

**Development of Future Recyclable Wind Blades with
Composites: A Solution to Landfill Waste**

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To my past self, who took the first step, my future self, who will carry this knowledge forward, and to my friends and colleagues at the University of Tennessee, whose support and camaraderie made this journey unforgettable.

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ABSTRACT

Thermosets comprise 80% of the market for reinforced composites with epoxy-based systems representing a substantial portion of wind turbine blade (WTB) manufacturing due to their superior mechanical strength, fatigue resistance, and chemical stability. As a result, WTB comprising of glass fiber reinforced polymers (GFRP) using epoxy resin systems are costly and difficult to manage at end-of-life. By modifying the composite with high-performance aniline-based epoxy curing agents, Recyclamines, wind blade waste can be reclaimed and reused to reduce the impact on landfills. In this study, an epoxy-based resin with Recyclamine, Epotec (EP), was used to produce nonwoven carbon fiber reinforced polymers (CFRP) using low-cost carbon fiber (LCCF), GFRPs, and hybrid composites through a traditional vacuum-assisted resin transfer molding (VARTM) process and compared to an epoxy resin and infusible methacrylate-based thermoplastic resin with thermoset-like processability, ELIUM (EL). Rheological and thermal analysis showed EP's suitability for WTB manufacturing, with low viscosity (~ 10 Pa.s), extended gel time (>200 min), and enhanced thermal stability, further improved by LCCF reinforcement. Mechanical testing showed that EP and nonwoven discontinuous LCCF significantly improved tensile and flexural strength and stiffness compared to EL with flexural strengths up to 858 MPa and tensile modulus reaching 48.2 GPa. Microscopy showed nonwoven composite failure was attributed to fiber pullout and debonding indicating enhanced stiffness compared to interlaminar shearing between hybrid and knitted fiber structures. Solvolysis demonstrated near 100% fiber recovery as 70.72% of the total mass was recovered as reusable fiber using low energy input conditions. The findings presented in this study displayed composite properties offered by LCCF, EP, and nonwoven fabrics for WTB manufacturing and recycling.

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LIST OF ABBREVIATIONS

BOM – Bill of materials

CFTF - Carbon Fiber Technology Facility

CFRP - Carbon Fibers Reinforced Polymer

CH₃COOH - Acetic Acid

CH₃COONa– Sodium Acetate

CT - Cycle time

DMA - Dynamic mechanical analysis

DSC - Differential Scanning Calorimetry

DTG - Derivative thermogravimetric

EfW - Energy-from-waste

El - Elixir

EoL - End-of-life

EP – Epotec

FEA - Finite elemental analysis

FRP - Fiber-Reinforced Polymer

GFRP - Glass Fibers Reinforced Polymer

GFF – Glass fiber Fabric

GHG - Greenhouse Gas

HT Temperature furnace

LCCF - Low-cost carbon fiber

LT - Low temperature

NaOH – Sodium Hydroxide

ORNL - Oak Ridge National Laboratory

PAN - Polyacrylonitrile

TCF - Textile grade carbon

TGA - Thermogravimetric analysis

SBF - Short Beam Flexure

SEM - Scanning Electron Microscopy

VARTM - Vacuum Assisted Resin Transfer Molding

WT - Wet laid

WTB- Wind Turbine Blade

Chapter 1: Introduction

The global shift and effort to decarbonize and achieve sustainable manufacturing and energy production has significantly increased the need for high-performance recyclable composites that can minimize the waste, cost, and energy associated with thermosets at end-of-life (EoL). While wind energy produces only 1-3% of greenhouse gas (GHG) emissions, the rapid development of wind power globally and a projected 3x installation increase of onshore and offshore turbines according to the Global Wind Report 2024 by the current volume to accommodate for the energy growth rate of 13.67% by 2030 has placed low-carbon intensive processes for recycling GFRP waste imperative [1-3]. This increase in addition to an approximate 2.4% annual decommission rate after a 20–25-year product life and an increase of rotor diameters to an estimated 270m and 350m for onshore and offshore respectively by 2030 requires more high-performance sustainable material to offset the cradle-to-grave impact of wind blades [3-5]. Current large-scale wind blade production comprises of a composition of thermoplastic coating, GFRP, CFRP, balsa wood, and resin [6]. These operating blades contain approximately 75-90% GFRP, typically E-glass fibers (i.e. borosilicate glass), by mass, and products such as the LM 37.3 P2 blades comprise of 86% GFRP indicating a large need for thermoset recycling [5, 7, 8]. However, with a high-volume content of fiber, dry areas void of resin between fibers can result in reduced mechanical and fatigue performance thus investigations into using lighter and stiffer alternatives such as carbon fibers have been conducted. Currently, companies Vestas (Aarhus, Denmark) and Siemens Gamesa (Zamudio, Spain) use CFRP for spar caps produced by a resin-infusion process to provide stiffness to the blade structure at critical loads [9, 10]. This adoption has led to a focus on hybrid composites as an alternative to pure GFRP or CFRP wind blades to reduce weight, increase the stiffness and failure strain, reduce damage to blades from lightning strikes, improve

vibration damping, and reduce transportation and installation costs of reinforced composites [11]. However, CF is expensive relative to GF, and it is difficult to make complex surfaces without a reduction in performance due to its strong directivity and low tangential strength. Hence, a need for a high-performance hybrid composite at a low cost that can be reclaimed at end-of-life.

This project aims to reduce manufacturing costs and environmental impact at EoL by investigating the performance of sustainable composite systems. Specifically, it evaluates the mechanical, thermal, interlaminar, and recycling properties of composites made using heavy 48k tow LCCF, EP, and discontinuous ½” and 1” inch nonwoven fibers. Since current wind blades are mainly produced using a resin infusion, each composite will be made using the VARTM process. The goal of this project is to establish the groundwork for introducing the Epotec (EP) and LCCF as an alternative to epoxy, vinyl ester, or polyester systems to address thermoset EoL operations. If successful, the use of both materials could decrease the cost of WTB production and reduce the impact of waste while providing a pathway to reducing the cost and waste of thermosets in alternative sectors.

Chapter 2: Background and Literature Review

This chapter reviews the background of wind blade manufacturing by exploring the cradle-to-grave analysis of the manufacturing and waste processes. This literature review will incorporate a detailed analysis of current materials, cost, and waste associated with wind turbine blade (WTB) construction and current recycling and energy recovery methods. Additionally, the Recyclamine Resin system and LCCF produced by the Carbon Fiber Technology Facility (CFTF) at Oak Ridge National Laboratory (ORNL) will be reviewed for this study.

2.1 Wind Blade Manufacturing

As the global projected energy demand is expected to increase by 13.67%, wind generation is expected to grow from 1,300 TWh in 2018 to 3,300 TWh by 2030, and WTB rotor diameters are expected to increase by 25% and 22.73% for offshore and onshore blades respectively by 2030, an annual increase in WTB waste of approximately 200,000 tons by 2033 will be realized as older generations of wind blades are decommissioned [3, 12]. As a result, the manufacturing process, materials, cost, and waste management need to be addressed and optimized for waste avoidance by creating long-lasting WTBs, the ability to be repurposed or recycled, and a decrease in the cost of production and end-of-life management.

2.1.1 Manufacturing and Materials

Since the introduction of WTB technology, glass fiber impregnation has been the predominant method of manufacturing using wet-hand lay-up technology in the 1970s for open molds on small to medium-sized blades (35-55 m). Since then, infusion and prepreg technology has been the predominant form of manufacturing allowing for better material consolidation, efficient production, and longer blades minimizing voids, wrinkles, unwet fibers, and air entrapment. Of the two, the most widely adopted process is the Vacuum Assisted Resin Transfer Molding

(VARTM) in which resin is injected under a vacuum at or below atmospheric pressure using a vacuum bag to allow low-viscosity resin to wet the fibers. In contrast, prepregs utilize “pre-impregnated” fibers containing the resin before heat and pressure are used to cure the fiber matrix. While prepregs offer less variation in mechanical properties, the cost of prepreg is substantially higher making VARTM the more common approach [14]. Therefore, this study will utilize VARTM to ensure adherence to the industry's most used manufacturing practices.

At present, the basic construction of WTBs includes skin, spar caps, webs, trailing edge panels, sandwich layers, and lightning conductor cables as shown in **Fig.2.1** [13]. A comprehensive overview of WTB manufacturing consists of a minimum of 69 processes encompassing the kitting, prefabrications, shell operations, and finishing operations not accounting for labor and material processes as detailed in **Table 2.1** [15]. Depending on the blade manufacturer, the material kitting will vary, but the VARTM process for producing prefabricated parts and shells is consistent with variations in material layup. This method consists of 1) Applying a release agent to the mold, 2) Layup of materials (discontinuous fibers, reinforcement, balsa wood, foam, anisotropic fibers), 3) Applying a layer of release film, distribution media, and bagging film, 4) Resin Infusion, 5) Demolding.

Modern WTB thermosetting composites account for >80% of the mass depending on the wind blade design. Wind Blades such as the LM 37.3 P2 shell and web composite matrix account for 86% of the total mass comprising of E-glass and polyester resin at 58% and 28% respectively [16]. However, epoxy-resin systems are becoming the predominant form of thermoset matrices with companies such as Siemens Gamesa's B45 and Vestas's V47 model comprising the total mass of GFRP thermosets of 90% (55% GF, 35% Epoxy Resin) and 82% (61% GF, 21% Epoxy Resin) respectively [17, 18]. The increase in blade length and the amount of GFRP presents

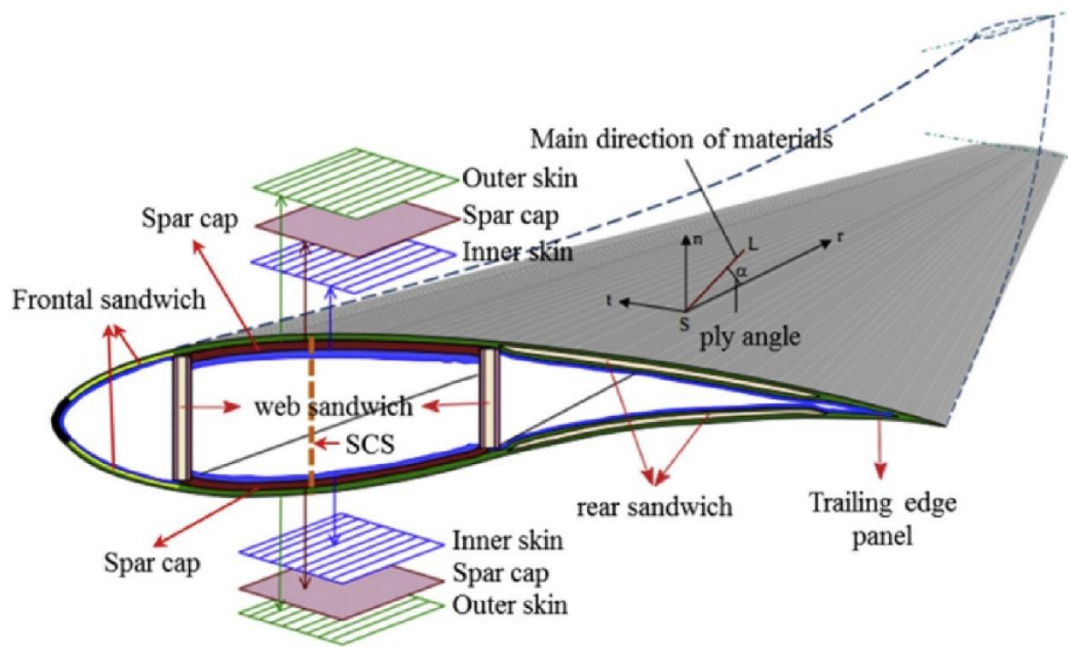


Figure 2.1: Schematic of WTB cross-section identifying the composite layup and structural components, including spar caps, sandwich panels, and skin layers. Material orientation and ply angles are indicated to highlight design optimization for mechanical performance. [13].

Table 2.1. Blade Making Operations [15]

<u>KNITTING</u> (4) Glass Kitting Core Kitting Resin Kits Consumable Kitting <u>PREFABRICATION</u> (23) Spar Mold Preparations Layup Infusion Prep Infusion Cure Monitor Demold & Prep Shear Webs Mold Preparations Layup Infusion Prep Infusion Cure Monitor Demold & Prep Root Prefabrication Mold Preparations Layup Infusion Prep Infusion Cure Monitor Demold & Prep Miscellaneous Small Parts Balance Boxes Root Flange Root Closeout Platform Root Closeout Brackets Root Band	<u>SHELL OPERATIONS</u> (23) Low Pressure Shell Mold Prep Initial Glass Layup Install Spar & TE Core Layup Glass layup Root Buildup Infusion Prep Infusion Cure Monitor Debug & Prep High Pressure Shell Mold Prep Initial Glass Layup Install Spar & TE Core Layup Glass layup Root Buildup Infusion Prep Infusion Cure Monitor Debug & Prep Bond & Blade Close Load Shear Web Shear Web Bond Close & Cure	<u>FINISHING OPERATIONS</u> (19) Trim LE & TE Trim Wet Lamination Work Over Laminations Web Reinforcement Root Closeout LPS Installation Post Cure Oven Drill Root Face & Drill Paint Preparation Root Closeout Install Barrel Nut & Bolt Root Band Paint Prep/Sand Vortex Gen Install Paint Load Carts Paint Application Paint Touchup Final Finishes Root Closeout Lid Zero Degree Jig Weigh & Balance Final Finishes
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issues with mechanical and loading conditions during use due to the weight and flexibility characteristics of the fiber matrix. Therefore, reinforcement of the structure using lighter stiffer materials such as carbon fiber that are 70% lighter and 20% stronger than GF in the spar of the WTB allows for longer blades and more stability, optimizing the maximum energy production, minimizing the mass, and the cost of energy (COE) [13, 19].

2.1.2 Cost of Materials and Manufacturing for Current WTBs

The cost of WTB manufacturing using VARTM can greatly differ between models; however, fixed and variable costs are associated with the manufacturing process. The variable costs include materials, labor, and utilities while fixed costs include equipment, tooling, building, maintenance, overhead, and capital. In addition, cycle time (CT) and labor hours greatly affect the cost of production.

An extensive overview of three blades, the 33.35-meter WindPACT study blade, the 63.7-meter IEA Wind Task 37 Land-Based WTB, and the 100-meter SNL-100-03 cost was conducted by Bortolotti *et al* including bill of materials (BOM) of the total composite, core, and coating costs with additional labor, cycle time, waste, and fixed costs [20]. The WindPACT and Wind Task blades utilize epoxy-based GFRP with unidirectional GF spar cap reinforcement while the SNL-100-03 utilizes epoxy-based GFRP with CF spar cap reinforcement. The total cost of variable and non-variable expenses was \$52,146.09, \$154,090.40, and \$547,723.35 respectively indicative of the exponential cost associated with blade length increase. While cycle time, labor hours, and material quantity increase respective to length, the substantial increase in the cost of the SNL-100-03 blade was greatly attributed to the unidirectional CF at \$30.00/kg relative to the \$1.87/kg of unidirectional GF. As shown in **Fig.2.2**, the fabric, core, and coating share in the BOM for the SNL blade is substantially more than the other blades with the carbon-

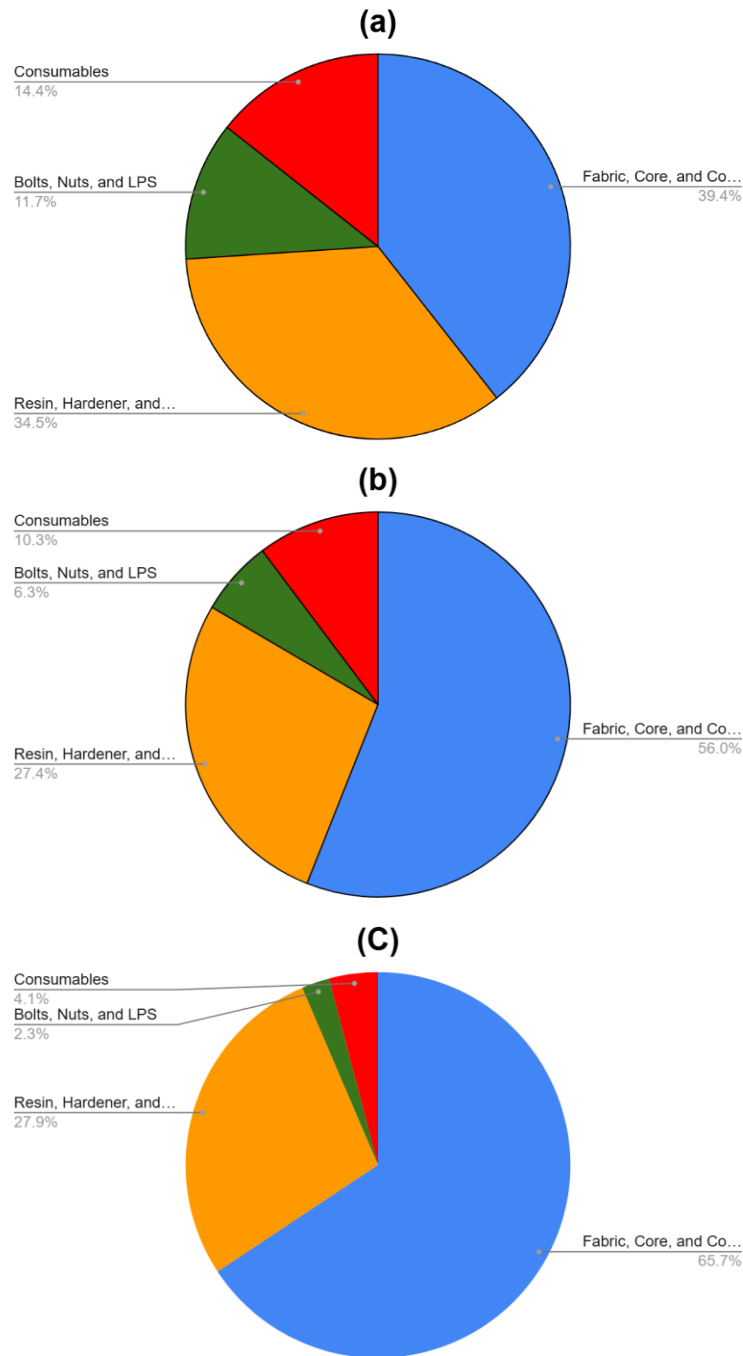


Figure 2.2: Breakdown of bill of materials (BOM) costs by component category for three wind turbine blades: (a) WindPACT Blade (\$24,577.65), (b) IEA Land-Based Reference Blade (\$73,638.82), and (c) SNL-100-03 Blade (\$327,375.63). Pie charts illustrate the relative share of fabric, resin, fasteners, and consumables in each design [20].

fiber share attributing to 60% of the total cost [20]. Indicating a need for reducing spar cap CFRP cost (per kg). Contradictory to this models CF cost, Sandia Nation Laboratories report on optimized carbon fiber composites in WTB design, baseline CF-pultruded 50k tow used in 61.5m turbine blade spar caps is \$17.98/kg reducing the cost by 40.1% however still in comparison to E-glass pultruded spar caps are \$2.65/kg, indicating a 578.49% increase in cost [21, 22]. As the global market shifts to higher production of blade lengths greater than 70 meters, CFRP spar cap usage is expected to grow as nearly 55% of the turbine manufactures utilize carbon fiber spar caps in the ≥ 70 m onshore and offshore production. The projected increase in CFRP for lightweighting and increased WTB stiffness is critical to improving the cost-to-energy production in the 3-5 MW and 8-10 MW sectors [21].

2.1.3 Waste Management

The end-of-life processes for WTB waste are characterized by four sectors: disposal, energy recovery, repurpose, and recycling. The disposal route and energy recovery are defined by landfill and incineration at energy-from-waste (EfW) facilities respectively. Solid municipal waste management using landfills and incineration are undesirable due to the loss of material, and energy recovery is minimized due to a high non-combustible fraction of material use for WTB construction operating at 15-24% electrical efficiency [1]. Additionally, byproducts such as ash after incinerations account for 60% of the WTB mass and are sent to landfills, and the emissions from the resin system, typically epoxy or vinyl ester, combustion can also produce harmful byproducts such as carbon monoxide and formaldehyde while biodegradable WTB materials in landfills can release methane overtime [23]. However, incineration can be scaled, and disposal is quick and more cost effective for manufacturers, making these methods the most common approach.

A more desirable alternative, repurposing, is cut or refashioned wind blade waste allocated as secondary material for products such as furniture, beams, polymer additives, pedestrian structures, thermal insulation, building facades, etc. This process requires little to no chemical change but requires significant cutting with negative side effects of extensive dust production, unknown mechanical properties, and an increase in cost of waste management. Additionally, repurposing is difficult to scale as the wind-blade geometry is curved and difficult to disassemble and costly to transport for various niche applications [12].

Therefore, the most desirable sector for WTB waste management is recycling to achieve a 100% sustainable circular economy that is profitable. Recycling of WTB requires the extraction of GFRP and CFRP, balsa wood, foam core, paint, steel fasteners, aluminum, and adhesives. As blades continue to increase in length and the fraction of composite material increases with the introduction of CFRP spar cap integration, separation and extraction of materials at near or virgin grade is critical for cost effectiveness and circularity. Recycling processes include mechanical, chemical, high-voltage fragmentation, pyrolysis, and co-processing into concrete. These processes, excluding chemical solvolysis, are easily scalable decreasing the cost with possible breakthroughs in fiber recovery and reuse of entire WTB.

