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# Improving Solvolysis Rate Via Under Glass-Transition Temperatures Quenching Followed by Roll Bending of Carbon Fiber Reinforced Polymer Composite

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## Abstract

*Reusing carbon fiber once used as reinforcement in composite structures involves a complex recycling technique. This recycling process requires a strategy to ensure that the recycled carbon fiber meets technical standards. Among various recycling processes, the solvolysis technique yields carbon fiber with high purity. However, solvolysis is also the most time-consuming method. This study proposes using quenching and roll-bending pretreatments, which increase the contact surface area between the composite and the solvent solution for expediting the solvolysis process of carbon fiber reinforced polymer (CFRP) composite. Quenching was performed by heating the composite to 75°C, 100°C, and 125°C before immersing it in water at room temperature. Roll bending was designed based on the principle of three-point bending and aimed to induce a strain in the composite that exceeds the strain of the epoxy resin. Quenching at 75°C, followed by roll bending, resulted in a 21% increase in the surface area of the composite. This increased area improved the solvolysis efficiency of CFRP composites by up to 21% after four days of immersion in a mixture of acetone and acetic acid.*

**Keywords***: solvolysis; glass transition temperature; quenching; surface area; delamination*

## 1. Introduction

The recycling research of epoxy resin CFRP composites has dominated the field of study. Thermosetting epoxy resins constitute up to 55% of the various polymers used as composite matrices, owing to their exceptional mechanical properties and resistance to environmental changes. However, these superior properties pose a challenge when determining suitable treatments for the recycling process. Furthermore, the thermosetting nature of epoxy resins, which cannot be melted, adds to the complexity of recycling.

In CFRP composite recycling, the primary objective is the recovery or reclamation of carbon fiber. The high cost of virgin carbon fiber motivates efforts to recycle used carbon fiber. Mechanical recycling methods and matrix decomposition methods have not been extensively developed due to their drawbacks, such as producing recycled carbon fiber of poor quality and low productivity. Pyrolysis and solvolysis methods have shown the most potential for recovering carbon fiber from composites without significant quality degradation. Numerous studies have explored the feasibility and effectiveness of these recycling methods. For example, Naqvi et al. conducted a study on the pyrolysis of CFRPs and found that the resulting carbon fibers exhibited properties comparable to virgin fibers. However, pyrolysis requires further research to address certain drawbacks, including feedstock and product uncertainty, energy consumption, and concerns regarding safety and environmental impact (Naqvi et al., 2018).

On the other hand, research on solvolysis for CFRP recycling reveals that this method can reclaim carbon fiber with a quality of up to 95% compared to virgin carbon fiber (Utekar et al., 2021). In addition, none of the composite components or solvents used in the solvolysis process are wasted. The carbon fiber can be recovered without significant defects, and the polymer matrix, solvent, or catalyst can be separated and reused for subsequent processes (Tian et al., 2022). This finding has motivated researchers to extensively investigate and advance the solvolysis method to overcome its primary limitation: the relatively longer recycling time than other methods. The use of solvents in the form of near- and

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supercritical fluids has not been able to produce recycling rates that are technically and economically acceptable (Pakdel et al., 2021).

The investigation of modifying the contact area between the solvent and the unused CFRP composite has not been extensively explored. In theory, a larger contact area accelerates the solubility of the epoxy resin in the solvent, similar to how dissolving sugar with larger grain sizes takes longer than smaller ones. The electrochemical method for fragmenting the epoxy matrix has not been widely developed to increase the contact area. It requires high electrical voltages, and the resulting carbon fiber often contains numerous defects. The crosslinked network formed within thermosetting polymer resin poses a significant challenge in matrix fragmentation (Mativenga et al., 2016). However, a pretreatment method can enhance the area through cracking, delamination, or fragmentation. In that case, there is potential to significantly speed up the solvolysis process and enable faster recycling of CFRP composites.

In a previous work (Sukanto et al., 2023), the effect of quenching treatment on CFRP composites was investigated at a temperature close to the glass transition temperature (Tg). The results showed a decrease in the ductility of the epoxy resin but an increase in the interlaminar shear strength (ILSS). However, an increase in ILSS is undesirable in composite recycling, so the quenching temperature needs to be below Tg. Furthermore, applying bending loading can induce cracks or fractures in the resin matrix without causing damage to the carbon fiber, as long as the quenching treatment effectively renders the epoxy resin more brittle. This article presents the results of solvolysis conducted on CFRP composites after pretreatment of quenching below Tg and continuous roll bending.

