Research article

Recycling of carbon fiber reinforced polymers in a subcritical acetic acid solution

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ABSTRACT

A novel single-stage solvolysis process is demonstrated for recycling carbon fibers from an epoxy-based composite material using 50 wt% acetic acid solution under subcritical conditions. The process yields 100% fiber recovery efficiency in less than 30 min at 300 °C. Qualitative SEM/EDS analysis of the fibers reveals that the recovered fibers are entirely free of resin, and the carbon fiber surfaces were not damaged. SEM images and gravimetric measurements of the composites treated at lower temperatures and short residence times show an initial increase in mass of the CFRP samples, suggesting a two-step process consisting of initial composite swelling due to uptake of solvent, followed by depolymerization and chemical decomposition of the polymer. FTIR and GC-MS analyses confirm resin decomposition and production of aromatic and aliphatic compounds.

1. Introduction

The use of composite materials dates back to the Mesopotamian era when people glued strips of wood at different angles and multiple layers to create plywood. Today, composite materials are used in automotive and aerospace industries, sports equipment, and other consumer products. Widely used in the aviation industry, carbon fiber reinforced polymer (CFRP) primarily consists of two components: the matrix and the reinforcement. The carbon fiber is used as reinforcement, providing strength to the composite. The matrix is generally a resin polymer or epoxy, which binds the carbon fiber. The two components are combined in a controlled proportion and process, yielding the desired material properties, *e.g.*, a high strength-to-weight ratio. CFRPs achieve tensile strength 3 to 5 times greater than stainless steel while being 30%–50% lighter. The ubiquitous use of CFRPs raises questions about their reuse and recycling practices from the perspective of environmental sustainability and the quality of the recycled feedstock [1, 2, 3, 4]. It is estimated that 30% of the total production is disposed of as waste, including the waste generated during the manufacturing process, constituting the composite's End-of-Life (EoL). Therefore, recycling most of the generated waste would significantly benefit both the environmental and economic aspects of the CFRP life cycle [2]. Recycled fibers can be reintroduced for non-structural applications in various industries, such as the automotive industry, sports, and consumer goods [5, 6, 7]. CFRP recycling depends

on developing secondary markets that can accept the fibers and other components from the process [8].

Current CFRP recycling technologies can be broadly categorized into mechanical, thermal, and chemical processes. Mechanical processes employ a grinding step to physically break the composite material and produce grain-size particles that can be introduced in manufacturing CFRP composites. Thermal techniques use high temperatures to vaporize the polymer matrix and leave the fibers as a product that can be recovered. Chemical techniques utilize various solvent-water mixtures that can chemically decompose the polymers, leaving the fibers intact for subsequent collection. Thermal and chemical processes are preferred over mechanical techniques due to their ability to recover long strands of fibers. The mechanical properties of the components manufactured using mechanically recovered fibers have inferior performance due to reduced fiber length [9].

Thermal recycling of CFRP by pyrolysis requires temperatures in the 450–700 °C range and an inert atmosphere to degrade and volatilize the polymer matrix, yielding clean carbon fibers [4, 10, 11]. In pyrolysis, the polymer matrix decomposes into two distinct product phases: gaseous low molecular weight (MW) compounds and high MW compounds such as oils and tars. Both product streams face subsequent discharge challenges, as the gaseous phase must be treated to meet emissions requirements, and the liquid phase is often classified as hazardous waste. Pyrolysis may also lead to the deposition or condensation of tar and char on the recovered fibers, reducing the quality of carbon fiber [4, 11]. The

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high MW species can be converted to low MW species via secondary pyrolysis at higher temperatures or fully carbonized [12], exposing the composite matrix to elevated temperatures can damage the fibers, reducing the mechanical properties of the recycled CFRP composite [13, 14, 15].