2.2 Recycling of Blade Material

Disposal strategies for WTB structures are difficult to reuse and recycle due to the thermoset polymer composite's molecular structure. Thermoset resins such as epoxy undergo an irreversible curing process during the infusion and the cross-linked structures formed between the resin and fiber matrix make it difficult to extract the fibers at EoL. Energy intensive methods shown in **Fig. 2.3** are categorized by mechanical, thermal, and chemical processes that are utilized to recover the material for reuse with aim to minimize environmental impact, reduce

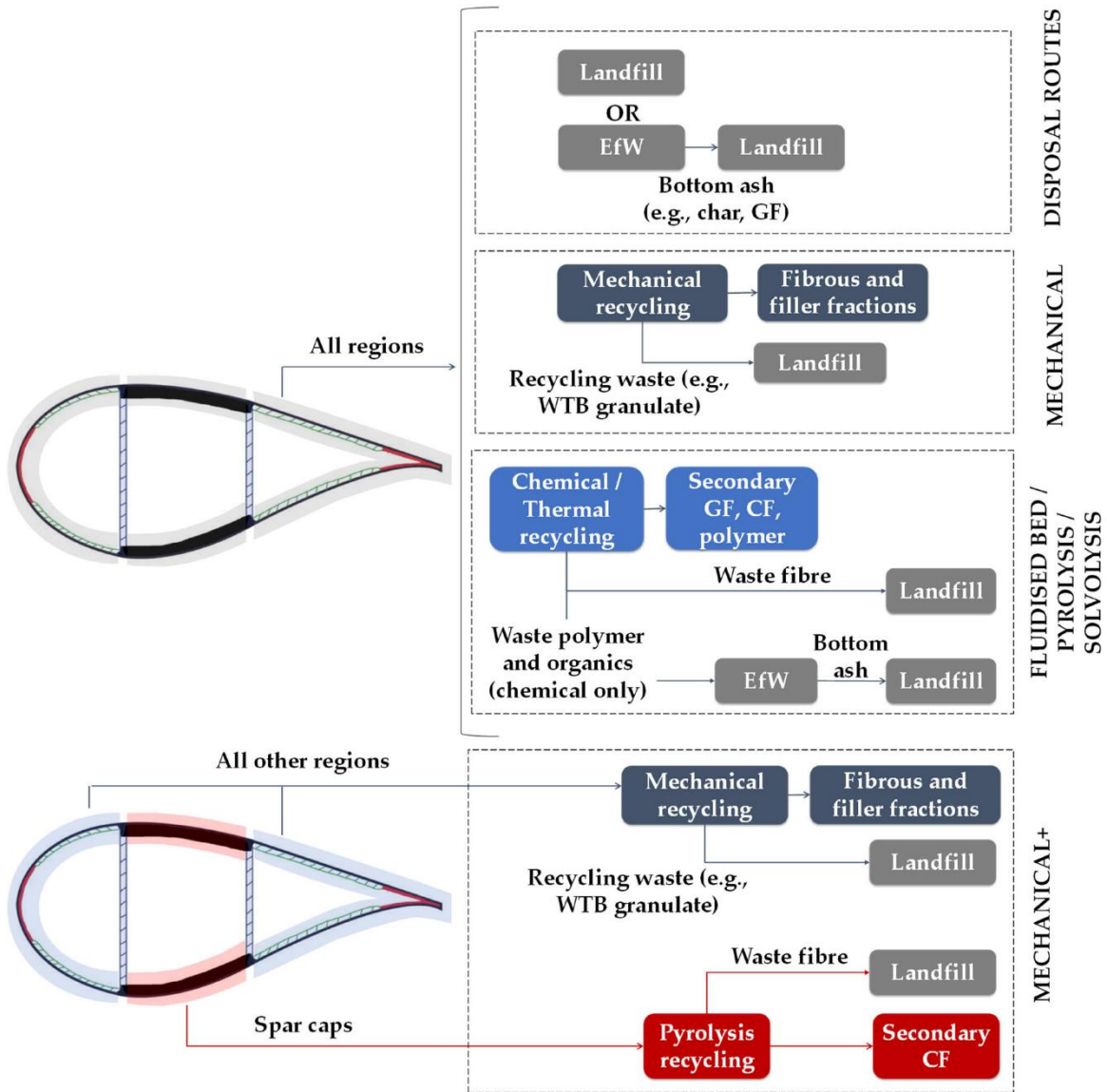


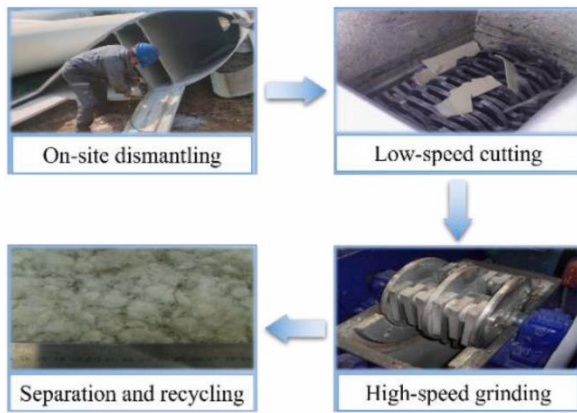
Figure 2.3: Overview of EoL processing for WTBs, including landfill, energy recovery, mechanical recycling, and advanced chemical/thermal recycling. The schematic highlights region-specific strategies, such as pyrolysis recycling for carbon fiber spar caps.[1].

damage to material during recovery, and achieve commercial viability. While difficult to extract the various CFRP and GFRP composites, minimizing the impact of the raw production of GF, CF, and resin by reutilization of the WTB waste has significant importance in the efforts for a low-carbon circular economy. This section provides a comprehensive overview of the WTB waste recycling use of each technology as shown in **Fig. 2.3**.

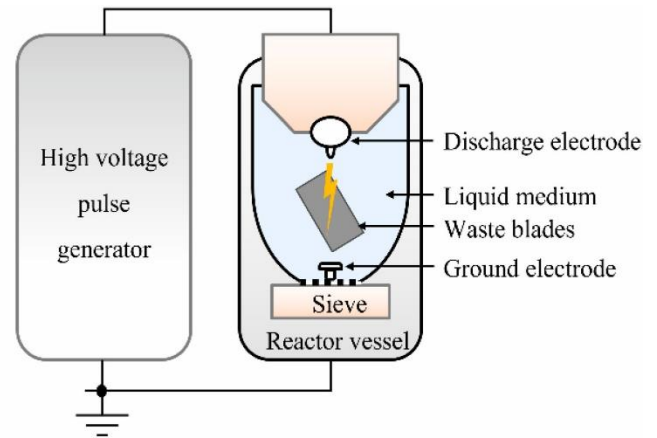
2.2.1 Mechanical Recycling of WTB

Currently, mechanical recycling is the most implemented industrialized standard of recovering wind blade waste. By means of mechanical crushing and grinding the composite material into smaller fragments or powder, shorter fibers and resin granules can be separated and through vibratory screening or centrifugal process and transported for second life applications. The reduction in fiber length lowers the mechanical properties of the second life materials but can reduce the transportation cost associated with WTB waste by mechanical disassembly at the wind plant [23, 24]. Additionally, mechanical recycling is used as a pre- or post-processing stage in other recycling technologies, as the cutting or crushing of WTBs is necessary for thermal and chemical recovery.

Mechanical recovery procedures consist of four stages of operation shown in **Fig.2.4**: 1) Disassembly and cutting of blade, 2) Low-speed shredding or crushing of bulk material, 3) High-speed grinding, 4) Separation. Material extracted at each stage can be repurposed for various applications and can be categorized as first, second or third grade treatment as define by Yiming et al. First grade treatment utilizes stage 1 of mechanical recovery to isolate regions of WTB by cutting material of various sizes and contours or separating large sections of the blade using line or circular saws respectively. These materials can be repurposed for local infrastructure projects and can reduce transportation costs. However, the first-grade treatment is difficult to scale, and



(a)



(b)

Figure 2.4: (a) Mechanical recycling process of wind turbine blades, including dismantling, cutting, grinding, and separation. (b) High-voltage pulse fragmentation setup for breaking down resin and fiber in a liquid medium. The method enables efficient material recovery for reuse [24].

the composites reuse opportunities are heavily restricted. Second grade treatment utilizes material produced after stage 2 in which 50-100mm fragments with metallic material removed can be preprocessed for second life applications such as road signs, skateboards, decorative, or low load bearing applications. Likewise, this method can reduce the impact of transportation and significantly reduce the energy requirement for reprocessing. Lastly, third grade treatment incorporates stages 1-4 to produce particles between 50-10 μ m by means of high-speed hammer or cutter mills. The fine powder can be implemented as additives for thermoplastics such as PLA, concrete, or construction panel reinforcement. While mechanical recycling significantly reduces the fibers mechanical performance, the low processing cost between 0.17-5.53 MJ/kg depending on the operations a facility capacity presents an excellent avenue for WTB circular economy advancement [25].

2.2.2 Thermal Recycling of WTB

Thermal recycling technology for composite materials is utilized to decompose the polymer from the fiber matrix allowing for the extraction of exposed fibers after combustion. Processes including pyrolysis, fluidized bed combustion, and microwave pyrolysis are currently adopted technologies for separation of fibers in thermoset composites.

Among the variations, pyrolysis as shown in **Fig. 2.5** is considered the predominant thermal recovery process for WTB waste. To extract the GF and CF from the thermoset, the composite structure after mechanical sectioning is exposed to oxygen-free temperatures between 450 to 700°C. This allows for the degradation of the polymer releasing pyrolysis oils such as aromatic hydrocarbons or phenolics during depolymerizations as well as pyrolysis gas including methane, carbon monoxide, hydrogen gas, carbon dioxide, and C₁ to C₄ hydrocarbons [25]. The

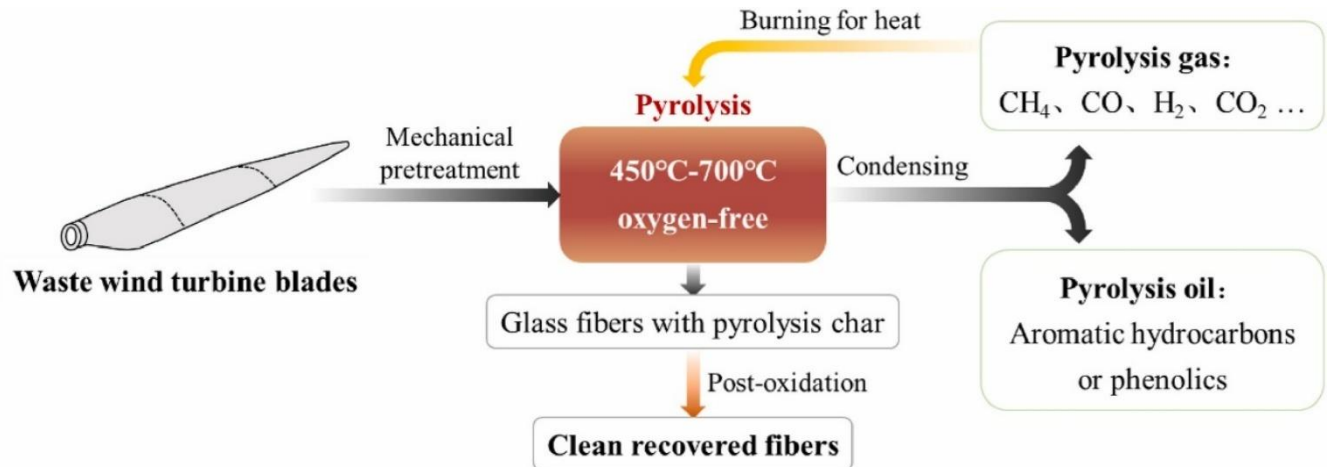


Figure 2.5: Pyrolysis-based recycling process for WTB waste, starting with mechanical pretreatment. Blades are thermally decomposed at 450–700 °C in an oxygen-free environment. The process yields pyrolysis gas, oil, and clean recovered glass fibers [24].

yield of both byproducts can be used as fuel for the continued combustion cycle of pyrolysis, and the CF, GF, and pyrolytic carbon char can be extracted at the end of the process.

Subsequently, post-oxidation of the solid products enables the recovery of pure CF or GF structures [24, 26].

Variations in temperature, heating rates, and modifications in atmospheric conditions during pyrolysis can significantly change the yield type and quantity, process time, and strength of reclaimed fibers. An investigation conducted by Ming-xin *et al* of a GF-epoxy wind blade material pyrolysis at 350, 450, and 550°C demonstrated that pyrolysis temperature increases cause elastic modulus loss of 19.02%, 70.71%, and 53.84% respectively with significant tensile strength decreasing from 10.51 MPa at 350°C to 2.59 and 2.78 MPa at 450 and 550°C respectively [24]. The elevated pyrolysis temperature decreases cycle time of resin degradation as temperatures above 500°C promote chemical bond cleavages but can drastically decrease the fiber performance and utility for second life application. Furthermore, atmospheric environments using additives such as N₂ and H₂O can accelerate the degradation of the resin while promoting pyrolysis gas and oil compound production while minimizing pyrolysis char. While scalable, this process is highly energy intensive, and the pyrolysis process is in complex with high inefficiency with significant degradation of mechanical performance for all thermal processes limiting the use for secondary applications.

2.2.3 Chemical Recycling of WTBs

Recycling and reuse technology can be accomplished via chemical processes such as hydrolysis, glycolysis, acid digestion and aminolysis [24]. End of life processing using solvolysis can enable the full recovery of fibers without degradation and loss of mechanical properties retaining full-length fibers as compared to thermoforming, mechanical grinding, and pyrolysis

[27]. The process works by using a reactive solvent under increased temperature and pressure to dissolve the polymer matrix by breaking the covalent bonds in the thermoplastic. However, this can require significant energy for processing thermosets due to the increased temperature, pressure, and time compared to thermoplastic materials which offer low processing temperatures [28].

Depending on the type of solvent used, significant operating conditions and cycle times can be affected as strong acids or alkalis can degrade thermosetting polymer bonds at low temperatures below 200°C and atmospheric pressure with high cycle times. However, highly concentrated acids have shown in multiple studies to decrease mechanical performance by nearly 70% with a resin decomposition rate of 99% indicative of high reclamation but similar secondary utility in comparison to mechanical or pyrolysis methods [29]. In response, a supercritical fluid method through solvents such as water, propanol, potassium phosphate, and diethylene glycol have shown degradation in fiber tensile strength between 7-10% while capturing nearly 91.6% pure GF [25]. While this process produces high quality fiber recovery, the technology requires high temperature, pressure, and advance equipment to operate supercritical fluid solvolysis directly influencing the cost in comparison to lower cost alternatives via mechanical grinding or pyrolysis. Additionally, harmful gases and the manufacturing impact of solvents for chemical recycling pose issues for reducing the carbon footprint associated with WTB material recovery. At the present, chemical solvolysis is under laboratory stages of development and do not offer large scale productions, but solvolysis offers a high potential for improvement and circular economic feasibility as thermosetting resin systems and innovative solvents at low impact and cost are becoming a desirable input to reclaim costly WTB material.

2.3 Recyclable Recyclamine Thermoset Resin (Aditya Birla)

As the development of alternative chemical solvolysis continues to progress for high-performance fiber recovery of wind blade GFRP and CFRP, recyclable high-performance thermosetting resin systems that possess comparable mechanical strength to epoxy or vinyl ester systems are critical for low-cost and impact circular EoL processing. An innovative solution to difficulties during EoL processing of WTB thermosets is a high-performance aniline-based epoxy curing agent, Recyclamine. The recyclamine curing agents enable cleavage points within the thermosetting epoxy as shown in **Fig.2.6** that can convert the system into a thermoplastic polymer under specific conditions. Thus, under the right conditions, the cross links in the thermoset polymer chain can be disintegrated allowing for the recovery and reuse of the fiber matrix and newly formed thermoplastic polymer for reuse as additives in other manufacturing processes such as additive manufacturing [30].

This system presents a strong case for integration and substitution of traditional epoxy systems that can allow for low-cost recycling and reuse of more than 90% of WTB composite materials. Therefore, this study will investigate the potential for Recyclamine for GF and CF composites with specific recyclamine molecule selection based on the processing properties and neat cured system mechanical properties.

2.4 Low-Cost Carbon Fiber (LCCF)

Carbon Fiber provides a much higher stiffness-to-weight ratio than glass fiber. In a finite elemental analysis (FEA) analysis of composite pressure vessels comparing E-glass, S2-fibreglass, basalt, and carbon epoxy-based laminates conducted by Aftab Afrathim *et al* [31] the CFRP ($\rho = 1560 \text{ kg/m}^3$) compared to GFRP ($\rho = 1800 \text{ kg/m}^3$) had a significant increase in tensile strength and Young's Modulus (E) and along the x-direction by 272% and 243% respectively

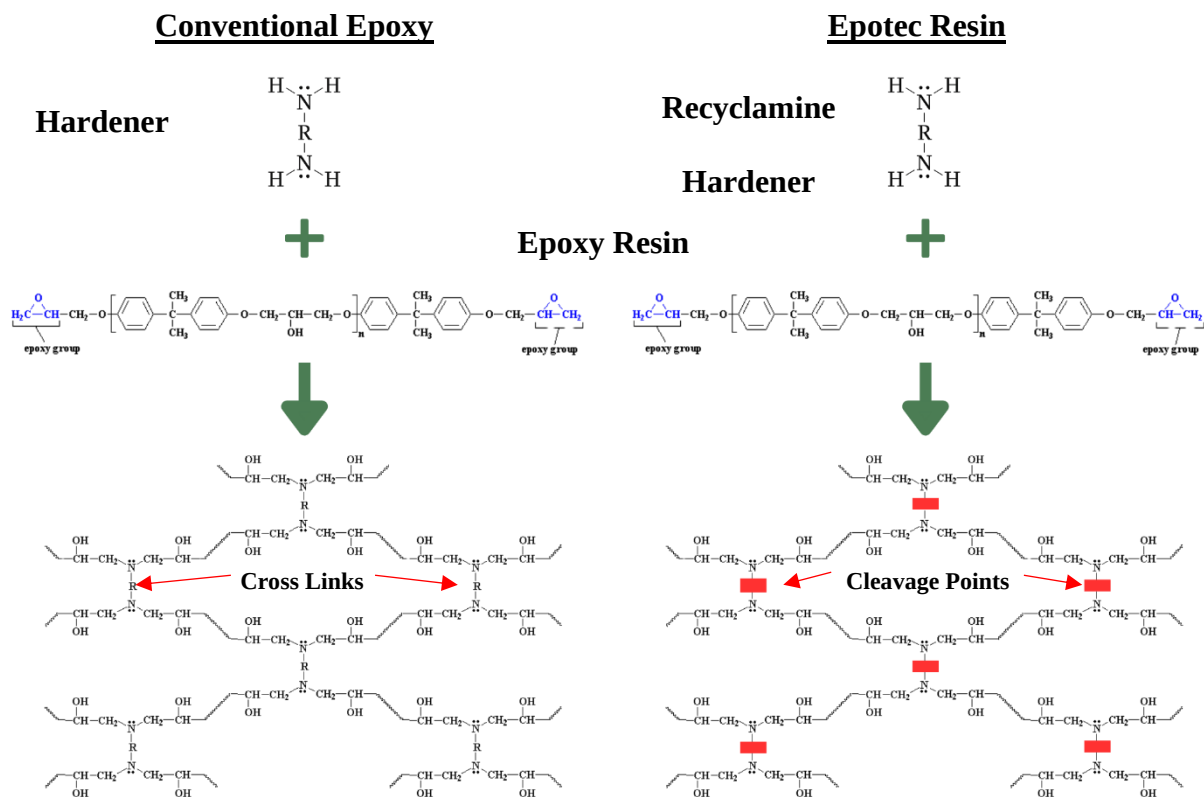


Figure 2.6: Comparison of conventional epoxy and EP Recyclamine resin. The left shows permanent cross-links formed with a standard hardener, while the right illustrates cleavable points in Recyclamine-cured structure, enabling chemical recyclability of the polymer [30].

while displaying negligible improved performance by 9.39% and 12.4% in the yz-directions [32]. This anisotropic behavior of CFRPs illustrates the drawbacks in the use of pure CF wind blades but significantly highlights the dual advantages of hybridized GFRP with CFRP by increasing mechanical performance and product durability while increasing the cost associated with manufacturing [33]. However, the cost of high-performance CF presents drawbacks in the potential use of increasing the volume fraction of CFRP in wind blade applications.

In response to the excessive cost associated with CF production, technologies for producing heavy tow textile carbon called low-cost carbon fiber (LCCF) are being implemented in industrial lab scale production volumes at ORNL's CFTF. The manufacturing process for LCCF depicted in **Fig. 2.7** determines the cost, performance, and chemical interactions with different resin systems based on a variety of factors and materials used. In the fiber production line, low-cost textile grade acrylic fibers are used costing \$2.24/kg compared to baseline precursor costs of \$3.63 resulting in a 38.3% reduction in cost [34]. The low-cost precursor undergoes pre-treatment via an oxidation oven at 250°C for 90 minutes. Subsequently, the acrylic fibers are oxidized and then heat treated through a low temperature (LT) and high temperature furnace (HT) in an inert nitrogen atmosphere at 700°C for 1.5 minutes and 1400°C for 1.5 minutes, respectively. Proprietary surface treatment and sizing of the heat-treated CF are then performed followed by quality inspection.

Variations in cost/kg of CF from polyacrylonitrile (PAN) precursor fibers depend greatly on the tow size from 3k to 100k with an inverse exponential correlation. The cost of a 3k tow is approximately USD 34.70/kg with a reduction of 68.59-69.74% in price for tow sizes of 48k, 50k, and 100k operating around \$10.9, 10.8, and 10.5 per kg, respectively. This cost estimation for CF production depicted in **Fig. 2.8** is largely associated with the precursor accounting for

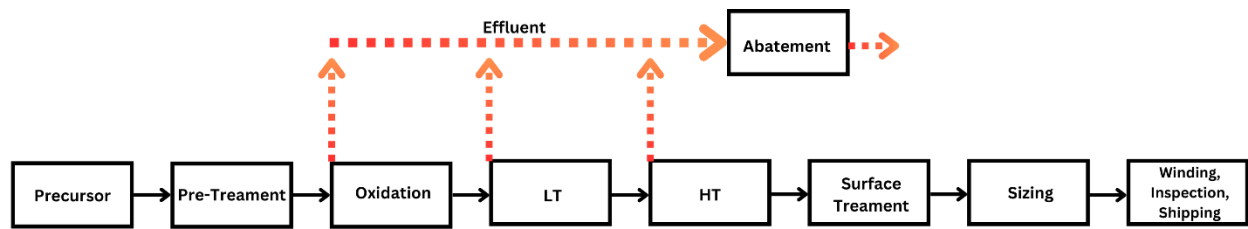


Figure 2.7: General stages of carbon fiber manufacturing, from precursor through pre-treatment, oxidation, and thermal processing (LT and HT), followed by surface treatment, sizing, and final inspection [34].

