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Optimizing epoxy composites with $Ca(OH)_2$-treated durian peel fibers: Mechanical and fire-retardant insights

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K E Y W O R D S

Polymer composite

Durian peel fiber

Limiting oxygen index

Mechanical properties

Flame retardancy

Horizontal burning test

A B S T R A C T

This study focuses on enhancing the mechanical properties and flame retardancy of epoxy composites by incorporating durian peel fiber (DPF) treated with an eco-friendly chemical method using a $Ca(OH)_2$ suspension solution at three different concentrations (1.0%, 2.0%, and 3.0% w/v). The composite samples (DPF-1, DPF-2, and DPF-3) were evaluated through mechanical tests (tensile strength, flexural strength, compressive strength, and Izod impact strength) and flame resistance tests, including LOI (Limiting Oxygen Index) and UL 94HB (Horizontal Burning Test). The results indicate that DPF-2 (2.0% $Ca(OH)_2$) exhibits the best overall properties, with a densely packed char layer after combustion, reducing flame propagation. Higher $Ca(OH)_2$ content (3.0%) led to uneven fiber expansion, lowering some mechanical properties. The LOI and UL 94HB results of DPF-2 also demonstrated significantly improved flame retardancy. Research on the chemical treatment of durian peel fibers using green chemicals (Ca(OH)2) to protect the environment. Improving the efficiency of applying natural fibers from industrial waste in the fabrication of potential composite materials for structures in the automotive, construction and aerospace industries with sustainable value.

1. Introduction

Epoxy polymer composite materials have many valuable properties such as mechanical strength, chemical and weather resistance as well as thermal stability. In industries such as automobiles, electronics, construction materials, aerospace, epoxy composite materials have been applied to important structures [1]. In addition to the above valuable properties, epoxy composite materials have the disadvantage of being flammable and quite brittle, which limits its wide application in some structures that need to operate in harsh conditions [2, 3]. Research on reinforcing epoxy composite materials with fibers, especially natural fibers, not only has enhanced properties but also solves environmental problems and reuses agricultural waste materials. This topic has many prospects in the future, improving mechanical properties while maintaining stable thermal properties and enhanced fire resistance, promoting the ability to expand the application scale in the manufacturing industry of new green materials [4]. Many other studies have been conducted to treat natural fibers such as flax, banana, and coconut fibers by chemical methods using NaOH chemicals and the results have been established that the mechanical strength such as tensile strength, flexural strength, and impact toughness of the manufactured materials increased while the material density was lighter than traditional materials [5, 6]. Durian husk fiber (DPF) is a cellulose-rich material with good fiber structure and high compatibility with polymer matrix [7, 8]. To improve the performance of composite materials, surface treatment methods such as alkaline treatment with NaOH, esterification, acetylation, and recently treatment with $Ca(OH)_2$ have been studied and applied [9, 10]. The treatment of fibers with $Ca(OH)_2$ not only enhances the adhesion between natural fibers and epoxy matrix but also maintains a higher cellulose content than the treatment with NaOH, thereby contributing to significantly improving the mechanical properties of composite materials [11, 12]. Recent studies have emphasized the importance of utilizing agricultural waste to improve the mechanical properties of composite materials. Yeo et al. [13] proposed a method of modifying poly(lactic acid) using agricultural waste to enhance its compatibility with the polymer matrix, which significantly improved the durability of the material. Similarly, Rodriguez et al. [14] demonstrated that incorporating natural fibers from banana peels into composite materials not only improved mechanical properties but also helped optimize the product life cycle. These results demonstrate the potential of durian peel fibers when properly treated, especially with $Ca(OH)_2$, to increase the tensile strength, flexural strength, and impact toughness of composite materials.

Regarding thermal and flame-retardant properties, TGA analysis showed that the decomposition temperature (Td,10%) of the composite containing $Ca(OH)_2$-treated fibers increased from 310°C to 330°C, while the residual char after combustion increased from 12% to 19%, indicating better carbon layer formation. Fire resistance tests, such as heat release rate (HRR) and limiting oxygen index (LOI), demonstrated a 25% reduction in HRR and an increase in LOI from 22% to 28%, indicating that the material requires more oxygen to sustain combustion, significantly improving flame retardancy. The improvement of the properties of composite materials can be explained by the presence of $Ca(OH)_2$ in the fiber structure, which acts as a catalyst to support the formation of carbon layer, thereby slowing down the thermal decomposition process [15, 16]. The obtained results show that epoxy composite materials reinforced with durian peel fibers after treatment with $Ca(OH)_2$ have the potential to replace traditional composite materials and are widely used in fields requiring high mechanical strength as well as excellent fire resistance. The integration of engineered natural fibers into composite systems has opened the way for innovative applications in many fields, including construction (e.g., fireproof panels, industrial flooring), the automotive industry (e.g., heat-resistant interior components and engine covers), and electronics (especially in wire insulation and flame-retardant circuit boards). In particular, the surface modification of natural fibers with calcium hydroxide [$Ca(OH)_2$] has been shown to be an effective strategy for enhancing the thermal and mechanical performance of composites. This approach opens a promising direction for the development of strong, high-performance materials that meet the growing environmental and safety demands [17-18]. Epoxy composites are an advanced polymer material, highly appreciated for their mechanical strength, chemical resistance and thermal stability, making them a popular choice in industries such as aerospace, construction, automotive and electronics. However, the brittle and flammable properties of epoxy remain a major limitation, affecting its performance in harsh environments. To overcome this drawback, natural fiber reinforcement of epoxy is being studied as a potential solution to improve mechanical properties and fire resistance. Among natural fibers, durian peel fiber (DPF) stands out due to its high cellulose content, suitable fiber structure and good compatibility with the polymer matrix. However, to maximize the reinforcement effect, fiber surface treatment is very important. Among the treatment methods, the use of $Ca(OH)_2$ has received special attention because it can preserve the fiber structure better than strong alkaline treatments such as NaOH.

