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# ENHANCING MECHANICAL PERFORMANCE OF EPOXY-BASED HYBRID COMPOSITES USING ALKALI-TREATED PINEAPPLE LEAF FIBER AND MWCNT

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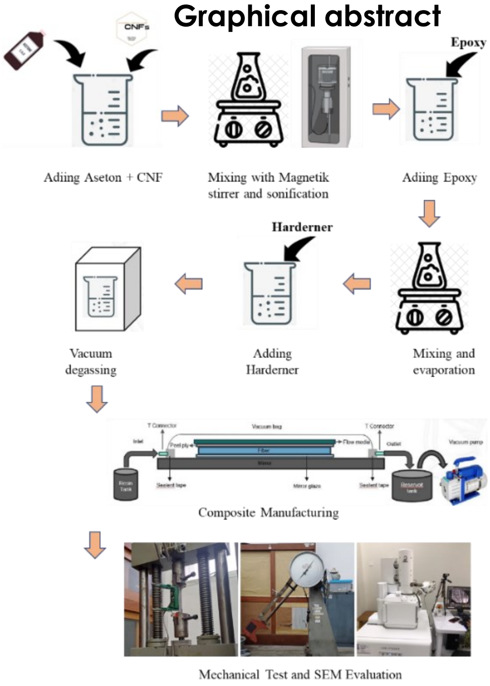
## Graphical abstract



## Abstract

The increasing interest in environmentally friendly materials in industries such as automotive, civil construction, and packaging has encouraged a lot of research related to environmentally friendly composites using natural fibers as reinforcement. This study is conducted to evaluate the performance of epoxy resin as a matrix with pineapple leaf fiber and multi wall carbon nanotube powder (MWCNT) as the reinforcements in hybrid composites. Pineapple leaf fiber is alkali treated by soaking the fiber in 5%wt alkali solution. The fibers are woven into unidirectional fiber sheets. A proper manufacturing process obtains quality unidirectional composites, which is carried out by configuring volume fraction ratio and matrix 30% and 70%, respectively. Hybrid composites, which consist of five fiber sheets with a configuration of pineapple leaf fibers, mixed with epoxy resin matrix and supplemented with MWCNT of 0.1, 0.3; 0.5 and 1 wt%, are composed by vacuum infusion method. The results show that alkali treatment improves the mechanical properties of epoxy/pineapple leaf fiber hybrid composites. The most optimal MWCNT content is obtained at 0.5%wt, resulted in a rise of tensile strength by 26.70%, bending strength by 17.82%, and impact toughness by 5.90%. Fiber alkali treatment and MWCNT addition to the hybrid composite increase water absorption. The existence of a good interface bond between pineapple leaf fibers and the matrix is indicated by the visible epoxy matrix and MWCNT particles which stick to the pineapple leaf fibers surface, proved by the FE-SEM observations.

*Keywords*: Hybrid Composite, Natural Fiber, MWCNT, alkali treatment, mechanical properties

## 1.0 INTRODUCTION

Research on composite materials has developed in recent decades to find various types of composite materials that have excellent mechanical, physical, and morphological properties for various applications. Researchers also strive in the development of biodegradable composites by applying nature materials. Natural fibers derived from plants show great potential for use in the food, automotive, and packaging industries. The potential is due to their excellent characteristics such as low density, high specific stiffness, good mechanical properties, biodegradability characteristic, environmental friendliness, toxicological harmlessness, good thermal and acoustic insulation sustainability [1-2].

Natural fibers have both positive and negative properties, including the ability of their structure to absorb moisture from their surrounding environment, resulting in the formation of hydrophilic and polar fibers in nature. Composite owns lower mechanical properties when natural fibers and matrix are combined. This happens when hydrophilic fibers are combined with hydrophobic polymers (thermosets and thermoplastics) [3]. This obstacle can be overcome through modifications such as chemical treatment with NaOH solution or (3- Glycidyloxypropyl) trimethoxysilane solution with various concentrations to improve the interfacial adhesion between the natural fiber and the composite matrix, in addition to improving the mechanical, physical, and thermal properties of the fiber [4]. Other modifications during acetylation can alter the fiber surface and increase its hydrophobicity [5].

