

# **Investigation of Chemical Extraction Parameters for Efficient Epoxy Matrix Removal from End-of-Life Carbon Fibre Composites**

**Tatiya Wannomai<sup>\*1</sup>, Kittiyaporn Peankoom<sup>1</sup>, Ornin Saengdee<sup>1</sup>, Kornthat Chattawutikorn<sup>1</sup>, Laurent Mezeix<sup>1</sup>, Komkrisd Wongtimnoi<sup>1</sup>**

<sup>1</sup>Faculty of Engineering, Burapha University, 169 Longhard bangsaen road, Saensuk, Mueang chonburi, Chonburi 20131, Thailand.

\*Corresponding author: tatiya.wa@eng.buu.ac.th

**Abstract** - This research investigates approaches for carbon fibre material recovery through combined physical and chemical processes to study fibre characteristics after carbon fibre material treatment, including examination of leachate from corrosion processes, to find sustainable carbon fibre material management solutions. This research examines processes used for carbon fibre material recycling through three pretreatment conditions: nitric acid, ethanol, and acetic acid. Subsequently, combined physical and chemical processes were employed. Physical processes included ultrasonic bath sonication and temperature application to remove coating materials (epoxy resin), while chemical treatment with acetone was utilized. The results showed percentage weight reduction, particularly when using acetic acid in the pretreatment process, which caused maximum corrosion and achieved the highest epoxy resin removal efficiency. Additionally, when examining leachate from material sheets soaked in water for one month, the fibre material showed increased pH values, turbidity, and TDS (Total Dissolved Solids). This may result from epoxy resin coating detaching and becoming microplastics, as epoxy resin contains reaction catalysts that may have water-soluble residual substances, thereby increasing TDS values. These findings suggest that without proper management or control of carbon fibre material usage, environmental impacts and health effects may occur, highlighting the importance of developing sustainable approaches for carbon fibre composite waste management.

**Keywords:** Carbon Fibre, Chemical Extraction, Epoxy Resin, Material Hardness, Waste Management

## **1. Introduction**

Carbon fibre originated from the development of carbon fibre materials arising from the United States military's demand for new materials that were lightweight and possessed high strength for aircraft construction [1]. Currently, carbon fibre is widely used in various fields including aerospace component manufacturing, automotive parts production, and sports equipment manufacturing such as bicycle frames, tennis rackets, and wind turbine blades. This is because carbon fibre possesses properties of high tensile strength, light weight, high strength, high temperature resistance, and high chemical resistance [2].

The internal composition of carbon fibre currently used consists of two parts: carbon fibre and epoxy resin. Typically, epoxy resin is applied as a coating to create a smooth surface and glossy appearance, while also preventing carbon fibre from breaking away as loose fibres. This is because the carbon fibre itself exists in fabric form that must be cut and shaped with molds, then molded to achieve the desired form. If epoxy coating is not applied properly, carbon fibres can break into small fragments, potentially causing lung cancer if inhaled [3].

Due to these two components of carbon fibre and their durable properties, they have been widely adopted for various applications. Currently, there are no proper sustainable disposal methods, with current practices focusing on landfilling and incineration, which adversely affect the environment [4]. For instance, carbon fibre waste that is barely biodegrade continues to increase significantly, while also resulting in the wasteful loss of high-value carbon fibre materials. [5]

In this research, the researcher recognizes the importance of preliminary studies for sustainable management of carbon fibre material components in composite material form through combined physical and chemical processes.

The main objectives of this study are: to investigate methods for separating carbon fibre components containing epoxy resin in their structure, to study the properties of carbon fibre and epoxy resin after treatment processes, and to examine leachate from carbon fibre materials both before and after treatment.