Chemical recycling of CFRP, or solvolysis, chemically de-binds the CFs from the polymer matrix by dissolution and depolymerizing the polymer. Acids, bases, alcohols, and other solvents have been explored with varied success [16, 17, 18]. Water has been used as a solvent at high-temperature, high-pressure (HTHP) conditions. CFRP recycling has been explored in both subcritical (<374 °C) and supercritical water (SCW) environments, which both can be effective at decomposing organic compounds [19, 20, 21] and recalcitrant chemicals [22]. Decomposition rates of organic compounds in SCW are typically faster than in subcritical water conditions [20, 23, 24], especially when an oxidant is added to promote oxidation in supercritical water [25, 26, 27, 28, 29].

Several promising chemical CFRP recycling studies used acids and solvents under subcritical conditions. Optimization of solvents and processes is required to increase the economic feasibility of the process [30]. Das et al. [31] used peracetic acid (acetic acid combined with H_2O_2) to decompose CFRP coupons at atmospheric pressure and moderate temperatures. They hypothesized that though peracetic acid is relatively weak, it is readily hydrolyzed in the aqueous solution. The tensile strength of the fibers was reported as similar to virgin fibers, and SEM/EDS analysis confirmed little-to-no damage of the fibers. This chemical oxidation process at atmospheric pressures may allow for the continuous recycling of fibers. Still, disposal or reuse of the liquid product remains challenging, and operating costs must be assessed against existing practices. Yu et al. [32] demonstrated that ethylene glycol (EG) could be used to decompose CFRP resin at 160–180 °C, and again the fibers retained >95% of the virgin fibers' tensile strength. A comprehensive review of CRFP recycling studies is presented by Liu et al. [33].

Exposure to weak organic acids has resulted in CFRP swelling. Xing et al. reported that CFRP composites delaminated into single layers at 160 °C–220 °C after 1 h of exposure to acetic acid [34]. Ballout et al. showed delamination of CFRP composites in formic acid [35]. The effectiveness of the weak organic acids for resin decomposition can be attributed to their hydrolyzation in aqueous solutions. Wang et al. reported that AlCl3/acetic acid decomposes epoxy resins polymer via selective cleavage of the carbon-nitrogen bond [36]. Other studies used amendments in acetic acid to promote polymer degradation [11, 37].

Chemical recycling can be used for a wide size range of composite materials. Once the resin matrix is decomposed, the RCFs can be collected and cleaned before reuse [38, 39, 40]. Fibers also tend to retain their original shape and tensile strength through chemical recycling [16, 41, 42]. Chemical recycling presents an opportunity to recover and reuse the polymer decomposition products captured in the liquid phase [43, 44, 45]. An improved understanding of promising CFRP chemical recycling conditions, chemistries, and reaction mechanisms is needed to advance the technology toward wider commercial adoption [46].

This work demonstrates a novel single-stage solvolysis process for recycling carbon fibers from an unknown epoxy-based resin composite using 50 wt% acetic acid solution under subcritical conditions. The process yields 100% fiber recovery efficiency in less than 30 min at T = 300 °C. The observed mechanism is a two-step process: (i) initial composite swelling due to solvent uptake and (ii) depolymerization and chemical decomposition of the polymer.

2. Materials and methods

2.1. Experimental apparatus

Experiments were carried out in a high-pressure batch reactor system comprising a 316 stainless steel vessel (High Pressure Equipment Co.;
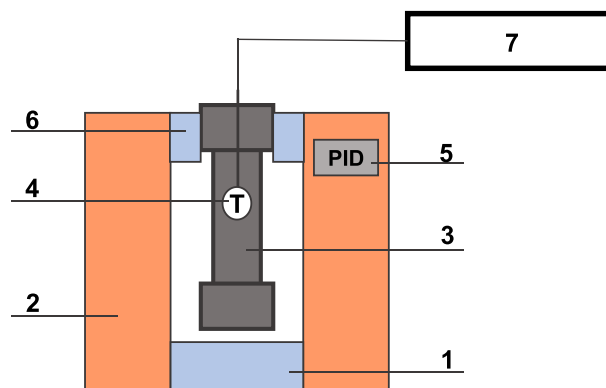

Figure 1. Schematic of a batch reactor system used for CFRP recycling consisting of (1) and (6) insulation, (2) electric tubular furnace, (3) reactor vessel, (4) internal thermocouple, (5) internal PID controller, and (7) DAQ.

