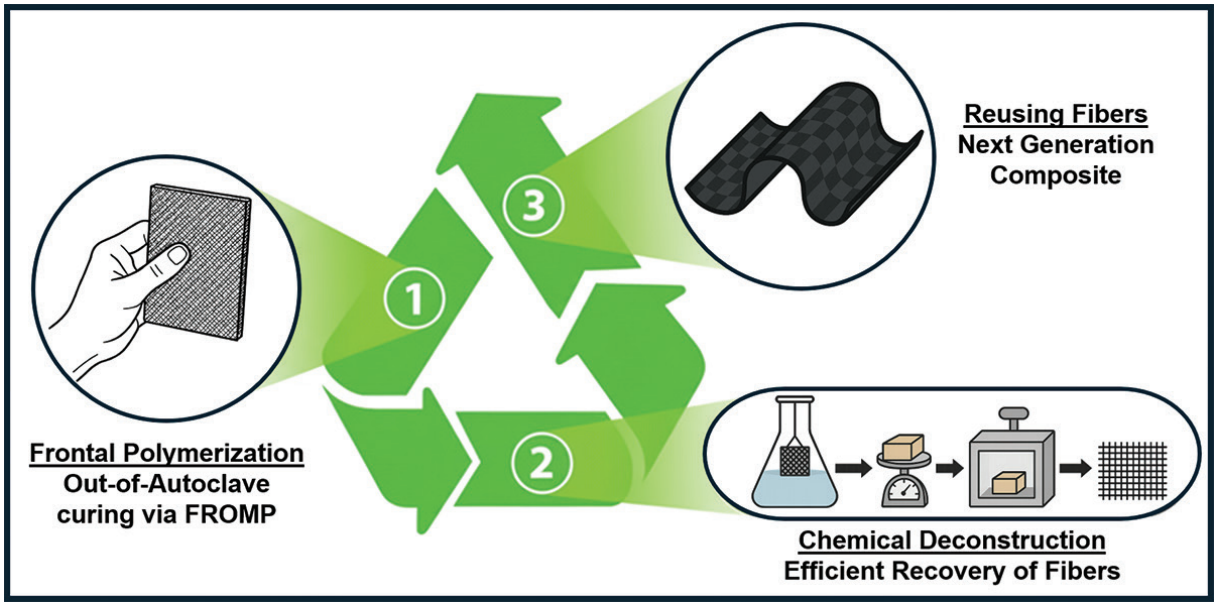

Recovery and Reuse of Carbon Fiber from Deconstructed Composites with A Dicyclopentadiene and 2,3-Dihydrofuran Copolymer Matrix

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ABSTRACT

Improving the life cycle of composite materials begins with low-energy manufacturing to achieve useful mechanical properties and often ends with either disposal or recycling. Thermoset composite recycling techniques typically involve burying, burning, solvent soaking, chopping, or grinding with only a small fraction of recycled material being used in down-cycled products. These resource-intensive processes are primarily driven by the environmental robustness of the composite and the difficulty of separating the reinforcement from polymer matrix. Prior work has demonstrated that dicyclopentadiene (DCPD) copolymerized with a cleavable comonomer, 2,3dihydrofuran (DHF), can be deconstructed in solution via DHF hydrolysis by hydrochloric acid (HCl) in cyclopentyl methyl ether (CPME). In this paper, we discuss the recovery and reuse of carbon fibers and fabrics from the deconstruction of composites having a copolymer matrix of poly(DCPD-co-DHF) which can be cured by frontal polymerization. We explore the kinetics of composite deconstruction as a function of comonomer concentration. Carbon fibers were successfully deconstructed, recovered, and reused in multiple composites without significant structural degradation observed. The surface properties of the recovered carbon fiber were analyzed by contact angle measurements and X-ray spectroscopy to confirm that the chemical composition of the fibers remains largely unaltered by the deconstruction process.