## 2. Material and Method

### 2.1. Materials

All glassware materials were applied to eliminate the contamination from plasticware with samples (epoxy resin is one of the plastic types that is easily eroded with some chemical reagents). Different sizes of plastic beakers, like 200, 500, and 600 ml, were used as lab-scale reactors. The composite material was cut into small pieces, which were 1.5cm. x 7cm. size (The fibre sheets were cut into both longitudinally and transversely styles). Gloves, a cutting board, and a saw were used during the cutting process. The saw was specifically chosen with a fine and 9-inch size, each. To completely mix the samples with prepared chemical solutions, an ultrasonic bath sonicator employed high-frequency sound waves to enhance chemical reactions through mechanical agitation, perfectly. Moreover, the lab oven was used to operate at precise temperatures of 50°C and 60°C for controlled heating applications such as drying samples and removing solvents. To evaluate and measure the resistance of the samples, the Innova hardness test machine was used to determine the strength and durability of the materials (samples) and Rockwell hardness test with steel ball were apply in this study. Other than that, to determine the differences of material surfaces and microstructures, Scanning Electron Microscopy (SEM) was used for analysing the samples before and after extraction.

### 2.2. Chemicals

These high-purity chemical reagents serve essential roles in laboratory procedures and sample preparation. The study investigates various pretreatment approaches to optimize the recycling of carbon fibre materials, comparing the effectiveness and characteristics of three distinct pretreatment processes in recovering and reusing carbon fibre components. Different three main chemical reagents were applied to compare the ability of extraction which were nitric acid ( $\text{HNO}_3$ ) at 65% concentration from QREC (represented a strong oxidizing acid commonly used for sample digestion), ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) at 95% concentration from L PURE (represented functions as a versatile solvent for extraction processes), and acetic acid ( $\text{CH}_3\text{COOH}$ ) at 99.8% concentration from QREC (acted as a weak acid serving as a solvent in organic synthesis reactions).

Moreover, this study conducted the treatment of carbon fibre materials coated with epoxy resin using the chemicals like acetone to compare their effectiveness in removing epoxy resin from the carbon fibre surface. Acetone ( $\text{C}_3\text{H}_6\text{O}$ ) at 100% concentration from VWR BHD CHEMICALS functions as an extremely effective degreasing agent commonly used for cleaning glassware, removing residual organic matter from surfaces.

### 2.3. Methodology

In this study, we separated the study and preparation into 5 steps. The first step was material and chemical preparation (to cut the sample into the appropriate size and to prepare chemical reagent concentrations according to the concentrations that were used in the experiment). Figure 1 shows the longitudinal and transverse cuts of the samples

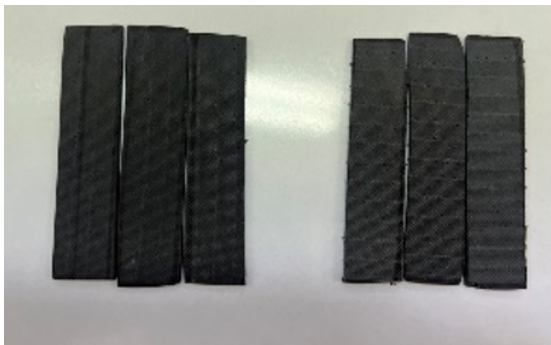


Fig. 1: the longitudinal and transverse cuts of the samples

The second step was pretreatment, which involved conditioning the carbon fibre by immersing the carbon fibre material in conditioning chemicals. The 6 carbon fibre sheets were paired by cutting direction - longitudinal and transverse cuts, with 1 pair each, resulting in a total of 3 pairs. Each pair was then immersed in 3 different types of chemicals. There were 3 different conditions, with immersion as follows: in nitric acid ( $\text{HNO}_3$  at 20% concentration), ethanol ( $\text{C}_2\text{H}_5\text{OH}$  at 95% concentration), and acetic acid ( $\text{CH}_3\text{COOH}$  at 99.8% concentration). All these conditions were carried out by immersion at room temperature for 2 hours. After 2 hours, the samples were washed with distilled water before proceeding to the next experimental step.