## 2. Materials and Methods

### 2.1 Materials

The matrix of the CFRP composite was composed of epoxy diglycidyl ether bisphenol A (DGEBA, Eposchon brand) with an epoxide functional group of 189±5 g/mol. The hardener employed was a cycloaliphatic amine known as Eposchon EP 555, primarily consisting of 3-aminomethyl-3,5/5 trimethylcyclohexylamine. This amine is equivalent to isophorone dimethyl amines, with a hydrogen equivalent weight of 86 g/mol and a viscosity of 0.5 – 1 poise at room temperature. As for the composite reinforcement, it consisted of woven Toray T300 carbon fiber. The woven texture of the carbon fiber was a 2 x 2 twill pattern, with five weft and five warp threads per 10 mm spacing. Each yarn comprised 3000 fibers, resulting in a 200 g/m2 density and an average thickness of 0.2 mm. Before being used in the composite, the epoxy resin and carbon fiber underwent no pretreatment and were used as received.

The CFRP composite was produced using hand-laying techniques with one layer of woven carbon fiber for ILSS testing and two layers for the solvolysis process. The composite matrix was prepared by mixing DGEBA and cycloaliphatic amine in a weight ratio 2:1, following the manufacturer's recommendation. The resulting composite thickness ranged from 1.5 mm to 2.1 mm. The composite was left to cure at room temperature for at least 48 hours. Subsequently, a post-curing treatment was carried out in an electric dryer oven at 60°C for 6 hours. This post-curing helps enhance the composite's mechanical properties and stability before undergoing further processes.

The solvolysis process involved a decomposing agent or solvent comprising a mixture of acetone (propanone, C3H6O) with a relative polarity of 0.355 and glacial acetic acid (vinegar, C2H4O2) with a relative polarity of 0.648. The acetone used had a purity of 99.5% and was sourced from the Sydney Solvent product, an industrial grade UN 1090 Hazchem 2YE PKG Group II. Additionally, the glacial acetic acid had a purity of 99.8% and was of technical grade (UN 2789) produced by Alliance Chemical. The acetone and acetic acid were mixed in a volume ratio of 1:4.

### 2.2 Method

Differential scanning calorymetry (DSC) testing is applied to the epoxy resin matrix to determine the glass transition temperature (Tg). By testing three samples, an average Tg was found to be 142ºC. Considering these results, CFRP composite specimens underwent quenching treatments at three temperature elevations: 75°C, 100°C, and 125°C. The heating process was conducted in an electric oven under atmospheric conditions, with a maximum heating rate of 25°C/minute. Calibration of the heating rate can be observed by increasing the temperature at certain time intervals. Once the target temperature was reached, it was maintained for 3 minutes to guarantee that the temperature of the specimens had reached a uniform condition at every point. Immediately after heating, the quenching process was carried out by immersing the composite in water at room temperature, which was set at 25 ± 2°C. The volume of water used for quenching was at least 60 times the composite volume to ensure that the quenching fluid did not experience a temperature rise exceeding 5°C during immersion (Wijang et al., 2015). A mercury or digital precision thermometer can be used directly to monitor the water temperature during quenching. The epoxy resin and composite were labeled according to the quenching treatment temperature variable to facilitate specimen identification, as detailed in Table 1.

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Roll bending was designed to induce new surfaces through cracks or fragments in the epoxy resin. The composite specimen was subjected to bending twice in opposite directions, applying tension and pressure to each surface. The curvature radius is adjusted by changing the vertical distance between the pressing and support rollers, as shown in Figure 1. This adjustment causes the specimen to deflect, and the deflection is set at 5.5 mm in the middle of the support rollers, which are placed 40 mm apart. The deflection was adjusted using a thread so that the depth can be guaranteed to be precise. With this setup, the pure bending model forms a radius of curvature of approximately 18 mm, sufficient to create strain and induce cracks in the epoxy resin.

The CFRP composites underwent roll bending before being cut to dimensions of 30 x 25 x 1.7 mm³ for the solvolysis process. The size of the soaking vessel was carefully selected to ensure complete immersion of all specimens during the solvolysis process. The vessel was made from ceramic glass material to ensure that the solvent did not dissolve it. The solvolysis treatment occurs without additional thermal energy or stirring. Thus, it must be carried out in a room where it is ensured that there are no significant changes in temperature and humidity. The immersion time was varied within the range of 1 to 4 days. After the solvolysis process, the carbon fiber was separated from the solvent and left to dry naturally in the open air for 24 hours. Subsequently, the drying process continued in an oven dryer at 50°C for 2 hours to ensure the water content in the composite was no more than 1%.

