G_{13} was assumed to be equal to G_{12} . Also, for the calculations of the estimated force and deflection, the estimated Mixed-mode IFT, $G_{(I/II)C}^{est}$ was considered as the average values of the G_{IC} and G_{IIC} of the composite measured in this present study. Six MMB tests were carried out to achieve a minimum of five valid tests by controlling the failure mode and maximum allowable deflection of the specimens according to the criteria of the standard.

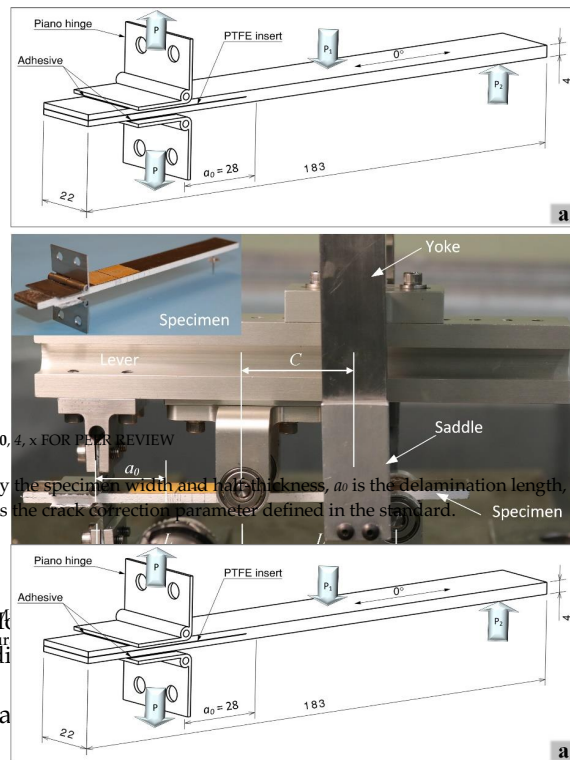


Figure 7. Mixed-Mode I/II delamination test setup (a) configuration and dimensions.

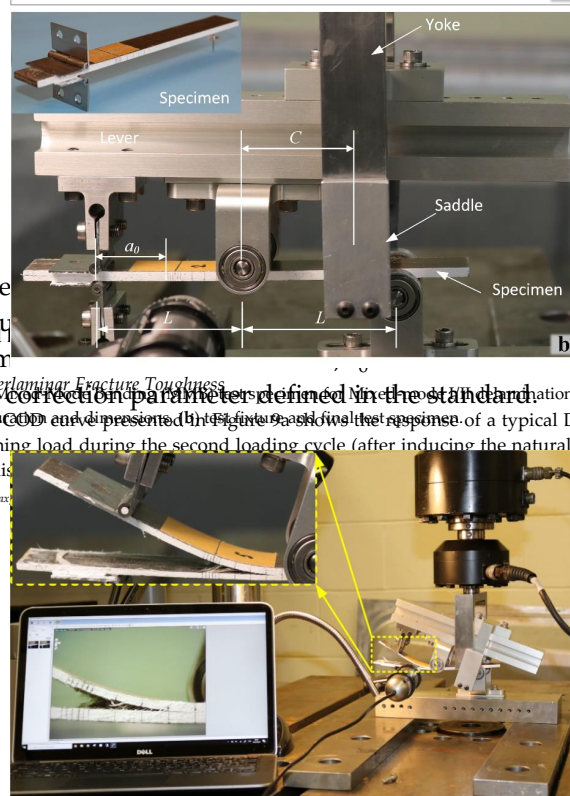


Figure 8. Mixed-mode I/II delamination test setup.