Keywords: Frontal Polymerization, Upcycling, Thermoset Composites Recycling, Carbon Fiber Surface Properties

DOI: 10.33599/SJ.v62no1.04

1. INTRODUCTION

Carbon fiber reinforced composites (CFRPs) have become indispensable in industries that require high-performance structural materials and exceptional strength-to-weight ratios, such as automotive, motorsport[1], aerospace[2], and space exploration[3]. In aerospace, CFRPs gained significant traction in the late 1990s, with their use steadily increasing across commercial and military aircraft. Notable examples include the Boeing 787 and Airbus A350, where composites constitute a substantial portion of the structure. This surge in adoption has spurred advancements in composite manufacturing and tooling techniques[4]. Commercially available manufacturing methods, such as vacuum infusion[5-7], pre-pregs, filament winding, Automated Fiber Placement (AFP)[8], and continuous fiber composites[9], have propelled the industry forward. However, as the lifespan of composite-based aircraft nears its end, critical challenges remain regarding their recyclability[10].

The production of CFRPs, particularly thermoset composites, is energy intensive. For instance, curing the Boeing 787 fuselage requires approximately 350 gigajoules of energy over eight hours[11]. To address this, rapid and energy-efficient manufacturing methods, such as frontal polymerization[12] and additive manufacturing[13-14], have garnered attention. Despite these advances, the recyclability of thermoset materials remains a complex scientific challenge.

Composites are broadly classified into thermosets and thermoplastics[15-17]. While thermoplastics are usually recyclable due to the reversibility of their physical cross-links, thermosets are typically not. The chemical crosslinked structure of thermosets provides superior mechanical properties but makes them resistant to conventional recycling methods like dissolution or thermomechanical degradation. Past research efforts have largely focused on reclaiming carbon fibers, the most valued component, at the end of a composite's lifecycle[18].

Traditional recycling of thermoset composites typically involves mechanical grinding or cutting, which is inefficient, energy-intensive, and sacrifices material properties[19]. These methods disrupt fiber orientation and result in discontinuous fibers that degrade many of the structural advantages of using them in CFRPs[20]. Instead, the fibers are used in mechanically down-graded products. Other fiber recovery approaches, such as pyrolysis[21] and solvolysis[22-24], alter the inherent properties of recovered fibers, reducing their future value and utility. Given that reinforcement fibers represent the most expensive component of composites, recovering them with intact length and orientation is both economically and environmentally impactful. However, existing methods often fail to achieve this efficiently.

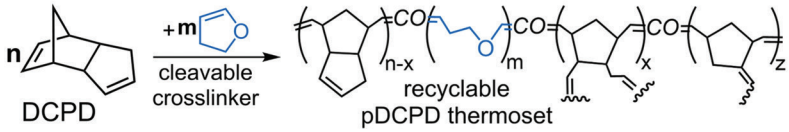

Figure 1. Copolymerization reaction between DCPD and DHF.

Frontal polymerization of dicyclopentadiene (DCPD) has shown promise as an energy-efficient method for manufacturing thermoset polymers and composites. Yet, the recyclability of thermosets like DCPD remains a challenge. This paper introduces an innovative approach to manufacturing composites with good thermomechanical properties which are also recyclable. By incorporating 2,3-dihydrofuran as a co-monomer into the DCPD polymer backbone, we achieve cleavable enol-ether linkages that can be broken down via acid hydrolysis by hydrochloric acid in a polar solvent like cyclopentyl methyl ether[25-26]. The chemical reaction is summarized in Figure 1. Upon exposure to acid, these ether linkages cleave to hydroxyl and other species, resulting in soluble oligomeric species[27-28]. Importantly, this process preserves the length and orientation of the carbon fibers. When in woven form, the structural integrity is maintained, and the fabric remains handleable for reuse in subsequent composite processing which helps to retain value and enable future uses.

2. EXPERIMENTATION

2.1 Materials and Methods

2.1.1 Thermoset Matrix

Ultrene 99 DCPD was sourced from Cymetech Corporation, KY, USA, with polymerization enthalpy of around 360 J/g. 2,3 dihydrofuran and the Grubbs' 2nd generation catalyst were sourced from Sigma Aldrich®. Given DCPD is solid at room temperature, the sourced DCPD was melted in an oven at 50°C and around 5% ethylidene norbornene (ENB) by weight with respect to DCPD added to keep the resin liquid at room temperature. Throughout this paper we refer to this DCPD/ENB mixture as DCPD.