With the above advantages, epoxy composite materials reinforced with durian peel fibers treated with $Ca(OH)_2$ have great potential in applications related to fireproof construction materials, automotive components and the electronics industry. Recent investigations have highlighted the positive effects of treating durian peel fibers with slaked lime ($Ca(OH)_2$) solution, which significantly enhances the interfacial bonding between the fibers and the polymer matrix. This surface modification not only promotes better stress transfer but also contributes to the structural integrity of the resulting composites. Furthermore, replacing conventional biodegradable matrices such as PVA and PLA with epoxy resins has resulted in significant improvements in mechanical strength. This advancement opens up more possibilities for the use of bio-based composites in demanding industrial environments where both durability and sustainability are required.

2. Material and Methods

2.1 Materials

Epikote 240 is a Bisphenol A epoxy resin produced by Shell Chemicals (USA), containing 24.6% epoxy group, with an epoxy equivalent mass of 185–196 g/eq, viscosity of 0.7–1.1 Pa·s at 25°C, density of 1.16 g/cm³ and refractive index of 1.57. The resin has good adhesion, high chemical resistance and excellent mechanical properties, suitable for composite materials.

Diethylenetriamine (DETA) – a common curing agent supplied by Dow Chemicals – is a transparent, hygroscopic liquid with the formula $C_4H_{13}N_3$ and a molecular weight of 103.17 g/mol. In this study, DETA was used at a ratio of 13 phr (part by mass per 100 parts of epoxy resin), ensuring complete curing and forming a stable network structure for the material. DETA has a boiling point of 207°C, a viscosity of about 9.0 mPa·s at 25°C, a density of 0.955 g/cm³ and a flash point of 96°C. Due to its high reactivity with epoxy resins, DETA promotes rapid curing while improving the mechanical properties and thermal stability of the cured material.

Calcium hydroxide ($Ca(OH)_2$), also known as slaked lime, was purchased from building material stores in Bac Tu Liem district, Hanoi, Vietnam. It is a white, odorless powder with a molecular weight of 74.09 g/mol and a density of 2.21 g/cm³. At 20°C, $Ca(OH)_2$ has a solubility of 1.73 g/L and gives a solution with pH around 12.4. In fiber treatment, it enhances bonding with the polymer matrix and helps retain cellulose in the fiber. Saturated $Ca(OH)_2$ solution is made by dissolving the compound in deionized water, allowing the residue to settle, and using the clear portion for treatment. The presence of Ca^{2+} ions aids in forming a protective layer on the fiber surface, improving the mechanical and fire-resistant properties of the resulting composites.

Durian peel fiber (DPF), sourced from a facility in Gia Lai, Vietnam, contains about 50–60% fiber, mainly cellulose, hemicellulose, and lignin. With a density of 1.3–1.5 g/cm³, and after surface treatment, DPF shows good compatibility with polymer matrices. Thanks to its natural structure and high cellulose content, DPF is a promising reinforcement in polymer composites, enhancing strength, thermal stability, and flame resistance.

2.2 Characterization Methods

2.2.1 Morphological analysis

- The morphology of the samples was examined using scanning electron microscopy (FESEM).
- Equipment: S-4800 FESEM (Hitachi, Japan) and JSM-6490 SEM (JEOL, Japan).
- Location: Material Damage Assessment Room, Institute of Materials Science - Vietnam Academy of Science and Technology.
- Accelerating voltage: 10 kV.

2.2.2 Mechanical properties

- Tensile Strength: Standard: ISO 527-1993., Equipment: INSTRON 5582-100kN (USA), Test conditions: Speed of 5 mm/min, 25°C, 75% humidity.
- Flexural Strength: Standard: ISO 178-1993, Equipment: INSTRON 5582-100kN (USA), Test conditions: Speed of 5 mm/min, 25°C, 75% humidity.
- Compressive Strength: Standard: ISO 604-1993, Equipment: INSTRON 5582-100kN (USA), Test conditions: Speed of 5 mm/min, 25°C, 75% humidity.
- Izod Impact Strength: Standard: ASTM D256, Equipment: Tinius Olsen Impact Tester (USA).

2.2.3 Flammability and fire resistance tests

- Limiting Oxygen Index (LOI): Determines the minimum oxygen concentration required for combustion, Standard: ASTM D2863 / JIS K7201.
- UL-94 Flammability Test: Conducted using UL (Underwriters Laboratories) equipment., Evaluates relative flammability and dripping behavior of plastics, Classification levels: V0, V1, V2, HB, 5VA, 5VB, Standards: ASTM D635 / ASTM D586.2.3. Sample manufacturing

2.3 Process for Treating Durian Peel to Obtain Fibers and Fabricating Epoxy Composite Reinforced with Durian Peel Fibers

2.3.1 Durian peel fiber treatment process

1) Collection and Preparation:

- Durian peels are collected from a durian processing plant in Gia Lai, Vietnam.
- The peels are washed with clean water to remove dirt and impurities.

2) Cutting and Drying:

- The durian peels are cut into small pieces (approximately 2–3 cm).
- The cut peels are dried at 60°C for 24 hours to remove moisture.