Erie, PA), a tube furnace (Barnstead Thermolyne Corp.; Dubuque, IA), and an internal K-type thermocouple to monitor the reaction temperature, as shown in Figure 1. The reactor vessel dimensions are 304.8 mm L x 25.4 mm D, with an internal volume of 50 mL. Fiberglass insulation was used to insulate the reactor vessel inside the furnace. A thermocouple data acquisition (DAQ) module with a proportional–integral–derivative (PID) controller was used to set the temperature during experiments.

2.2. Reagent purity and preparation

A thermoset CFRP composite with an unknown composition was used for all experiments. The total fraction of resin in the composite matrix was determined by thermogravimetric analysis (TGA, TA Instruments, Q50). It was found to be in the range of 26–32 % of the total mass of the original CFRP. The composite sample was cut into 50 mm × 10 mm x 2 mm coupons. An aqueous solution of 50 wt% glacial acetic acid (99%, Fischer Scientific) and 50 wt% DI water (resistivity 18.2 MΩ-cm) was used in all tests.

Note that initially, several solvents were screened: NaOH (concentrations: 0.1 M, 0.5 M, and 1 M); 10% IPA; 10 wt% ethanol; and acetic acid (concentrations 10 wt%, 20 wt%, 40 wt%, and 60 wt%) at 300 °C for 60 min. The liquid products from all the experiments suggested some decomposition of the resin; however, the fiber imaging showed that the resin was not removed from the composite in most cases. The experiment with 40 wt% and 60 wt% acetic acid cases showed the best resin removal. Thus, to study the temperature and residence time trends, 50 wt% acetic acid solution was used.

2.3. Data collection and analysis

The resin removal rate (R_R) for the composite matrix was calculated based on the mass of CFs recovered at the end of each experiment, as shown below:

$$R_R = \frac{W_{CFRP} - W_s}{W_{CFRP}} \times 100$$

where W_{CFRP} is the mass of the CFRP composite before the start of the experiment, W_s is the mass of the RCFs collected after drying the remained solids.

The chemical composition of the liquid samples was analyzed using ATR-FTIR (ThermoFisher Scientific, Nicolet iS 10 FTIR Spectrometer) and GC-MS (Agilent Technologies, MS: 5973; GC: 6890 with 7683 autosampler). The liquid samples were preprocessed before analyzing in the GC-MS. An aliquot of 5 mL was mixed with 5 mL of dichloromethane (DCM) to extract the organic solvents present in the sample. Sodium hydroxide (ACS reagent, ≥99.0%, anhydrous, granular, Sigma Aldrich) was added to the extracted liquid to remove the water from the organic

extracts. The organic compounds in the solution were identified by comparing the mass spectra with the inbuilt NIST database of the instrument.

2.4. Experimental conditions

During each experiment, 35 mL of acetic acid solution and a 5 g CFRP coupon were loaded into the batch reactor and sealed. The reactor was then placed inside the tube furnace, heated to the target temperature (T = 200–300 °C), and held for the desired residence time (τ_{res} = 0–120 min). The residence time datum (τ_{res} = 0 min) was set when the target experimental temperature was reached in the reactor. On average, the heat-up time was ~80 min. Expansion of the solution during heating resulted in an autogenic pressure rise to P ~ 10 MPa, keeping all reagents in a compressed liquid phase, i.e., below the transition to the supercritical mixture (critical point of water is P > 22.1 MPA, T > 374 °C). After the target exposure time was reached, the reactor was cooled rapidly by immersion in cold water until it returned to room temperature. The reactor contents were then collected and filtered using Grade 1 Whatman filter paper (Millipore Sigma, Burlington, MA, US). The liquid consisting of solvent and decomposed resin was stored in a Nalgene vial for further analysis. The recycled carbon fibers (RCFs) were then rinsed with DI water and cleaned in an ultrasonic bath for 15 min in IPA (99.9 %, Sigma Aldrich) to complete the removal of liquid residues from the recovered fibers. The cleaned fibers were then dried in a desiccant dehumidifier for 24 h. Figure 2 shows the example of the recovered sample after 30 min exposure at 300 °C. The image is a typical example of the clean fibers, the SEM images for the same condition are presented in Figure 5.