The tested formulations contained a mole ratio of 5% to 15% DHF relative to DCPD and 250 parts per million (ppm) of catalyst, also relative to DCPD. The combined polymerization enthalpy was measured using differential scanning calorimetry (DSC) to increase from 390 to 410 J/g as the ratio of DHF to DCPD was increased from 5 to 15%. The temperature of the front for neat resins during frontal polymerization was 190-200°C[37].

2.1.2 Carbon Fiber Laminates

The carbon fiber laminates were made using commercially available Hexcel® AS4C woven fabrics with a 3K 2x2 twill weave with 6 layers of fibers stacked together. They were processed by

vacuum assisted resin transfer molding (VARTM) as shown in Figure 2. The infused fibers were pressed in a hot press at 2 tons (20 kN) of pressure to ensure consistent fiber volume fraction.

The selected dimensions for the stacked fabrics were approximately 6-inch x 6-inch (152 x 152 mm). The VARTM setup was assembled in-house using disposable infusion tubes, peel-ply, and nylon films. Tubes of 4 mm inner diameter × 6 mm outer diameter were fed into 5 inch (127 mm) long, 3/8-inch (9.5 mm) outer diameter spiral cut cable wrap and attached to the fiberglass on each side by vacuum tape. These tubes facilitate the transfer of resin into the VARTM setup. One tube end is submerged into the resin, and the other tube end is attached to a vacuum pump. At the completion of the infusion, excess resin is collected into a resin trap attached to the vacuum pump.

A resistive surface heater sourced from OMEGA® was used to initiate the polymerization front through the thickness of the infused fibers. A thermocouple was used to measure the temperature of the fabric to ensure that the polymerization reaction was initiated. The surface heater was ramped from room temperature until the thermocouple read 200°C in approximately 2 minutes at which time the heater was turned off. The distinctive rise in thermocouple temperature associated with the polymerization exotherm was observed (~85-90°C). The composite was fully cured in less than 5 minutes, with DSC thermograms showing no residual enthalpic peak after curing.

3. RESULTS AND DISCUSSION

3.1 Composite Properties

We manufactured carbon fiber composite laminates ranging from 5% to 15% mole fraction DHF to DCPD. The fiber volume fraction was characterized using optical microscopy (Keyence VHX7000) and compared to values calculated using the manufacturer's reported density for the fabric and the recovered mass of the fiber post deconstruction.

No significant voids were observed by optical microscopy with a 500x zoom and fiber volume fraction was consistently higher than 50%. Values for fiber volume fraction obtained through the recovered mass method were slightly lower than those obtained by microscopy. We can attribute this to the fact that the calculated V_f through microscopy was done on a complete fiber tow perpendicular to the plane of focus (highlighted red in Figure 3), ignoring the resin pockets present at the intersection of two tows in a weave (highlighted green in Figure 3). Despite there being no significant losses observed during processing, the observed decrease in V_f could potentially also be related to loss of fiber material during deconstruction and drying.

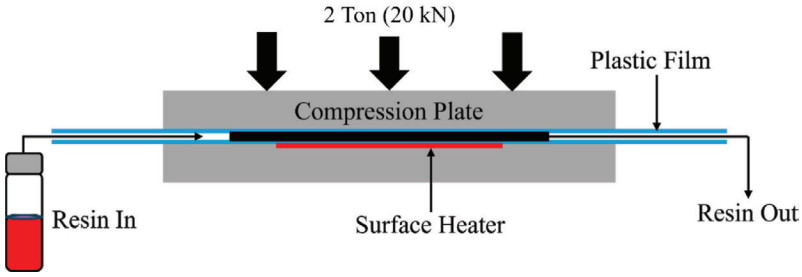

Figure 2. Vacuum Assisted Resin Transfer Molding (VARTM).