The most common method to decrease surface tension and improve adhesion of the fiber-matrix interface is alkali treatment. The purpose of this treatment is to remove hemicellulose, to break the fiber in fibrils, and to produce dense cellulose chains as a result of internal strain relief. This treatment improves the mechanical properties of the fiber [6]. Alkali treatment also removes certain parts of hemicellulose, lignin, pectin, wax and oil (weak boundary layer) from the cellulose surface [7]. Alkali treatment can also remove the impurity material covering the fiber surface of the fiber bundle, known as covering material. This results in an effective surface area of the fiber for matrix adhesion and improved fiber dispersion in the composite. The surface roughness of the treated fiber causes better bonding of the matrix and fiber, which can further improve the bonding of the matrix and fiber interface by increasing the bonding area where mechanical interlocking occurs [8].

The mechanical properties of natural fiber composites should be improved by nanoparticle reinforcement addition. The addition is vital because even though natural fibers have been chemically treated, natural fiber composites are still relatively weak. A nanoparticle reinforcement which is proven to increase the strength of the polymer matrix is carbon nanotube (CNT). CNT is manufactured from carbon and has a nanometer structure that resembles a tube. It possesses high aspect ratio, low density, high water resistance, high thermal and electrical conductivity, and high elastic modulus [9]. According to [10], the application of carbon nanotubes (CNT) as nano reinforcement in epoxy/bamboo composites improved mechanical properties, including tensile strength by 6.67%, bending strength by 5.8%, and impact toughness by 84.5%. This improvement in mechanical properties is indicated by an increase in interfacial bonding due to the addition of CNT.

In this study, the vacuum resin infusion method is used for the fabrication of epoxy hybrid composites reinforced with woven pineapple leaf fibers and MWCNT. The effects of chemical treatment and MWCNT addition to the hybrid composites on mechanical properties and morphology are investigated.

## 2.0 METHODOLOGY

### 2.1 Materials

Pineapple (ananas comosus), is well known due to its various function in each part of the fruit as shown in Figure 1(a). In this study, pineapple leaf will be processed to form a yarn, In this study, pineapple leaf will be processed to form a yarn, which is displayed in Figure 1(b). The reason of using this leaves is it has stronger mechanical properties, compared to other fibers [11]. The resin used in this research is epoxy resin with material basis bisphenol A – Epichrlorohydrin, with a density of 1.16 g/cm3, while the hardener used is Cycloaliphatic Amine type which has a density specification of 1.01 g/cm3. MWCNT with a crystallinity index > 95% is obtained from Ossila, UK. Based on the material specification from the manufacturer the calculated aspect ratio (length to diameter) is 2500, so that considered as high aspect ratio material.

**Figure 1** (a) Pineapple plant, (b) pineapple leaf fibers

### 2.2 Fiber Treatment

Pineapple leaf fibers obtained are spun into yarn. After the yarn from pineapple leaf fibers is obtained, the alkalization process is carried out by using 5% NaOH for 2 hours as in Figure 2. Then the pineapple yarn is rinsed thoroughly with distilled water until it reaches neutral pH and then dried using an oven at a temperature of 100°C for 24 hours. On the other hand, the alkalized pineapple leaf fiber yarn is then woven into fabric sheets with a unidirectional direction measuring 40 x 40 cm.

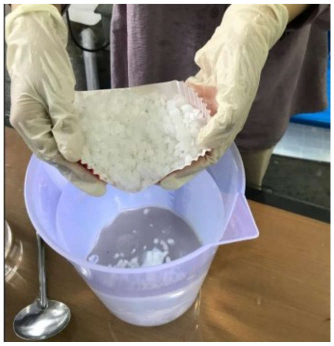


**Figure 2** Alkali treatment

## 2.3 Fabrication Hybrid Composite

The unidirectional hybrid epoxy/pineapple leaf fiber composite is manufactured by using the vacuum infusion process method. Initially, epoxy resin is mixed with hardener and then stirred for 5 minutes. The woven pineapple leaf fibers are arranged on a glass plate with a size of 100 x 100 cm in 5 layers and then vacuum bags are arranged above the peel ply and flow net. Vacuum bags are attached with sealent tape. The resin infusion hub is installed in the center of the spiral vacuum bag tubing and then connected to the vacuum pump and resin tube. After that, the epoxy and hardener mixture is flowed into the mold. Curing is carried out at room temperature for 24 hours. The dried hybrid composite mold is then disassembled to be cut into pieces according to the standard size used for various tests.