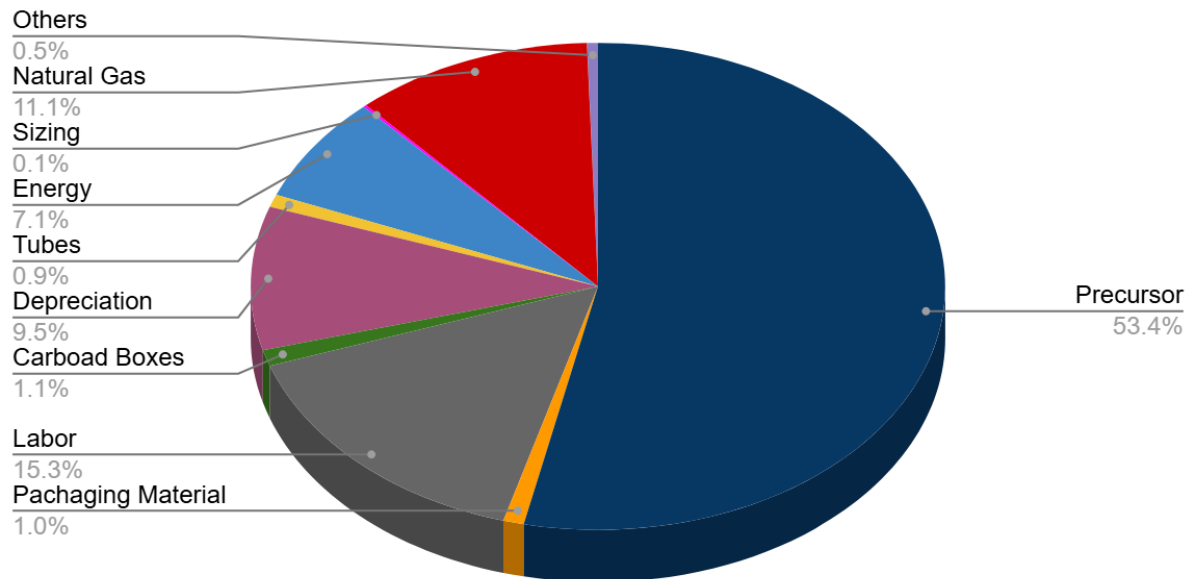


Figure 2.8: Cost breakdown for 50k tow carbon fiber production at a baseline price of \$10.81/kg. The precursor accounts for the largest share (53.4%), followed by labor (15.3%) and natural gas (11.1%). Minor contributions come from depreciation, energy, and packaging materials [35].

53.4% of the total cost while labor (15.3%), depreciation (9.51%), energy (7.06%), and natural gas (11.13%) account for the large majority of other operations [35].

Due to the disproportionate cost of CF precursor, LCCFs acrylic precursor adoption plays a crucial role in the reduction of raw materials. With estimates of \$7.82/kg after full utilization of CFTFs manufacturing line can significantly change to cost-to-energy ratio for WTB production and EoL processing. However, as shown in **Fig. 2.9** the mechanical properties of LCCF diminish narrowing the effective utilization for WTB applications. Although the mechanical properties are negatively affected, post-production spar cap cost scenarios utilizing pultrusion using LCCF, adding 5% to the total cost, have shown to decrease the baseline cost of \$16.44/kg by 33-49%. These improvements in cost-performance ratio for CF production can drastically improve the impact of WTB manufacturing and aid in the goal for sustainability while being cost effective.

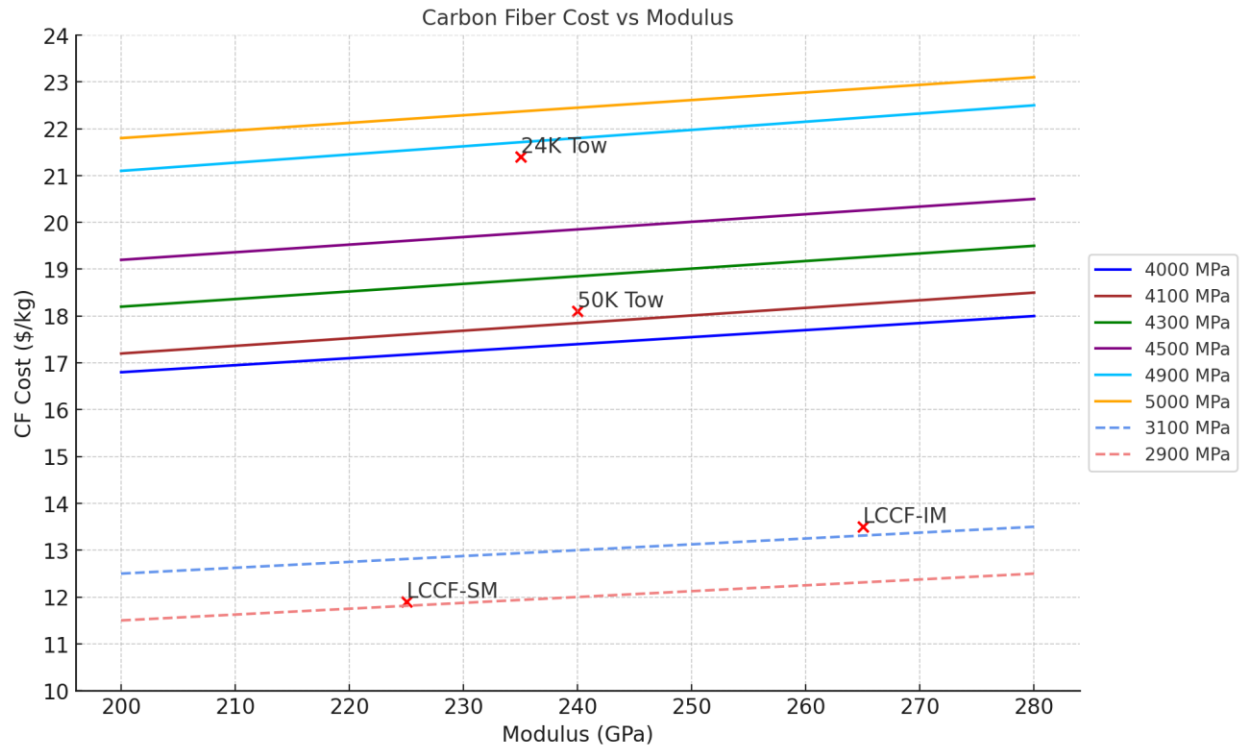


Figure 2.9: Comparative cost-to-strength performance of heavy-tow LCCF and heavy-tow baseline commercial CF depicting the reduction in cost of LCCF-SM and LCCF-IM fibers produced at CFTF [34].

Chapter 3: Material and Methods

3.1 Materials

3.1.1 Textile Grade Carbon Fiber (LCCF)

The corresponding textile grade carbon (TCF) fiber was manufactured and obtained from the CFTF. The fibers were derived from a PAN precursor and obtained as discontinuous fibers at 1” and ½” and biaxial $\pm 45^\circ$ knit mats. The relevant fiber properties for the LCCF are depicted in **Table 3.1**.

3.1.2 Fiber Glass

The corresponding biaxial 806 GEC X2400 $\pm 45^\circ$ knitted fiberglass used in this study were provided by the Composites One suitable for design flexibility and adopted use in wind blade manufacturing.

3.1.3 Aditya Birla Chemicals Epotec YDL 5567 / THR 9369

The thermoset resin and recyclable amine crosslinker hardener, Recyclamine, provided by Aditya Birla Chemicals known as Epotec YDL 5567 and THR 9369 respectively, was utilized for this project. The resin system is suitable for this project for its recyclability and high mechanical strength. This polymer has a resin density of 1.10-1.20 g/cm³, hardener density of 0.90-1.00 g/cm³, and initial mix viscosity of less than 300 mPa.s using ISO 2811-1 and ASTM D 2196-18^{§1}/ISO 2555 respectively. The mechanical properties of the neat cured system are displayed in **Table 3.2**.

3.1.4 Elium[®] 191 XO/SA

The thermoplastic resin, Elium 191 XO/SA, provided by Arkema Global through MDF at ORNL, was utilized in this project to compare recyclable resin system mechanical properties for potential use in WTB manufacturing with mechanical properties displayed in **Table 3.3**.

Table 3.1: LCCF Mechanical Properties and Cost Comparison of Industry Baseline and GF properties [34]

Material	UTS [MPa]	UCS [MPa]	E [GPa]	Cost
Industry Baseline (Pultrusion)	2427.3	-1649	157.6	\$16.44
LCCF (full utilization)	1508.5	-1315.0	160.6	\$8.38 (full utilization)
				\$11.01 (current)
Fiberglass Infusion	1169.7	-743.5	42.8	\$2.06

Table 3.2: Epotec YDL 5567/THR 9369 Neat Cured System

S.No.	Property	Test Method	Unit	Specification
1.	Tensile strength	ISO 527-2	MPa	70 - 80
	Tensile elongation		%	4.0 - 7.0
	E- modulus		GPa	3.0 - 3.5
	Ultimate Tensile strength		MPa	65 - 75
	Ultimate Tensile elongation		%	5.0 - 8.0
2.	Flexural strength	ISO 178	MPa	120 - 140
	Flexural elongation		%	5.0 - 8.0
	E- modulus		GPa	3.0 - 3.5
	Ultimate Tensile strength		MPa	110 - 130
	Ultimate Tensile elongation		%	6.0 - 10.0

Table 3.3: Elium 191 XO/SA Neat Cured System

S.No.	Property	Test Method	Unit	Specification
1.	Tensile strength	ISO 527-2	MPa	47.1
	E- modulus		GPa	2.0 - 7.0
	Ultimate Tensile elongation		%	4.0 - 50
2.	Flexural strength	ISO 178	MPa	80 - 140
	E- modulus		GPa	2.0 - 8
	Ultimate Flexural elongation		%	4

The acrylic based resin system is highly compatible with glass, carbon, and natural fibers with excellent wettability during infusion processes making it an idea choice WTB composite manufacturing. This polymer has a resin density of 1.178 g/cm^3 and initial mix viscosity of less than 100 mPa.s using ISO 1675 and ISO 2555 respectively.

3.1.5 Acetic Acid Lab Ally Essential Chemicals

The acetic acid (CH_3COOH) glacial 99% lab grade used for this study functioned as the solvent for epoxy degradation in the composite matrix provided by the Recyclimine cleavage points in the thermoset.

3.1.6 Sodium Hydroxide

The sodium hydroxide (NaOH) used for this study functioned as a coagulation agent for separating acetic acid from the thermoplastic solution created by composites matrix.

3.1.7 VARTM Equipment

The resin infusion process for panel manufacturing using both resin systems required: a smooth glass surface, sealant tape, vacuum bag film, release film (Peel Ply or PTFE (Teflon)), breather cloth, distribution media, vacuum tubing, spiral tubing, and a vacuum pump with resin trap **(Figure 3.1)**.

3.2 Methods

This section is comprised of three main parts that encompass the research done. The first section explains how the composites were manufactured using wet-laid and VARTM processes. The second section illustrates the comparison in the mechanical, thermal, and structural performance in a lab-scale environment of the two resin systems (EL and EP) for various fiber structures and layering sequences. The last section depicts a preliminary study providing methodologies and proof of concept for Epotec recycling through solvolysis.

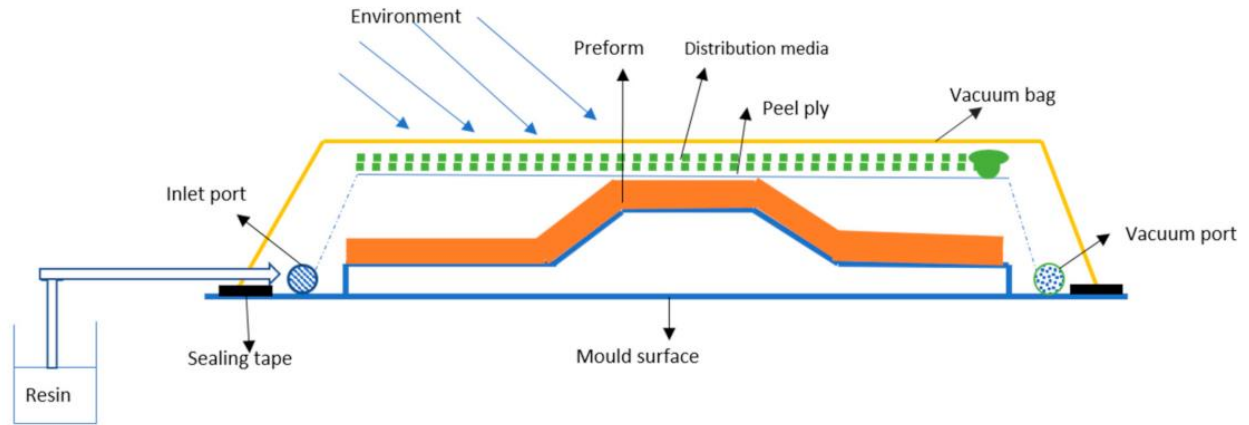


Figure 3.1: Schematic of Vacuum-Assisted Resin Transfer Molding (VARTM) setup. Resin is drawn into the fiber preform under vacuum through distribution media and peel ply. The system includes inlet/vacuum ports, vacuum bag, sealing tape, and mold surface [36].

3.2.1 Composite Manufacturing

To evaluate the mechanical and thermal properties associated with EP and LCCF, traditional VARTM was used for the manufacturing of the GFRP and CFRP composites. The panels produced from LCCF and GF consisted of three types of fiber structures: 1) Discontinuous LCCF at 1/2" and 1" fiber length, 2) Stitched 45° GF, 3) Stitched 45° LCCF. Different stacking sequences were manufactured as shown in **Table 3.4** to address the use of nonwoven mats, textile PAN, and hybrid composite systems using LCCF to decrease the cost-to-performance ratio. Nonwoven discontinuous mats were produced through a wet laid (WT) technique and incorporated as a low-cost alternative to textile mats or pultrusion.

3.2.1.1 Wet Laid Process for Nonwoven Discontinuous Fiber Mats

Non-woven fabrics consist of discontinuous fibers with random orientation that provide isotropic materials properties to the composite material. Traditional methods of processing like extrusion compression molding or injection molding cause fiber degradation during processing and various fiber consolidation. The wet laid (WL) technique used in this study with process conditions and equipment provided by Endeavor Composites provide a cost-effective alternative to anisotropic textile production or pultrusion methods for composite technology and may present considerable opportunities for WTB technology and cost reduction. As seen in **Fig.3.2**, the WL process consists of the dispersion of fibers in water using two stirring paddles in figure 8 pattern with additives including surfactants, dispersing agents, and flocculent agents to provide enhanced web formation and full fiber dispersion. Following dispersion, the web formation is extracted after the filtration of water and transferred to a dryer to solidify the matrix and remove moisture [37].

Using the optimized WL process depicted in the study provided by Hicham *et al*, LCCF mats were produced at the Fibers and Composites Manufacturing Facility (FCMF). Each mat

Table 3.4: Panel Manufacturing of Epotec and Elium composite panels using LCCF and GF

Material	Resin System	Fiber Type	Abbreviation	Stacking Sequence	Curing
Low-Cost Carbon Fiber (LCCF)	Elium	½"	EL_1/2" LCCF	6 layers	24h RT, 2h 80°C post-cure
		1"	EL_1" LCCF		
	Epotec	½"	EP_1/2" LCCF	6 layers	24h RT, 8h 80°C post-cure
		1"	EP_1" LCCF		
		Textile (45°)	EP_4GFF	4 layers	
		Textile GF & 1/2" CF	EP_2GF_4CF1/2"	2GF/4CF	
		Textile GF & Textile CF	EP_GF/CF	GF/CF/GF/CF	
Hybrid			EP_2GF_2CF	CF/CF/GF/GF	

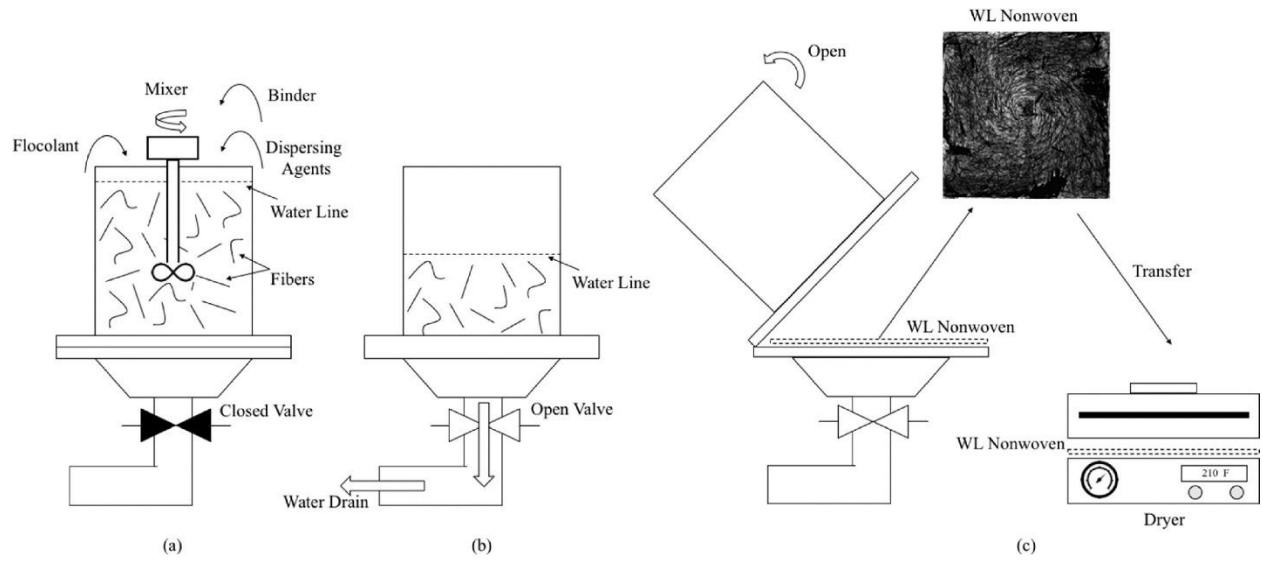


Figure 3.2: Wet-laid system process utilized by Endeavor Composites for this project: a) Fiber distribution, b) Drainage and web formation, c) Transfer and drying of WL mat [37].

achieved full dispersion in 20 minutes and produced 12x12” 200GSM mats using 1” and ½” LCCF. This study explores the wettability, mechanical, and thermal properties of the WL nonwoven CF mats for both resin systems to investigate the potential for further use in low-cost recyclable composite manufacturing.

3.2.1.2 Composite Manufacturing using VARTM

Vacuum assisted resin transfer molding (VARTM) was implemented for each mat produced in this study. Each 12x12” panel consisted of the stacking sequence defined in **Table 3.4** and consisted of a process illustrated in **Fig. 3.3** and **Fig. 3.4**. A glass pane was cleaned using acetone with mold release applied to the area where the fabric was placed. Sealant tape was placed around the infusion area with a spiral and semi-permeable spiral tube placed at both ends of the panel for resin infusion and vacuum port respectively. A Teflon release fabric was placed over the fiber mats followed by breather cloth and distribution media to guide flow. Subsequently a vacuum bag film was placed over the entire area and bonded to the sealant tape, followed by a leak/vacuum drop test for 1 hour to ensure maintained pressure throughout the infusion and curing process. Lastly the resin and hardener were mixed and infused through the fibers, and once infused, the system was closed off with the pump maintaining pressure three various curing conditions to evaluate the best Epotec composite performance. The three curing conditions included: 1) A room temperature (RT) cure for 48-hour under vacuum, 2) A 24-hour RT cure with an 8-hour 80°C post cure, 3) 24-hour cure at 80°C. The Elium composite was kept constant at a 24-hour RT cure and 2-hour 80°C post cure.

3.2.2 Experimentation

To assess the viability of Epotec as an improvement or alternative to epoxy and thermoplastic resins (EL), the various samples listed in **Table 3.4** were subjected to mechanical and thermal

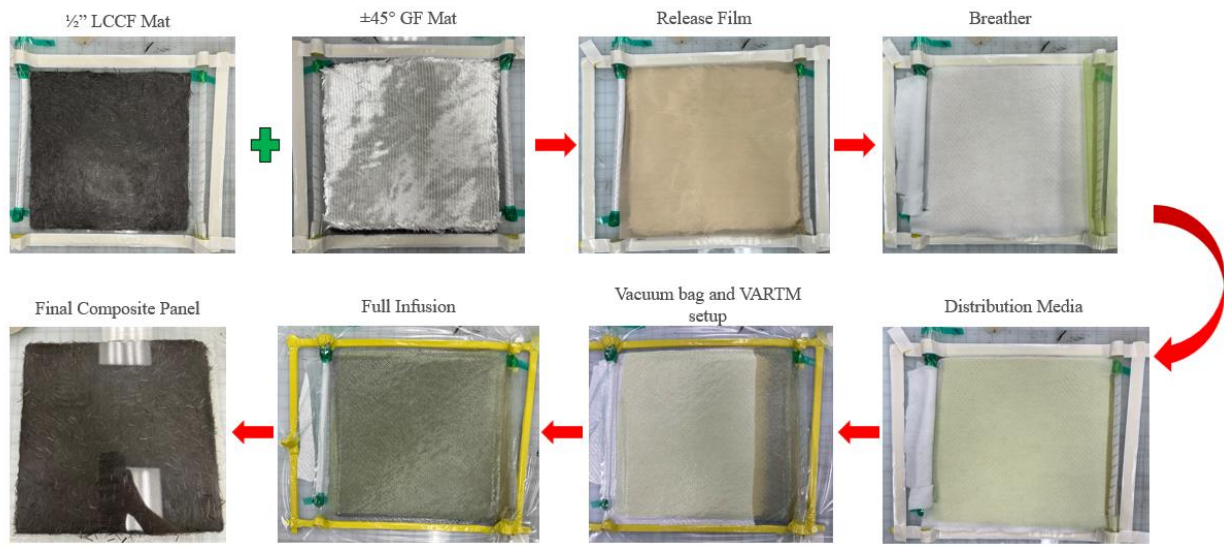


Figure 3.3: VARTM process for 12×12” composite panels using EP, EL, and epoxy resin systems. Sequential layup includes LCCF mat, glass fiber, release film, breather, distribution media, and vacuum bagging. Enables full resin infusion, producing the final composite panel.