Dynamic Mechanical analysis was performed in 3-point bend (3PB) setup with constant amplitude in a constant strain mode experiment. DMA samples with a thickness of ~2 mm, a width of ~3 mm, and length of 30 mm, were prepared by cutting the composite laminate using a diamond blade. The DMA instrument was pre-loaded to 2 N of force with a ramp rate of 2°C per minute from 0°C to 250°C. The glass transition temperature (Tg) was obtained as the peak in tan(δ). 6-ply laminates with 10% DHF were tested, yielding a glass transition temperature of around 145-150°C and storage modulus (E') at 25°C of over 35 GPa. In comparison, the frontally polymerizable deconstructable composites that use a 7-membered silyl ether cleavable comonomer (iPrSi-7) linkages with pDCPD demonstrated by Lloyd et. al[29], obtained a carbon fiber laminate with stiffness of around 36.5 GPa and had a significantly lower glass transition temperature of around 100°C. The 6-ply laminates manufactured with DHF concentrations ranging from 5% to 15%, showing expected decrease in Tg as the fraction of DHF in the neat polymer is increased as demonstrated by Davydovich et al.[30]. As shown in Figure 4, the glass transition temperature of pDCPD-co-DHF is between that of neat DCPD and DCPD-iPrSi-7.

3.2 Composite Deconstruction

To deconstruct the composite laminate and recover the fibers, we suspended a square piece of composite measuring 20 cm x 20 cm in the 3M HCl in

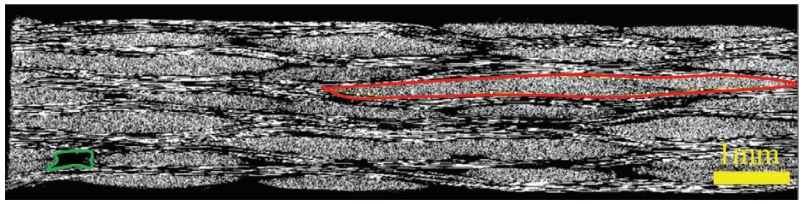

Figure 3. Optical microscopy of the cross-section of 5% DHF in DCPD.

Table 1. Fiber volume fraction (V_f).

DHF: DCPD Mole Ratio	V_f (Microscopy)	V_f (Mass Loss)
5%	60.59 ± 2.11	55.19 ± 4.61
10%	65.92 ± 1.81	50.15 ± 3.93
15%	64.68 ± 2.14	55.29 ± 4.51

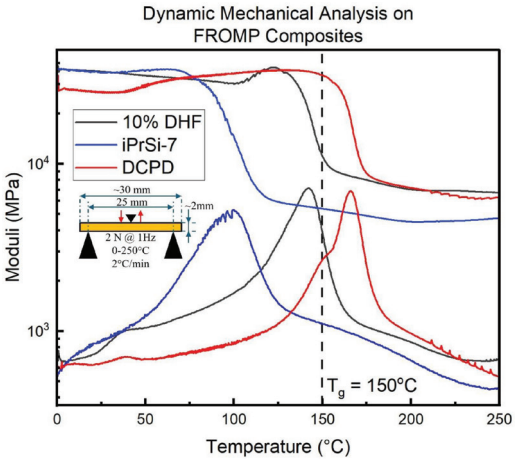

Figure 4. Dynamic mechanical analysis on 10% DHF samples.

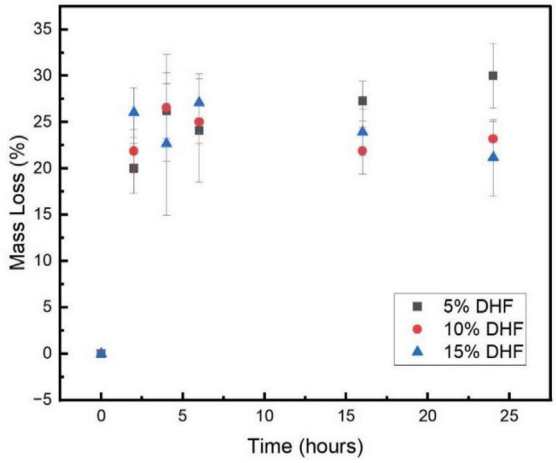

Figure 5. Composite mass loss as a function of time during deconstruction.