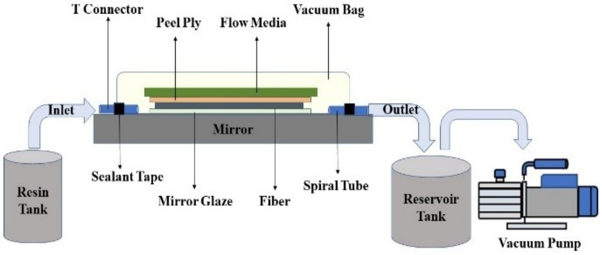


**Figure 3** Schematic illustration of preparation of vacuum infusion manufactured

Epoxy/pineapple leaf fiber/MWCNT hybrid composites are also manufactured by using vacuum infusion method. MWCNT with variations of 0.1; 0.3; 0.5; 1%wt, mixed with acetone and stirred using a magnetic strirrer for 90 minutes at 300 rpm. The amount of acetone used for the mixture is 100 ml. After that, MWCNT nanocellulose and acetone are sonicated for 30 minutes at 45°C. Then epoxy is added to the mixture and stirred using a magnetic strirer for 300 minutes at 750 rpm and heated at 80°C to remove acetone from the mixture. After that, hardener is mixed into the mixture. The ratio of epoxy and hardener used is 2:1 and the mixture is stirred for 2 minutes and then undergoes vacuum degassing for 2 minutes. Figure 4 and Table 1 describe a manufacturing process of hybrid composites and hybrid composite compositions, respectively.

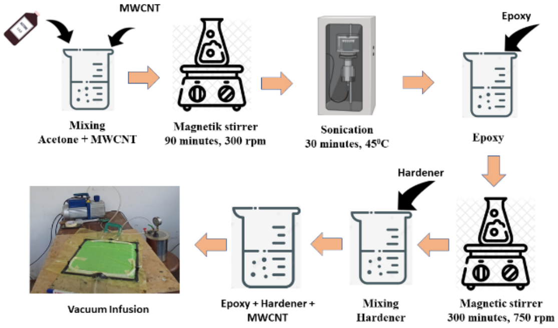


**Figure 4** Diagram of hybrid composite manufacturing process

**Table 1** Formulation Hybrid Composites

| No | Sample | Compositions (%wt) | | | | |
|---|---|---|---|---|---|---|
| | | Composite | Epoxy | Hardener | Pineapple Leaf fiber | MWCNT |
| 1 | S1 | E | 46,7 | 23,3 | 0 | 0 |
| 2 | S2 | E/UPLF | 46,7 | 23,3 | 30 | 0 |
| 3 | S3 | E/APLF | 46,7 | 23,3 | 30 | 0 |
| 4 | S4 | E/APLF/0,1 MWCNT | 46,6 | 23,3 | 30 | 0,1 |
| 5 | S5 | E/APLF//0,3 MWCNT | 46,5 | 23,2 | 30 | 0,3 |
| 6 | S6 | E/APLF//0,5 MWCNT | 46,3 | 23,2 | 30 | 0,5 |
| 7 | S7 | E/APLF/1 MWCNT | 46 | 23 | 30 | 1 |

Note : E = Epoxy
UPLF = Untreated pineapple leaf fiber
APLF = Alkaline pineapple leaf fiber

## 2.4 Water Absorption Behavior

Seven composite specimens with size 24 x 76 mm are tested for water absorption with reference to the ASTM D 570-98 standard. All composite specimens have been recorded with the initial data of wet weight of the test specimens which have previously been dried using oven at 100°C for 60 minutes and conditioned weight of the test specimens where the specimens are soaked for 120 minutes in distilled water. The water absorption value of the composite has been determined by Equation (1).

$$\text{Increase in weight}\ \% = \frac{\text{wet weight} - \text{conditioned weight}}{\text{conditioned weight}} \times 100 \qquad (1)$$

## 2.5 Density and Void Content Measurement

Density testing is a test of the physical properties of composite specimens, which is the ratio between mass (m) and volume (V). This test refers to ASTM D 1896. Density testing aims to determine the mass density of the composite material. Actual density can be calculated by using the Archimedes principle as Equation (2), and theoretical density can be calculated by Equation (3).