3. Results and Discussion

3.1. Mode I Interlaminar Fracture Toughness

The load-COD curve presented in Figure 9a shows the response of a typical DCB specimen to the crack opening load during the second loading cycle (after inducing the natural pre-crack in the first cycle). This curve shows that the UD-FFREC DCB specimen has a perfect linear behavior up to 95% of the P_{max} , followed by a short nonlinear curve up to the P_{max} point. Upon reaching the peak

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are respectively the specimen width and half thickness, a_0 is the delamination length, L is the half-span length, and χ is the crack correction parameter defined in the standard.

mode I/II delamination:

defined in the ASTM D6671:

$$(5)$$

$$(6)$$

$$(7)$$

where G_{IC} and G_{IIC} are the critical load, in this study are respectively the specimen length, and χ is the crack correction parameter defined in the standard.

mode IFT, respectively, P_C is the lever length, b and h are the specimen thickness and height, L is the half-span

3.1. Mode I Interlaminar Fracture Toughness
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3.1. Mode I Interlaminar Fracture Toughness

The load-COD curve presented in Figure 9a shows the response of a typical DCB specimen to the crack opening load during the second loading cycle (after inducing the natural pre-crack in the first cycle). This curve shows that the UD-FFREC DCB specimen has a perfect linear behavior up to 95% of the P_{max} , followed by a short nonlinear curve up to the P_{max} point. Upon reaching the peak point, which differs from the point at which the delamination initiates, the load drops slowly and continuously with crack propagation, indicating that stable crack propagation is happening. Figure 9b confirms unique behavior for all of the DCB DCB specimens and also shows good repeatability of the tests for a NFRPC FRPC DCB. This behavior of the DCB DCB specimens is in good agreement with the observations of Ravandi et al. [62–64] for UD FRPC DCB specimens which were stiffened by GFRP/KFRP fabric on both sides, as well as with those of Ben Abdou et al. [51] [57] for epoxy-UD (UD 0/0₄₅) FRPC laminates stiffened with fibers and those of Saidine et al. [68] for woven flax/epoxy composite. Investigating woven/vinylester DCB specimens, Almansour et al. [47] also observed an initial linear response followed by a nonlinear curve up to the peak load. However, this is in marked contrast to the observations of Rajendran et al. [66,67] for the flax fabric/epoxy DCBs, who reported a high nonlinear response up to the maximum load. Ravandi et al. [62] and Rajendran et al. [67] tested epoxy composite DCBs made of relatively similar flax fabrics supplied by the same supplier with the specifications as described in the Introduction part. However, they reported an entirely different load-displacement response and IFI values; the average G_{IC} value reported by the former ($\approx 2000 \text{ J/m}^2$) is much higher than that of the latter (363 J/m^2).