## 3. Results and Discussion

## 3.1 Results

### 3.1.1 Epoxy resin infrared spectrum

The infrared spectrum of the thermoset epoxy resin without quenching, obtained from Fourier Transform Infrared or FTIR testing, is displayed in Figure 2. The spectrum exhibits characteristic patterns of the polymer material (Nandiyanto et al., 2019), with specific epoxy functional groups detected at peak wavenumbers 828, 958, 1181, 1297, 1609, and 2927 cm$^{-1}$ (Maity et al., 2008). During the quenching treatment of the epoxy resin up to 125°C, no peak shift was observed in the infrared spectrum, but changes in the absorbance were evident, as obtained in previous research (Sukanto et al., 2023). The thermosetting polymer crosslink network emerges from the fingerprint zone at wavenumbers 1181 and 1297 cm$^{-1}$ as C–N bonds. The epoxy crosslink, acting as a secondary amine and hydroxyl group, is formed through the reaction between the exposed epoxide ring and the primary amine group belonging to the hardener. Additionally, the crosslink is formed as a tertiary amine group, characterized by the presence of a $-CH_2-$ bond group at the peak of the wavenumber 732 cm$^{-1}$.

**Table 1**. Specimen identification.

| Specimen | Description |
|---|---|
| T0 | The specimen without quenching |
| T1 | The specimen was quenched at 75°C |
| T2 | The specimen was quenched at 100°C |
| T3 | The specimen was quenched at 125°C |

### 3.1.2 Roll bending of CFRP

After quenching treatment, the CFRP composite exhibits a distinct crack pattern when subjected to three-point roll bending. The alterations in ductility and ILSS are crucial factors in the formation of cracks and delamination. In the composites without quenching treatment (Figure 3(a)), there are fewer epoxy resin cracks and the cracks that occur may not always cause fractures in the epoxy resin. Composites with low ILSS and ductility (Figure 3(b), (c), and (d)) have the potential to exhibit open delamination and resin fragmentation. Delamination is more significant in creating new areas than cracks or fractures. As a result, low ILSS has the potential to form larger areas than low-strain epoxy resin. The exposed surface area is formed from cracks or fractures in the outer layer of the epoxy resin, propagating along the interface between the epoxy resin and the carbon fiber. However, the occurrence of cracks or fractures in the epoxy resin's outer surface does not

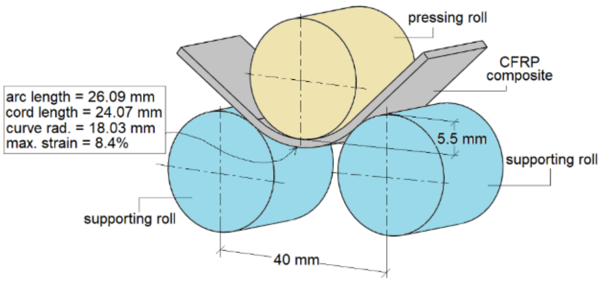


**Figure 1.** The scheme of roll bending.

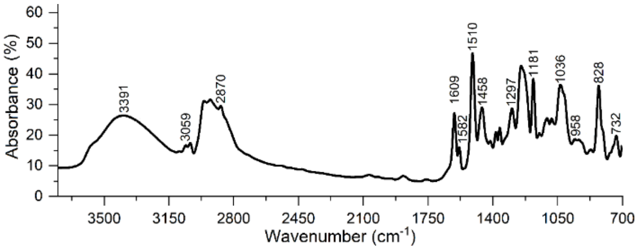
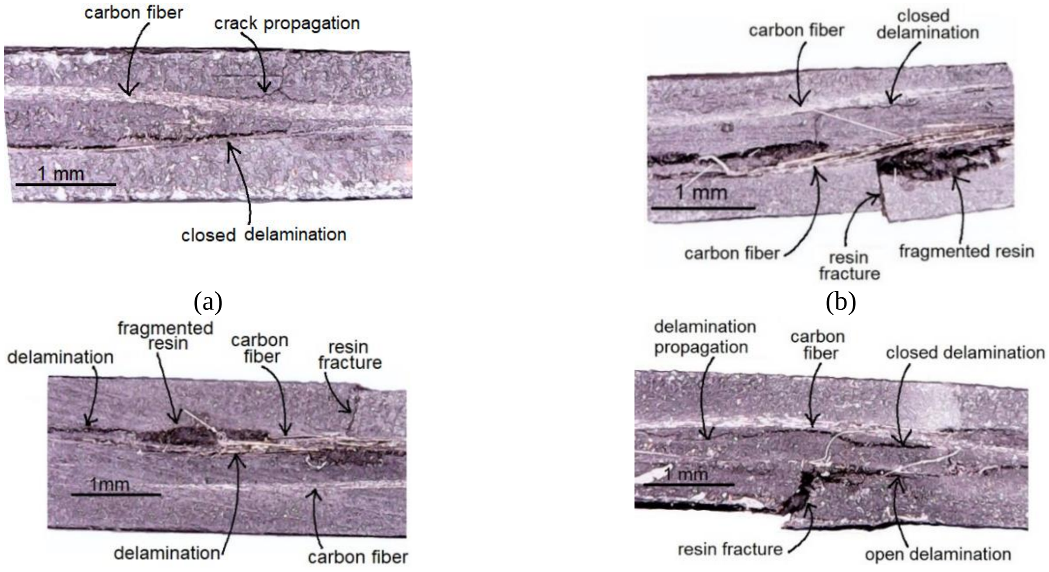