$$\rho_{\text{actual}}=\frac{W_{\text{air}}}{W_{\text{air}}-W_{\text{fluid}}}\times\rho_{\text{fluid}} \qquad (2)$$

$$\rho_{\text{theory}}=\frac{1}{\left(\frac{W_{\text{fiber}}}{\rho_{\text{fiber}}}+\frac{W_{\text{matrix}}}{\rho_{\text{matrix}}}\right)} \qquad (3)$$

where: $\rho_{\text{actual}}$ – density actual of hybrid composite, $\rho_{\text{fluid}}$ – density actual of aquades, $w_{\text{air}}$ – weight of air, $w_{\text{fluid}}$ – weight of aquades, $\rho_{\text{theory}}$ - density theoretical of hybrid composite, $w_{\text{fiber}}$ - weight of pineapple fiber, $w_{\text{matrix}}$ - weight of matrix, $\rho_{\text{fiber}}$ - density of pineapple fiber, $\rho_{\text{matrix}}$ - density of matrix.

Porosity is the volume fraction of voids to the total volume of the material, expressed in percent (%). The quality of the composite material decreases as the number of the porosity rises. If the actual density ($\rho_{\text{actual}}$) is measured and the theoretical density ($\rho_{\text{theory}}$) is calculated, the % porosity can be determined by Equation 4.

$$\%\text{void content}=\frac{\rho_{\text{theory}}-\rho_{\text{actual}}}{\rho_{\text{theory}}}\times 100\% \qquad (4)$$

where: $\rho_{\text{actual}}$ – density actual of hybrid composite, $\rho_{\text{fluid}}$ – density actual of aquades, $w_{\text{air}}$ – weight of air, $w_{\text{fluid}}$ – weight of aquades, $\rho_{\text{theory}}$ - density theoretical of hybrid composite, $w_{\text{fiber}}$ - weight of pineapple fiber, $w_{\text{matrix}}$ - weight of matrix, $\rho_{\text{fiber}}$ - density of pineapple fiber, $\rho_{\text{matrix}}$ - density of matrix.

### 2.6 Mechanical Testing

The mechanical tests for this experiment comprise of five tests. The tests are tensile, flexural, and impact test. Tensile and flexural tests are carried out by using Tokyo Testing Universal Testing Machine, while the impact test is carried out by using Charpy impact machine. The tensile, flexural, and impact tests follow ASTM D638-99, ASTM D7264, and ASTM D5942-96, respectively. The first two tests crosshead speed are 5 mm/min and 2 mm/min, respectively. Flexural test applies three points bending method, with the specimen size is 10 mm x 15 mm x 5 mm and the span length is 80 mm. Impact test specimen size is 80 × 10 × 5 mm. Each test will be executed ten times to obtain a range of data. Once the experiment data are collected, the average value in the same test are calculated to obtain the average.

### 2.7 Field Emision Scanning Eclectron Microscopy (FE-SEM) Analysis

A field emission scanning electron microscope (FE-SEM) machine JEOL brand type JSM- 6510LA type is used to observe the surface morphology of hybrid composite materials which has undergone tensile test. The observed materials comprise epoxy/pineapple leaf hybrid composites without treatment and with treatment, and epoxy/pineapple leaf fiber/MWCNT hybrid composites. Prior to observation, the samples are coated by sputtering method using gold coating to make the samples become conductive and can interact with electrons on the FE-SEM machine.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Water Absorption Behavior

Water absorption behavior of the composite non-hybrid E/UPLF, and composite hybrid E/APLF, E/APLF/0.1 MWCNT, E/APLF/0.3 MWCNT, E/APLF/0.5 MWCNT, E/APLF/1 MWCNT are shown in Figure 5. The test illustrates of the composite non-hybrid and composites hybid are directly proportional with water absorption which increases rapidly at the initial time of immersion, then approaching saturation at the end of time. The test results display the water absorption ability of UPLF hybrid composite 0.940% and APLF hybrid composite of 0.624%.

Another fact from the experiment result, the addition of MWCNT increase the composite ability to absorb water. To illustrate, the water absorption in the E/APLF/0.1MWCNT, E/APLF/0.3MWCNT, E/APLF/0.5MWCNT, E/APLF/1MWCNT are 1.023%, 1.313%, 1.217%, and 1.177%, respectively, whereas these results are higher compared to the E/UPLF and E/APLF. The water uptake in the MWCNT should be lower as the its concentration increases [12] because of strong barrier in the carbon nanotube which likely reduces the composite ability to absorb water [13]. However, in some occasions, higher concentration of carbon nanotubes tends to create agglomeration, resulting in microcracks and microvoids in the epoxy [14]. Therefore, the cracks and voids allow water to the composite, which increase the absorption ability.