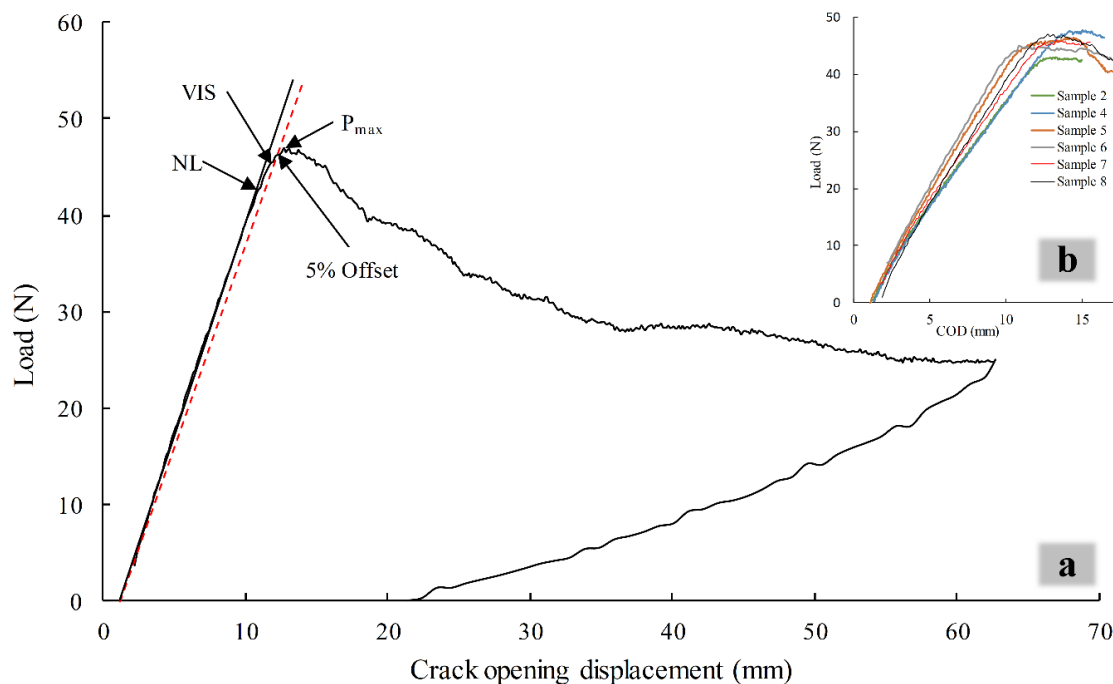


Figure 9. Load vs. crack opening displacement (COD) of the DCB specimen: (a) Typical loading/unloading cycle and initial G_{IC} value measurement points, and (b) overall initial behavior.

The calculated G_{IC} values for a typical DCB specimen are drawn as a function of delamination length (l) (Figure 10) to generate the delamination resistance curve, known as an R-curve, aimed to determine the initiation and propagation values of G_{IC} . As can be seen in Figure 10, the G_{IC} value determined at NL is the minimum followed by the VIS and 5% Offset points, which happened before the P_{max} point for all DCB tests on the load-COD curve (Figure 9a). Figure 10 shows that G_{IC} increases by delamination growth to a maximum value and then drops to stabilize at a plateau value. Several authors observed similar R-curves, and this is a commonly occurred phenomenon for UD-FRPC DCB specimens when the delamination grows parallel to the fibers between two UD plies that is attributed to the development of fiber bridging across the crack [45,53,57,62,64,78]. This resistance-type fracture behavior of UD DCB composites is an incidence of properly implantation of the delamination insert and validates the tests. The scale of the fiber bridging is much more extensive for UD-NFRPCs compared to their synthetic fiber counterparts [59,62–64,68]. The authors related this phenomenon to

similar R-curves, and this is a commonly occurred phenomenon for UD-FRPC DCB specimens when the delamination grows parallel to the fibers between two UD plies that is attributed to the development of fiber bridging across the crack [45,53,57,62,64,78]. This resistance-type fracture behavior of UD DCB composites is an incidence of properly implantation of the delamination insert and validates the tests. The scale of the fiber bridging is much more extensive for UD-NFRPCs compared to their synthetic fiber counterparts [59,62–64,68]. The authors related this phenomenon to the high strength of the longer synthetic fibers which tend to remain unbroken and have an ability where regular fibers are composed of shorter mechanical fibers with irregularity. They also believe that the higher G_{IC} value determined for UD-FRPCs compared to these UD glass epoxy composites is associated with the larger scale of fiber bridging.