The third step was physical treatment process, this involved using an ultrasonic device (with demineralized water) to remove the physical components of the carbon fibre material, such as the epoxy resin surface adhered to the carbon fibre. The treatment took 30 minutes and 1 hour of sonicate to see the different effects of time after the pretreatment process. The carbon fibre sheets were dried in an oven at  $60^\circ\text{C}$  for 1 hour. After drying until the samples were completely dry, they were weighed and recorded properly. (If the carbon fibre sheet samples were not completely dry and water evaporation was incomplete, the drying time should be extended or the temperature should be increased.)

The fourth step was the chemical treatment process, which involved using chemical to decompose and separate the carbon fibre from the epoxy. The used chemical was acetone ( $\text{C}_3\text{H}_6\text{O}$  at 100% concentration). After completing the physical treatment process, the carbon fibre sheet samples were placed in a beaker containing 100 ml of pre-prepared solution (acetone) and heated (in ultrasonicate) at  $50^\circ\text{C}$  for 1 hour. After 1 hour, the samples were cooled, then the solid and liquid phases were separated. Only the solid portion was used for further experimentation.

The last step was to analyse for physical characteristics using Scanning Electron Microscopy (SEM) and hardness testing. The separated solid portion was washed with distilled water and dried in an oven at  $60^\circ\text{C}$  for 1 hour. After the drying time was complete, the samples were weighed and prepared to check the physical properties.

Other than checking physical properties of the treated materials, we also studied an improper carbon fibre management. We simulated improper management conditions by taking 4 carbon fibre sheet samples - 2 sheets cut longitudinally and 2 sheets cut transversely - and immersing them into prepared water for a period of 1 month. Before immersing the sample sheets, pH values, turbidity, and total dissolved solid (TDS) values of the water were measured both before and after the experiment to study the impacts on an environment.

### 3. Results and Discussion

According to the methodology section. The results and discussion will be divided into 3 categories which are an improper carbon fibre management, results and discussion of using different chemical reagent for the pretreatment (which are nitric acid, ethanol, and acetic acid), and results and discussion of using different chemical reagent for main chemical extraction (which is acetone). Using different time length are also discussed.

#### 3.1 Improper carbon fibre management

An important study about how carbon fibre composites degrade when exposed to water and what materials leach out, which has real environmental implications. Experimental results of a study on improper carbon fibre management, conducted by submerging carbon fibre material sample sheets in water for a period of 1 month. After the 1-month period, physical changes to the carbon fibre sheets were observed, along with monitoring the shedding of epoxy material and carbon fibres into the soaking water. The water from the soaking process was then tested for turbidity and TDS values for further environmental impact studies. The results are then separated into 3 classifies