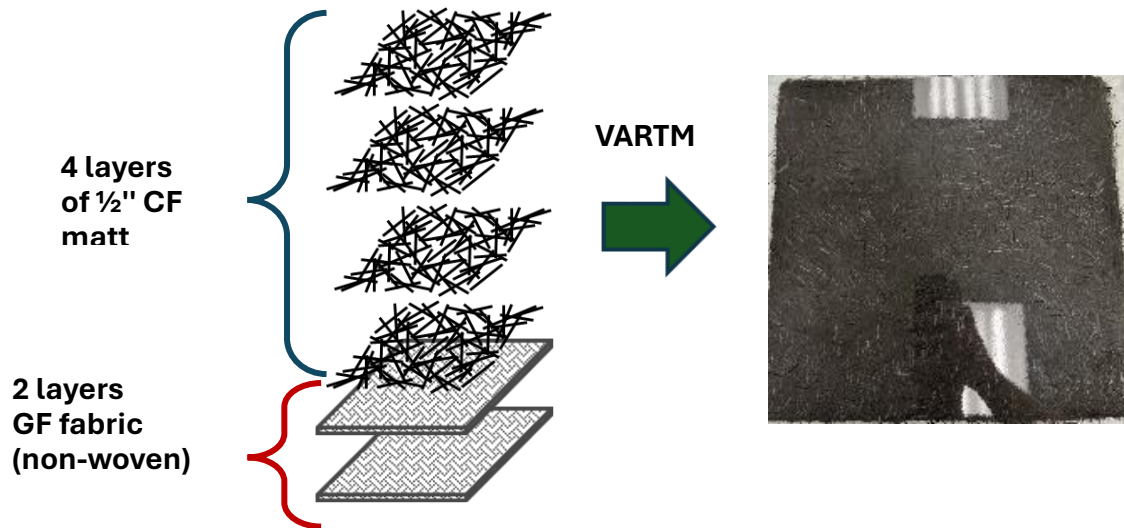


Figure 3.4: VARTM stacking sequence for hybrid composite layup. The laminate consists of 2 layers of $\pm 45^\circ$ knitted glass fiber and 4 layers of $\frac{1}{2}$ ” nonwoven LCCF. The same process was implemented for each composite with differing stacking sequences.

testing including microscopy for analysis of fiber failure and interlaminar shearing.

Additionally, a recycling study was conducted on neat cured Epotec and hybrid fiber reinforced composites using Epotec resin, acting as a preliminary trail for further evaluation. This section details parameters and equipment used for analysis.

3.2.2.1 Tensile Equipment and Testing D638

Type 1 tensile samples were cut by the OMAX 2626 JetMachining center CNC as seen in **Fig.3.5**, and a minimum of 6 samples were evaluated for strength, strain, and modulus of elasticity. The testing

The testing parameters for each composite structure had a breaking threshold of 2.224N, break sensitivity of 90%, data acquisition of 10Hz, and test speed of 2 mm/min. The thickness and width of the composite were recorded and input for strain and stress calculation based on data provided by an Epsilon Model 3542 extensometer as shown in **Fig.3.6**.

Tensile Strength

Equation 3.1

$$\sigma_f = F^{tu} = \frac{p^{max}}{A}$$

Tensile Strain

Equation 3.2

$$\sigma_i = \frac{p_i}{A}$$

Modulus of Elasticity

Equation 3.3

$$\epsilon_i = \frac{\delta_i}{L_g}$$

3.2.2.2 Flexural Testing D790-17

A minimum of five flexural samples were cut by method of OMAX 2626 JetMachining center CNC water jet with settings set for carbon-fiber laminate and a quality of. For hybrid composites, ten samples were cut allowing for flexural analysis of both sides in tension and

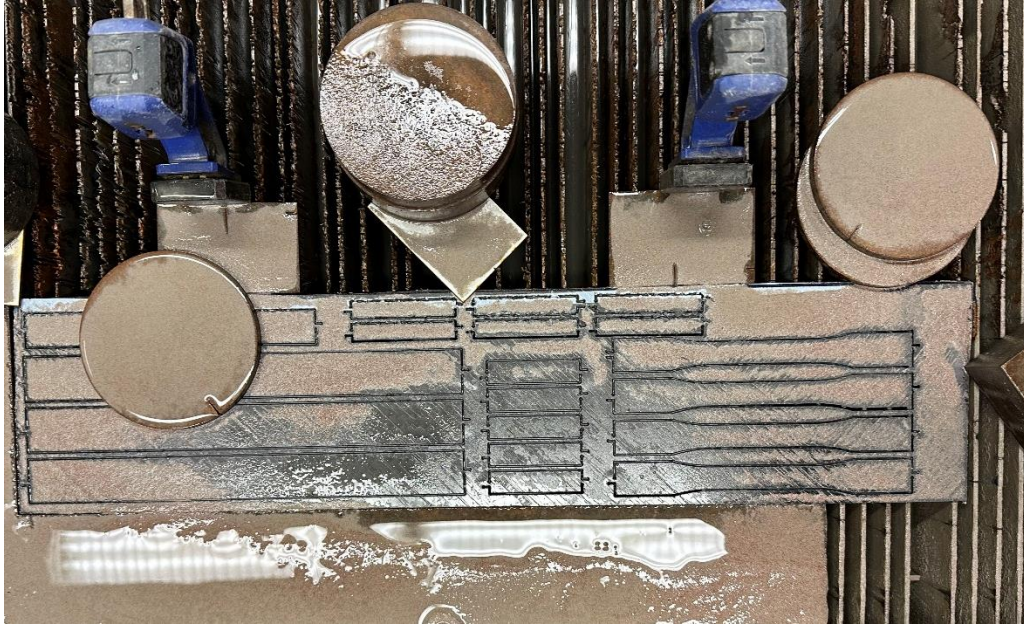


Figure 3.5: Tensile, flexural, and DMA sample cutting using the OMAX 2626 JetMachining center CNC water jet.

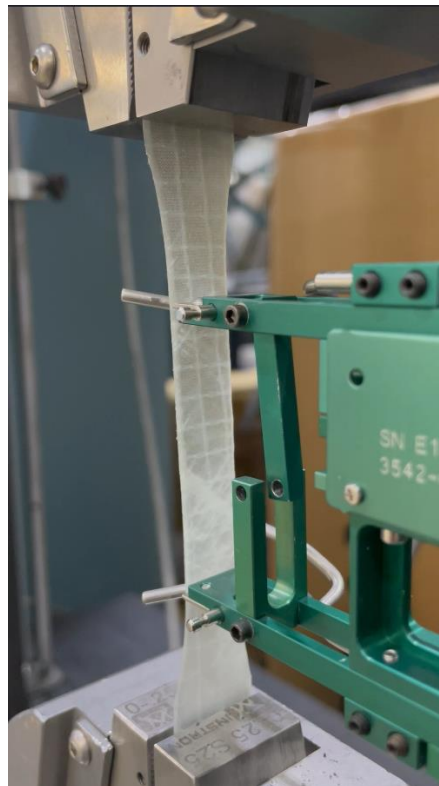


Figure 3.6: Tensile test setup for composite specimens. The specimen is clamped and loaded axially while strain is measured using an extensometer. This setup enables evaluation of mechanical properties such as tensile strength and modulus.

compression during 3-point bending operations. For this data set, the samples were tested on a 50 kN Test Resources frame (product #313Q, Test Resource, Minnesota, USA) and deflection recorded using deflectometer shown in **Fig. 3.7**. In accordance with ASTM Standard D790-17 samples dimensions cut to specification listed in equations **3.4-3.6** and tested at the calculated rate of crosshead motion detailed in equation **3.7**.

Sample Dimensions

Equation 3.4 $16d + 12.7 = \text{sample length [mm]}$

Equation 3.5 $16d = \text{span length [mm]}$

Equation 3.6 $12.7 = \text{width [mm]}$

Crosshead Rate of Motion

Equation 3.7
$$R = \frac{zL^2}{6d}$$

Midspan Deflection

Equation 3.8
$$D = \frac{rL^2}{6d}$$

Flexural Stress

Equation 3.9
$$\sigma_f = \frac{3PL}{2bd^2}$$

Flexural Strain

Equation 3.10
$$\varepsilon_f = \frac{6Dd}{L^2}$$

Modulus of Elasticity

Equation 3.11
$$E_f = \frac{L^2m}{4bd^3}$$



Figure 3.7: Test Resources 3-point bending setup with deflectometer. The composite specimen is supported on two ends (span length) while load is applied at the center. A deflectometer measures the tensile side mid-span displacement to determine flexural properties.

3.2.2.3 Dynamic Mechanical Analysis (DMA)

DMA was conducted on a minimum of 5 samples with a width of 9.5mm and length of 59mm cut by method of OMAX 2626 JetMachining center CNC water jet. The samples were tested in a DMA 850 instrument in accordance with ASTM D4065 standard test for plastic in flexion on a single cantilever beam as shown in **Fig. 3.8**. The thermoset composites were measured and compared for storage modulus (E'), loss modulus (E''), $\tan \delta$ (E''/E'), and glass transition temperature (T_g).

3.2.2.4 Scanning Electron Microscopy (SEM)

SEM was conducted on EP and EL ½” and 1” LCCF composites at 10 μm for each sample and 2 μm and 20 μm for ½” LCCF EP and El composites respectively. Operating conditions for each sample are depicted in **Table 3.5**.

3.2.2.5 Thermogravimetric Analysis (TGA)

TGA analysis was conducted on 3 samples: 1) Aditya Birla Epotec neat Resin, 2) ½” LCCF/Epotec Composite, 3) ½” LCCF/Elum Composite. Each sample had a mass of 10-15mg and was tested with a TGA5500 instrument in accordance with ASTM E1131. The experimentation used inert N_2 gas with a temperature ramp of 10.00°C/min to 900.00°C. The weight % drop and derivation weight (%/C) were recorded and compared for each sample.

3.2.3 Recycling EP-Based Composites

The Epotec resin system was evaluated for recycling and reclamation of materials through three different methods. The first method assessed the ability to dissolve the resin system in diluted acetic acid solution and reclaim the Recyclamine thermoplastic polymer by exposing the solution to a diluted sodium hydroxide solution to coagulate the thermoplastic. This process utilized a 441.05g block of cured neat resin dissolved in a 1:1 water to acetic acid solution at 80°C for 4-

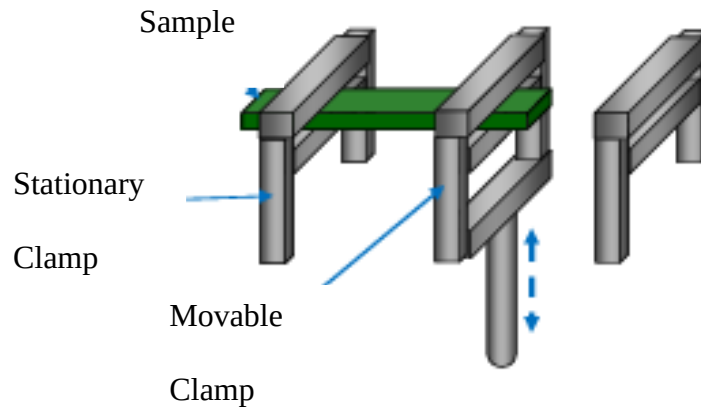


Figure 3.8: Dynamic Mechanical Analysis (DMA) single cantilever clamp setup per ASTM D5418. The composite sample is fixed at one while the other is deflected by a movable clamp. This configuration enables measurement of viscoelastic properties under dynamic loading.

Table 3.5: SEM Analysis Testing Specifications for ½” and 1” LCCF Composites

Sample	Resolution (μm)	EHT (kV)	WD (mm)	Mag	Signal A	Signal B	Mix Signal
Epotec 1/2” LCCF	10	1.00	4.0	100KX	In-Lens	SE2	0.1556
Epotec 1/2” LCCF	2	1.00	4.2	500KX	In-Lens	SE2	0.1556
Elium 1/2” LCCF	20	1.00	6.0	500X	In-Lens	SE2	0.1556
Elium 1/2” LCCF	10	1.00	5.0	100KX	In-Lens	SE2	0.1556
Epotec 1” LCCF	10	1.00	5.7	100KX	In-Lens	SE2	0.1556
Epotec 1” LCCF	10	1.00	6.5	100KX	In-Lens	SE2	0.1556

hours. The EP/Acetic acid solution was then transferred to a high surface area glass dish and exposed to a $6\pm0.5:1$ ratio of water to sodium hydroxide to coagulate the resin. To determine fiber separation in the EP composite, the second study used LCCF non-woven laminate which was dissolved in a borosilicate glass with the acetic acid solution from method 1 in container on an 80°C hotplate with stirrer for 24 hours. Lastly, the final study conducted established fiber recovery of hybrid knitted LCCF and GF using the procedure established in the second study while minimizing stirring speed and changing the duration to 2 hours.

Chapter 4: Results and Discussion

4.1 Thermo-Mechanical and Rheological Characterization of Neat and Fiber-Reinforced Resin Systems

4.1.1 Manufacturing Observations

The composites manufactured using VARTM as detailed in **Table 4.1** resulted in an average resin content of 31.32wt% with a standard deviation of 6.21% . The ½” LCCF nonwoven composites averaged nearly 37wt% for both Elium (EL) and Epotec (EP) while 1” LCCF composites averaged 33wt%. Surface quality between resins and fiber type was left unaffected between samples, with smooth surface finishes attributed to the glass panel and rough surfaces attributed to the bagging and distribution media as shown in **Fig. 4.1** and **4.2**. No polishing was conducted on the samples prior to experimentation and material consolidation with minimal voids were achieved with RT cure and post-curing process at 80°C.

4.1.2 Rheological Behavior

To determine the manufacturing conditions for the use of EP YDL 5567/THR 9369 in WTB applications, neat resin thermal and mechanical properties were evaluated. Among these, oscillatory shear flow, complex viscosity (η^*) was compared between traditional epoxy by Trimer, EP, and EL. As shown on **Fig. 4.3**, the complex viscosity (Pa.s) before curing for EP operated around 10 Pa.s while epoxy was found to be at 0.4 Pa.s operating within the standard viscosity for WTB manufacturing between 0.1-0.5 Pa.s. Elium in contrast to uncured resin property documentation of 100 mPa.s had a significantly higher viscosity measured at 400 Pa.s before curing making it considerably challenging for long wind blade VARTM. Furthermore, while epoxy demonstrated improved flow conditions, the operating time before curing, 9-10 minute, was considerably smaller than that of EL and EP resin at 50 and >200 minutes

Table 4.1: EP Composite Fiber/Resin Content

Composite	Stacking Sequence	Fiber Mass (g)	Post Infusion Mass (g)	Resin wt%
EP_1/2" LCCF	6 layers	98.	156.7	36.82
EP_1" LCCF		111.57	164	33.42
EP_CFF		108	172	37.21
EP_4GFF	4 layers	387.25	505.2	23.35
EP_GF/CF	GF/CF/GF/CF	236	310	23.87
EP_2GF_4CF1/2"	2GF/4CF	246.05	369.62	33.43

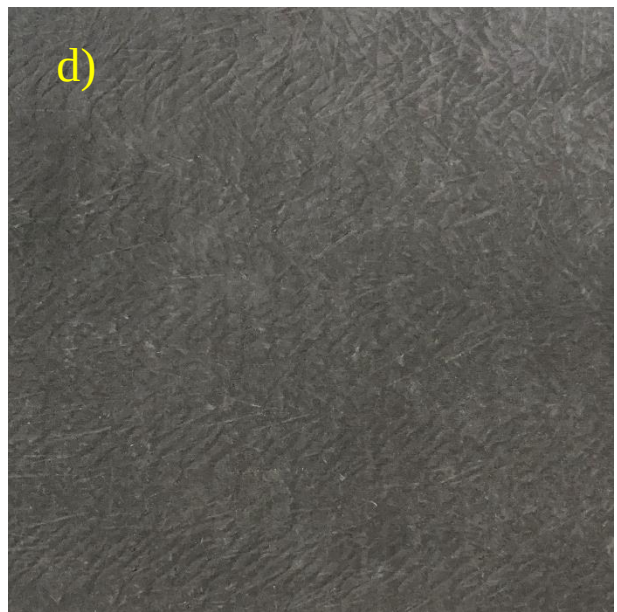
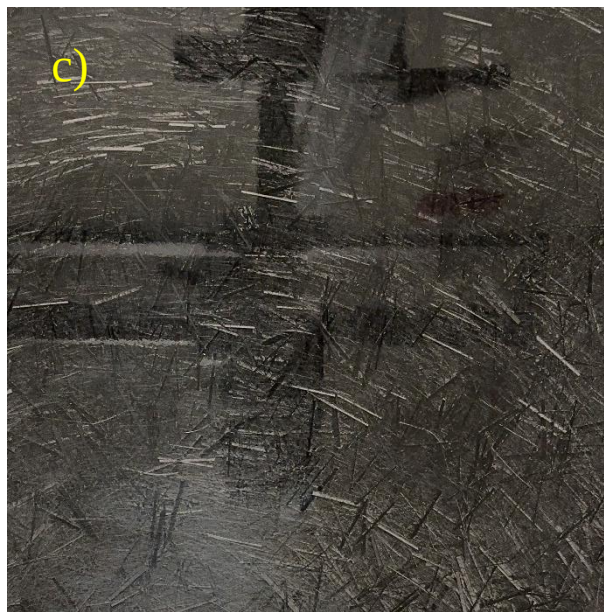
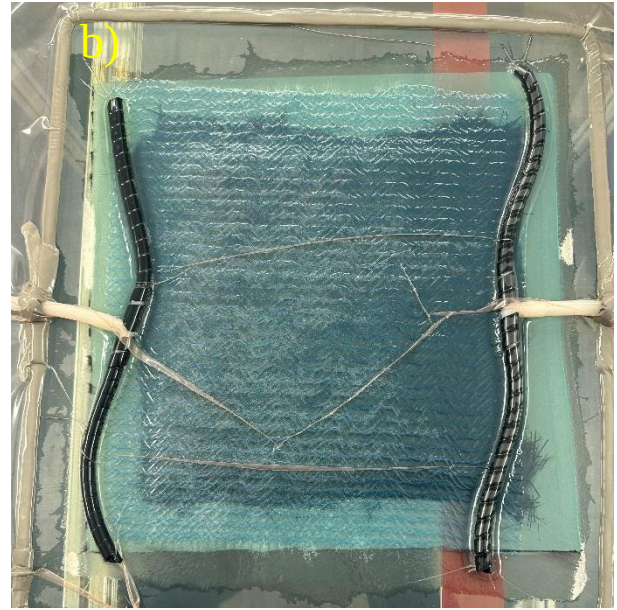
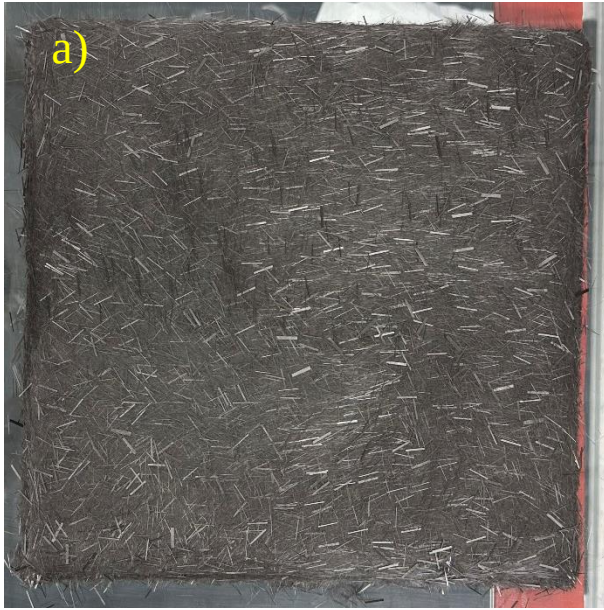


Figure 4.1: Composite surface evaluation post-VARTM process. (a) shows the initial LCCF layup; (b) depicts vacuum infusion in progress. (c) and (d) compare surface finishes on the glass-side and vacuum bag-side, respectively.

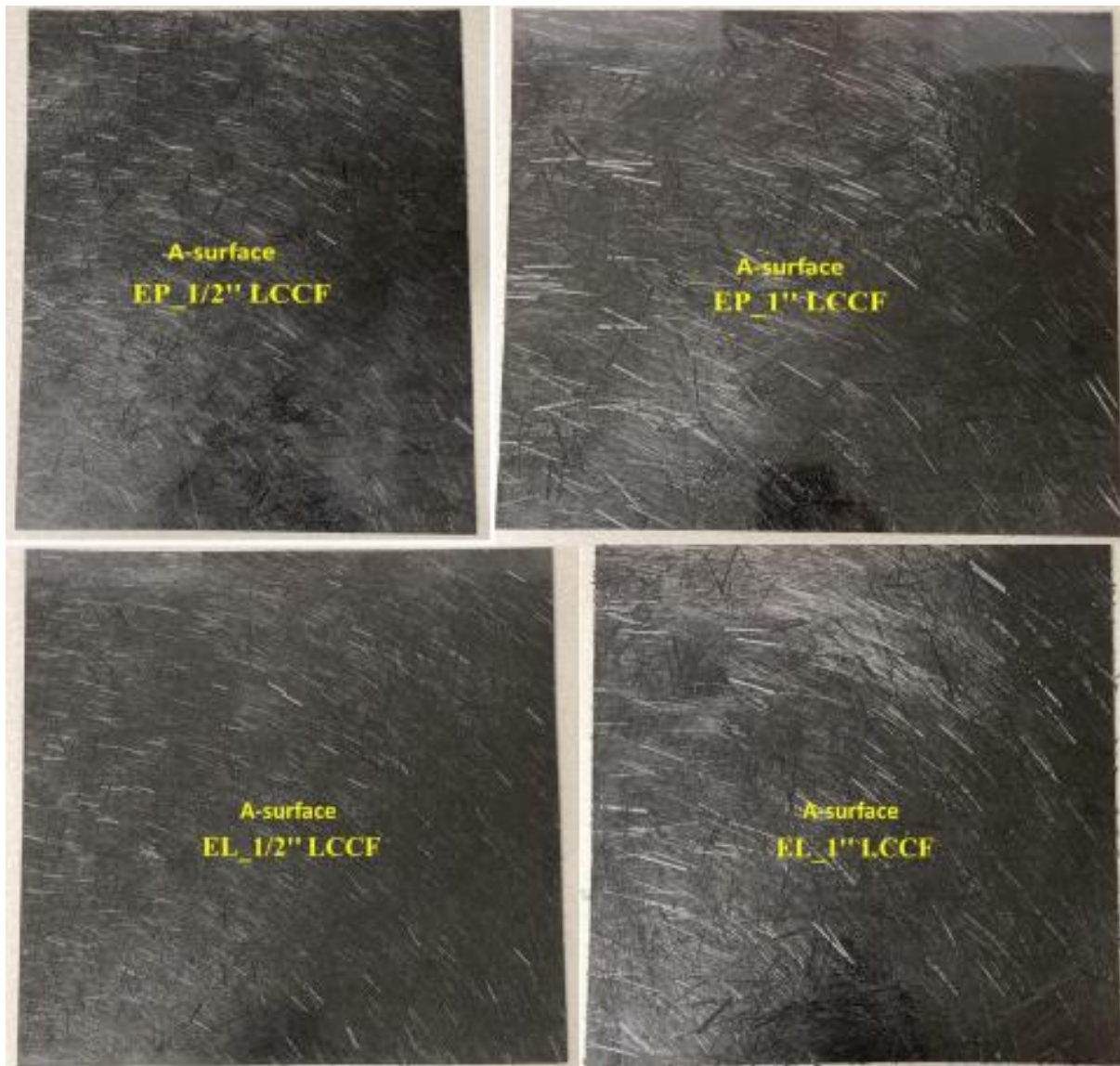


Figure 4.2: Surface quality comparison of EP- and EL-based composites using $\frac{1}{2}$ " and 1" LCCF mats. Top row shows EP-based composites; bottom row shows EL-based variants. Surface finish does not vary with resin system, but fiber length variation is visible.