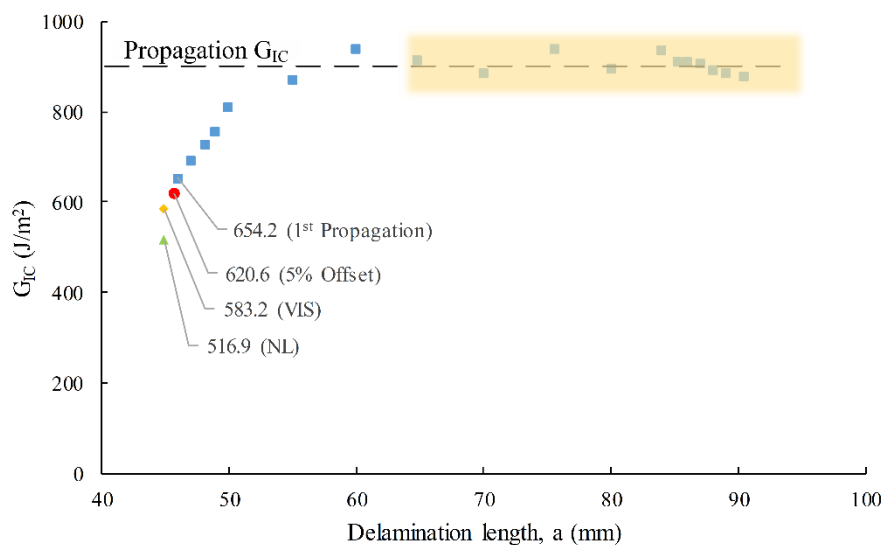


Figure 10. Crack Resistance Curve (R-Curve) of a typical UD-FFRPC DCB, the plateau region is highlighted.

The G_{IC} values, determined based on different definitions of onset of delamination, are presented in Table 1. However, combining some damage detection techniques, such as Radiography, strain gauge, and AE, with DCB tests, reveal that the delamination starts in the interior of the specimen width very close to NL point on the load-COD curve [53,68,79], and as can be seen in Figure 10, this point is far from propagation values, thus is considered as initiation G_{IC} value, as recommended by ASTM D5528. The plateau value of G_{IC} in the R-curve, the average value within the highlighted zone in Figure 10, is considered as the propagation G_{IC} value and summarized in Table 1.

Table 1. Mode I fracture toughness values for UD FFRPC.

Mode I G _{IC}	G _{IC} (NL Point)	G _{IC} (VIS Point)	G _{IC} (5%/Max)	G _{IC} (Propagation)
Average (J/m ²)	574	641	644	903
Standard deviation	38	31	26	55
Coefficient of variation (%)	6.6	4.8	4.0	6.1

The relevant DCB tests conducted for FFRPCs and their corresponding G_{IC} values available in the literature are summarized in Table 2. As can be seen in Table 2, the most similar tests have been performed by Ravandi et al. [64] for an analogous UD-FFRPCs (0₁₆)₁₆ laminate containing a similar UD reinforcement with a lower surface density (110 g/m²) compared to 200 g/m² used in the under-study 0₁₁₂ laminate. However, the obtained initiation G_{IC} value at VIS point (641 J/m²), as well as the propagation G_{IC} value (903 J/m²) in the present study, are about 20% lower than those reported by these authors. Considering that they stiffened their DCB specimens with GFRP, this difference may be explained by referring to the work of Almansour et al. [47], where replacing the out-most layers of flax fabric composites by the 4-fold stiffer basalt fiber plies, equivalent to stiffening flax composite DCBs, augmented the G_{IC} values by around 19%. Therefore, the obtained results in the current study are in excellent agreement with their results. The experiments of Bensadoun et al. [57] for the cross-

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Overall, considering the differences in reinforcements architecture/source, matrix type, processing, testing, and data reduction method, the obtained results in the present study are generally consistent with the literature data given in Table 2. Therefore, the determined and validated values for the initiation and propagation G_{IC} for UD-FFRECs can be considered as the material properties of these composites and be confidently applied in future research and engineering fields.

Table 2. Mode I interlaminar fracture toughness tests and values for FFRPCs.