**Figure 2.** Epoxy resin infrared spectrum without quenching treatment

always lead to delamination. Delamination or cracks can also arise at the interface without being preceded by a crack or fracture in the epoxy resin (closed delamination). In contrast, open delamination and resin fragmentation involve the formation of more expansive surfaces and cracks that may cut the carbon fiber, a phenomenon not observed in closed delamination.

### 3.1.3 CFRP composite degradation by solvolysis

Solvolysis of CFRP composites without quenching for up to 4 days (96 hours) still left the epoxy resin undecomposed and bonded to the carbon fibers, as depicted in Figure 4. However, it was observed that fragments of the epoxy resin matrix detached from the carbon fibers during immersion. After subjecting the



(a) (b)

(c) (d)

**Figure 3**. Macroscopy images of composite CFRP after roll bending (a) T0, (b) T1, (c) T2 dan (d) T3.

**Figure 4.** Images of composite solvolysis result after roll bending without quenching (T0).

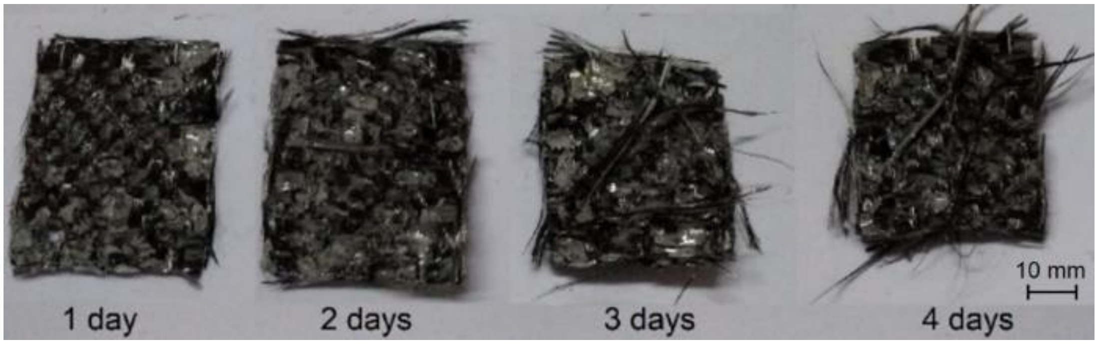


**Figure 5.** Images of composite solvolysis result after roll bending with quenching at 100ºC (T2).

composite to quenching and roll bending, all the epoxy resin mass attached to the carbon fibers is degraded and no longer bound to the carbon fibers, as illustrated in Figure 5.

Epoxy resin decomposition results in a reduction in the total weight of the composite after solvolysis. This weight loss serves as the basis for calculating the efficiency of the solvolysis process using Equation (1).

$$E_S = \frac{m_1 - m_2}{m_1 \cdot f_m} . 100\% \qquad (1)$$

$E_S$ is the solvolysis efficiency, and $m_1$, $m_2$, and $f_m$, respectively, represent the weight of the composite before the solvolysis, the weight of the composite after the solvolysis, and the weight fraction of the epoxy resin.

Higher efficiency values indicate that a greater mass of epoxy resin is degraded and released from the composite during the solvolysis process. The efficiency results are summarized in Table 2. The addition of 24 hours (a day) of solvolysis time increased solvolysis efficiency in all composites with quenching. Among the composites subjected to quenching, immersing for 96 hours (4 days) at 75°C (T1) achieved the highest efficiency, reaching 79%.

### 3.2 Discussion

The effect of rapid cooling on the quenching process does not allow the epoxy resin to reach an equilibrium condition, so the atoms that make up the polymer chain cannot adjust their position to fill the void (Oliveira & Creus, 2004). This condition results in thermo-mechanical degradation characterized by softening or decomposition of the polymer. This degradation is also related to the reduction in weight of the epoxy resin, which at each surface depth has a different degradation rate (Hancox, 1998). This surface degradation can increase hydrogen absorption when the epoxy resin is dipped in water. In addition, water in the resin impairs the adhesion properties, degrades the chemical bonding and crosslinking of the polymer network, and damages the dimensional due to swelling.