##### 3.1.1. Results of Scanning electron microscopy: SEM

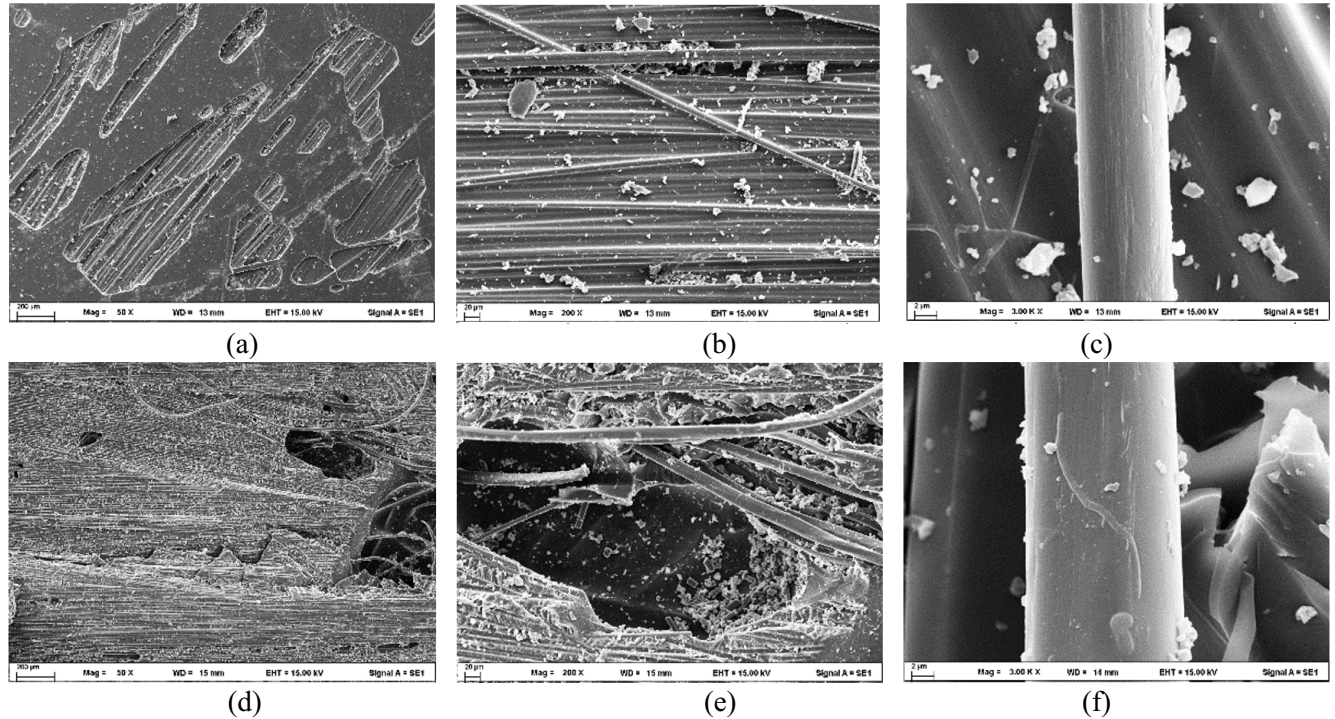


Fig.2: SEM of soaked sample in water for a month (a and d) 50x magnification, (b and e) 200x magnification, (c and f) 3000x magnification, respectively

After a month of soaking, the SEM results showed that soaked sample in water had minimal degrading effect on the carbon fibre composite - the material maintained its structural integrity even after prolonged water exposure. The Fig.2 (a, b, and c) figures represent the longitudinal cuts and the (d, e, and f) figures represent the transverse cuts, respectively. Both of the cutting styles show similar results indicating that the different of cutting style does not affect the internal structure of the fibre. Additionally, we could clearly see small pieces of epoxy resin attached with the fibre statedly.

### 3.1.2. Results of Hardness

Table 1 Hardness results

Results	Hardness (kgf/mm <sup>2</sup> )					Average (kgf/mm <sup>2</sup> )	SD (kgf/mm <sup>2</sup> )
	1	2	3	4	5		
Longitudinal cuts	69.80	52.40	52.70	49.55	50.00	54.89	8.45
Transverse cuts	64.15	52.10	48.75	39.40	43.85	49.65	9.43