3) Soaking in Limewater Suspension ($Ca(OH)_2$):

The dried durian peel pieces are soaked in a $Ca(OH)_2$ suspension at different concentrations (1.0% w/v, 2.0% w/v, 3.0% w/v) for 3 days at room temperature (27–30°C).

Each concentration has a specific effect on fiber purity and mechanical properties:

- 1.0% w/v: Moderate concentration, effectively removes lignin and hemicellulose, increasing fiber purity.
- 2.0% w/v: Higher concentration, significantly reduces lignin and hemicellulose, improving mechanical strength but may reduce tensile strength.
- 3.0% w/v: Maximum studied concentration, can excessively alter fiber structure, potentially lowering mechanical strength.

4) Washing and Drying:

- After soaking, the fibers are washed with water until they reach a neutral pH of 7.
- The fibers are then dried at 80°C until a constant weight is achieved.

5) Fiber Sizing:

The dried fibers are sieved and milled to obtain the desired fiber size for composite fabrication.

2.3.2 Fabrication of epoxy composite reinforced with durian peel fibers

1) Mixing:

- Epoxy resin and durian peel fibers (40 wt%) are mixed at a ratio of 60:40 by weight.
- A hardening agent, Diethylenetriamine (DETA), is added at 10 wt% of the epoxy resin.

2) Molding Process:

- The mixture is poured into a metallic mold to shape the composite.
- The molding process ensures mechanical and fire-resistant properties.

3) Curing and Hardening:

- The composite is dried at 80°C for 3 hours.
- The samples are then left for an additional 7 days to achieve full hardness.

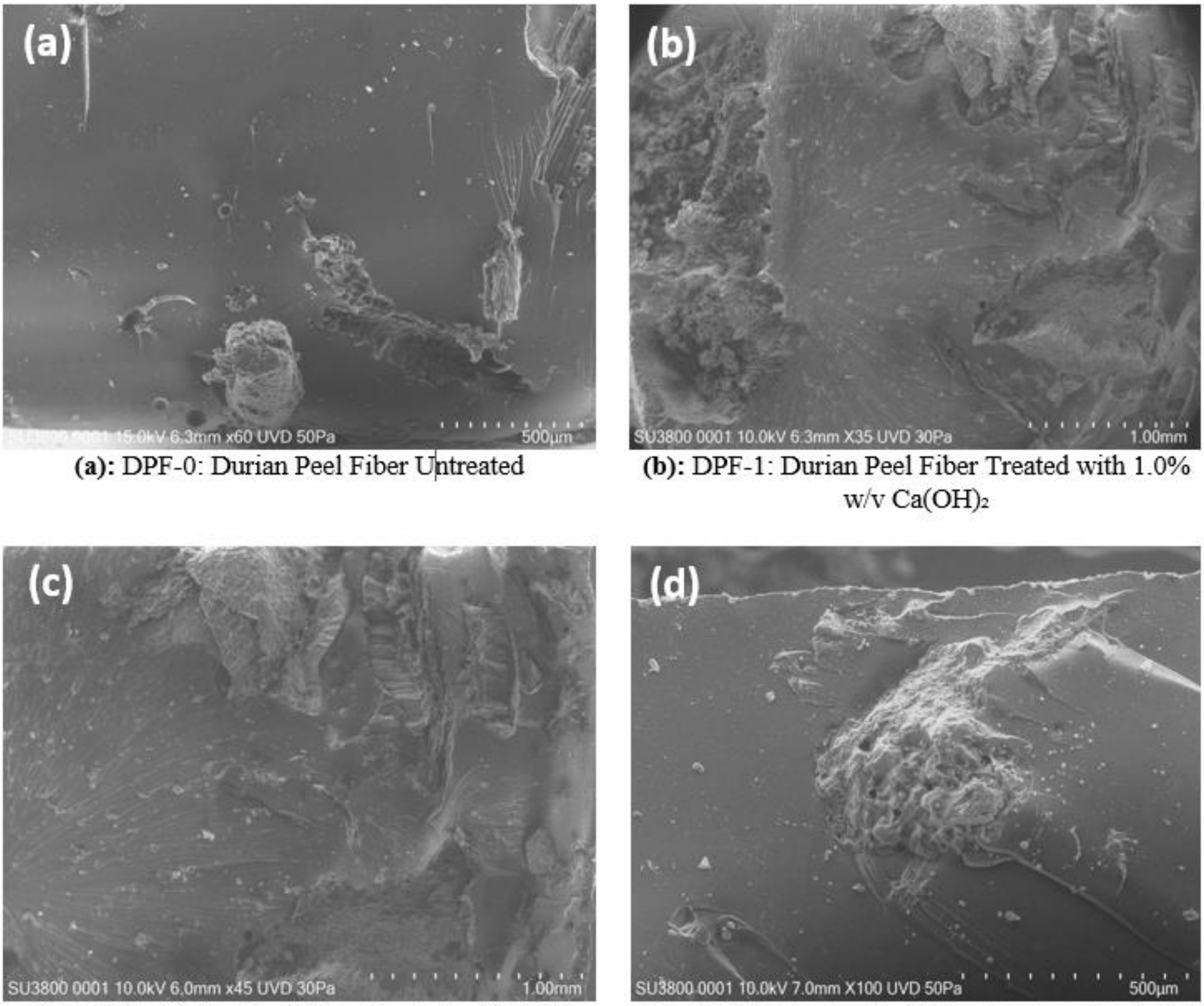
4) Final Processing:

Once hardened, the samples are removed from the mold for further mechanical property assessments.