The infrared spectrum provides insights into water interaction with the epoxy resin network and the diffusion mechanism during water absorption. Water exhibits three energetic vibrations associated with stretching (3800 – 3600 $cm^{-1}$) and bending O–H bonds (1650 – 1590 $cm^{-1}$). Once absorbed into the epoxy resin, water may exist as highly mobile free (± 3600 $cm^{-1}$) or held together by hydrogen bonds (± 3300 $cm^{-1}$) molecules (González et al., 2012). The infrared spectrum displays a wide wavenumber with peaks around 3400 $cm^{-1}$ and sharp peaks around 1630 $cm^{-1}$, indicating the stretching and bending modes of hydroxyl groups that are adsorbed on the surface of the epoxy resin, particularly in carboxyl or alcohol functionalities (Du et al., 1999). The absence of a C=O bond peak (1700 $cm^{-1}$) in the infrared suggests that the hydroxyl is bound in the alcohol compound within the epoxy resin. These hydroxyl groups are linked to each other through hydrogen bonds and facilitate the acceleration of thermomolecular reactions occurring during the complex curing process of the epoxy resin (Swier et al., 2005). Water absorption in epoxy resin is influenced by its chemical formula, but the quenching treatment also alters the water molecule content. The presence of water within the epoxy resin increases the mobility of the macromolecular chains. This effect is commonly observed in amine hardener-based epoxy materials. The increased mobility of the epoxy resin chain, in turn, reduces the glass transition temperature and yield stress (Le Guen-Geffroy et al., 2019). Under such conditions, a thermal shock can promote cohesive failure, decreasing the epoxy resin's mechanical strength (Fan & Li, 2012).

**Table 2**. Solvolysis efficiency of CFRP composites.

| **Specimen** | **Solvolysis efficiency, $E_S$ (%)** | | | |
|---|---|---|---|---|
| | **1 day** | **2 days** | **3 days** | **4 days** |
| T0 | 55 | 58 | 63 | 65 |
| T1 | 67 | 73 | 75 | 79 |
| T2 | 66 | 71 | 74 | 76 |
| T3 | 58 | 66 | 68 | 72 |

The heating stage during the quenching within free air allows the epoxy resin to be oxidized. The oxidation of epoxy resins is typically characterized by the reduction of hydroxyl groups or the appearance of carbonyl compounds in carboxylic groups, which can be observed and analyzed using infrared spectra at 1550 – 1610 $cm^{-1}$ (Nandiyanto et al., 2019). Oxidation of hydroxyl also causes intra-molecular dehydration in epoxy resins, further oxidizing to form carboxylic groups (Park & Blount, 1957). The increased mobility of epoxy resin molecules, when heated and followed by immersion in water, provides an opportunity for the addition of hydroxyl groups because there is sufficient energy for water molecules to diffuse into the epoxy resin (Gibhardt et al., 2022). Dispersed water molecules can more easily access other polar groups with weak bond energies or low enthalpy values (Elkebir et al., 2020). Hydroxyl groups can be attributed to the thermal degradation and oxidation of epoxy resins dominated by the weakest atomic bonds (Wolfrum et al., 2009).

The quenching procedure generates two opposing effects. First, debonding occurs due to differences in the thermal expansion coefficient of composite members. This effect leads to composite deformation, thermal stress, and micro-cracks at the carbon fiber and epoxy resin interface. Second, post-curing at high temperatures