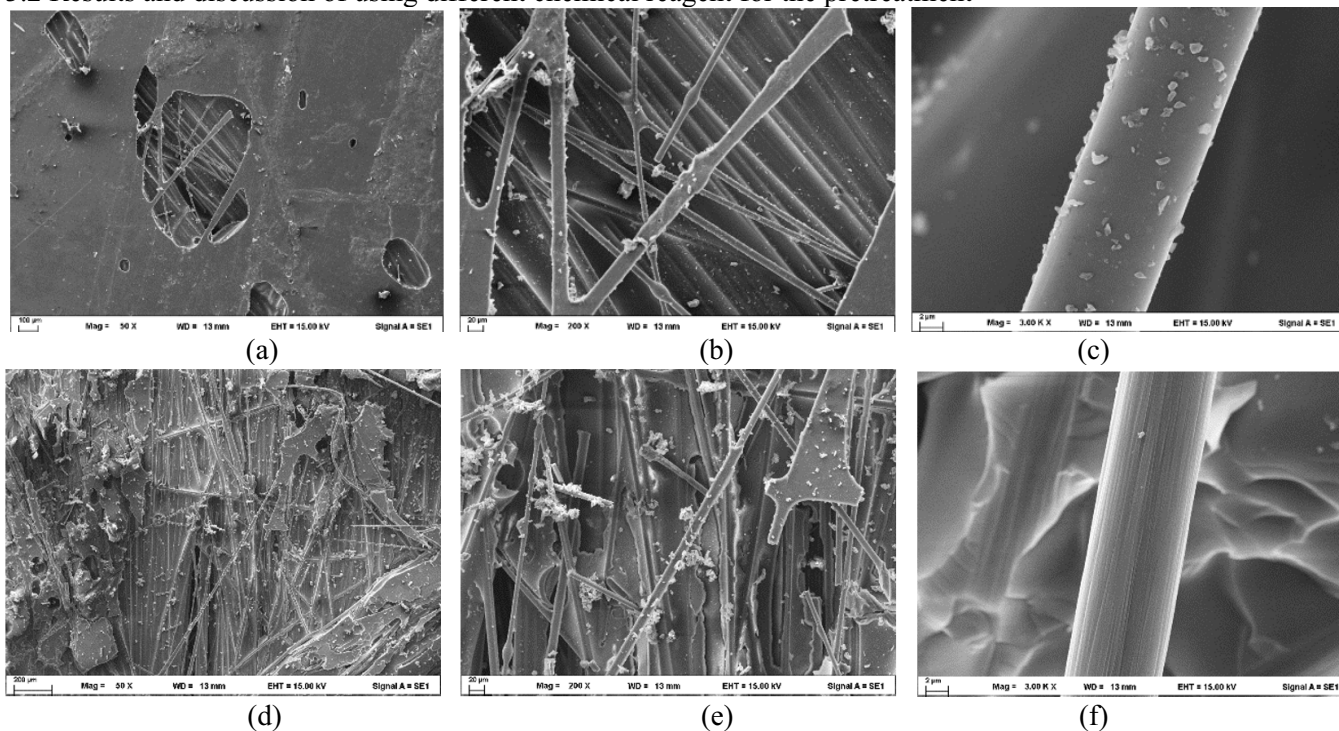
Table 1 shows the results of a month-soaked carbon fibre in water, both cutting styles did not show significant different results of hardness. However, according to the hardness result of carbon raw material (72.44 kgf/mm<sup>2</sup>), when compared to the hardness value of the carbon fibre material sheet that has not undergone any treatment process, which is at 72.44 kgf/mm<sup>2</sup>, there is a significant difference. Therefore, carbon fibre sheets that have been soaked in water for extended periods, even without the need for chemical soaking, can result in reduced hardness values of the specimens.

Table 2 Leaching condition results

Leaching conditions						
Results	pH		Turbidity (NTU)		TDS (mg/l)	
	Before	After	Before	After	Before	After
Longitudinal cuts	7.80	7.36	0	1.58	0	22.1
Transverse cuts	7.86	7.36	0	1.58	0	22.1

Table 2 shows the results of leaching condition like pH, Turbidity, and TDS. pH does not seem to be different with before soaking condition, slightly decreasing in small digits. However, turbidity and TDS, according to the results, it is particularly significant because it shows visible contamination - the black particles floating in the water represent carbon fibre microparticles that could contaminate water sources if released into the environment and also possible to accumulate in food chains over time. This visual evidence supports the study's premise that improper carbon fibre waste management could affect to the real environmental and health risks.