3. Results & discussion

3.1. Analysis of recovered fibers

All recovered fibers were dried and weighed to assess resin decomposition during each experiment. Figure 1 shows an image of the recovered fibers from one experiment, while Table 1 shows the resin removal percentage for all conditions in the experimental matrix.

Figure 3 shows the resin decomposition trends at various temperatures. For all residence times at 200 °C, 225 °C, and 250 °C, the overall weight of the RCF coupon increases, with a maximum weight gain after 90 min for exposure at 225 °C and 250 °C. This trend is followed by a loss

Figure 2. Photograph of recovered fibers after exposing the composite to a 50 wt% acetic acid solution at 300 °C for 30 min.

Table 1. Resin removal rate (%) after exposure to 50 wt% acetic acid solution at various temperatures and exposure times. Positive values indicate a mass decrease, while negative values indicate a weight gain, indicating swelling of the polymer matrix.

Temp. (°C)	Resin removal rate (%)				
	0 min	30 min	60 min	90 min	120 min
200	0	-3.96	-3.61	-4.40	-5.99
225	-4.35	-4.49	-6.53	-18.6	-3.92
250	-6.82	-3.39	-2.04	-16.52	3.98
275	-6.00	1.01	15.81	20.92	25.71
300	2.08	28.49	31.92	32.97	31.71

of mass at longer residence times, indicating that the exposed resins undergo an initial weight gain phase, likely due to solvent uptake into the polymer matrix, which has been previously reported [36, 47, 48, 34]. After the initial swelling step, we infer that the polymer begins to degrade in earnest, likely due to a combination of decomposition and depolymerization mechanisms. The temperature window from 200 °C and 250 °C show relatively to be a slow swelling process (>60 min), while higher temperatures encourage a faster swelling process, followed by more rapid decomposition and depolymerization, which can be seen in the weight loss of coupons exposed to the solution at 275 °C and 300 °C. Very little resin remained after treatment at 300 °C at τ_{res} > 30 min. We hypothesize that the swelling process plays a crucial role in the ability of the acetic acid solution to degrade the polymer matrix efficiently, and it may be possible to leverage this effect for more rapid decomposition by soaking the CFRP at a lower temperature before thermal treatment. Interestingly, the heating process (τ_{res} = 0 min experiments) resulted in no mass gain for T = 200 °C, about 5–7 % gain for T = 225 °C–275 °C, and mass loss when the sample was heated up to T = 300 °C. It is apparent this process is temperature-dependent, and it may be that some "activation" temperature is required for the acetic acid solution to penetrate the CFRP matrix.

SEM images of the composite matrix and recovered fibers indicate that 100% resin removal is achieved after exposure to the 50 wt% acetic acid solution at 300 °C Figure 4 (a-c) shows SEM images of the CFRP composite before the exposure, while Figure 5 (a-f) shows SEM images of the fibers recovered after the 300 °C exposure experiments. EDS analysis showed no other elements on the recovered fiber surfaces besides carbon, see Figure 6.

A limitation of this paper is that fiber tensile strength at various temperatures and exposure times was not investigated. Several authors examined the effect of the recycling condition on fiber mechanical
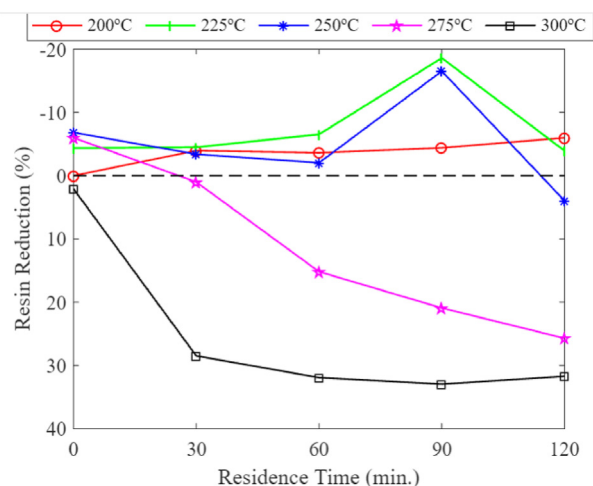