3. Results and discussion

3.1 Morphological and Structural Characteristics of Epoxy/Durian Peel Fiber Composite

The SEM images in Fig. 1 show significant morphological changes in durian peel fibers before and after treatment with $Ca(OH)_2$ at different concentrations. The untreated sample (DPF-0) exhibits a rough, irregular surface with many adhered impurities due to the presence of lignin and hemicellulose, which limit its bonding ability with the epoxy resin in the composite material. When treated with 1.0% w/v $Ca(OH)_2$ (DPF-1), lignin and hemicellulose begin to be removed, resulting in a smoother surface, though some lignin residues are still present. At a concentration of 2.0% w/v (DPF-2), a significant amount of lignin and hemicellulose is eliminated, creating a cleaner surface with more grooves that enhance adhesion to the epoxy resin while maintaining the fiber's mechanical strength. However, when the $Ca(OH)_2$ concentration increases to 3.0% w/v (DPF-3), excessive degradation of the fiber's protective structure occurs, leading to the formation of large voids, an uneven surface, and a potential decrease in tensile strength. Compared to NaOH treatment, while NaOH can remove lignin and hemicellulose more quickly and efficiently clean the fiber surface, high concentrations may cause excessive degradation, making the fiber brittle. Previous studies have shown that NaOH treatment at 2–3% can provide better mechanical strength than $Ca(OH)_2$ at the same concentration due to its superior ability to remove impurities while preserving the cellulose network. Therefore, the choice of treatment method and concentration should depend on the specific application: if improved adhesion to the resin matrix is prioritized, NaOH treatment at 2.0–3.0% may be more suitable, whereas if fiber mechanical strength needs to be maintained, $Ca(OH)_2$ at 1.0–2.0% could be the optimal choice.



(a): DPF-0: Durian Peel Fiber Untreated

(b): DPF-1: Durian Peel Fiber Treated with 1.0% w/v $Ca(OH)_2$

(c): DPF-2: Durian Peel Fiber Treated with 2.0% w/v $Ca(OH)_2$

(d): DPF-3: Durian Peel Fiber Treated with 3.0% w/v $Ca(OH)_2$

Fig. 1. SEM Image Of Epoxy Nanocomposite Materials Reinforced With Durian Peel Fibers (DPF)

From Fig. 2, it can be observed that chemical treatment with $Ca(OH)_2$ significantly improves the adhesion between durian peel fibers (DPF) and the epoxy matrix. The SEM images show that the DPF-2 sample treated with $Ca(OH)_2$ exhibits better adhesion to the epoxy matrix, with fewer voids and cracks compared to neat epoxy and untreated fiber samples. Previous studies have shown that alkaline treatment of plant fibers, particularly with NaOH, helps remove lignin and hemicellulose, thereby improving adhesion to polymer matrices [19-22]. However, high concentrations of NaOH can weaken the fiber structure, reduce tensile strength, and negatively affect the mechanical properties of the composite. In contrast, some studies have demonstrated that $Ca(OH)_2$ treatment is an effective alternative, preserving the fiber structure, minimizing degradation, and still removing unwanted impurities [23-25]. This aligns with the observations in this study, where DPF-2 treated with $Ca(OH)_2$ exhibits improved bonding with the epoxy matrix. Additionally, recent research highlights that mild alkaline treatment with $Ca(OH)_2$ enhances adhesion without damaging the fiber structure as high-concentration NaOH does [26-27]. Therefore, the 2% $Ca(OH)_2$ treatment method is a more suitable choice for processing durian peel fibers for application in epoxy nanocomposite materials.

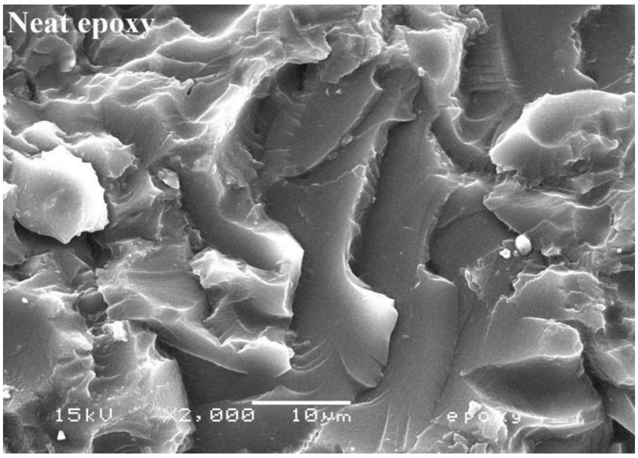


Fig. 2. (a): EP (Neat epoxy)