### 3.2 Results and discussion of using different chemical reagent for the pretreatment





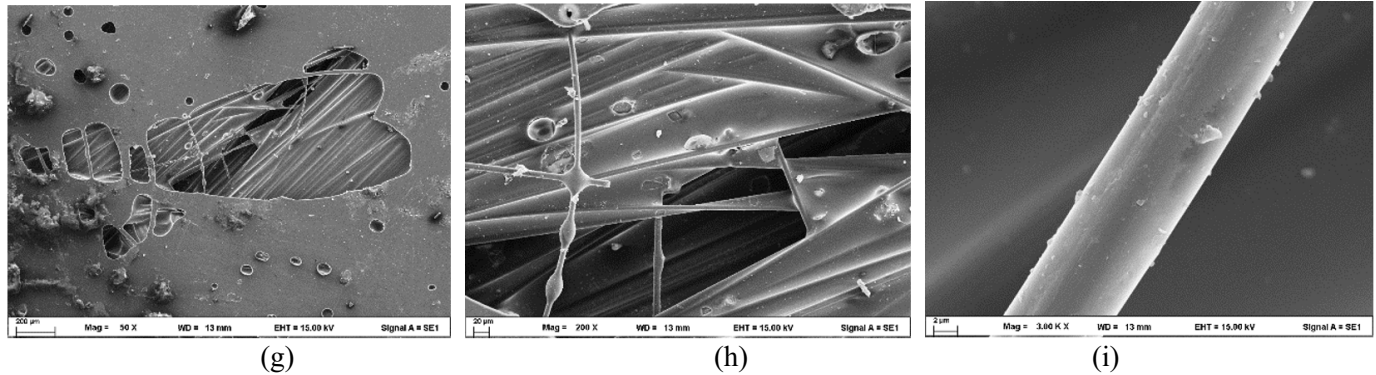


Fig. 3: SEM of pretreatment sample in nitric, acetic, and ethanol (respectively) for 2 hours (a, d and g) 50x magnification, (b, e and h) 200x magnification, (c, f and i) 3000x magnification, respectively.

Fig. 3, from the SEM analysis results of the process where carbon fibre material samples underwent pretreatment by soaking in acetic acid ( $\text{CH}_3\text{COOH}$  at 99.8% concentration) for 2 hours, it can be observed that acetic acid was able to corrode the surface of the carbon fibre material sample sheet extensively. The carbon fibre sheet was corroded much more severely when compared to nitric acid treatment. The acetic acid corroded uniformly across the entire carbon fibre sheet, rather than creating localized pitting corrosion like nitric acid. However, by soaking in ethanol ( $\text{C}_2\text{H}_5\text{OH}$  at 95% concentration) for 2 hours, it can be observed that ethanol did not cause significant corrosion like nitric acid. It only corroded the surface slightly, creating small pits scattered across the carbon fibre sheet sample. The 3000x magnification represents the close-up view of the damaged for each chemical, the result reviews that acetic acid condition can remove epoxy resin from the fibre better than strong acid and ethanol. The SEM results have a good compatibility with weight loss for each condition which is shown in Table 3

Table 3 Weight loss of each (pretreatment) chemical condition.

Condition	Before) g(	After) g(	Loss) g(	Loss) %(
Acetic (T)	3.37	3.19	0.17	5.2
Acetic (L)	3.40	3.17	0.2	.69
Ethanol (T)	3.23	3.15	0.02	0.8
Ethanol (L)	3.22	3.17	0.02	0.7
Nitric (T)	3.18	3.15	0.07	2.3
Nitric (L)	3.41	3.39	0.05	1.5

Note: T refers to Transverse cut, L refers to Longitudinal cut.

Table 4 Hardness results

Acetone							
Conditions	Hardness (kgf/mm <sup>2</sup> )					Average (kgf/mm <sup>2</sup> )	SD (kgf/mm <sup>2</sup> )
	1	2	3	4	5		
Acetic (T)	73.15	62.25	66.60	51.85	61.25	63.02	7.81
Acetic (L)	53.00	59.05	41.35	69.55	55.45	55.68	10.20
Ethanol (T)	70.55	58.90	57.20	58.95	55.30	60.18	5.99
Ethanol (L)	61.75	62.65	58.70	57.45	56.40	59.39	2.71
Nitric (T)	44.00	52.50	67.15	60.40	66.15	58.04	9.77
Nitric (L)	71.05	64.35	76.65	68.80	60.90	68.35	6.08

Table 3 indicates that acetic acid caused the most severe material degradation (up to 6.89% weight loss), while ethanol had minimal impact (less than 1% weight loss). This correlates with the SEM observations showing acetic acid's aggressive corrosion effects compared to ethanol's minimal surface pitting.