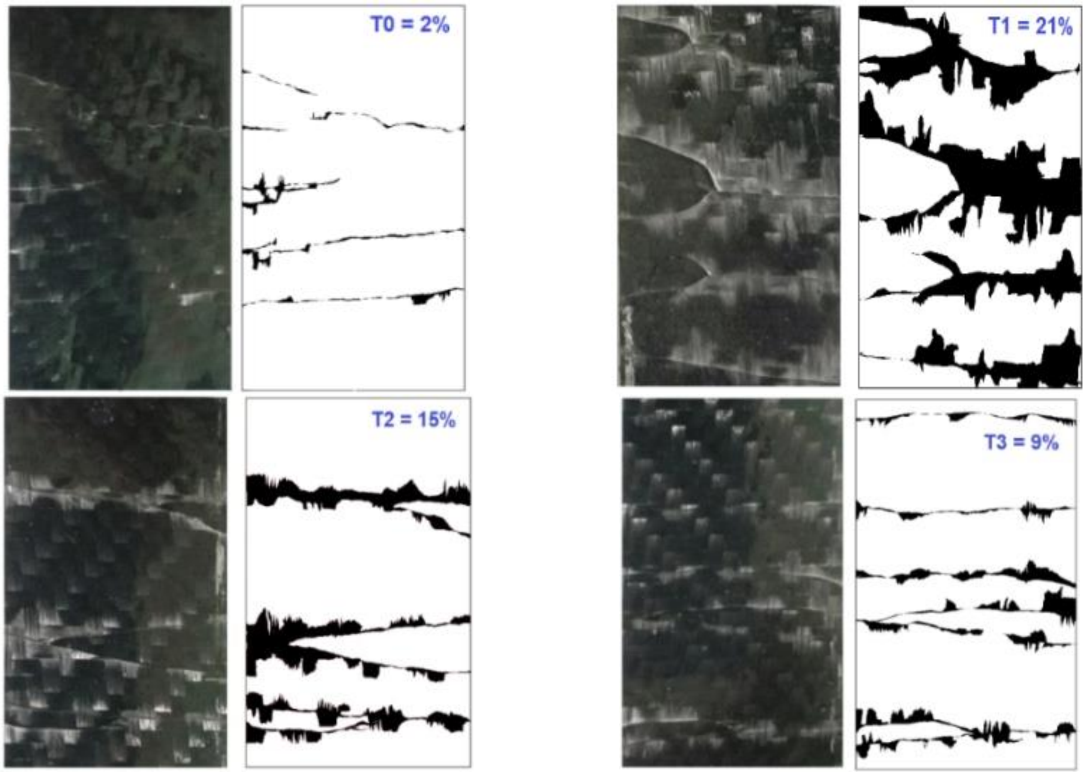


**Figure 6**. The quantification of the new surface area as a result of roll bending was analyzed with ImageJ software. Cracks and delamination are represented as black areas with random shapes and patterns.

increases the crosslink density, enhancing the composite's interlaminar shear strength (Azimpour-Shishevan et al., 2019). However, the temperature variation during quenching significantly affects ILSS due to changes in the viscosity of the epoxy resin in the interface region. This change in viscosity plays a crucial role in determining the mobility and interdiffusion distances of epoxy resin molecules (Ballout et al., 2016). The low viscosity at high temperatures also facilitates complex chemical reactions between carbon fiber and epoxy resin, improving ILSS in CFRP composites (Zhu et al., 2014). The interaction between carbon fiber and the matrix resin during thermal treatment is a complex phenomenon but plays an important role. This interaction can lead to the formation or strengthening of interfaces. The interfacial zone formed typically exhibits a different composition, microstructure, and properties than the epoxy resin matrix. The strength of interfacial bonds in amorphous epoxy resins is governed by interatomic bonds that are dependent on reaction crosslinks. Local chemical reactions can strengthen interfacial interactions, but excessive progressive reactions might create brittle bonds (Ray, 2006). The phenomenon of these two reactions may be revealed through mechanical loading.

The roll-bending process causes the formation of cracks and delamination, leading to the creation of new surfaces on the CFRP composite. These additional surface areas result from changes in the properties of the epoxy resin, which becomes more brittle and experiences a decrease in ILSS after undergoing the quenching treatment. A decrease in the ductility of the epoxy resin leads to an increase in the number of cracks in the composite. On the other hand, a reduction in ILSS results in broader areas of delamination (Sukanto et al., 2023). The area quantity approach was used, and ImageJ software was employed to analyze macro photos of the CFRP composite surfaces, assuming uniform thickness across all sections. The color thresholds of cracks and delamination areas were highlighted using contrasting color settings, as depicted in Figure 6. The additional surface areas resulting from the roll bending treatment after quenching at 75°C, 100°C, and 125°C were found to be 21%, 15%, and 9%, respectively. In contrast, roll bending on composites without quenching resulted in a minor increase in area of only 2%.