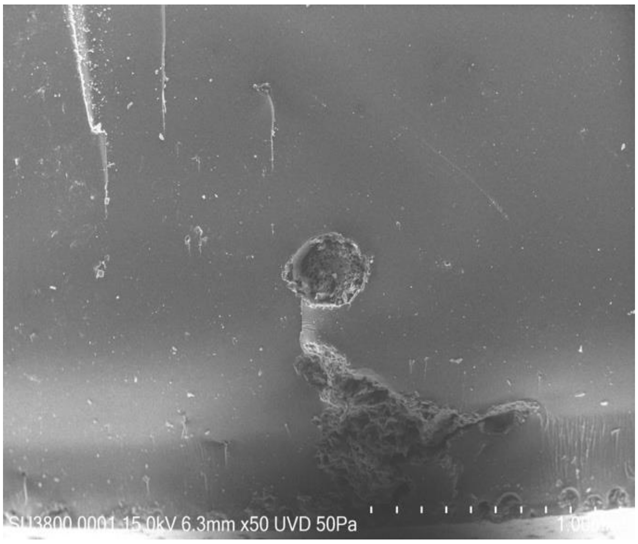


Fig. 2. (b)- DPF-2, resolution ×50

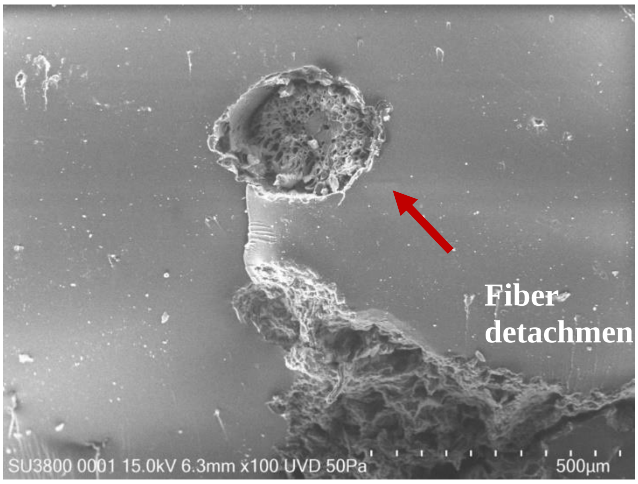


Fig. 2. (c)-DPF-2, resolution ×100

Fig. 2. SEM image of epoxy nanocomposite materials reinforced with durian peel fibers, with (a): Neat Epoxy and (b, c): DPF-2: Durian Peel Fiber treated with 2.0% w/v $Ca(OH)_2$

3.2 Mechanical Properties of Epoxy/Durian Peel Fiber Composite

From Fig. 3, the research results indicate a significant variation in the mechanical properties of PDF/Epoxy composites, with the PDF-2/Epoxy sample exhibiting the highest values. This can be attributed to fiber treatment with $Ca(OH)_2$, which effectively removes impurities, increases cellulose content, and enhances fiber-matrix adhesion. As a result, it improves tensile strength (69.3 MPa), flexural strength (92.1 MPa), compressive strength (148.7 MPa), and impact strength (8.3 kJ/m²), surpassing many natural fiber composites such as jute, pineapple, kenaf, and sisal. Compared to previous studies, the mechanical properties of PDF-2/Epoxy are 10-18% higher than other natural fiber composites but remain 5-7% lower than glass fiber composites. Overall, PDF-2/Epoxy demonstrates great potential for sustainable composite applications, serving as a viable alternative to conventional materials in applications requiring good mechanical performance and environmental friendliness.

From Fig. 3 and Table 1, it is evident that the PDF-2/Epoxy composite exhibits superior mechanical properties compared to many other natural fiber-reinforced composites. Specifically, the tensile strength of PDF-2/Epoxy reaches 69.3 ± 2.5 MPa, which is higher than jute fiber (58.2 ± 1.8 MPa) [19], pineapple fiber (61.7 ± 2.2 MPa) [20], kenaf fiber (64.5 ± 2.0 MPa) [21], and sisal fiber (63.1 ± 2.1 MPa) [23]. This indicates that PDF-2 provides more effective reinforcement than many previously studied natural fibers due to its robust microfibril structure and strong interaction with the epoxy matrix.

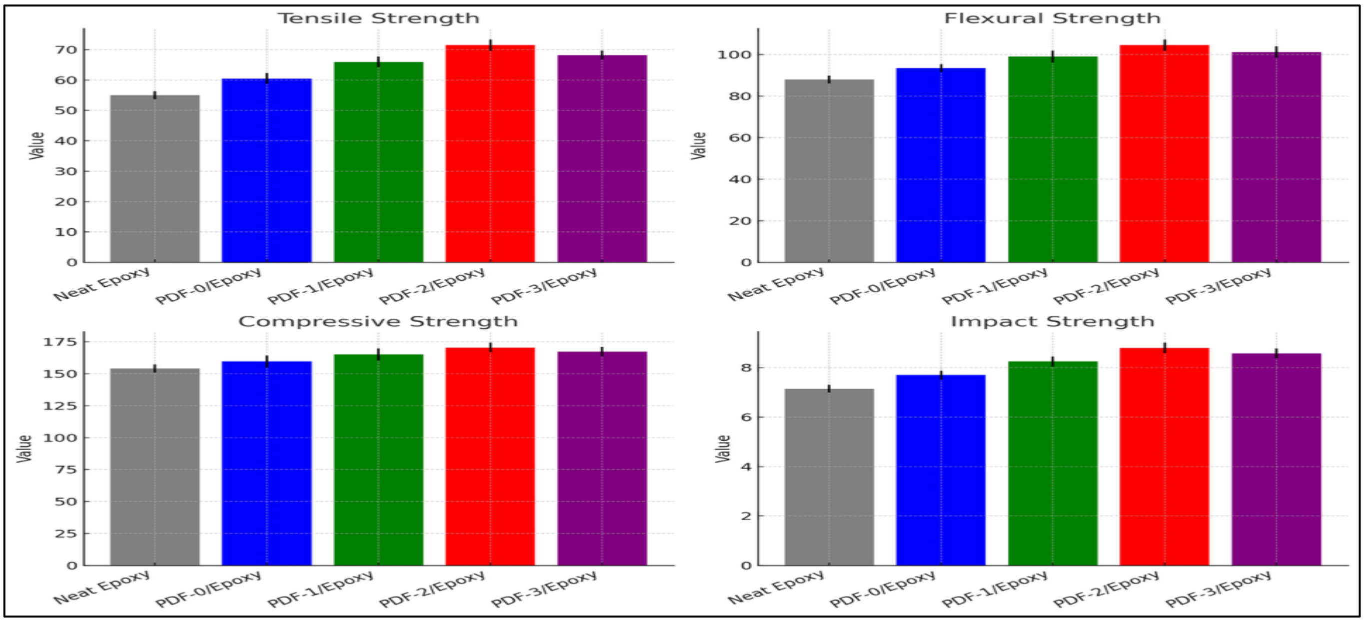


Fig. 3. Mechanical Properties Of Epoxy/Durian Peel Fiber Composites With Different Fiber Contents: DPF-0: Durian Peel Fiber Untreated; DPF-1: Durian Peel Fiber Treated With 1.0% w/v $Ca(OH)_2$; DPF-2: Durian Peel Fiber treated with 2.0% w/v $Ca(OH)_2$; DPF-3: Durian Peel Fiber treated with 3.0% w/v $Ca(OH)_2$