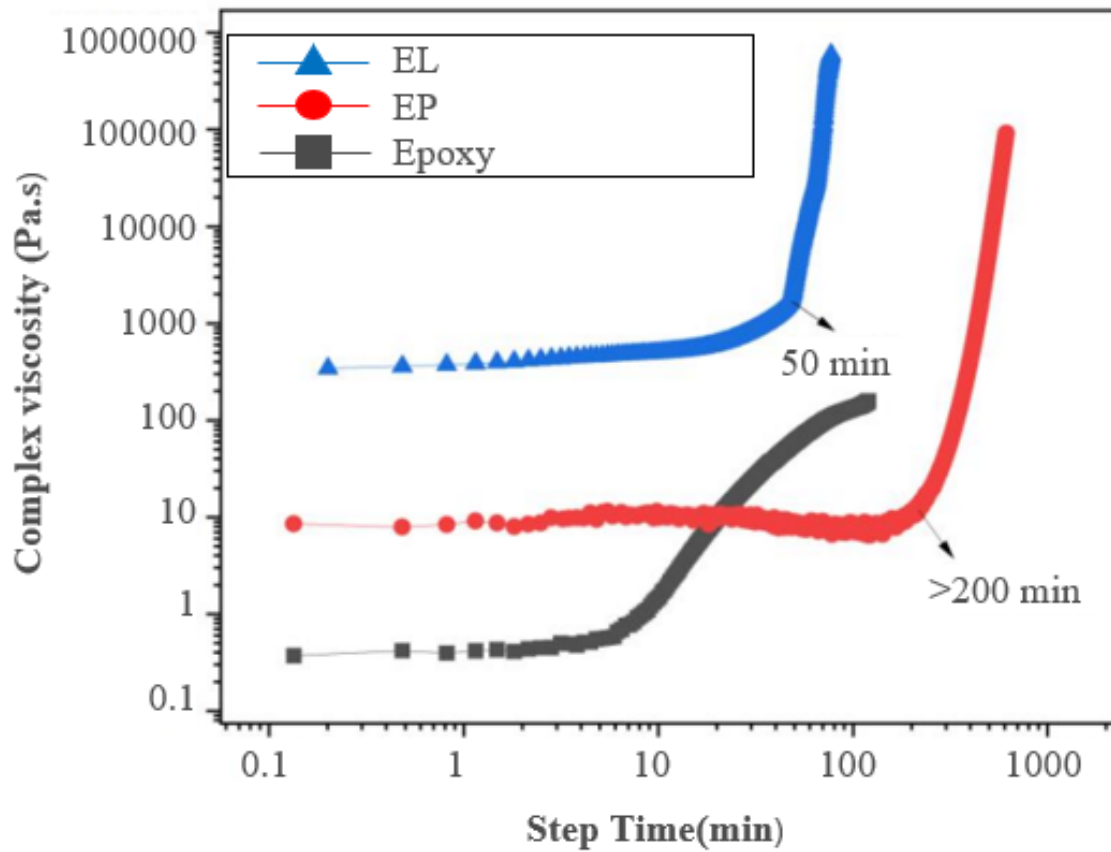


Figure 4.3: Complex viscosity comparison of Epoxy, EP, and EL resin systems over time. Epoxy exhibits the fastest rise in viscosity, gelling at ~50 minutes, while EL shows the slowest cure (>200 minutes). Longer processing windows of EP and EL resins offer improved flow and infusion characteristics for VARTM.

respectively. With a significant increase in time before curing compared to epoxy and EL along with a significantly low complex viscosity compared to the thermoplastic alternative (EL), EP exhibited desirable material properties for VARTM offering a higher processing window, lower viscosity, and recyclability for WTB composites.

4.1.3 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed on neat EL and EP resin systems, as well as on a composite made from EP and ½” discontinuous LCCF. All samples were tested from 40°C to 900°C at a heating rate of 10°C/min in a nitrogen atmosphere. The weight loss and derivative weight loss curves are shown in **Figures 4.4** and **4.5**.

4.1.3.1 Neat Resin Comparison: Elium vs. Epotec

Figure 4.4a presented the weight% loss as a function of temperature. EP demonstrates superior thermal stability compared to EL as indicated by its delayed onset of decomposition. Epotec exhibits a 5 wt.% loss at approximately 300°C, while Elium reaches the same loss at only 200°C. Additionally, Epotec retains a higher weight percentage at elevated temperatures, showing more gradual degradation behavior. The derivative thermogravimetric (DTG) curves in **Figure 4.4b** further emphasized this distinction. The primary degradation peak of Elium occurs at 306°C, while Epotec shows a broader and slightly higher peak at 370°C, with a secondary shoulder peak at 670°C indicating a second stage of thermal decomposition. The slower degradation rate and multiple degradation steps of Epotec showed significant improvement compared to Elium. It should be noted however, that the thermal energy required for pyrolysis will be directly associated with the degradation temperature and rate of thermal decomposition.

4.1.3.2 Neat Epotec vs EP/LCCF Composite

Figure 4.5 compared the neat EP resin with the EP and ½” discontinuous LCCF based

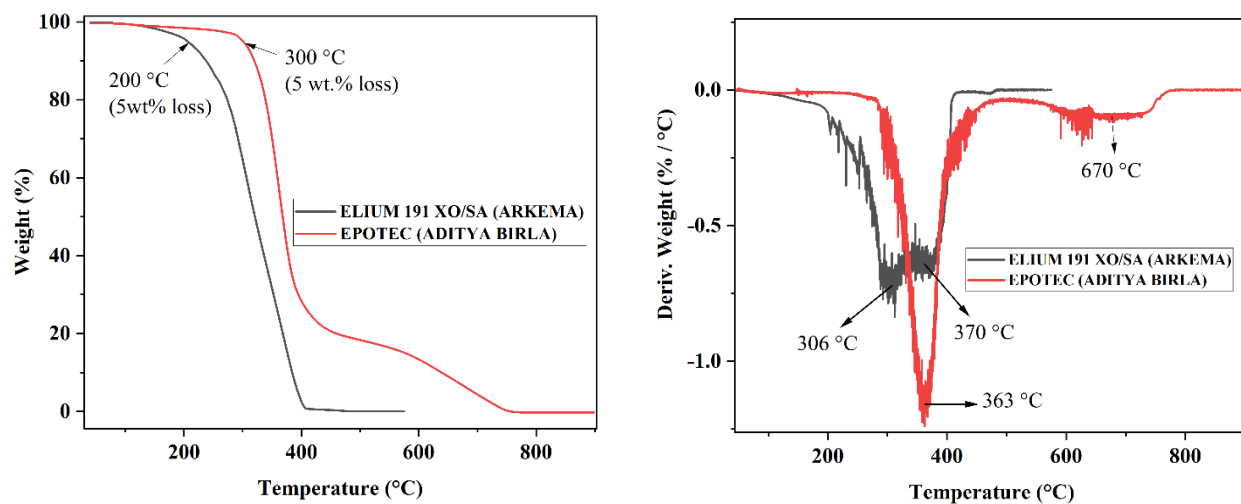


Figure 4.4: TGA of neat EL and EP resin from 40°C-900 °C at 10 °C/min: a) Weight percent drop vs. time, b) Derivative of Weight % as a function of temperature.

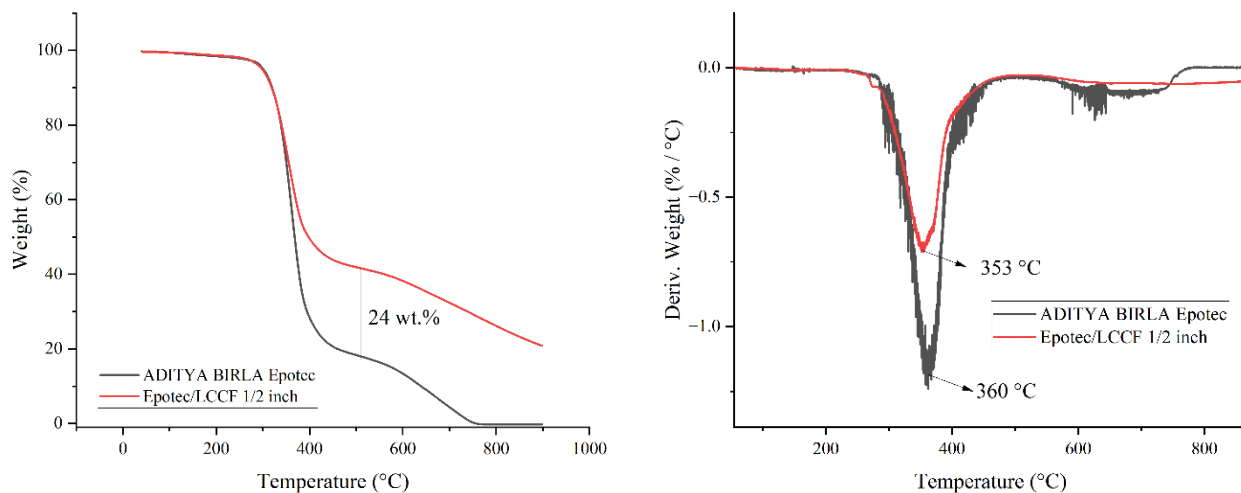


Figure 4.5: TGA comparison of neat EP resin and nonwoven 1/2" fiber from 40°C-900 °C at 10 °C/min: a) Weight percent drop vs. time, b) Derivative of Weight % as a function of temperature.

composite. The weight loss curve in **Figure 4.5a** revealed that the composite retained significantly more mass throughout the thermal cycle. At 900°C, the EP/LCCF composite exhibited approximately 24 wt.% greater residual mass than the neat EP, indicating the contribution of the thermally stable carbon fiber to the overall structure.

The DTG curves in **Figure 4.5b** showed that both the neat EP and the EP_1/2”LCCF composite had similar peak degradation temperatures, with EP and EP_1/2”LCCF peaking at 353°C and 360°C respectively. This suggested that the presence of LCCF did not significantly alter the degradation mechanism of the resin matrix. However, the increased char residue confirmed the composite’s improved thermal resistance. Furthermore, while epoxy demonstrated improved flow conditions, the operating time before curing, 9-10 minute, was considerably smaller than that of EL and EP resin at 50 and >200 minutes respectively. With a significant increase in time before curing compared to epoxy and EL along with a significantly low complex viscosity compared to the thermoplastic alternative (EL), EP exhibited desirable material properties for VARTM offering a higher processing window, lower viscosity, and recyclability for WTB composites.

4.1.4 Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) was conducted to evaluate the viscoelastic behavior of the neat EL and EP resin systems, ½” and 1” discontinuous LCCF composites using both resin systems, and hybrid composite structures using EP. Key properties analyzed included storage modulus (E'), loss modulus (E''), mechanical loss factor ($\tan \delta$), and glass transition temperature (T_g).

4.1.4.1 Neat Resin DMA

Figure 4.6 displayed the DMA results for neat EL and EP resins. The EP resin exhibited a

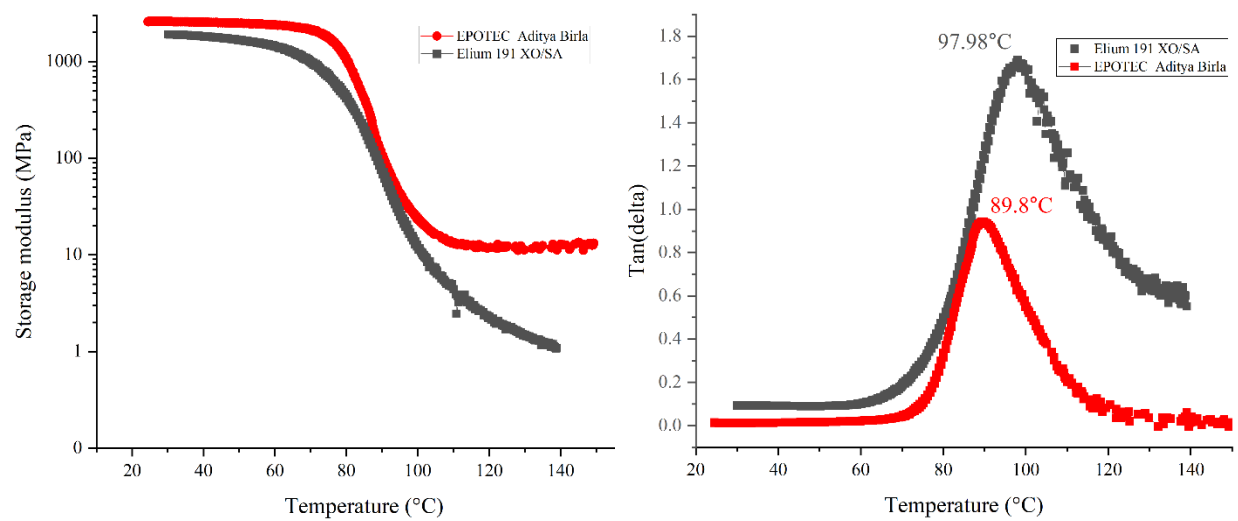


Figure 4.6: DMA results for neat EPOTEC (EP) and Elium (EL) resin systems. Left: EP resin exhibits higher storage modulus across temperatures, indicating greater stiffness. Right: Tan δ peaks at 89.8°C for EP and 97.98°C for EL, reflecting differences in T_g.

slightly higher storage modulus than EL, measuring approximately 1200 MPa compared to 1100 MPa. This suggests that EP provided a stiffer matrix under dynamic loading conditions.

In terms of damping behavior, EL demonstrated a significantly higher $\tan \delta$ peak of approximately 1.7, indicating greater molecular mobility near T_g , compared to EP, which exhibited a $\tan \delta$ peak of 0.9. The glass transition temperature (T_g), determined at the peak of the $\tan \delta$ curve, was also higher for EL at 97.98°C compared to 89.8°C for EP. The combination of a higher storage modulus and lower $\tan \delta$ in EP indicated reduced damping and increased rigidity relative to EL under similar thermal conditions. Therefore, making EP resin suitable for increasing the thermos-mechanical performance of WTB composite structures.

4.1.4.2 Effect of Fiber Length and Resin

Figure 4.7 illustrated the influence of fiber length ($\frac{1}{2}$ " vs. 1") and resin type (EL vs. EP) on the viscoelastic properties of LCCF-reinforced composites. Incorporating fibers significantly enhanced the storage modulus in both systems. Compared to their neat counterparts, EP- and EL-based composites with $\frac{1}{2}$ " LCCF exhibited a 650% and 718.18% increase in storage modulus, respectively. However, increasing fiber length from $\frac{1}{2}$ " to 1" resulted in no significant increase in modulus for either system.

A notable trend was observed in the T_g values of the composites. For EP-based composites, T_g increased from 93.3°C to 106.7°C for EP_ $\frac{1}{2}$ "LCCF and EP_1"LCCF, respectively. Similarly, EL-based composites exhibited an increase in T_g from 111.06°C to 115.83°C when fiber length increased from $\frac{1}{2}$ " to 1". These shifts indicated that longer fiber lengths improved thermal resistance and restricted polymer chain mobility near the glass transition further establishing the use of long discontinuous fiber in WTB application to increase resistance in instances of lightning strikes.

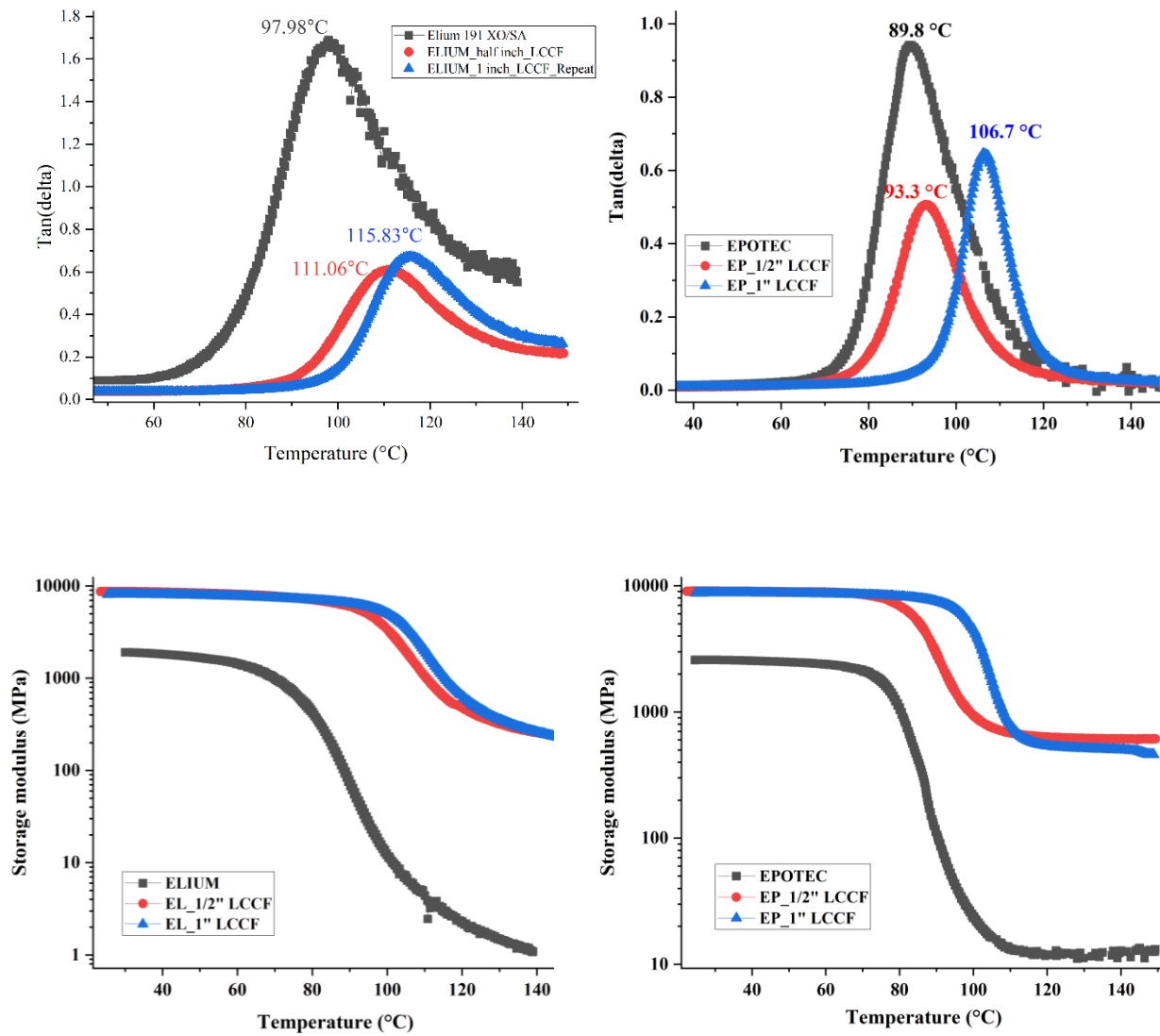


Figure 4.7: Comparative evaluation of neat resin and effects of nonwoven fiber length for EL (EL) and EP (EP) composites: a) Representative curves for loss factor and Tg of EL composites), b) Representative curves for loss factor and Tg of EP composites, c) Representative curves for storage modulus of EL composites d) Representative curves for storage modulus of EP composites.

Additionally, both EP and EL composites showed an increase in the $\tan \delta$ peak height with fiber length. EP_1”LCCF showed a 40% increase in $\tan \delta$ compared to EP_1/2”LCCF and EL_1”LCCF showed a 16.67% increase in damping peak compared to EL_1/2”LCCF. This observation shows longer fibers likely contribute towards increased deformation before fracturing, thus, improving the thermos-mechanical attributes offered by nonwoven LCCF. Furthermore, all composite systems showed an overall reduction in $\tan \delta$ relative to their neat resin counterparts, reflecting decreased viscoelastic damping and increased structural thermos-mechanical performance due to fiber reinforcement.

4.1.4.3 Hybrid Composite DMA

Figure 4.8 compared two EP-based hybrid composites: a four-layer alternating (EP_GF/CF) composite and a four-layer 1/2” LCCF and two-layer GF composite (EP_2GF_4CF1/2”). Results showed that EP_2GF_4CF1/2” exhibited a higher T_g of 103.9°C and marginally higher $\tan \delta$ of 0.56 compared to EP_GF/CF with a T_g of 98.8°C and $\tan \delta$ of 0.54. Similarly, EP_2GF_4CF1/2” had a storage modulus of about 10000MPa compared to EP_GF/CF at 5500 MPa resulting in an 81.8% higher value. This reduction in storage modulus and $\tan \delta$ for hybrid GF and CF kitted composites might be attributed to less efficient interfacial load transfer due to poor interfacial adhesion, stiffness mismatching, and/or wettability. Further evaluation of the composites failure and microstructure are evaluated to confirm this theory. However, this reduction is further emphasized by the results found in **Fig. 4.9** comparing the storage modulus, T_g , and $\tan \delta$ of 4-layer GF (EP_4GFF) to EP_GF/CF. The DMA showed an increased storage modulus 11000 MPa and exhibited a lower T_g of 93.9°C and $\tan \delta$ of 0.35 confirming LCCF contributed to thermal performance. In addition, uniform GF significantly improved the storage modulus compared to EP_GF/CF and reduced damping, suggesting that EP_GF/CF improved T_g

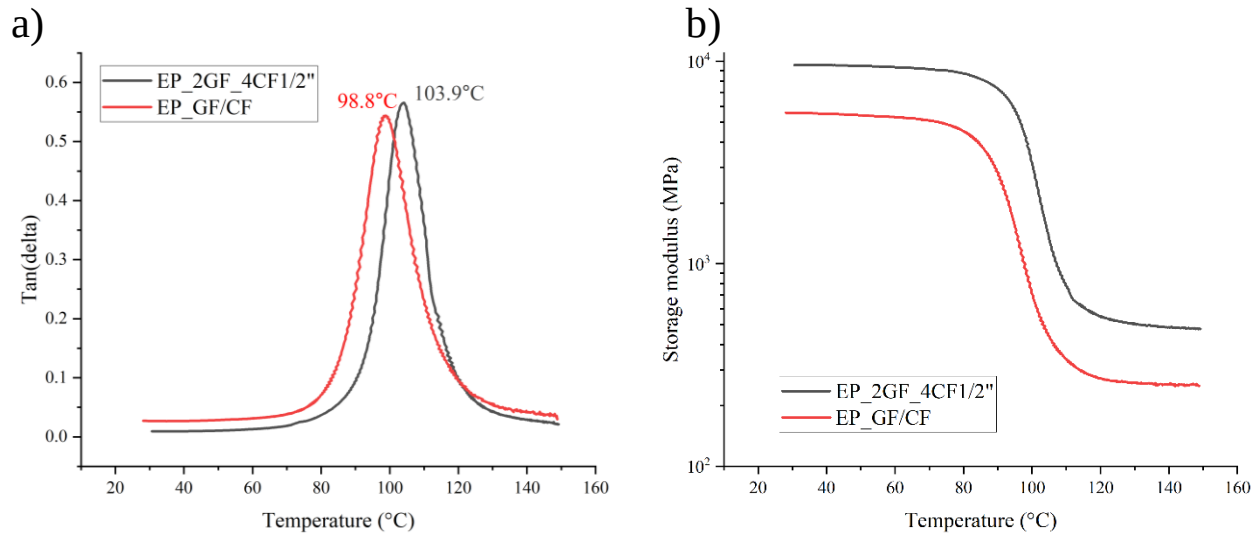


Figure 4.8: Hybrid Composite DMA analysis of four-layer alternating GFF and CFF (red) and four-layer 1/2" LCCF, 2-layer GF (black) using EP: a) Loss factor analysis, b) Storage modulus analysis.