CPME solution using nylon cable. Deconstruction was observed and the residual mass was weighed after 2, 4, 6, 16, and 24 hours, as shown in Figure 5. After each cycle, the residual mass was thoroughly washed with tetrahydrofuran (THF) and water repeatedly until the acid was removed as indicated by a neutral pH by litmus paper.

After recovering, the residual mass was dried in a vacuum chamber with a solvent trap. Initial and final weights were measured to characterize the percentage mass loss of the composite as a function of time. A polyethylene mesh was used to support the fibers and preserve the fabric weaves during the deconstruction process.

The laminate appeared fully deconstructed to fibers after around 6 hours and were reclaimed after 24 hours. Thermogravimetric analysis (TGA) was carried out on the reclaimed fibers to ensure there was no residual resin left. No mass loss was observed in the TGA up to 600°C, suggesting no residual resin was present. Optical microscopy also showed no significant signs of residual matrix and no significant indication of fiber breakage or misalignment (Figure 6).

Figure 6. Optical microscopy on recovered fibers showing no significant resin deposition.

3.3 Reuse of Recovered Fiber Fabrics

Since the recovered carbon fabrics remained intact and handleable, they were used directly to make a new composite using the same VARTM process. The recovered fiber fabrics were subjected to a drying and heating process at 150°C. For this second infusion study into the recovered fabrics, composite samples with 10% and 15% DHF were used. As illustrated in Figure 7b (10% DHF), the woven sheets can be removed and reused with minimal manual intervention, although there is a small potential risk of fiber misalignment. The integrity of the preserved fiber weave is shown in Figure 6 and 7.

3.3.1 Surface Properties of Recovered Fibers

Furthermore, to understand the effect of deconstruction on the surface properties of the

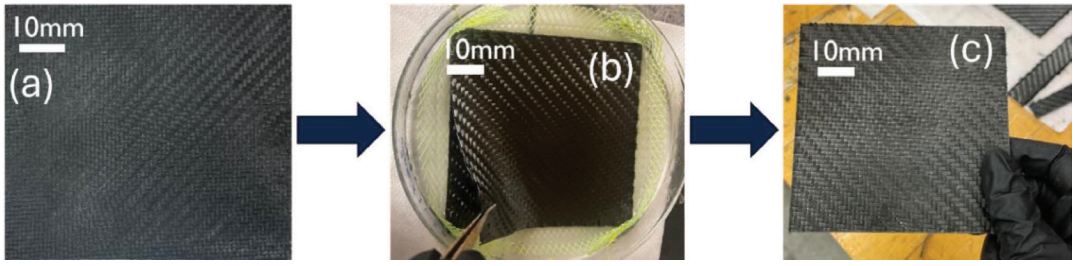

Figure 7. (a) Generation 1 composite laminate, (b) deconstruction and recovery of woven fiber fabrics, and (c) reuse of the fabrics for generation 2 composite laminate.

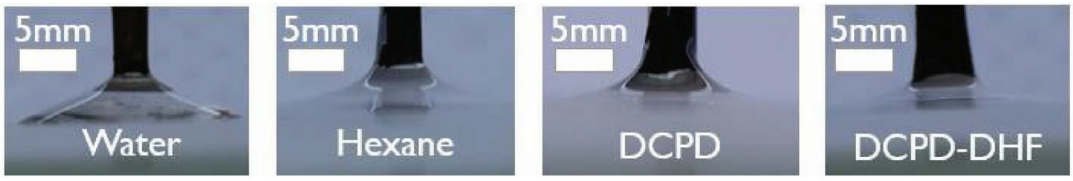

Figure 8. Images of bundled fibers contact angle test.

fibers, tow based contact angle tests were carried out and Young-Dupre equation was used to characterize the surface energy. Contact angle tests are relatively simple tests used to determine surface energy which dictates the wetting or adhesive nature of the surface. We hypothesize that changes in the surface energy of the fibers following deconstruction would impact fiber-to-resin adhesion across subsequent generations. This phenomenon has also been documented in the context of epoxy based composite materials[31-32]. For the contact angle testing, we used individual carbon fiber tows, removed from the recovered fiber fabrics. The chosen chemicals were water (polar solvent), hexane (non-polar solvent), liquid DCPD and DCPD-DHF (10%) as the simulated resins[33]. The magnified contact angle is shown in Figure 8. All tests were done at room temperature inside a fume hood.