Figure 3. Resin removal rate at various experimental temperatures. The lower temperature experiments show sample mass gain before the weight reduction at the longer residence times.
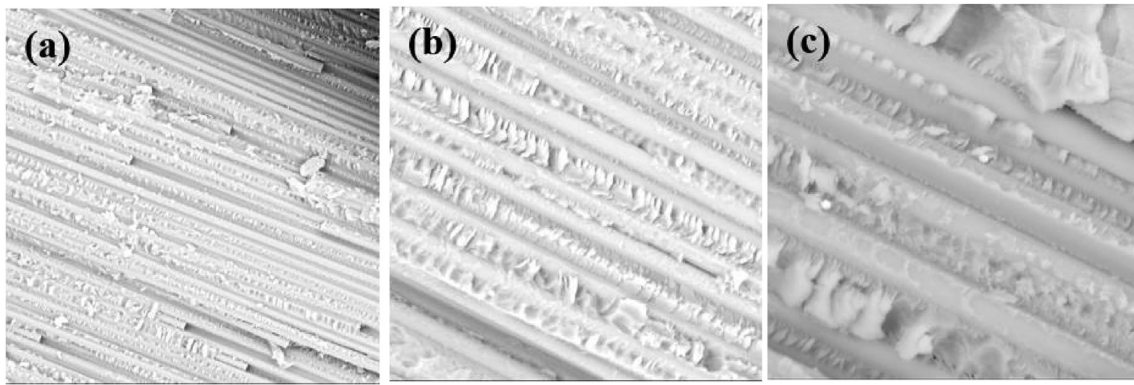
Figure 4. SEM images of CFRP composites before exposure (magnification: 850x, 2250x, and 4000x respectively).
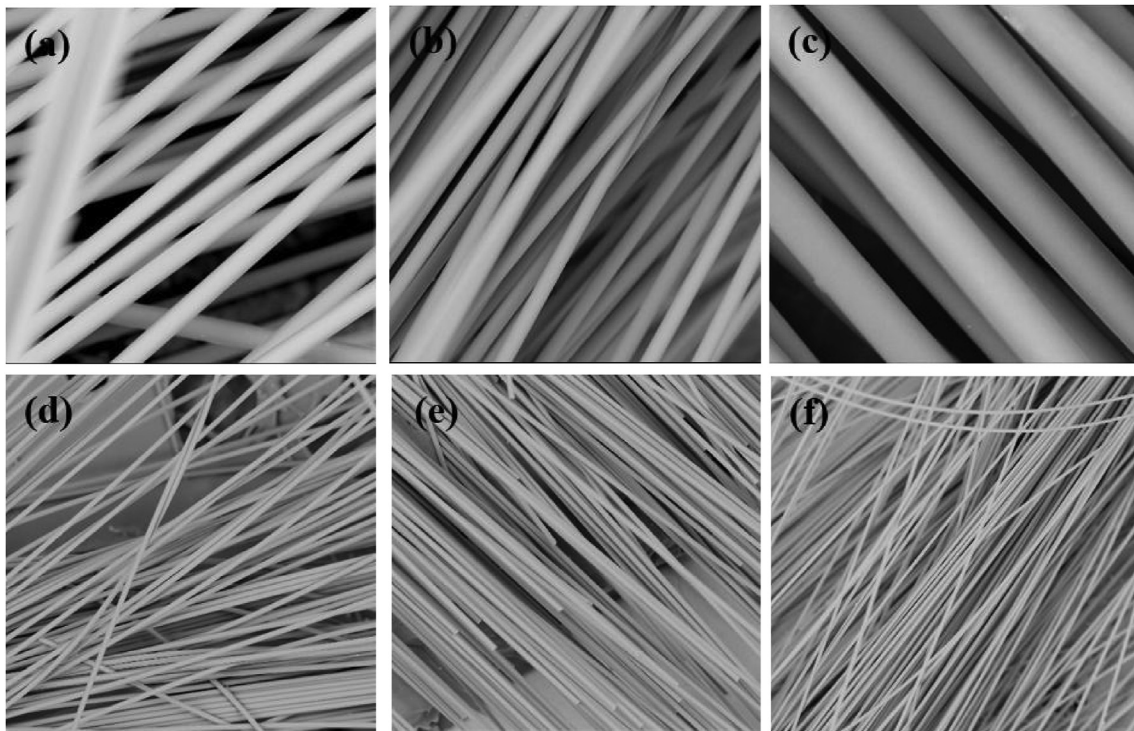