	Current Study	Ravandi et al. [62]	Ravandi et al. [64]	Bensadoun et al. [57]	Bensadoun et al. [57]	Almansour et al. [47]	Rajendran et al. [66,67]	Li et al. [61]	Chen et al. [45]	Saidane et al. [68]	Zhang et al. [59]	Vo Hong et al. [65]
Reinforcement	UD-flax (200 g/m ²) [0] ₁₂	4 × 4 W-flax fabric (500 g/m ²)	UD-flax (110 g/m ²) [0] ₁₆	0°/0° flax-plyies [90,0] _{2s}	W-flax fabrics	(±45°) W-flax fabric	2 × 2 W-flax fabric (200 g/m ²)	UD-T-flax fabric (200 g/m ²)	UD-T-flax fabric	2 × 2 W-flax fabric	UD-T-flax fabric	UD-flax
Matrix	Epoxy	Epoxy	Epoxy	Epoxy	Epoxy	Vinyl ester	Epoxy	Epoxy	Phenolic resin	Epoxy	Phenolic resin	Gliadin powder
Stiffener	-	CFRP	GFRP	GFRP	GFRP	-	-	-	-	-	-	GFRP
Composite fabrication	RTM ‡	VARI †	VARI †	RTM ‡	RTM ‡	VI ±	Hand layup	CM +	VARI †	CM +	CM +	Hand layup
V _f (%)	41	31	40	40	40	31	44	60	73	40	67	40
Test/Data reduction method	ASTM D5528/MBT	ASTM D5528/MBT	ASTM D5528/MBT	ASTM D5528/MBT	ASTM D5528/MBT	ASTM D5528/MBT	CC	ASTM D5528/CC *	ASTM D5528/CC	ASTM D5528/MBT	ASTM D5528/-	ISO 15024/MBT
Damage initiation point	NL	-	VIS	NL	NL	VIS	SG detected/P _{max}	-	VIS	AE detected	-	NL
G _{IC} (J/m ²) Initiation	574	≈2000	771	496	457–754	3579	363/485	-	≈440	1079	280	50–60
G _{IC} (J/m ²) Propagation	903	≈3200	1250	663	1151–1597	11789	-	1400	≈580	≈2400	550	450–550

† Vacuum-assisted resin infusion, ‡ Resin transfer molding, ± Vacuum infusion, + Compression molding, * In the paper mentioned MTB (by mistake), AE: Acoustic Emission, W: Woven, UD: Unidirectional, T: Twisted, SG: Strain gauge, CC: Compliance Calibration, MBT: Modified Beam Theory.

3.2. Mode II Interlaminar Fracture Toughness

Figure 11 presents the load-displacement curve of a typical ENF fracture test. As can be seen, the composite shows an overall linear response with a clear and sudden load drop, from which, respectively, the compliance of the specimen and the peak load can be determined. This is a normal and known behavior exhibited by ENF where, under an in-plane shear loading, the surfaces of the mid-plane plies slide over each other and lead to unstable crack growth and sudden load drop [54]. This failure allows only the measurement of crack initiation IFT. Almansour et al. observed an analogous behavior for woven flax/vinyl-ester [48] and non-woven flax mat/vinyl-ester [83], whereas Rajendran et al. [66,67] reported a complete nonlinear load-displacement curve for woven flax/epoxy composites. The experience of the latter, likewise to their DCB tests, is in stark contrast with the observations of previously cited authors (Almansour et al.) and many other authors [84–86].

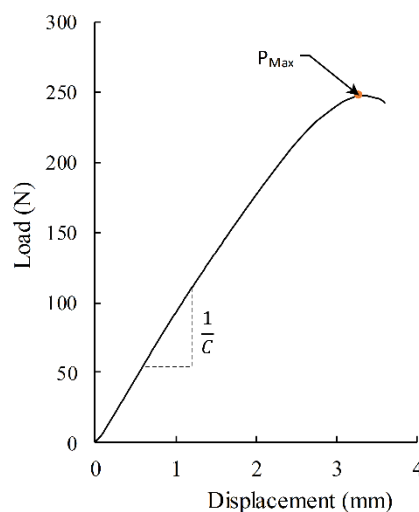


Figure 11. Load-displacement curve of ENF test and illustration of compliance (C) and Peak determination.

The Mode II interlaminar fracture energy, G_{IIC} , was calculated using the data reduction method explained in the previous section, and the averaged results of five or six NPC and NPC tests are presented in Table 3. As can be noticed, considering the variations, the averaged results are in the same range and overlapping, however, NPC results have a larger variation.