Table 4, it can be observed that acetic acid produced the lowest hardness values, with an average of both cutting patterns at 59.35 kgf/mm<sup>2</sup>. Nitric acid showed the highest hardness at 63.20 kgf/mm<sup>2</sup>, while ethanol was at 59.8 kgf/mm<sup>2</sup>. This demonstrates that the chemicals used in the pretreatment process affect the hardness of the specimen samples. Additionally, when considering different cutting patterns of the specimens, this can also influence the hardness of the treated specimens. The resulting specimen hardness may be attributed to the epoxy resin that remains attached to the specimen after undergoing the treatment process. Therefore, the reduced hardness values compared to the untreated specimen (72.44 kgf/mm<sup>2</sup>) may indicate that epoxy resin can be successfully removed through the chemical extraction process.

#### 4. Conclusion

Chemical extraction methods represent a promising approach for carbon fibre recovery from epoxy composite waste. While significant technical challenges remain, ongoing research in process optimization, environmental sustainability, and economic viability continues to advance the field. This experiment is essentially asking: *"If we throw carbon fibre materials in water, what bad stuff happens to the environment?"* The answer helps create better environmental protection policies. Demonstrating that long-term water exposure alone (without chemicals) is sufficient to degrade the material's structural integrity. According to the results of the pretreatment in this study, this comparative analysis shows different degradation mechanisms with different chosen chemicals. Acetic acid can potentially cause uniform and extensive surface corrosion, while nitric acid causes localized pitting corrosion. However, ethanol could only minimally surface pitting.

The progressive softening and brittleness with longer acid exposure demonstrate how chemical pretreatment can dramatically alter carbon fibre's structural integrity. These changes result from acid reacting with both the surface and internal structure of carbon fibres, highlighting the critical importance of controlling exposure time and acid concentration in the epoxy resin extraction process. Hence, the hardness reduction from the baseline 72.44 kgf/mm<sup>2</sup> suggests successful chemical extraction of epoxy resin, with acetic acid being the most effective solvent for this purpose.

#### Acknowledgements

This report was supported by the Faculty of Engineering, Burapha University's fund, Thailand. The material (carbon fibre material) was supported by Asst. Prof. Dr. Komkrisd Wongtimnoi, Department of Materials Engineering, Burapha University. The hardness test was performed under the supervision of Asst. Prof. Dr. Sunisa Khamsuk, Department of Industrial Engineering, Burapha University.

#### References

- [1] J.H. Taylor, G. Troisi, S.M. Soltani, 2024, "Application of chemically-activated recycled carbon fibres for aqueous-phase adsorptions - part I: Optimisation of activation process," Chem. Eng. J. Adv., vol. 18, pp. 100591-100605.
- [2] J. Jiang, G. Deng, X. Chen, X. Gao, Q. Gao, C. Xu, L. Zhou, 2017, "On the successful chemical recycling of carbon fibre/epoxy resin composites under the mild condition," CST., vol. 151, pp. 243-251.
- [3] C. Morin, A. Loppinet-Serani, F. Cansell, C. Aymonier, 2012, "Near- and supercritical solvolysis of carbon fibre reinforced polymers (CFRPs) for recycling carbon fibers as a valuable resource: State of the art," J. Supercrit. Fluids., vol. 66, pp. 232-240.
- [4] L. C. Lopez, C. Coletti, S. M. Ruano, G. Cultrone, 2024, "Use of recycled carbon fibre as an additive in the manufacture of porous bricks more durable against salt crystallization," Ceram. Int., vol. 50, pp. 9682-9696.
- [5] Y. Wei, S.A. Hadigheh, 2024, "Enhancing carbon fibre recovery through optimised thermal recycling: Kinetic analysis and operational parameter investigation," Matter. Today Sustain., vol. 25, pp. 100661-100677.