Figure 5. SEM images of recovered carbon fibers after exposure at 300 °C. (a), (b), and (c) show strands of fibers with 100% resin removal (magnification: 2500x) while (d), (e), and (f) show multiple layers of clean fibers (magnification: 590x).
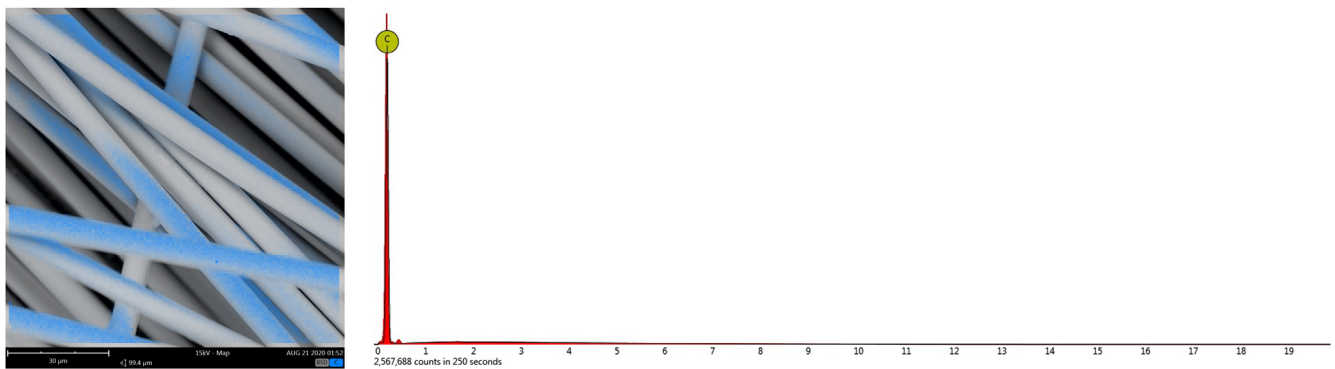