$$W = \gamma(1 + \cos\theta) \quad (1)$$

Equation 1, shown above, is the Young-Dupre equation for characterization of surface energy using contact angle, where, W is the surface energy, γ is the surface tension of the solvent used, and θ is the contact angle the bundled fibers make with the solvent. To incorporate polar and dispersive components, the surface energies of both the liquid and solid are often divided into their polar (γ_p) and dispersive (γ_d) parts (where $\gamma = \gamma_p + \gamma_d$) and measured with water and hexane, respectively. Table 2 summarizes the numerical results for each and is plotted in Figure 9 with the total surface energy.

From Figure 9, it is evident that the surface energy of the recovered fibers does not change significantly across generations and is like the virgin fibers, suggesting wettability and adhesion of the fibers before and after deconstruction will be consistent.

To understand the chemical reactivity of the fibers with the matrix during the deconstruction process, X-Ray Photoelectron Spectroscopy (XPS) was done on the virgin unsized, sized, and the recovered fibers. XPS can identify and quantify the elemental composition of the surface, including carbon (C), oxygen (O), nitrogen (N), and other

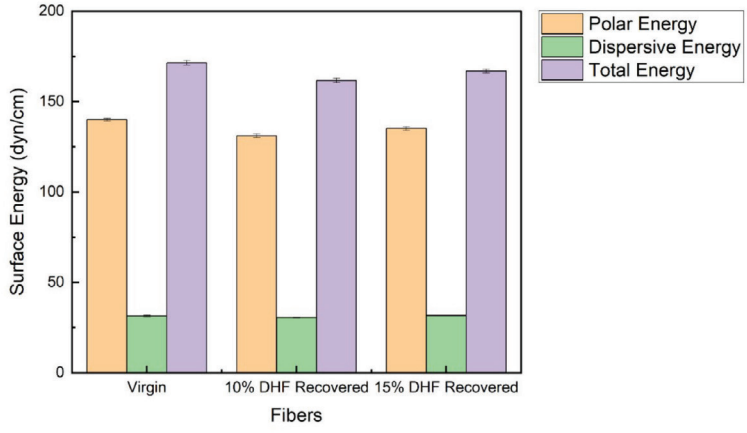

Figure 9. Surface energy characterized through surface angle test.

Table 2. Surface energy values obtained through Young-Dupre equation.

	Polar Energy (J/m²)	Dispersive Energy (J/m²)
Virgin	139.99 ± 0.79	31.40 ± 0.48
10% DHF Gen 1	131.11 ± 1.01	30.49 ± 0.19
10% DHF Gen 3	135.27 ± 0.67	31.69 ± 0.08
15% DHF Gen 1	135.16 ± 0.99	31.62 ± 0.14
15% DHF Gen 3	138.21 ± 0.35	31.60 ± 0.34

elements that might be present on the carbon fiber surface. This helps in determining the degree of oxidation, contamination, or functionalization that the fibers might have undergone. This test was done to determine if the surface chemistry of the fibers changes as a function of deconstruction, which in-turn affects the resin-fiber adhesion and mechanical properties of the composite[34-35]. This test was done at the US Air Force Research Laboratory, Wright Patterson AFB, Dayton OH by Dr. Tyson Back. The XPS source was a monochromatic Al K-alpha rays at 1486.7 eV with step size of 0.1 eV. The compared XPS samples were:

- Vacuum Dried: AS4C-GP (general purpose) sized fibers were baked in an oven at 150°C for 3 hours followed by an overnight drying cycle inside a vacuum chamber connected to a Schlenk line. This was done to ensure no impurities or moisture was present
- AS4C-unsized: used as provided by manufacturer
- AS4C-GP Sized: used as provided by manufacturer
- 15% DHF: Recovered AS4C-GP sized fibers from 15% DCPD-DHF matrix, followed by drying cycle in dynamic vacuum overnight provided by a Schlenk line

The percentage composition of the O-C-N elements on the commercially sourced carbon fibers are in agreement with the data on the same fibers published by Vautard et al.[36]. However, there is a higher percentage of chlorine on the surface of the reclaimed fibers, which we hypothesize to be from the hydrochloric acid solution used for deconstruction. It is anticipated this amount of chlorine would not contribute significantly to the thermomechanical properties of the recovered fibers or the composite laminate made using them.