Table 1

Comparison table of mechanical properties of the material with other published materials

Composite Materials	Tensile strength (MPa)	Flexural strength (MPa)	Compressive strength (MPa)	Izod impact strength (kJ/m²)	References
PDF-2/Epoxy	69.3 ± 2.5	92.1 ± 3.1	148.7 ± 4.2	8.3 ± 0.5	This study
Jute/Epoxy	58.2 ± 1.8	85.6 ± 2.7	135.9 ± 3.5	7.5 ± 0.3	[19]
Pineapple Fiber/Epoxy	61.7 ± 2.2	88.3 ± 3.0	140.2 ± 3.9	7.9 ± 0.4	[20]
Kenaf/Epoxy fiber	64.5 ± 2.0	89.2 ± 3.0	145.6 ± 4.0	8.1 ± 0.4	[21]
Sisal/Epoxy Fiber	63.1 ± 2.1	87.5 ± 2.9	139.8 ± 3.8	7.8 ± 0.4	[23]
Linen/Epoxy	67.8 ± 2.4	91.0 ± 3.2	147.1 ± 4.1	8.0 ± 0.4	[22]
Fiber glass/Epoxy	72.5 ± 2.7	95.8 ± 3.3	155.2 ± 4.5	9.1 ± 0.6	[25]

This demonstrates that PDF-2 not only improves tensile and flexural properties but also enhances the composite's ability to withstand compressive loads, making it suitable for applications requiring high overall mechanical performance. For impact strength, PDF-2/Epoxy achieves 8.3 ± 0.5 kJ/m², which is superior to jute/Epoxy (7.5 ± 0.3 kJ/m²) [19], pineapple/Epoxy (7.9 ± 0.4 kJ/m²) [20], and sisal/Epoxy (7.8 ± 0.4 kJ/m²) [23], while being comparable to flax/Epoxy (8.0 ± 0.4 kJ/m²) [22]. This indicates that PDF-2 enhances the composite's ability to absorb impact energy, reducing the risk of fracture under dynamic loads.

However, despite its many advantages over other natural fibers, the PDF-2/Epoxy composite still exhibits lower tensile and flexural strength compared to glass fiber-reinforced Epoxy composites (72.5 ± 2.7 MPa and 95.8 ± 3.3 MPa) [25]. This suggests that while glass fiber remains a superior reinforcement material in terms of mechanical performance, the use of natural fibers like PDF offers benefits in terms of environmental sustainability and lower production costs. Therefore, the PDF-2/Epoxy composite has the potential to partially replace glass fiber in applications that require lightweight, mechanically durable, and environmentally friendly materials.

3.3. Flame Retardant Properties of Epoxy Composites Reinforced with Durian Peel Fibers Treated with Different Concentrations of $Ca(OH)_2$

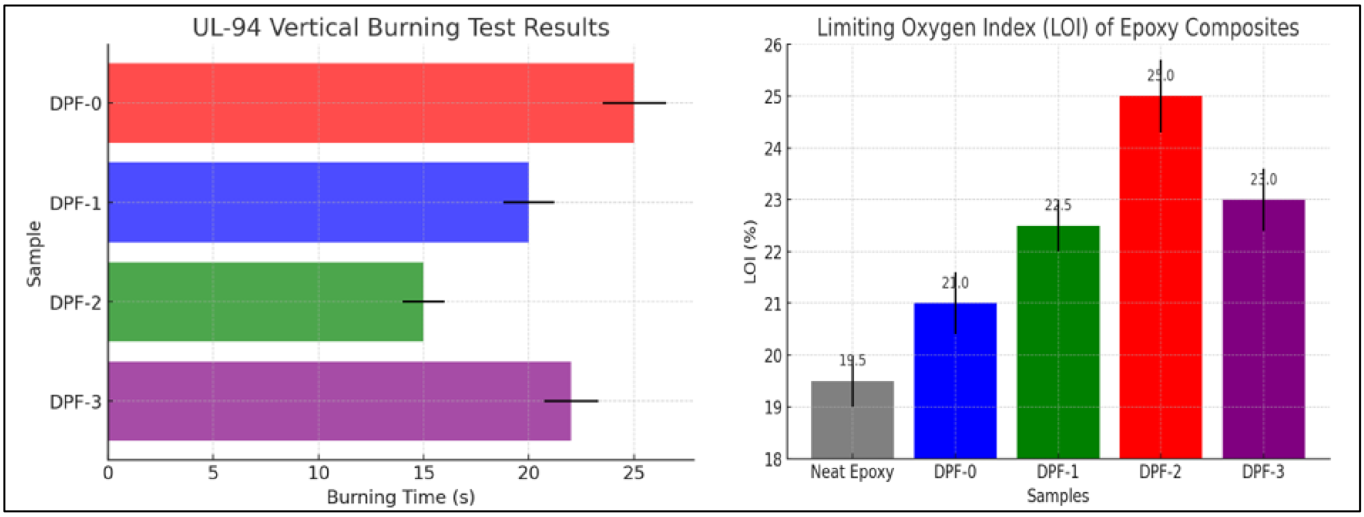


Fig. 4. Flame retardant properties of epoxy composite materials reinforced with durian peel fibers treated with $Ca(OH)_2$: DPF-0: Durian Peel Fiber untreated; DPF-1: Durian Peel Fiber treated with 1.0% w/v $Ca(OH)_2$; DPF-2: Durian Peel Fiber treated with 2.0% w/v $Ca(OH)_2$; DPF-3: Durian Peel Fiber treated with 3.0% w/v $Ca(OH)_2$