Furthermore, the solvolysis by immersing the CFRP composite in an acid solution demonstrated the dominance of backbone bond digestion of epoxy resin over decomposition (Ma & Nutt, 2018). The solvent,

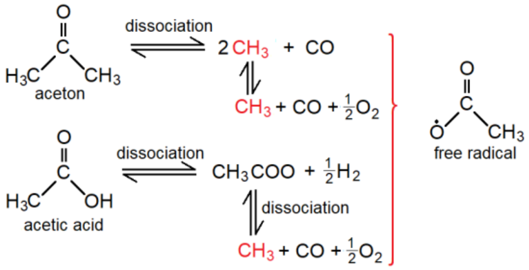


**Figure 7.** The dissociation mechanism is likely caused by radical formation from acetone and acetic acid.

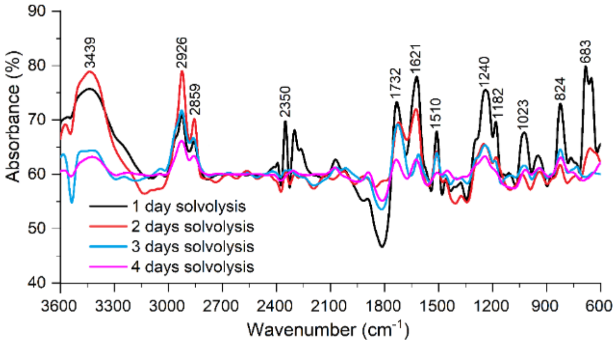
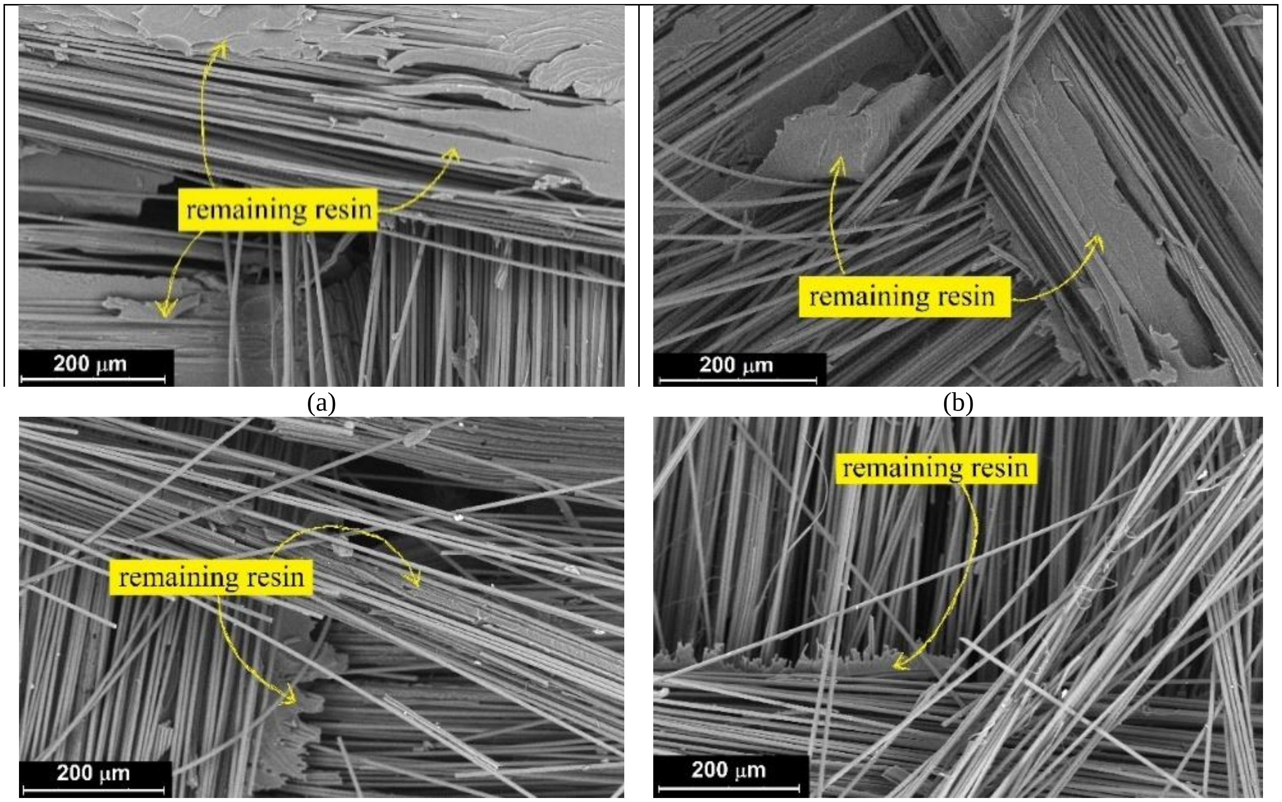