We hypothesize that the physiorbsed and chemisorbed H_2O is from the fibers wetting and drying successively post recovery, -OH, C-O-R and C-O-C from deconstructed oligomers and ring opened dihydrofuran and C≡N and pyrrolidine from AS4C surface chemistry. While there are slight

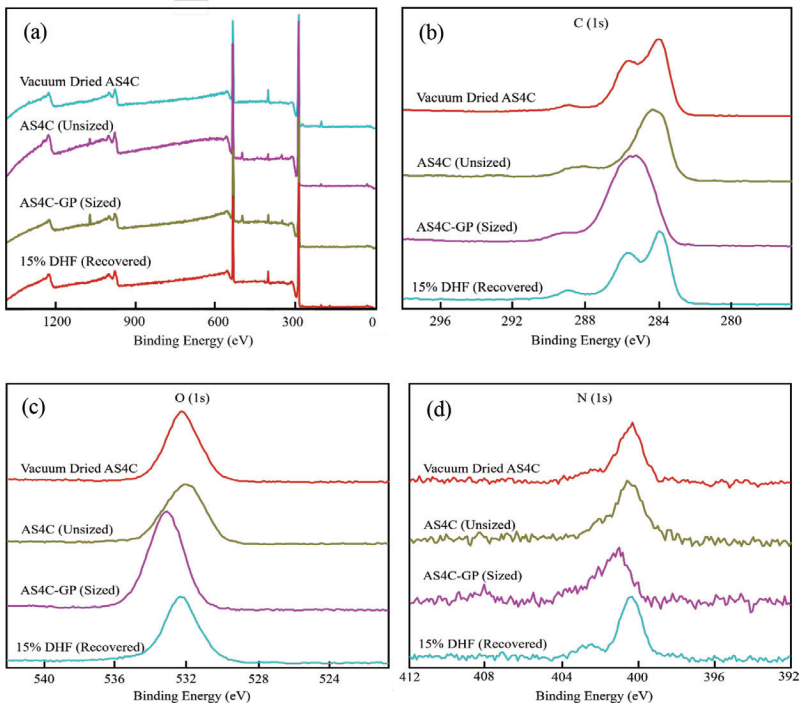

Figure 10. XPS data for (a) atomic abundance and binding energy (eV) for (b) carbon, (c) oxygen, and (d) nitrogen.

Table 3. Atomic abundance by mass (%) of various tested fibers using XPS.

	Vacuum Dried	AS4C-Unsized	AS4C-Sized	15% DHF
Carbon	80.49	79.33	80.74	78.73
Oxygen	15.52	16.94	17.78	15.98
Nitrogen	3.42	2.81	0.94	3.44
Sodium	-	0.54	0.14	-

Table 4. Chemical species corresponding to binding energies of C, N, O.

C (1s)		O (1s)		N (1s)	
Binding energy (eV)	Component Peak	Binding energy (eV)	Component Peak	Binding energy (eV)	Component Peak
289.7	Physiorbsed H_2O	533	R-OH, C-O-C	400.5	Pyrrolidine, pyridine
286.4	C-O-R, C≡N	534	Chemisorbed H_2O	-	-
284.5	Csp2	-	-	-	-

traces of deconstructed fragments and acid, we believe that it is not enough to lead to a drastic decrease in mechanical properties or impact resin-fiber adhesion. Preliminary dynamic mechanical analysis was done on the second-generation composite laminates and initially indicated retention of the storage modulus and a slight shift in glass transition temperature which will be investigated further. The second infused composite was deconstructed again to create a three generation of fiber fabric (virgin, first reclamation, second reclamation). The properties of these fiber fabrics will be studied further.