Table 3. Mode II fracture toughness values for UD FFREC.

	G_{IIC} (NPC)	G_{IIC} (NPC)	G_{IIC} (NPC)	G_{IIC} (Average)	G_{IIC} (Average)
Mean (J/m ²)	401	401	378	390	390
Standard deviation	18	18	7	18	18
Coefficient of variation (%)	4.5	4.5	1.9	4.6	4.6

There are very limited similar ENF test data available in the literature for comparison and validation purposes. In fact, there is no available ENF test being conducted and treated exactly in the same way for UD FFRECs; nevertheless, the closest ones are summarized in Table 4.

In Table 4, the obtained G_{IIC} value for the under-study UD-FFREC laminate is placed within the range of those reported for similar tests. However, it is clearly lower than that of the closest material, i.e., [90,0]_{2s} laminate with 0°/0° UD-flax/epoxy plies in the midplane [57]. This difference can not only be associated with the variation existing within the properties of natural fibers, it can also be due to the difference in the reinforcement configuration of the laminates, the GFRP tabs used for stiffening [90,0]_{2s} laminate and the different data reduction method employed for calculation of G_{IIC} . The results of Almansour et al. [48,83] show that replacing the out-most plies of the flax composite with high-stiffness basalt plies, which is equivalent to tabbing ENF specimens, augmented the G_{IIC} value of the flax composites up to 62%. Therefore, tabbing can be one of the reasons for obtaining higher value for the compared composite. In addition, for DCB tests, it is proven that alterations in the off-center plies (orientation and material) of the UD laminates with the same delamination interface layers affect G_{IIC} values [68,80,81]. There is no research result for ENF tests; however, this might be true for Mode

Table 4. Mode II interlaminar fracture toughness tests and values for FFRPCs.

	Current Study	Bensadoun et al. [57]	Almansour et al. [48]	Almansour et al. [83]	Rajendran et al. [67]	
Reinforcement	UD-flax (200 g/m ²)	0°//0° UD-flax plies in [90,0] _{2s}	W-flax fabrics	(±45°) W-flax fabric	Non-woven flax mat	W-flax fabric (200 g/m ²)
Matrix Stiffener	Epoxy -	Epoxy GFRP	Epoxy GFRP	vinyl ester -/basalt	vinyl ester -/basalt	Epoxy -
Composite fabrication	RTM	RTM	RTM	VARTM	Hand layup + CM	Hand layup
Fiber content	V _f = 0.41	V _f = 0.40	V _f = 0.40	100 Wt.%	V _f = 0.23	V _f = 0.44
Test /Data reduction method	ASTM D7905/CC/SBT	SBT	SBT	SBT	SBT	CC
G _{IIC} (J/m ²)	378/612	728	1315-1872	266/430	1940/2173	962

UD: Unidirectional, W: Woven, RTM: Resin transfer molding, VARTM: Vacuum-assisted resin transfer molding, CM: Compression molding, SBT: Classical Simple Beam Theory, CC: Compliance Calibration.