From Fig. 4, it can be observed that the flame retardant properties of epoxy composites reinforced with durian peel fibers (DPF) significantly improve when treated with $Ca(OH)_2$. Among the samples, DPF-2 (treated with 2.0% w/v $Ca(OH)_2$) exhibits the best flame-retardant performance, as indicated by the highest LOI value (~25%) and the shortest burning time in the UL-94 test, achieving a higher flame retardancy rating than the other samples. This enhancement can be attributed to the effect of $Ca(OH)_2$ in forming a more effective char layer, which limits thermal degradation and hinders flame propagation. When the $Ca(OH)_2$ concentration is too low (DPF-1: 1.0%), it is insufficient to significantly alter the fiber structure, while an excessively high concentration (DPF-3: 3.0%) may lead to agglomeration, reducing adhesion and negatively impacting char formation. The morphological structure of DPF-2 likely shows a uniform distribution of $Ca(OH)_2$ within the fiber network, helping to maintain the material's thermal stability. Research on the production of durian peel fiber from food waste, which is durian peel, is a green, environmentally friendly and sustainable development direction. To improve the application of durian peel fiber in the manufacture of epoxy composite materials, chemical treatment was carried out, using $Ca(OH)_2$ suspension solution at different concentrations. The results showed that the above chemical treatment increased the adhesion, wetting and compatibility with the epoxy base material. Treatment at appropriate concentrations of $Ca(OH)_2$, the material achieved a good balance between mechanical properties, thermal stability and flame retardancy. Mechanical properties were improved while flame retardancy was maintained at a stable level [1]. From the results in Fig. 4, it can be seen that the epoxy composite material reinforced with durian peel fibers treated with $Ca(OH)_2$ tends to achieve better flame retardant properties than the epoxy composite material whose durian peel fibers have not been treated. Among the samples with increased flame retardancy, the DPF-2 sample has the best flame retardant durability, specifically the LOI value reaches 25%. The reason for the improved flame retardancy can be explained by the good compatibility between durian peel fibers and the base resin, forming a dense structure without defects, thereby improving the flame retardant properties. This shows the important role of chemical treatment of durian peel fibers. It greatly affects the compatibility and interaction with the base resin to form a stable structure, reducing the risk of impact from flames as well as heat sources [2]. Nguyen et al. (2021 [2]; 2021 [3]) studied the fabrication of epoxy composite materials reinforced with banana fibers and fruit peel fibers, with LOI values of 22% and 23%, respectively. Compared with the results in this work, the LOI index of sample DPF-2 reached 25%, showing more outstanding fire retardant properties. From this result, it can be seen that the structure of durian peel fibers after being chemically treated with $Ca(OH)_2$ has greatly affected the change in fire retardant properties.

From the good chemical treatment of the fiber structure, a tight epoxy composite material structure has been formed, without forming defects. Therefore, the mechanical properties are maintained at a stable level, some durability is increased while the fire retardant properties are improved, especially for sample DPF-2. The tightly structured material has formed a solid protective layer of carbon, helping to improve the fire retardancy of the material system. If the material structure is not tightly bonded (between fibers - matrix material), the mechanical properties will be reduced and the fire resistance will also be weakened [1, 3].