**Figure 8.** Infrared spectra comparison of epoxy resin after solvolysis for one to four days.

composed of acetic acid and acetone, creates a polar compound capable of dissolving other polar compounds, particularly the DGEBA-amine epoxy resin system. The acetone and acetic acid combination facilitates hydrogen bond interactions (Rassing, 1972). With sufficient excitation energy, acetone undergoes separation, producing methyl anions and acetyl radicals (Zhou & Schlegel, 2008). At higher excitation energies, acetone migrates into two methyl radicals (*$CH_3$) and carbon monoxide (CO) (Maddox, 1990). Methyl radicals react with oxygen to produce acetoxy free radicals ($CH_3OO$*), which have high reactivity properties (Hoare & Walsh, 1957). Acetyloxy radicals are also created from acetic acid, which separates into acetate anion ($CH_3COO$) and hydrogen gas. Subsequent dissociation converts the acetate anion into a methyl radical (*$CH_3$) and carbon monoxide gas (Lu & Continetti, 2004). The formation of methyl radicals impacts the displacement of hydrogen



(a) (b)

(c) (d)

**Figure 9**. SEM images of carbon fiber resulted from CFRP composite solvolysis, (a) T0, 4 days; (b) T3, one day; (c) T2, 3 days; (d) T1, 3 days.

atoms from carboxyl acid clusters (Wenthold & Squires, 1994). The prediction of acetoxy radical formation is illustrated in Figure 7. Acetoxy radicals act as crosslink chain breakers in epoxy resins by oxidizing C–OH groups, forming carbonyl compounds.

The C–N bond with the lowest energy exhibits the highest likelihood of bond breakage due to free radical penetration and possesses the most potent capacity to attract other atoms. The breaking of the C–N bond is identified by forming C–O bonds between the free radical and epoxy resin molecules. The C–O bond between the free radical molecule and the epoxy resin is unstable because the O atom has a high electron cloud, making it prone to binding with the H atom, which has the lowest electronegativity. The transfer of H atoms from epoxy resin molecules to free radical molecules leads to the formation of epoxy resin residues and acetic acid after undergoing a rearrangement process to achieve molecular stability (Wright & Ingold, 2000). The presence of carbonyl compounds resulting from the solvolysis process of CFRP composites is detected by the appearance of a C=O bond at an infrared wavenumber of 1732 cm$^{-1}$, as shown in Figure 8. The soaking time of the solvolysis process influences the reduction of hydroxyl groups at the wavenumber 3439 cm$^{-1}$. Stabilizing acetoxy free radicals into acetic acid, which removes hydrogen atoms from the epoxy resin, significantly reduces hydroxyl groups. The infrared spectrum also displays symmetrical and asymmetrical C–H bonds in the CH3O and CH2O groups at 2926 cm$^{-1}$ and 2859 cm$^{-1}$, respectively.

The C–H bond allows for overlap with symmetric methyl compounds and bending overtones, indicating that the benzene ring remains intact and is not collapsed due to the solvolysis process (Das et al., 2018). The unbranched saturated ether group of the tertiary amine bond is detected through a symmetrical C–O–C bond at 824 cm$^{-1}$. Based on the changing infrared absorbance, the ether groups significantly decreased after 2 days of immersion. The bonds of the crosslink groups are also altered. The amine functional groups with N–H bonds at 683 cm$^{-1}$ and 1510 cm$^{-1}$ and C–N bonds at wavenumber 1240 cm$^{-1}$ show changes in the infrared peaks that weaken as the immersion time is extended. This spectrum indicates that the immersion of the CFRP composite in a solvent solution destroys the epoxy resin by breaking the crosslink bond network. The reduced C–N bond also suggests the breaking of the interfacial bond between the carbon fiber and the epoxy resin, resulting in a cleaner carbon fiber surface as the soaking time increases and removes epoxy resin residues (Wang et al., 2015).

Solvolysis efficiency determines the quality of carbon fiber by assessing the remaining epoxy resin after immersion in a solvent. The SEM images in Figure 9 illustrate the results of the solvolysis of CFRP composites. When the solvolysis efficiency is below 70%, epoxy resin still predominantly attaches or bonds to the carbon fiber surface and is also found between the yarns. However, at a higher solvolysis efficiency, no traces of epoxy resin are visible on the carbon fiber surface. The remaining portion of the epoxy resin tends to accumulate at the junctions between the webbings due to using carbon fiber reinforcement in mat form.

## 4. Conclusion

The sequence of quenching and roll-bending applied to CFRP composites has been proven to accelerate the recycling process via solvolysis. The mechanical properties degradation of epoxy resin obtained by quenching facilitates the occurrence of cracks and delamination when the CFRP composite is subjected to roll bending to increase the composite's surface area. The quenching sequence at a temperature of 75°C, roll bending, and solvolysis processes shows a 21% improvement in recovering carbon fiber from CFRP composites. The critical point is that the creation of additional surface area resulting from the quenching and roll-bending processes can be applied to all composites with a thermosetting matrix without the need to identify their composition in detail.

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