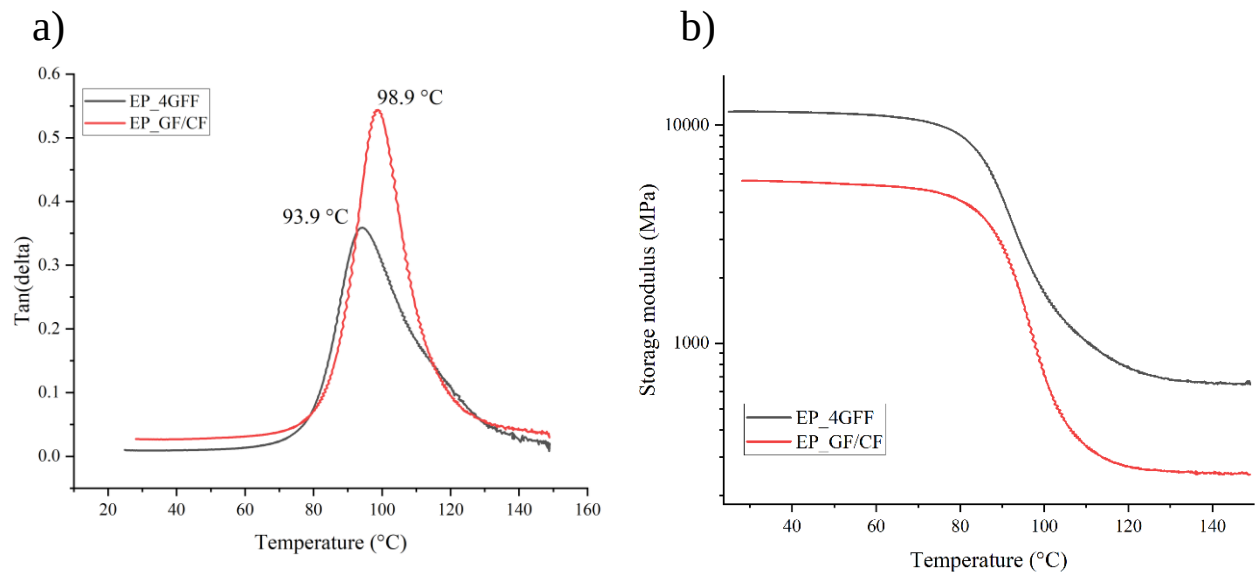


Figure 4.9: Hybrid Composite DMA analysis of four-layer alternating GFF and CFF (red) and four-layer GF (black) using EP: a) Loss factor analysis, b) Storage modulus analysis.

and damping significantly reduced the stiffness of the composite due to the heterogeneous fiber-matrix.

When compared to ½” and 1” LCCF composites depicted in **Fig. 4.10**, EP_1”LCCF showed a significant increase in T_g and $\tan \delta$ by 13.63% and 85% respectively compared to EP_4GFF without compromising the storage modulus. This indicates that randomly oriented fibers enhanced damping capacity and thermal resistance with fiber length playing a critical role enhancing damping as EP_GF/CF offered a midway point between the two composite structures. These finds showed that EP_4GFF exhibited the highest overall storage modulus highlighting improved material properties in uniform composites. However, EP_1”LCCF retained the materials modulus more effectively at elevated temperatures while improving damping, indicating improved thermo-mechanical performance was achieved used LCCF technology and emphasizing the need for CF in WTB composite structures to improve thermal durability.

4.2 Mechanical Characterization of Nonwoven and Hybrid LCCF/GF Composites

4.2.1 Flexural and Tensile Characterization

Mechanical testing was conducted to assess the tensile and flexural properties of composite laminates reinforced with LCCF and various stacking sequences of knitted glass fiber fabric (GFF) and CF. The study focused on two categories of reinforcement architectures: 1) nonwoven ½” and 1” LCCF reinforced matrix systems using EL and EP, and 2) hybrid laminate structures integrating discontinuous and continuous fiber layers. All samples were fabricated as defined in **Table 3.4** and tested in accordance with ASTM standards D638 and ASTM D790-17.

4.2.1.1 Nonwoven ½” and 1” LCCF Composites using EP and EL

The mechanical behavior of nonwoven LCCF composites with EP and EL resin systems and two different fiber lengths (½” and 1”) was evaluated under tensile and flexural loading as shown in

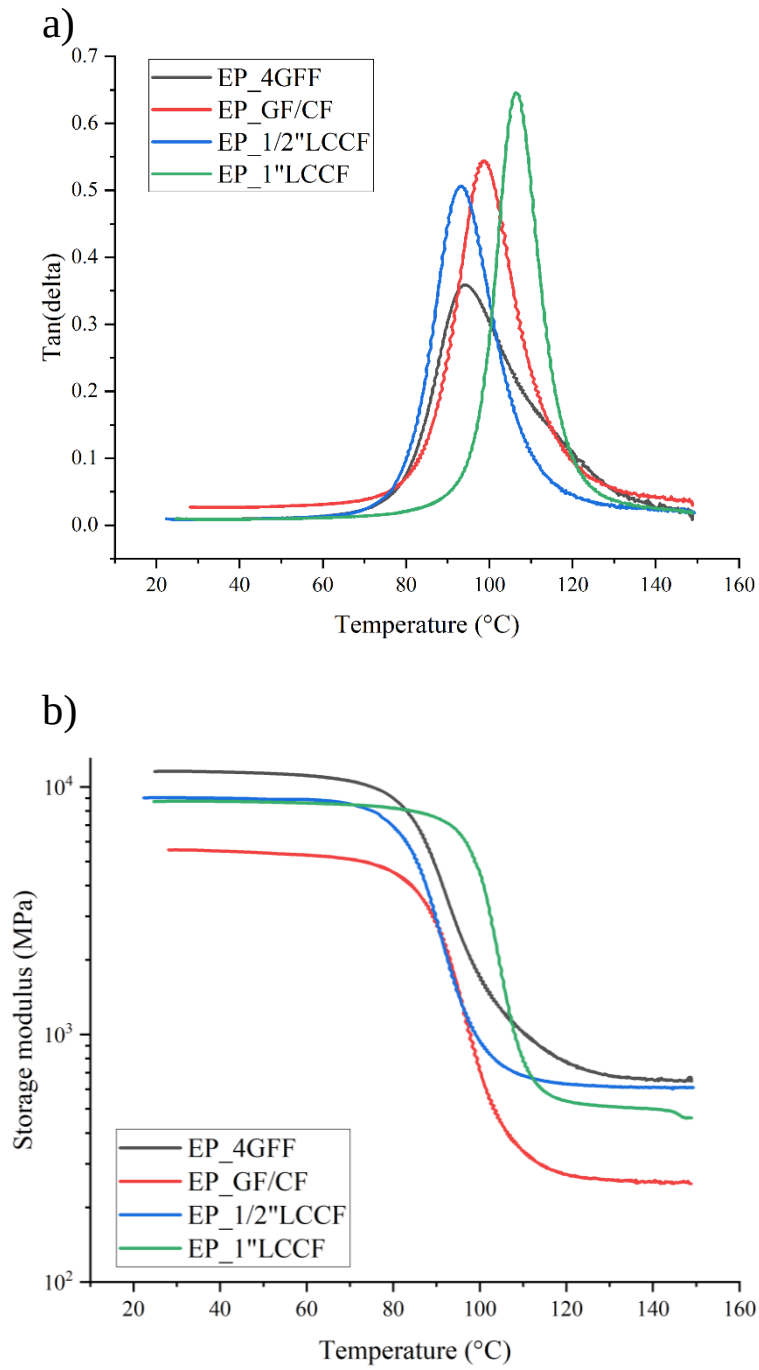


Figure 4.10: DMA comparison of nonwoven ½" and 1" LCCF composites and hybrid composites using EP: a) Loss factor analysis, b) Storage modulus analysis.

Fig. 4.11 and 4.12.

In flexural testing, the EP_1" LCCF sample displayed the highest mechanical performance, with a flexural strength of 797.02 MPa and modulus of 116.35 GPa. This surpasses both EP_½" LCCF (453.05 MPa, 24.8 GPa) and EL_1" LCCF (478.23 MPa, 21.03 GPa), indicating that longer fiber reinforcement and EP resin system significantly improved the overall stiffness and strength. However, EL_½" LCCF showed comparable flexural strength (462.68 MPa) to the EL_1" LCCF and EP_1/2" LCCF with a higher modulus (33.59 GPa) indicating that ½" fibers increased EL-based composite stiffness with negligible affects to strength like due to improved fiber wettability.

Tensile tests displayed similar trends as EP_1" LCCF achieved the highest tensile strength at 134 MPa, slightly exceeding EL_½" LCCF (131.6 MPa). The increased strength observed significantly smaller standard deviation, indicated signs of better part consolidation between these composites. Furthermore, both ½" and 1" EP composites exhibited a higher Young's Modulus (23.5 GPa, 22.43 GPa) than EL at (20.05 GPa, 20.05 GPa), respectively. This observation further emphasizes the improved stiffness characteristics EP composites provide in comparison to EL composites. In addition, EP_½" LCCF directly cured at 80°C opposed to a 24-hour RT cure and 8-hour 80°C post cure demonstrated significantly reduced properties by 36.29% and 46.06% for tensile strength and modulus respectively, indicating that curing conditions critically affect mechanical integrity.

4.2.1.2 Hybrid Composites LCCF and GF composites

Hybrid composite laminates were fabricated using strategic combinations of knitted GF, LCCF, and nonwoven LCCF mats using EP. The influence of stacking sequence, fiber type, and fiber architecture on mechanical performance was evaluated under both flexural and tensile

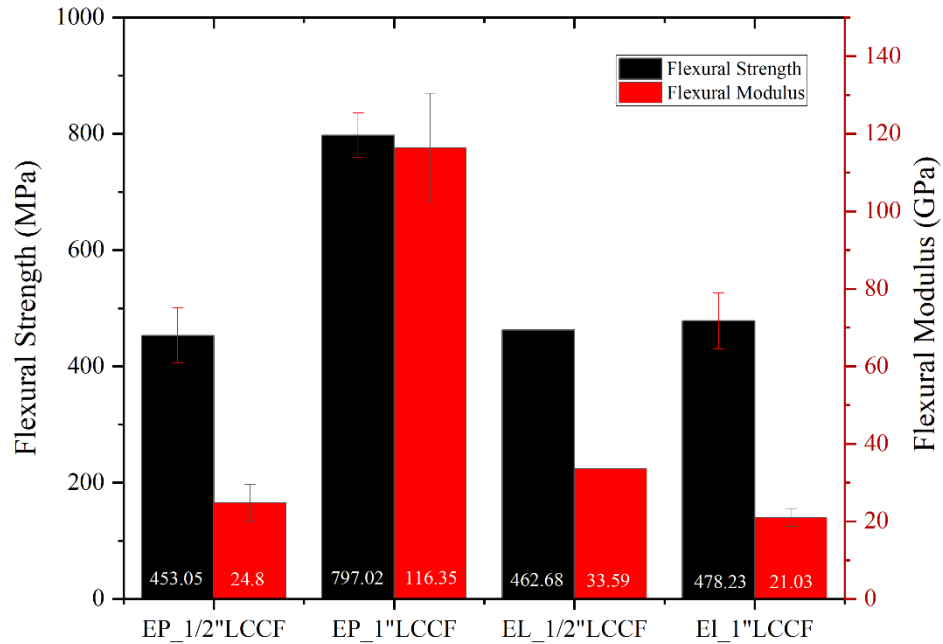


Figure 4.11: Flexural strength and modulus of nonwoven 1/2" and 1" LCCF laminates with EP and EL resins. EP_1" LCCF shows the highest flexural strength (797.02 MPa) and modulus (116.35 GPa). Overall, EP-based composites outperform EL-based ones in strength and stiffness.

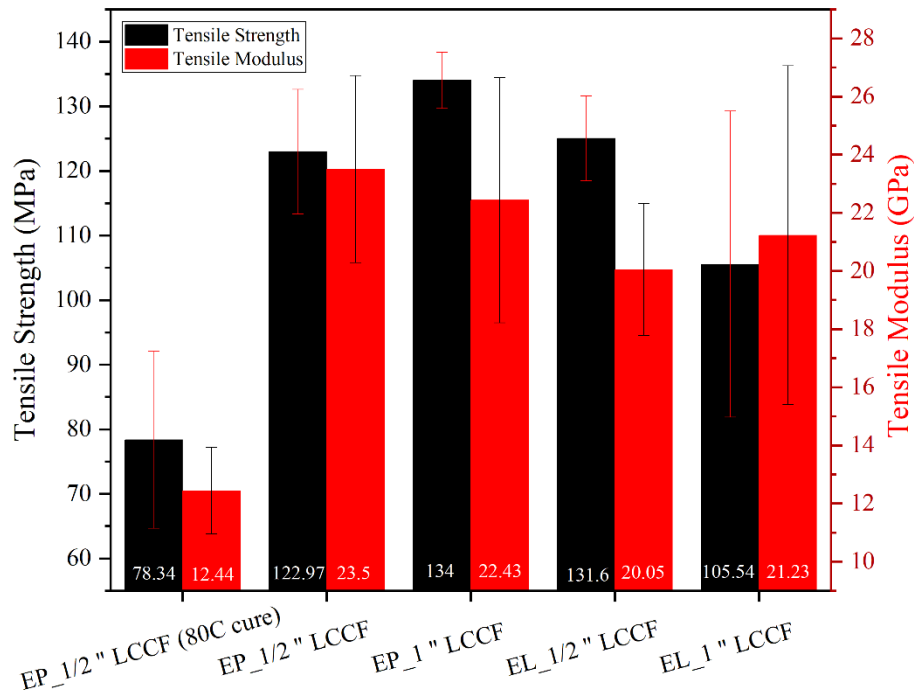


Figure 4.12: Tensile strength and modulus of nonwoven 1/2" and 1" LCCF laminates using EP and EL resins. An additional EP_1/2" LCCF laminate cured at 80°C for 24 hours is included for comparison displaying reduced performance. EP_1" LCCF exhibits the highest tensile strength (134 MPa), while EL-based composites show slightly lower values.

conditions. The EP_2GF_2CF flexural analysis was evaluated for CF in both tension (GF_Top) and compression (CF_Top). The tested configurations are depicted in **Table 3.4**.

As shown in **Fig. 4.13**, the hybrid system EP_2GF_4CF $\frac{1}{2}$ ” exhibited the highest flexural strength (858.38 MPa) and flexural modulus (24.49 GPa), indicating improved mechanical performance using nonwoven LCCF compared to knitted LCCF. Furthermore, significant improvement in stiffness was observed based on the integration of nonwoven $\frac{1}{2}$ ” LCCF and knitted LCCF as all samples resulted in an increase in flexural modulus compared to EP_GFF. Additionally, the LCCF when under compression depicted in EP_2GF_2CF(CF_Top) and EP_2GF_4CF $\frac{1}{2}$ ” offered significantly higher flexural modulus improving the overall stiffness by 195.04% and 268.37% respectively when compared to CF in tension. However, LCCF in compression didn’t significantly improve flexural strength as EP_GFF (279.29 MPa) outperformed both EP_2GF_2CF samples. Therefore, the reduced strength was likely attributed to suboptimal load transfer possibly due to fiber orientation or interface behavior between the woven layers. This indicates that interface behavior and load transfer were likely improved by using discontinuous fiber for hybrid composite structures under flexion.

Tensile properties shown in **Fig.4.14** further established mechanical improvement using discontinuous LCCF as EP_2GF_4CF $\frac{1}{2}$ ” resulted in the highest tensile strength and modulus of 103.4 MPa and 48.2 GPa, respectively, emphasizing the contribution of short, randomly oriented LCCF in resisting axial deformation by improving EP_GFF modulus by 175.43% . In contrast, EP_GF/CF exhibited the lowest tensile modulus at 12.6 GPa, despite having a comparable strength (86.3 MPa) to EP_4GFF. This suggests that interfacial mismatch between knitted LCCF and GF reduced stiffness efficiency, while all-glass laminates achieved better cohesion and modulus retention indicating poor interfacial performance.

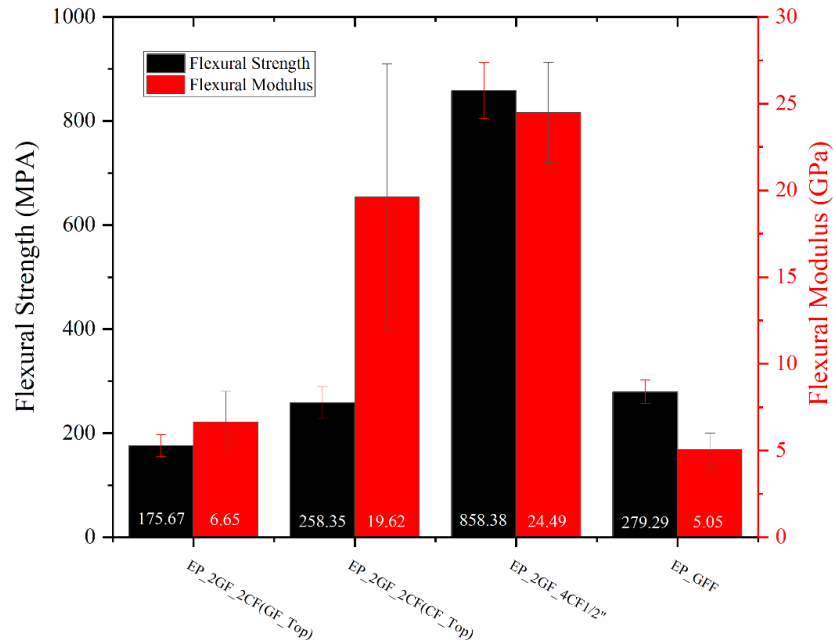


Figure 4.13: Flexural strength and modulus of hybrid LCCF and GF laminates with EP resin. The EP_2GF_4CF($\frac{1}{2}$ ") laminate shows the highest flexural strength (858.38 MPa) and modulus (24.49 GPa).

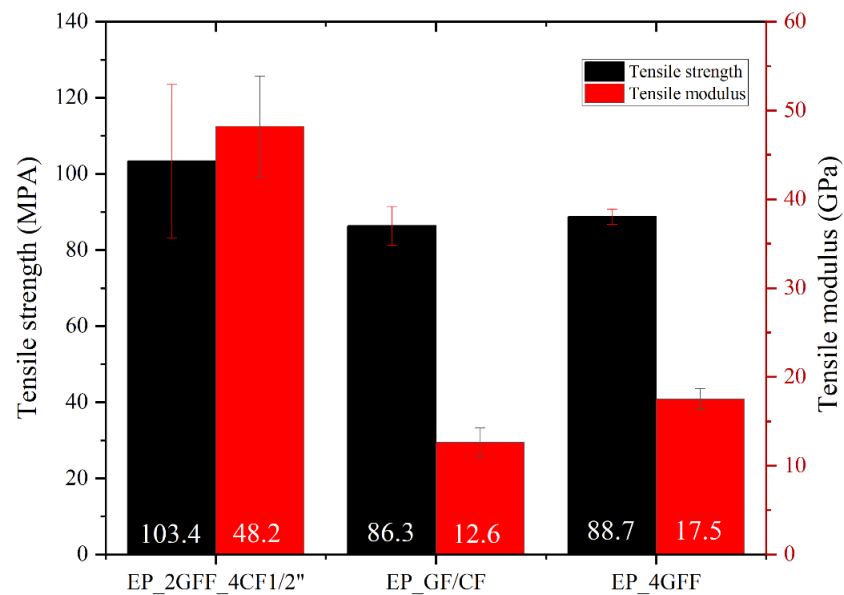


Figure 4.14: Tensile strength and modulus of hybrid LCCF and GF laminates with EP resin. The EP_2GFF_4CF($\frac{1}{2}$ ") laminate exhibits the highest tensile modulus (48.2 GPa) and strength (103.4 MPa). Discontinuous nonwoven LCCF significantly improves tensile performance.

4.2.1.3 Overall Comparative Analysis Nonwoven and Hybrid Composites using EP and EL

A full comparison of all composite systems is presented in **Fig. 4.15-**. Among the tested composite, EP_1”LCCF demonstrated the lowest overall flexural strength-to-stiffness ratio, achieving a flexural modulus of approximately 116 GPa and a corresponding flexural strength of 797 MPa. While EP_2GF_4CF½” improved the flexural strength by 7.65% compared to EP_1”LCCF, the composite's flexural modulus was reduced by 79.3% significantly improving damping but compromising stiffness. Additionally, the mass of EP_1”LCCF reduced the overall mass by 55.63% resulting in a 2.093:1 strength-to-weight ratio between EP_1”LCCF and EP_2GF_4CF½”. Overall, both the ½” and 1” nonwoven materials using EP and EL resulted in an increased flexural modulus compared to hybrid and GF composites while improving flexural strength evident by the 207% increase in flexural strength between EP_GFF and EP_2GF_4CF½”.

Furthermore, tensile observations showed significant improvement of tensile strength in nonwoven ½” and 1” LCCF panels using EP and EL compared to hybrid and GF composites. EP_1”LCCF showed the highest tensile strength while EP_2GF_4CF½” improved the tensile modulus by 114% indicating hybrid discontinuous LCCF and directional kitted GF provide improved stiffness while maintaining a high tensile strength. This response demonstrated the efficacy of combining nonwoven LCCF and GF for developing stiff composites that are lightweight and high strength at a low cost.