In Table 4, the obtained G_{IIC} value for the under-study UD-FFREC laminate is placed within the range of those reported for similar tests. However, it is clearly lower than that of the closest material, i.e., [90,0]_{2s} laminate with 0°//0° UD-flax/epoxy plies in the midplane [57]. This difference can not only be associated with the variation existing within the properties of natural fibers, it can also be due to the difference in the reinforcement configuration of the laminates, the GFRP tabs used for stiffening [90,0]_{2s} laminate and the different data reduction method employed for calculation of G_{IIC}. The results of Almansour et al. [48,83] show that replacing the out-most plies of the flax composite with high-stiffness basalt plies, which is equivalent to tabbing ENF specimens, augmented the G_{IIC} value of the flax composites up to 62%. Therefore, tabbing can be one of the reasons for obtaining higher value for the compared composite. In addition, for DCB tests, it is proven that alterations in the off-center plies (orientation and material) of the UD laminates with the same delamination interface layers affect G_{IIC} values [68,80,81]. There is no research result for ENF tests; however, this might be true for Mode II delamination, as well. Furthermore, the ASTM D7905 standard is followed in the current study; however, Bensadoun et al. used the classical simple beam theory method (SBT). While using different data reduction methods yields variations in the obtained G_{IIC} values, for instance, applying the classical SBT used in their study to the test data of the current study, a higher value (612 J/m²) is obtained, Table 4. The findings of some authors [57,87] revealed that woven reinforcements result in higher G_{IIC} values compared to UD ones; thus, the difference between the G_{IIC} values of UD laminates and those of woven-fabric reinforced composites given in Table 4 is reasonable. Some authors believe that normally G_{IIC} values are higher than G_{IC} [57,88], whereas some observed the opposite [47,48,67], and some believe that both can be true for laminated composites [89]. As listed in Tables 3 and 4, depending on the applied data reduction method, both cases can happen for the UD-FFREC. For the most similar instance ([90,0]_{2s} laminate) discussed above, using ASTM D7905 standard for G_{IC}, and SBT for G_{IIC} results in the same trend.

3.3. Mode II Interlaminar Fracture Toughness

The mixed-mode loading behavior of the composite is plotted in Figure 12. For clarity, a single curve specimen is presented in Figure 12a and shows that in general, MMB specimens of FFREC exhibit an initial linear behavior followed by a nonlinear curve up to the peak load, after which, a plateau type curve with gradual load drop can be seen till the end of the test. Having plotted the curves of all 5 MMB specimens in Figure 12b, it can be seen that they exhibit a similar and consistent response with acceptable repeatability for the natural fiber composites.

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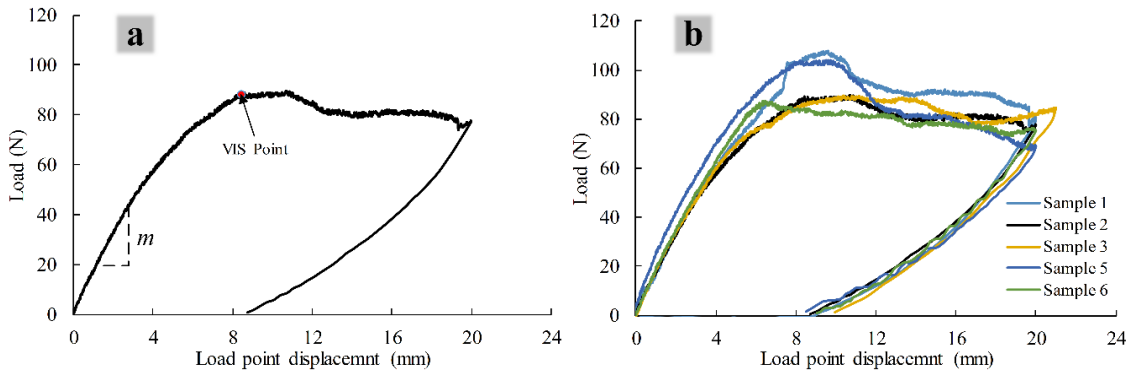


Figure 12. Load-displacement curves of MMB tests: (a) determination of m and P_C , and (b) all curves of the tests.

The slope of the load-displacement curve (m) and the critical load (P_C) need to be determined for the calculation of mixed-mode I/II fracture energy of the composite. It was directly calculated from the linear part of the curves, as shown in Figure 12a. However, there are no definite instructions for the standard NLE, VLS, VIF, 5% and 5% points as defined for calculations of $G_{I/II}$ and the standard deviation should be used. The largest horizontal primary part of the load-displacement curve and the 5% offset points are used for the peak of the curve, which is that the yield stress to be the failure point and using the corresponding load and displacement result as the estimated fracture energy. On the other hand, the VLS and P_C points, it was observed that the failure happened before the peak load. This was also observed by Rajendran et al. [67], Rajendran strain (ϵ) gauge to use at the gauge of failure. Accordingly, the VLS point is considered as the point of delamination. Based on the associated load on this point, it is found that this point for all the tests, the maximum deflection was small deflection, which was the deflection that was determined based on the criterion of the standard deviation and they were validated.