Figure 6. EDS analysis of the recovered fibers treated at 300 °C for 30 min.
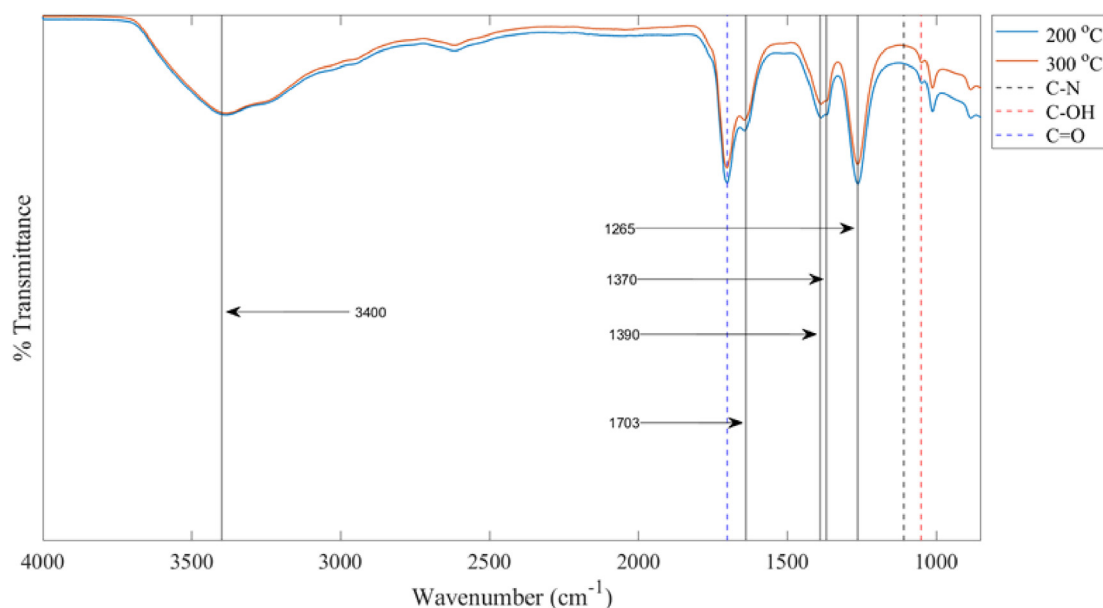
Figure 7. ATR-FTIR spectra of liquid products from CFRP decomposition experiments in 50 wt% acetic acid solution at 200 °C (blue) and 300 °C (red).

properties. Recent reports suggest that the reclaimed fibers have similar chemical structure and graphitization to virgin fibers and comparable tensile strength [49, 50, 51].

3.2. Analysis of liquid products

The liquid products from each experiment were analyzed using ATR-FTIR to determine their composition. Figure 7 shows the ATR-FTIR spectra of the liquid products recovered after exposure at 200 °C and 300 °C for 30 min. Peaks at 1390 cm^{-1} and 1703 cm^{-1} indicate the presence of phenolic, carbonyl, and aliphatic ketones in the recovered liquids [52, 53, 54]. Higher temperature treatment shows the formation of additional C=O bonds indicated by the peak at 1703 cm^{-1}. The peaks corresponding to alkyl aryl ethers, amines, and aliphatic nitro compounds can be observed at 1265 cm^{-1}, 1640 cm^{-1}, and 1370 cm^{-1}, respectively. The formation of species with similar functional groups has been previously reported in the degradation of DGEBA epoxies which are generally used for aerospace applications [31, 32, 52, 53, 54, 55].

The solvolysis process of epoxy-based resin composite material in the mixture of 50 wt% acetic acid solution under subcritical conditions was characterized as a function of temperature and residence time. Two a two-step process was observed: (i) initial composite swelling due to solvent uptake and (ii) depolymerization and chemical decomposition of the polymer. At the lower temperatures, the mass was increasing, indicating the uptake of the solution into the CFRP matrix. At T = 250 °C, the resin CFRP started to lose mass after 90 min showing the onset of depolymerization. At higher temperatures, swelling and depolymerization occur significantly faster, and the process yields 100% CF recovery in less than 30 min at 300 °C. The absence of an absorption peak in the C–N bond at 1110 cm-1 suggests that the polymer decomposition may proceed via C–N bond cleavage as previously reported in CFRP treatment by acetic acid with alkali amendment [36] and by the mixture of acetic acid and H2O2 [31]. Since the actual composition of the epoxy in this study was not known, we cannot fully determine the decomposition route.

4. Conclusions

The recycling of aerospace-grade CFRP composite material in hot-compressed 50 wt% acetic acid solution is demonstrated. We show that complete resin decomposition and recovery could be achieved in an acetic acid solution without an alkali catalyst. Decomposition is characterized as a function of exposure time and temperature; the range T = 200–250 °C promotes acetic acid-induced swelling of the polymer, and temperatures above 250 °C yield rapid polymer degradation and depolymerization. A 100% resin decomposition and fiber recovery are demonstrated at 275 °C (residence times ~120 min) and 300 °C (residence times ~30 min). Analysis of the liquid products reveals the presence of aromatic and aliphatic compounds, which could potentially be recovered for reuse.

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