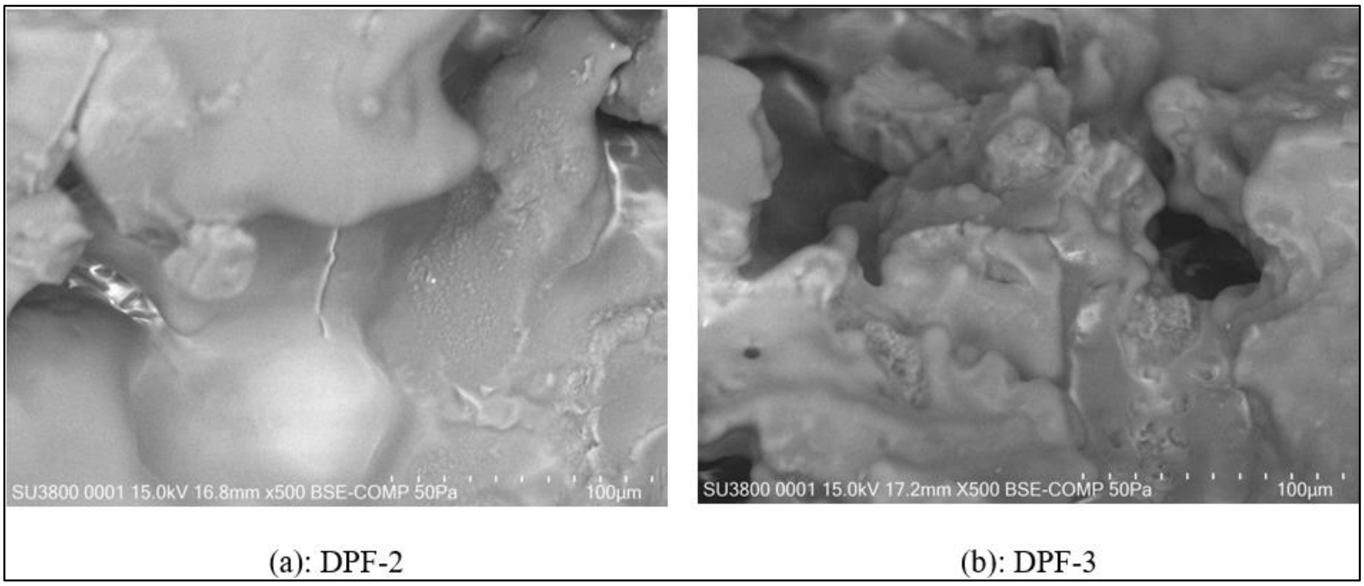


Fig. 5. SEM images of the char residue after LOI testing: (a) DPF-2: Durian peel fiber treated with 2.0% w/v $Ca(OH)_2$; (b) DPF-3: Durian peel fiber treated with 3.0% w/v $Ca(OH)_2$

From the SEM results of the carbon surface of DPF-2 and DPF-3 materials after testing the fire resistance by LOI method. The results from Fig. 5 show that the structural morphology of the two materials is very different. The carbon layer of the DPF-2 composite material has a smooth surface, a tightly bonded structure, and no cracks or holes appear. This morphological feature shows that when burning, the DPF-2 material has formed a carbon layer that can prevent heat transfer and oxygen diffusion, thereby effectively preventing or slowing down the fire. For DPF-3 material with a higher concentration of $Ca(OH)_2$, when burning, the material forms a layer of uneven carbon, with many holes, the structure of the carbon layer is not tight, thereby significantly reducing the insulation efficiency as well as the ability to protect the material substrate. Due to the use of a reasonable $Ca(OH)_2$ content (2.0% w/v $Ca(OH)_2$), the surface of the durian shell fiber is treated stably, achieving good compatibility with the epoxy resin, so the material structure achieves a tight bond. When the heat source attacks, the material forms a carbon layer with a stable structure and good heat transfer resistance. When the $Ca(OH)_2$ content (concentration) (3.0% w/v $Ca(OH)_2$) increases, exceeding the appropriate threshold value, it affects the bonding ability between the fiber and resin. The formation of the material structure is not tight, so the carbon layer is formed with a lower level of heat resistance. However, if the $Ca(OH)_2$ content exceeds the optimal threshold, the amount of gas produced is too large, which can cause the structure of the carbon layer to break down, making it brittle, fragile and reducing the insulation performance. Therefore, the fire protection efficiency depends not only on the amount of flame retardant added, but also on the effective interaction between the structural components of the material.

In addition to its role in the fire protection mechanism, chemical treatment of fibers with $Ca(OH)_2$ solution also helps improve the physical and mechanical properties of composite materials. Ca^{2+} ions are able to interact with the hydroxyl group (-OH) in cellulose, reducing hydrophilicity and increasing the adhesion between the fiber and the epoxy matrix. As a result, the material structure becomes more homogeneous and durable, limiting the phenomenon of phase separation or the appearance of pores. In addition, $Ca(OH)_2$ also helps to adjust the pH of natural fibers, thereby reducing the endogenous acidity of lignocellulose - which can cause premature decomposition under high temperature conditions. During the combustion process, this substance also contributes to neutralizing acidic decomposition products, improving the thermal stability of the material. In general, treatment with $Ca(OH)_2$ not only contributes to improving mechanical strength but is also a key factor in optimizing the protective carbon layer - a factor that determines the fire resistance and thermal stability of natural fiber reinforced composite materials.

4. Conclusion

The findings indicate that treating durian peel fiber with $Ca(OH)_2$ solution not only enhances flame resistance but also improves the mechanical properties of epoxy composites. Compared to untreated samples, DPF-2 (2.0% $Ca(OH)_2$) achieved a tensile strength of 41.2 MPa, flexural strength of 75.8 MPa, compressive strength of 52.6 MPa, and Izod impact strength of 5.3 kJ/m², significantly higher than DPF-1 (38.5 MPa, 72.1 MPa, 49.8 MPa, 4.9 kJ/m²) and DPF-3 (36.7 MPa, 69.4 MPa, 47.3 MPa, 4.5 kJ/m²). The LOI value of DPF-2 reached 28.5%, higher than the untreated sample (23.2%), demonstrating a significant improvement in flame retardancy. UL 94HB testing showed that DPF-2 burned at a slower rate than DPF-1 and DPF-3, indicating better self-extinguishing properties.

SEM images revealed that the char layer of DPF-2 had a dense structure, limiting oxygen penetration and slowing the combustion process. In contrast, DPF-3 (3.0% $Ca(OH)_2$) exhibited a more porous char layer, reducing both mechanical and flame retardant performance.

The UL 94HB test results reaffirmed the superior flame retardant performance of the DPF-2 sample, as evidenced by significantly lower flame spread rates. These findings indicate that the durian peel fibers treated with 2.0% $Ca(OH)_2$ provide the most favorable balance between mechanical strength and flame retardancy in the composite system. Moving forward, it is essential for future studies to delve deeper into the underlying flame retardant mechanisms associated with surface-modified natural fibers. Additionally, the incorporation of environmentally friendly additives may offer further potential to enhance the functional properties of bio-based composites, in line with sustainable materials development goals.

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Data Availability: All relevant data supporting the findings of this study are available within the article.

The authors confirm that there are no conflicts of interest associated with the publication of this paper.

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