4. CONCLUSIONS

Dihydrofuran is a commercially available enol ether that provides cleavable functionality when incorporated into poly(dicyclopentadiene) (pDCPD). The resulting thermoset demonstrates exceptional potential as a frontally polymerizable system with good thermomechanical properties. Contact angle measurements and X-ray spectroscopy analyses confirm that the chemical composition of the fibers remains largely unaltered before and after the deconstruction process. This study highlights that employing frontal polymerization followed by chemical deconstruction offers the potential for a more energy-efficient life cycle than conventional thermo-chemomechanical methods for the manufacturing and recycling of carbon fibers.

Notably, three generations of carbon fiber fabrics were successfully deconstructed and recovered. While investigation continues, no significant structural degradation has been observed. The deconstruction of the thermoset matrix into oligomeric products facilitates the reuse of carbon fibers as intact woven fabrics without necessitating fiber discontinuity and re-orientation which enables simple reuse of fabrics in deconstructable matrices over multiple generations.

5. ACKNOWLEDGMENTS

This work was supported as part of the Regenerative Energy-Efficient Manufacturing of Thermoset Polymeric Materials (REMAT), an Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science, Basic Energy Sciences at the University of Illinois at Urbana Champaign under award DE-SC0023457. Special thanks to Dr. Tyson Back, Air Force Research Laboratory, for conducting the XPS test in the facility. Lastly, the authors would like to thank the Undergraduate Research Opportunity Program (UROP), UIUC for partially funding undergraduate research in the Summer of 2024.

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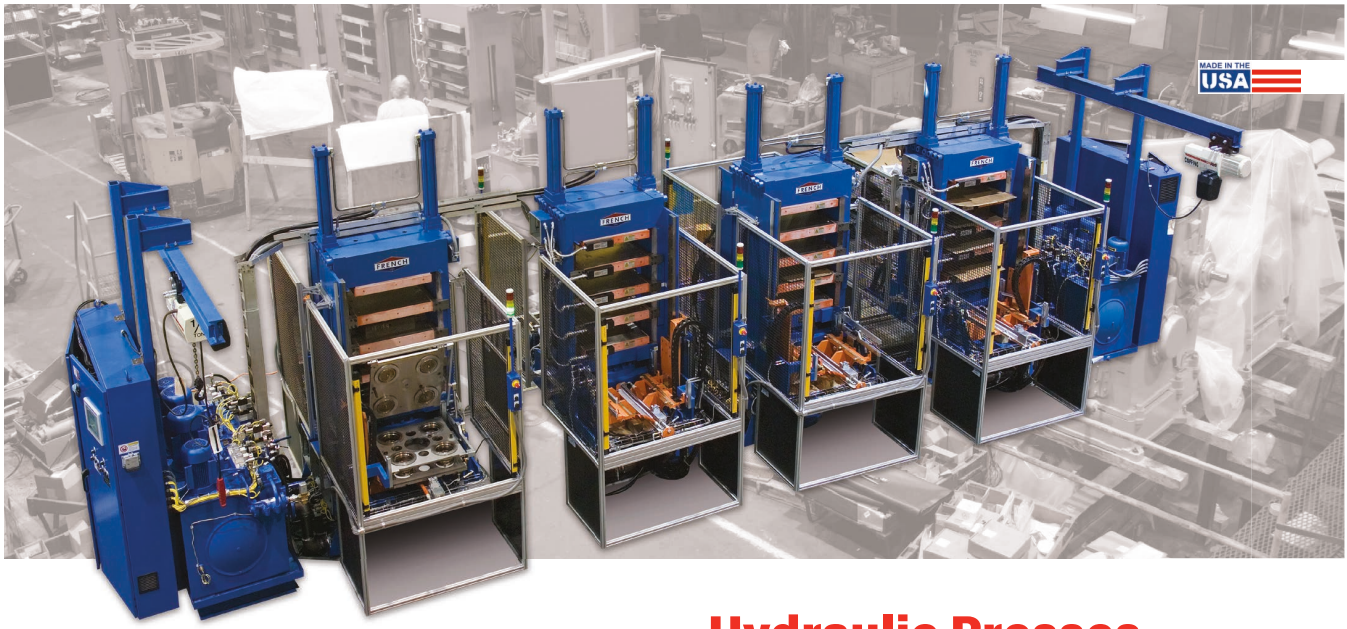

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