4.2.2 Microscopy

4.2.2.1 Optical Microscopy

Optical microscopy imaging of EP samples was evaluated to identify interlaminar fiber properties, fracture formations, and differences between stacking sequences of LCCF and GF.

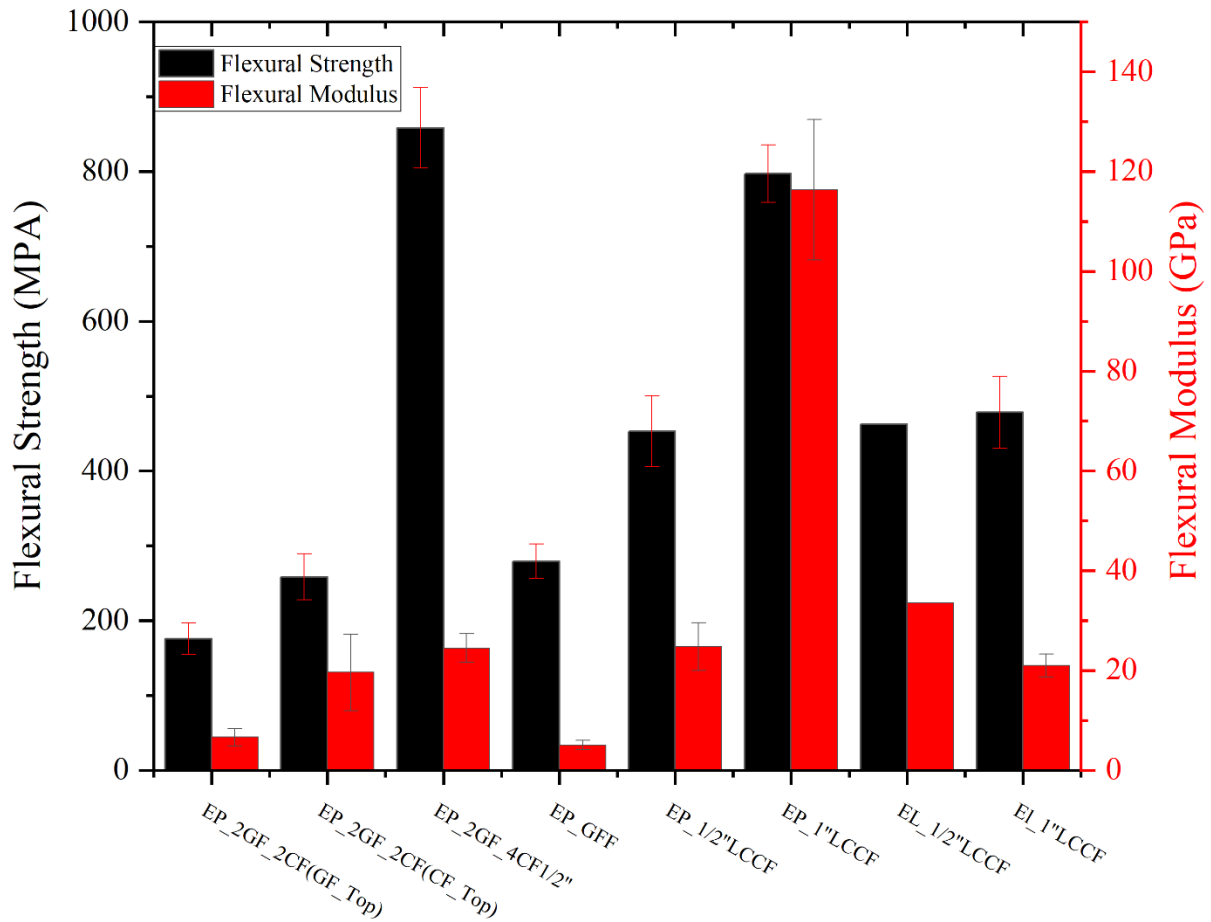


Figure 4.15: Flexural strength and modulus comparison between nonwoven and hybrid thermoset composites. Hybrid laminates with optimized stacking (e.g., EP_2GF_4CF1/2") and EP_1\"LCCF show superior mechanical performance. Nonwoven EL-based composites exhibit lower values, highlighting the effect of resin type and fiber architecture.

Therefore, the four samples evaluated under optical microscopy were: 1) ½” LCCF (4-layers) & GFF (2-layers), 2) Alternating CFF and GFF (4-layers total), 3) GFF (4-layers), and 4) ½” LCCF (4-layers) & CFF (2-layers). As shown on **Fig.16**, there was significant delamination between layers in all samples. However, the failure due to tension displayed two distinct forms of failure dependent upon the material and fiber structure. Firstly, the nonwoven ½” LCCF displayed significant fiber breakage at failure with minimal delamination at both the GFF and CFF. Furthermore, the ½” LCCF_GFF sample showed minimal signs GFF fracture indicating an increase in composite stiffness and resistance to elongation. Likewise, the ½” LCCF_CFF had similar fracture lines in the nonwoven layers, but delamination occurred between the CFF layers. Secondly, predominant composite failure due to significant interlaminar shearing occurred in the GFF and alternating CFF and GFF samples depicted in **Fig. 4.16** and **Fig. 4.17a-b**. Fracture occurred in the 45/45° plane, however significant structural failure can be attributed to the delamination occurring between CF and GF. Both results indicate that failure in tension for nonwoven materials was likely due to fiber fracture as seen in **Fig. 4.17d** and surface bonding between CF and GF is improved by using discontinuous CF compared to kitted mats. Therefore, the addition of nonwoven LCCF significantly improved the hybrid matrix stiffness with failure occurring due to fiber fracture.

4.2.2.2 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to investigate the fracture morphology of the tensile-tested EP- and EL-based nonwoven LCCF composites. The micrographs in **Fig.4.18** reveal critical insights into failure mechanisms, resin-fiber interfacial behavior, and the influence of fiber length and matrix type on the fracture surface morphology. Across all configurations, two dominant failure mechanisms were observed: fiber pull-out and matrix-fiber debonding, both

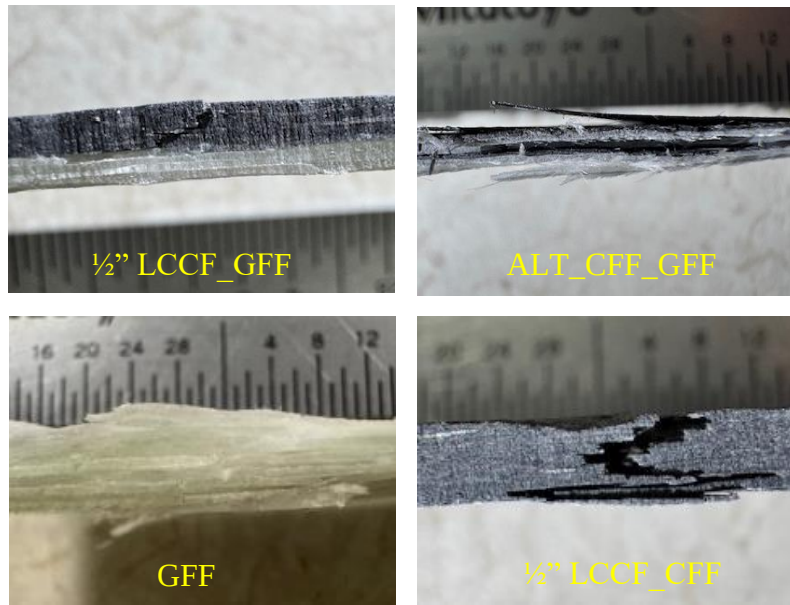


Figure 4.16: Observable damage in tensile samples of various composite laminates. Failure modes differ by architecture: delamination and interlaminar shearing observed in all samples while 1/2" LCCF layers showed matrix cracking.

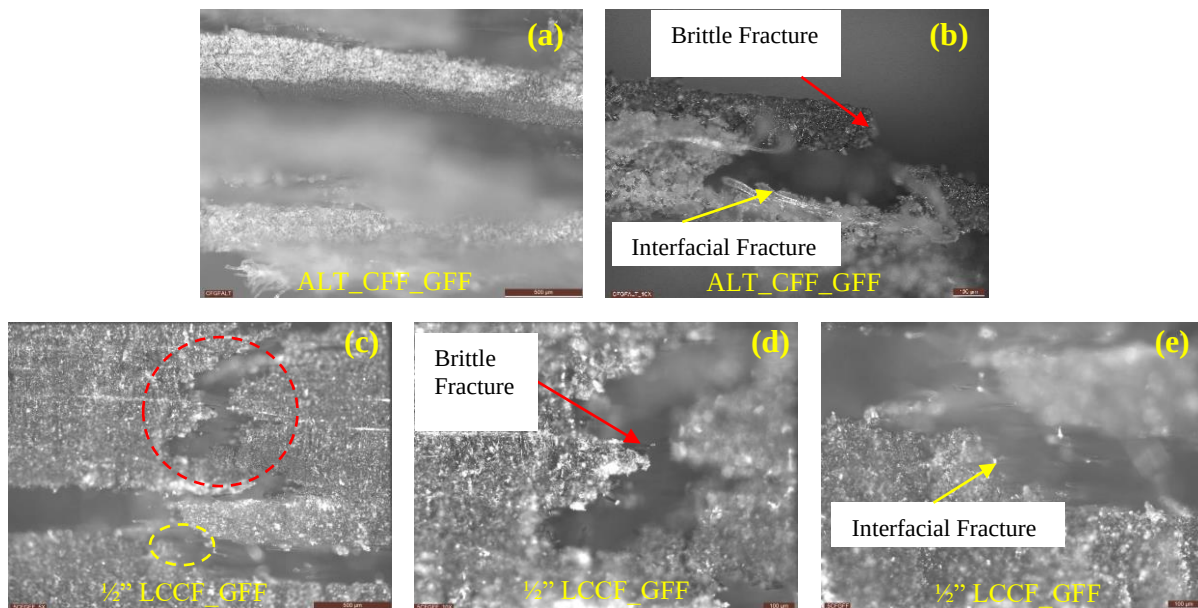


Figure 4.17: Optical microscopy observations of tensile fracture area of alternating CFF(2-layers)/GFF(2-layers) with and 1/2" LCCF(4-layers)/CFF(2-layers) EP composite structures. Observed matrix fracture in LCCF components and interlaminar shearing between GF and CF.

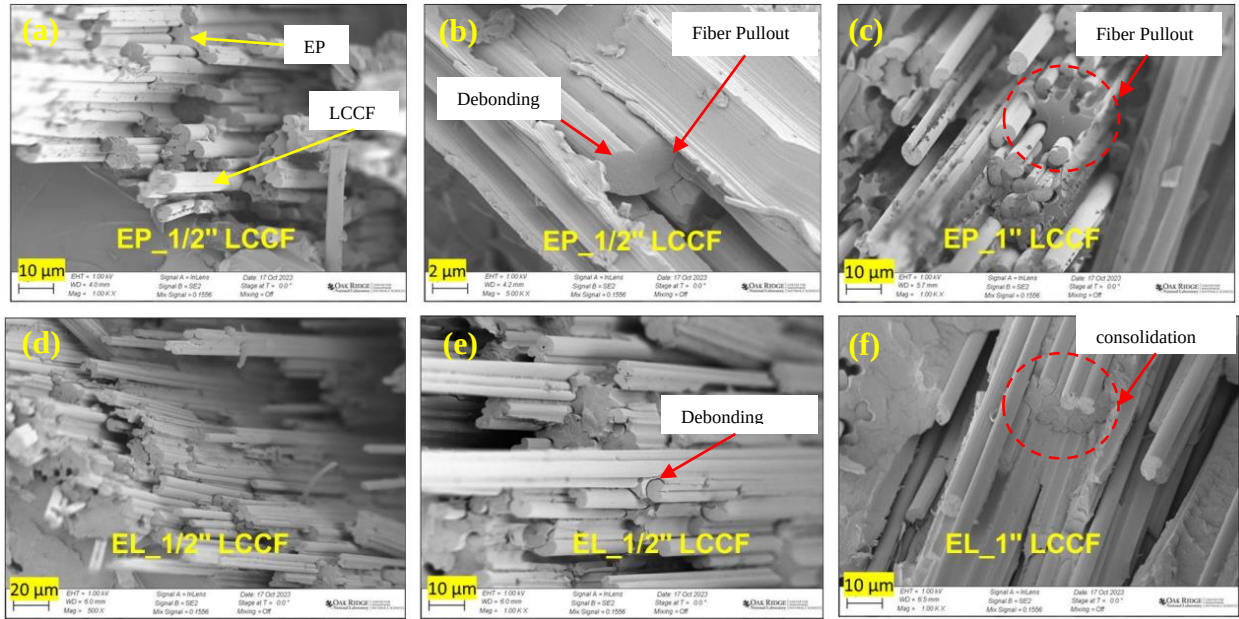


Figure 4.18: SEM images of tensile fracture surfaces for 1/2" and 1" LCCF composites with EP and EL resins. EP samples show fiber pull-out, debonding, and better consolidation in 1" LCCF. EL samples exhibit more interfacial debonding, especially in the 1/2" LCCF configuration.

of which are characteristic of high-stiffness composite systems that resulted in rapid crack propagation without significant deformation likely attributed to the high tensile modulus. In the EP_½” LCCF sample shown in **Fig.4.18a** and **4.18b**, significant fiber pull-out is evident, alongside clean debonding zones where fibers have separated from the matrix, leaving well-defined sockets. These features reflect an interfacial failure mechanism, wherein the stress at the fiber-matrix interface exceeded the cohesive strength of the bond, despite good initial fiber wetting during processing. The minimal resin residue on the pulled-out fibers suggests strong matrix rigidity but poor ductility, resulting in low energy absorption before failure. **Figure 4.18c** emphasized this finding as EP_1” LCCF showed extensive fiber pull-out cavities within the EP matrix. The presence of larger fiber socket clusters reflects both the increased surface area of longer fibers and the amplified shear stress at the interface. While this configuration achieved the highest tensile and flexural modulus among all samples, the mode of failure remains predominantly interfacial, with debonding preceding fiber fracture. Nonetheless, organized fracture surface implies excellent fiber-resin consolidation as seen in **Fig.18f**, reinforcing the mechanical findings of strong stiffness but limited toughness.

In the EL-based systems, similar trends in fracture behavior were identified. Fiber pullout was still present, reinforcing that interfacial debonding remained the primary failure mode even in the more ductile EL resin. These observations correlate with the tensile results, where EL composites displayed slightly lower stiffness but comparable strength for EL_½”LCCF. Therefore, this investigation indicated that cohesive strength between the fiber and matrix was the predominant mode of failure under tension and EP improved the overall composites strength. This observation is further emphasized by the manufacturing conditions as EP_½”LCCF (80C

cure) displayed significantly reduced tensile properties likely due to poor fiber/matrix interface bonding.

Overall, for discontinuous fiber, primary failure more fiber pullout with some fiber fracture. While with continuous fabric, the composites fail through interlaminar regions. Hybrid continuous fabric were more prone to interlaminar failure. Whereas hybrid CF discontinuous/GF nonwoven showed a significant improvement in wettability at the interface. In this case, failure occurred through fiber pull-out or breakage at the discontinuous fiber layers while continuous GF layers maintain stiffness leading to considerable improvement than all fabric composites.

4.3 Recycling

4.3.1 Thermoplastic Recovery

To assess the recyclability and reclamation potential of the EP resin system utilizing Recyclamine technology, a solvolysis-based recovery procedure was developed and evaluated under mild thermal and chemical conditions. The process, illustrated in **Fig. 4.19** and **4.20**, demonstrated the ability of the cured EP resin to be depolymerized and converted into a reusable thermoplastic product through acid-base extraction and precipitation techniques.

As shown in **Figure 4.19a**, a cured 441.05 g block of neat EP resin was weighed and subsequently transferred to a 1:1 aqueous acetic acid solution. The resin was placed in a 2-L borosilicate glass beaker and maintained at 80°C for 4 hours under constant magnetic stirring to promote uniform dissolution (**Fig. 4.19b**). Upon heating, the resin gradually broke down into a viscous, amber-colored solution (**Fig. 4.19c**), indicating successful depolymerization of the crosslinked thermoset into soluble linear fragments. After complete dissolution, the EP/acetic acid solution was transferred to a high surface area glass container and allowed to cool (**Fig. 4.19d**).

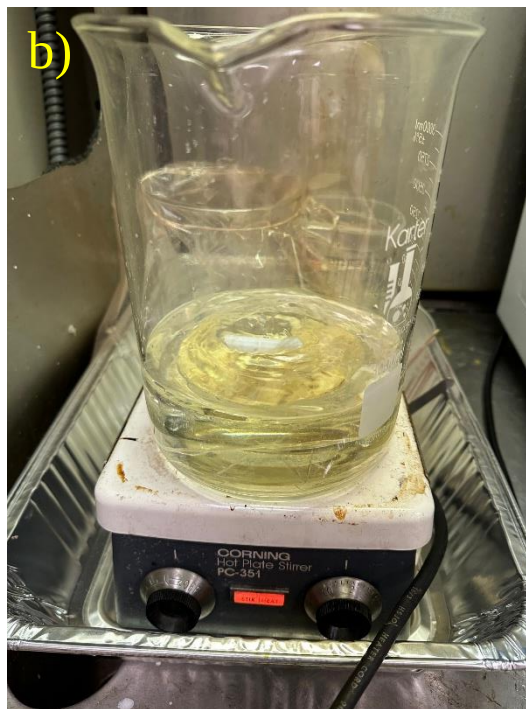
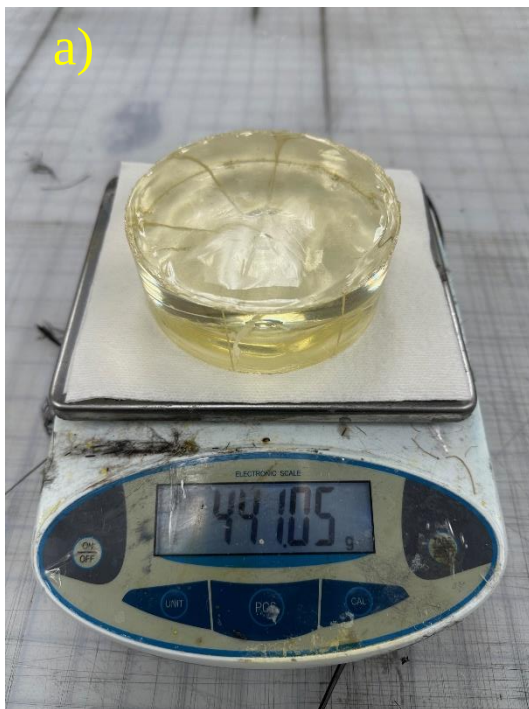


Figure 4.19: Solvolysis of neat EP resin using aqueous acetic acid at 80°C.(a–b) show resin weighing and initial dissolution setup with stirring.(c–d) illustrate resin breakdown and recovered liquid phase for further analysis.



Figure 4.20: Coagulation of solvolized Recyclamine using NaOH neutralization. (a) shows NaOH being added to the recovered solution; (b–c) depict phase separation and coagulated resin. This step enables recovery of the thermoset matrix for potential reuse or further processing.

To induce coagulation of the thermoplastic product from the solvolyzed solution, a diluted NaOH solution in a $6 \pm 0.5:1$ water-to-NaOH ratio was added shown in **Fig. 4.20a**. Upon introduction of the base, immediate coagulation of the polymer chains was observed, resulting in the formation of a soft, rubber-like precipitate as seen in **Figure 4.20b**. Over time, this precipitate consolidated and separated from the aqueous phase into a cohesive, ductile thermoplastic layer (**Fig. 4.20c**). The recovered material exhibited uniform thickness and transparency with a golden hue, confirming the formation of a reusable thermoplastic derivative of the original cured EP system.

4.3.2 Fiber Recovery

To investigate the effectiveness of chemical solvolysis for fiber reclamation from cured EP-based thermosets, a discontinuous EP_1”LCCF was evaluated. The composite was subjected to acetic acid dissolution under controlled thermal and stirring conditions. Shown in **Fig. 4.21a**, a section of the cured 1” LCCF/EP laminate was cut into a rectangular specimen approximately 80 mm $\times$ 100 mm. The sample was immersed in a 1:1 volume ratio solution of deionized water and glacial acetic acid and placed on a magnetic stirring hotplate set to 80°C for a duration of 24 hours.