The obtained average mixed-mode I/II fracture energy value was $G_{(I/II)C} = 414 \text{ J/m}^2$ with a 15% coefficient of variation. The variation of results seems relatively high, however, considering the nature of the fiber composite, the complexity of this test, the subjective visual solution for detecting the initiation of failure is reasonable. Based on the knowledge of MMB tests reported by Rajendran et al. [67] and the data literature for flax/epoxy composites, these values are considered to be in the range of $G_{(I/II)C}$ (400–600 J/m²) for woven flax/epoxy composites. Comparison of this result with the literature shows that the obtained value is in the range of the previous studies. It should be noted that the used data for the factor of method, mixed-mode (mode mixture ratio), $G_{II}/G_{(I/II)C}$ and G_{IC} method in this study was different with the literature. Furthermore, in the previous section, it was seen that the woven flax/epoxy composites produced higher $G_{I/II}$ and produced higher $G_{I/II}$ than the other composites. In the literature, the authors reported that the $G_{I/II}$ of the flax/epoxy composite is higher than the other composites. Considering that there is no comparison data for comparison, the obtained data of the mixed-mode I/II and $G_{I/II}$ fracture toughness, which were determined and validated earlier, this value of the mixed-mode fracture energy is only for the woven flax/epoxy composite. Therefore, it is considered as the representative of the representative property of the material properties used in the present design and numerical simulation studies. The B-K delamination prediction criterion was used and fitted to the obtained data to determine the B-K fitting parameter, η , and to predict the interlaminar toughness for various mixed-mode ratios [90]:

$$G_{(I/II)C} = G_{IC} + (G_{IIC} - G_{IC}) \left(\frac{G_{II}}{G_{(I/II)C}} \right)^\eta \quad (8)$$

where $G_{II}/G_{I(III)C}$ is the mixed-mode ratio, and η is the B-K fitting parameter. Using Equation (8), $\eta = 0.35$ is computed for the prediction of the $G_{I(III)C}$ with other mixed-mode ratios.

4. Conclusions

The present study investigated the interlaminar fracture energy of unidirectional flax epoxy composites in Mode I, Mode II, and Mixed-mode I/II delamination failure. The composite laminates were manufactured via RTM method and were tailored to have a constant $V_f = 0.44$, and a 13 μm -thick PTFE was placed in the midplane to induce pre-crack, it was then located by X-ray in the laminate. All the DCB, ENF, and MMB tests were conducted and validated according to corresponding ASTM standards to evaluate the fracture energies in Mode I, Mode I, and Mixed-mode I/II, respectively. For Mode I, $G_{IC} = 574$ and $G_{IC} = 903 \text{ J/m}^2$ were obtained respectively for crack initiation and propagation values. The obtained value based on the standard method for Mode II delamination is $G_{IIC} = 378 \text{ J/m}^2$, which in contrast to the results of previous authors, calculated via the SBT data reduction method, is lower than G_{IC} . However, when following the SBT method, $G_{IIC} = 612 \text{ J/m}^2$ is obtained, which is consistent with their results. MMB tests resulted in $G_{(III)C} = 414.4 \text{ J/m}^2$ for Mixed-mode I/II fracture energy. Despite the limited, and in some cases lack of data available in the literature for comparison, the results are compared to the existing similar data and confirmed. In view of the fact that there is limited or no interlaminar fracture energy for these composites, the obtained values are valuable material properties to be further used in the design engineering field and numerical simulation methods.

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