During the solvolysis process, visible degradation of the resin matrix was observed within the first few hours of exposure shown in **Fig. 4.21b-c**. Continuous agitation ensured uniform temperature and acid contact with the composite surface, allowing the resin to dissolve away from the fiber bundle interfaces. Following the 24-hour treatment, the remaining solid residue was filtered and rinsed thoroughly with deionized water to remove excess acid and solubilized matrix. The resulting material, shown in **Figure 4.21d**, consisted of a dense mat of liberated 1”

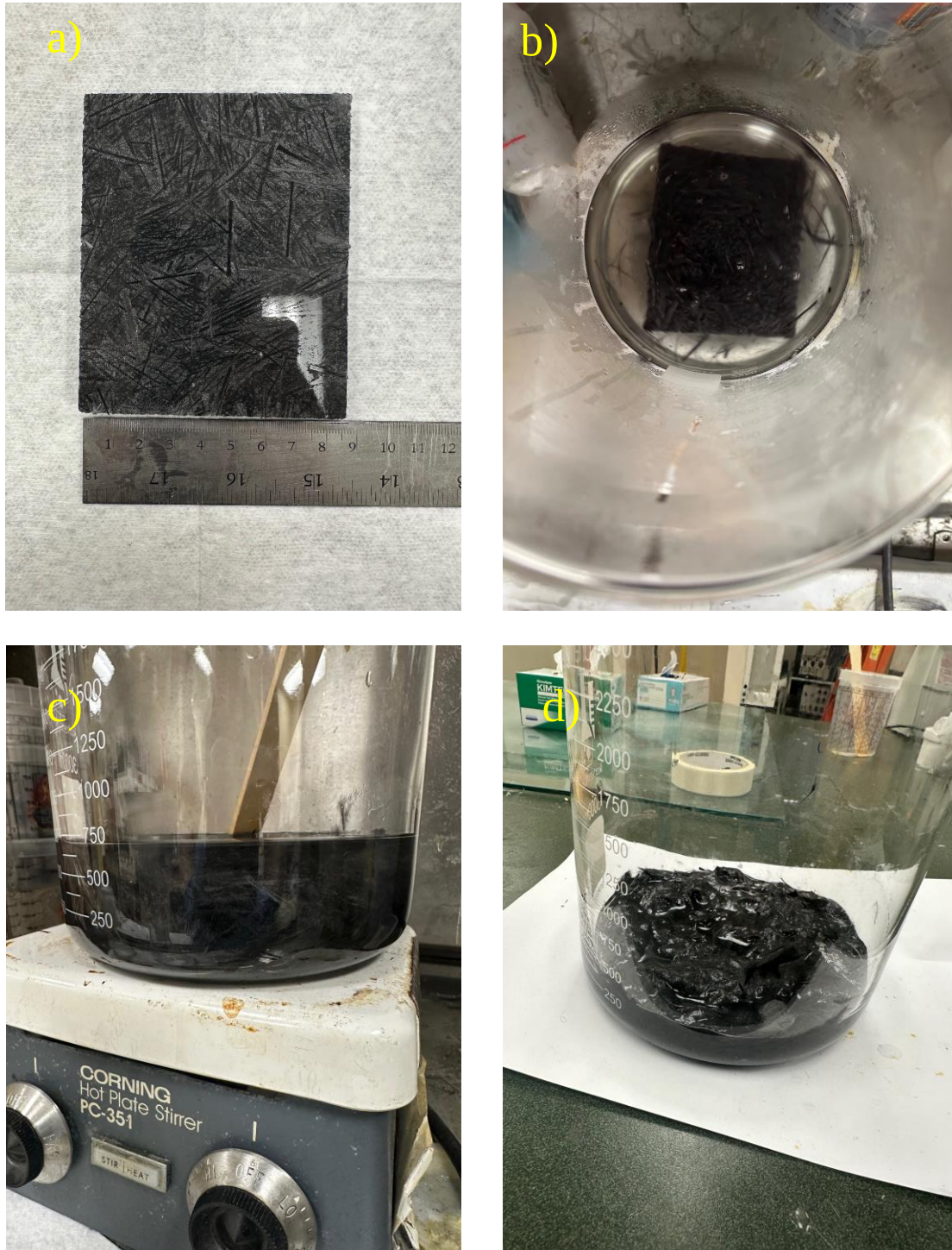


Figure 4.21: Solvolysis recovery of discontinuous 1'' LCCF from EP composite matrix. (a–b) show the composite sample before and during immersion; (c–d) depict resin dissolution and fiber liberation. Recovered LCCF retains structure, demonstrating feasibility of matrix-fiber separation for recyclability.

LCCF without observable fiber damage. The reclaimed fibers were sufficiently separated to indicate potential for reuse in recycled or secondary composite applications.

4.3.3 Matrix Recovery

This study focused on the solvolysis and recovery of GF and CF matrixes from hybrid composite tensile specimens composed of two knitted GF and LCCF (2GF_2CF). The objective was to evaluate both the efficiency of fiber separation and the chemical recovery of the thermoplastic Recyclamine derivative.

As shown in **Fig. 4.22**, 2GF_2CF samples total initial composite mass was 15.7548g. The samples were exposed to the same processing conditions as the EP_1”LCCF composite for 2 hours while reducing the stirrer rotation speed to minimize structural damage to the woven knitted fibers. Once removed from the solution, each layer could be easily separated with minimal impact to the knitted structure. Once rinsed and dried, the total fiber mass was recorded at 11.1413g. Thus, 70.72% of the total composite mass was reclaimed as fiber matrix indicating nearly full degradation of the EP resin from the matrix. Overall, GF represented 79.3% of the total mass while LCCF represented 20.7% of the total mass when extracted.

Following fiber removal, the reclamation of Recyclamine thermoplastic by precipitating the dissolved resin from the aqueous acetic acid solution was conducted. As shown in **Fig.4.23a**, the acetic acid solution was first filtered to remove residual solids, then an aqueous NaOH solution was subsequently added dropwise into the filtered acetic acid solution (**Fig. 4.23b-c**). Upon mixing, a coagulation reaction occurred, resulting in the formation of a suspended white thermoplastic precipitate within a solution of sodium acetate (CH_3COONa) and water, as seen in **Fig.4.23d**. This chemical response confirmed the precipitation of the depolymerized Recyclamine resin into a recoverable thermoplastic form. The precipitate formed a soft,

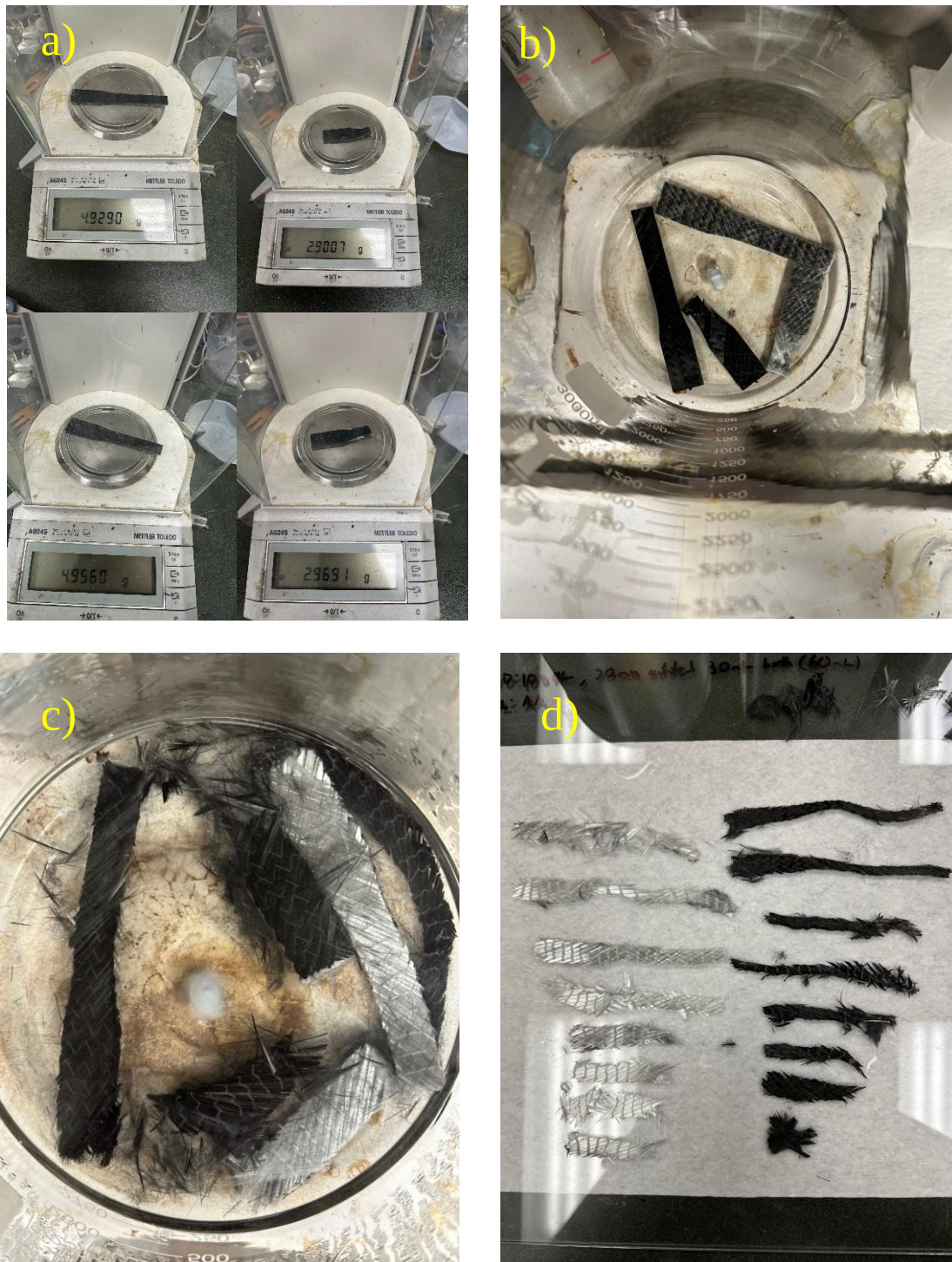


Figure 4.22: Solvolysis of hybrid tensile specimens containing knitted LCCF and GF layers (2GF_2CF). (a) shows initial mass measurements; (b–c) depict immersion and progressive matrix dissolution. (d) displays recovered LCCF and GF tows.

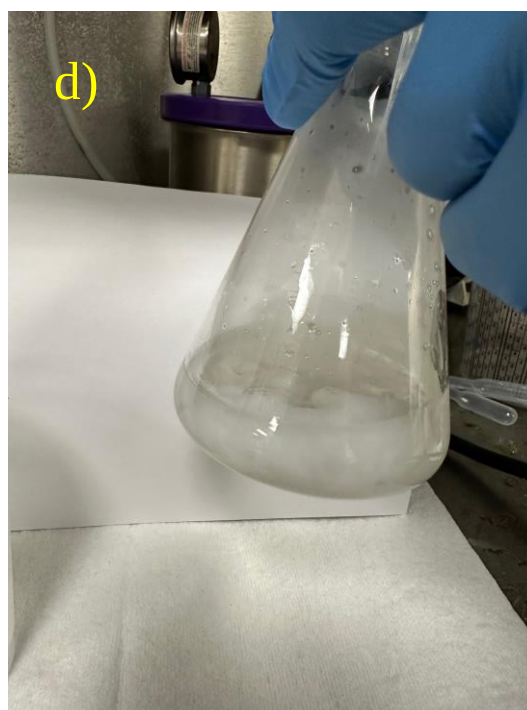
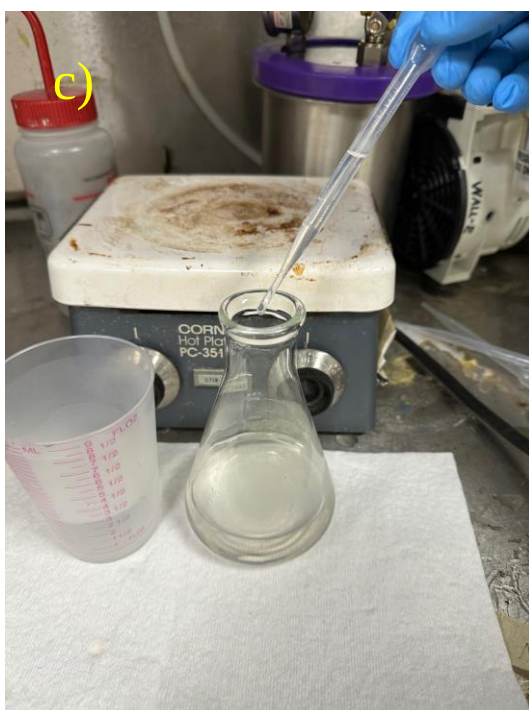
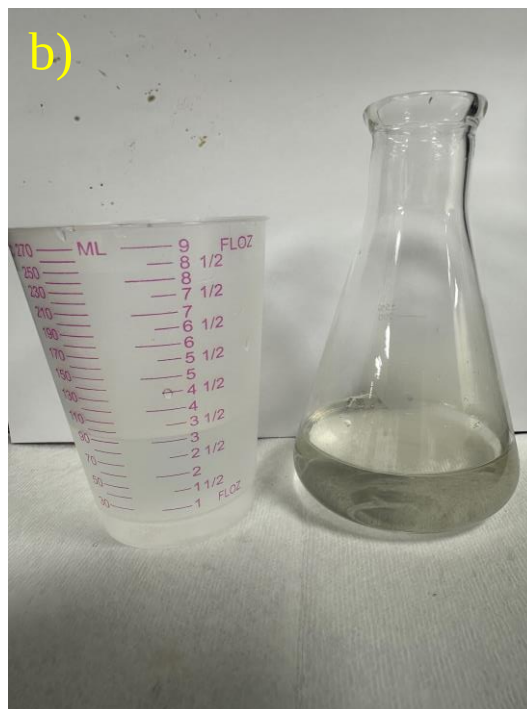
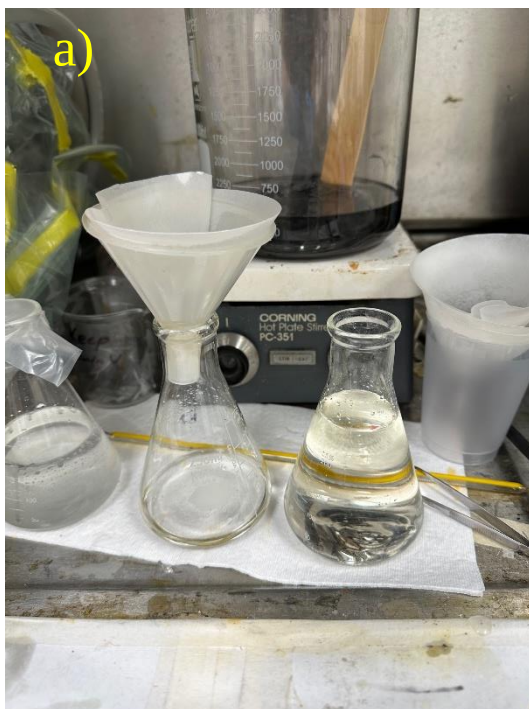


Figure 4.23: Reclamation of thermoplastic recyclamine in acetic acid solution a) filtration, b) NaOH solution and filtered acetic acid solution, c) combination of two solutions, d) coagulation caused by chemical reaction creating thermoplastic suspended in solution comprising of CH_3COONa and H_2O .

suspended network within the aqueous phase and was easily separated by decantation or vacuum filtration. Overall, the experiment demonstrated that the matrix recovery process using the EP resin system enabled efficient fiber reclamation through solvolysis and the chemical recovery of its Recyclamine-derived thermoplastic under mild, scalable, and low-cost processing condition.

4.4 Discussion

Results depicted in this study provide insight into the integration of LCCF reinforcement and EP resin systems into WTB composite. Offering improved mechanical performance and thermal stability compared to EL, EP showed desirable traits for WTB composites improving the tensile and flexural strength by 66.7% and 8.06% while improving the rigidity of the material under thermal loading in DMA. Thermal stability increased by 50% in TGA analysis highlighting EP's effectiveness for potential lightning structures in WTB operations and UV resistance.

Furthermore, the integration of discontinuous nonwoven fiber technologies and LCCF showed improved wettability, mechanical performance, stiffness, thermal resistance, and interlaminar adhesion while offering lightweighting potential at a reduced cost compared to baseline CF production. Uniform GF composite tensile and flexural strength was improved by 20.1% and 207.3% with the integration of discontinuous LCCF while increasing the tensile and flexural modulus by 175.4% and 384.9% respectively and reducing the mass by 55%. This demonstrates the potential use for improving stiffness at a reduced cost for WTB manufacturing while simultaneously offering a recyclable composite at end-of-life. Additionally, the use of discontinuous LCCF showed significant improvement in interlaminar adhesion at failure and with improved wettability and stiffness indicated by fiber pullout and debonding observed in SEM and optical microscopy emphasizing the enhanced performance under load and capacity for long WTB product life during operation.

With proven reclamation of the thermoplastic and intact fiber matrix recovery from EP-based composites using acetal solvolysis, thermoset materials with EP used in WTB can be recovered at a low cost and used for second-life applications retaining effective mechanical properties. While this study does not discuss the properties of reinfused LCCF, integration of recovered EP thermoplastic, and full-scale adoption of materials in WTB construction, the study highlighted the promising potential of integrating EP, LCCF, and nonwoven fibers as a precursor to further improvement and analysis of innovative technologies.

Chapter 5: Conclusions

As wind turbine blades (WTBs) continue to grow in size and demand increases for high-performance, cost-effective, and recyclable materials, the limitations of traditional glass fiber-reinforced thermosets have become more pronounced. Compared to older generations, current WTB designs must balance structural demands with manufacturing cost, weight, and end-of-life (EoL) waste management. These challenges underscore the need for innovative material systems that offer enhanced mechanical properties, thermal stability, and recyclability without compromising processability or affordability.

This study provides a promising material solution to address these evolving requirements by introducing low-cost carbon fiber (LCCF) and Recyclamine-based Epotec (EP) resin technology. The three main objects of this study were to evaluate EP resin properties compared to a recyclable thermoplastic resin system, Elium (EL), and epoxy, evaluate the integration of nonwoven discontinuous LCCF and woven continuous fabric using EL and EP, and lastly conduct preliminary evaluation on the recycling characteristic of EP.

Rheological analysis was conducted on neat EP, EL, and traditional epoxy. EP exhibited a significantly improved processing window compared to both EL and traditional epoxy systems. Rheological analysis revealed EP had a low complex viscosity (~ 10 Pa.s) favorable for vacuum-assisted resin transfer molding (VARTM), while EL exceeded 400 Pa.s, making it difficult to process at scale. Additionally, EP maintained a long gel time (>200 minutes), allowing for large-part infusion without premature curing, which is critical in wind blade fabrication.

Thermogravimetric analysis revealed that EP offers superior thermal stability compared to the EL with decomposition beginning at a significantly higher temperature ($\sim 300^\circ\text{C}$ for EP vs. $\sim 200^\circ\text{C}$ for EL) and a broader degradation profile, including a secondary breakdown phase at

670°C. This indicates that EP can withstand elevated temperatures (example: lightning strike) encountered during wind blade service life without rapid degradation. Additionally, the composite's peak degradation temperature (~360°C) and residue content were higher compared to neat resin, suggesting that the inclusion of LCCF enhanced thermal stability as well as increased char formation and long-term thermal durability. These findings, combined with EP's low viscosity and extended gel time, reinforce its suitability for VARTM processing and its potential to serve as a thermally robust, recyclable matrix system for next-generation wind turbine blade applications.

Dynamic mechanical analysis (DMA) demonstrated that EP offer favorable viscoelastic properties for wind turbine blade applications, especially when reinforced with LCCF. Although neat EL resin showed higher damping ($\tan \delta$) and glass transition temperature (T_g), EP resin exhibited restricted molecular mobility showcasing a higher storage modulus (1200 MPa), indicating its potential to enhance structural rigidity under dynamic loading. Incorporating LCCF fibers into both resin systems significantly increased the modulus by over 650% with minimal difference between ½" and 1" fiber lengths, though T_g increased with longer fibers, confirming improved thermal resistance. Among EP-based composites, 1" LCCF laminates achieved a T_g of 106.7°C and an 85% increase in damping over EP_4GFF, while maintaining comparable stiffness, highlighting the improved wettability of the discontinuous fiber in stress management. Furthermore, EP_2GF_4CF1/2" hybrid structures outperformed alternating GF/CF layups in stiffness and T_g , suggesting improved interfacial load transfer and compatibility with nonwoven LCCF. Collectively, these findings reinforce the viability of EP and LCCF systems to deliver thermomechanical performance essential for reliable, lightweight, and structurally resilient wind blade components.

Flexural and tensile testing revealed that composites reinforced with LCCF and EP consistently outperformed those made with EL or traditional GF fabrics in both strength and stiffness. Among nonwoven LCCF composites, the EP_1” LCCF sample achieved the highest flexural performance, reaching 797.02 MPa in flexural strength and significantly high 116.35 GPa flexural modulus, while also demonstrating the highest tensile strength at 134 MPa. These improvements highlight the effectiveness of longer discontinuous fibers in enhancing structural rigidity when paired with EP. In hybrid laminates, the EP_2GF_4CF½” configuration displayed the greatest flexural strength (858.38 MPa) and tensile modulus (48.2 GPa), emphasizing the beneficial role of randomly oriented LCCF layers in combination with knitted GF. Despite slightly reduced stiffness compared to the pure LCCF laminates, these hybrids achieved significant gains in damping and strength-to-weight ratio, with EP_1” LCCF reducing mass by over 55% while maintaining high mechanical integrity. Poor interfacial load transfer in some hybrid configurations, such as EP_GF/CF, limited stiffness, but the overall trend demonstrated that incorporating nonwoven LCCF, either alone or in hybrid structures, improved composite performance. These findings confirm that EP resin and LCCF enable the production of lightweight, high-strength, and cost-effective composites suitable for next-generation wind blade applications.

Microscopy analysis provided valuable insight into the failure mechanisms and interfacial behavior of EP- and EL-based LCCF composite systems. Optical microscopy revealed that nonwoven ½” LCCF laminates, both in standalone and hybrid configurations, primarily failed through matrix fracture with minimal interlaminar shearing, indicating improved interfacial bonding and matrix stiffness compared to alternating woven carbon and glass fiber layups, which exhibited pronounced interlaminar shearing and delamination. These observations suggest that

discontinuous LCCF enhances through-thickness bonding and restricts fiber sliding during tensile loading. Scanning electron microscopy (SEM) further supported these findings, identifying fiber pull-out and matrix-fiber debonding as the primary failure modes across all samples. EP composites showed clean fiber sockets and rigid fracture surfaces, pointing to strong initial wetting but brittle matrix behavior under stress. Longer 1” LCCF fibers exhibited larger pull-out cavities and amplified interfacial stress, correlating with a high mechanical stiffness. EL-based composites showed similar interfacial failure patterns although the composite was slightly more ductile. Overall, microscopy confirmed that cohesive interfacial failure governed composite performance and that EP resin systems, especially when combined with nonwoven LCCF, improved structural bonding and mechanical consistency, making them promising candidates for high-performance wind turbine blade applications.

Recycling trials confirmed the successful recovery of both fibers and matrix material from EP-based composites using a mild, scalable solvolysis process enabled by Recyclamine. Through acid-base extraction, the cured EP resin was effectively depolymerized in an aqueous acetic acid solution and converted to a reusable thermoplastic precipitate via NaOH-induced coagulation. Fiber recovery from EP_1”LCCF composites showed no visible damage, with fibers fully separated after 24 hours of mild thermal and chemical treatment, supporting their reuse in future composite applications. Similarly, hybrid GF/LCCF composites (2GF_2CF) underwent successful matrix degradation and separation, yielding a nearly 100% fiber reclamation rate as 70.72% of the original total mass was recovered as recycled fiber with preserved fiber structure. Matrix recovery from these samples demonstrated the repeatable reclamation of Recyclamine-derived thermoplastic from the acetic acid solution, illustrating the possibility for closed-loop recyclability of the EP resin composites. These findings validate that EP enables efficient fiber

and matrix reclamation under low-energy conditions, reinforcing its potential for circular economy integration in WTB manufacturing and end-of-life material reuse.

Based on this study, the introduction of EP and integration of LCCF and nonwoven materials could significantly reduce the cost of carbon fiber reinforcement by 33-49% in WTB manufacturing. Additionally, these materials demonstrate significant potential in reducing the impact associated with EoL processing based on minimal energy requirements for solvolysis and excellent fiber reclamation as an alternative to epoxy or EL. Although these materials show promising potential, further research is needed to improve the recycling and reclamation process and investigate the integration of EP, LCCF, and nonwoven materials in production WTBs.

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Vita

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