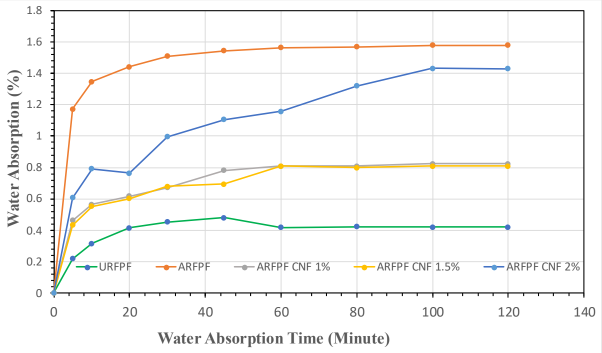


**Figure 5** Water absorption behavior of hybrid composites E/UPLF, E/APLF, E/APLF/0.1MWCNT, E/APLF/0.3MWCNT, E/APLF/ 0.5MWCNT, E/APLF/1MWCNT

The decrease in water absorption happens due to well dispersed and evenly distributed MWCNT during the vacuum infusion process. The increase in water absorption of fibers treated with alkali and with the addition of nanofillers such as cellulose nanofiber (CNF) and carbon nanotubes (CNT) was also reported by previous researchers [15-16], namely alkali treatment and the addition of nanofillers which provide a larger surface area as it attached on the surrounding area of the fibers so that the fibers can more easily contact with water, and strengthen the binding ability between fibers and water. with the addition of nanofillers such as cellulose nanofiber (CNF) and carbon nanotubes (CNT)

## 3.2 Density and Void content

Table 2 shows the experimental and theoretical densities. It can be seen that both experimental density and theoretical density of E/APLF composites increase compared to E/UPLF composites. This is likely due to the alkali treatment of the fibers. This treatment removes hemicellulose, lignin, and other impurities on the fiber surface causing fiber shrinkage resulting in an increase in fiber density [17,18]. Then, the addition of 0.1 MWCNT %wt to the E/APLF composite decreases the experimental density compared to the E/APLF composite. This may be due to agglomeration in the composite. However, the addition of 0.3, 0.5 increases the experimental density as well as the theoretical density of the composite. Excessive addition of MWCNT does not guarantee an increase in density and mechanical properties, as seen with the addition of 1% MWCNT. Table 2 shows that the experimental density value of the 1% MWCNT hybrid composite decreases due to agglomeration and the matrix not being evenly distributed as the reinforcing material is added, resulting in trapped air voids during the mixing, stirring, and hybrid composite production processes. The highest experimental density and theoretical density are obtained with the addition of 0.5 MWCNT %wt. The rising density is likely influenced by the addition of MWCNT which causes the voids between the matrix and the fiber is filled by homogeneously dispersed MWCNT. The increase in density indicates a good interface bond between the fiber and matrix.

**Table 2** Result of theoretical density, experimental density, and void content measurement of epoxy/pineapple leaf fiber/MWCNT specimen

| Sample | $\rho_{theoritical}$ (gr/cm$^3$) | $\rho_{experimental}$ (gr/cm$^3$) | Void content (%) |
|---|---|---|---|
| E | 1,16 | 1,159 | 0,08 |
| UPLF | 1,186 | 1,165 | 1,696 |
| APLF | 1,231 | 1,185 | 3,836 |
| E/APLF/0,1MWCNT | 1,232 | 1,179 | 4,564 |
| E/APLF/0,3 MWCNT | 1,2321 | 1,2093 | 1,849 |
| E/APLF/0,5 MWCNT | 1,2323 | 1,2143 | 1,285 |
| E/APLF/1 MWCNT | 1,2327 | 1,2139 | 1.580 |

The experimental and theoretical densities are used to calculate the porosity of the composites. The highest composite porosity is obtained in E/APLF/0.1MWCNT hybrid composite (4.56%). This high porosity indicates that the hybrid composite has more voids.

Voids can occur as a result of the alkali process, which removes non-cellulose components from the surface of the fibers, such as hemicellulose, lignin, and wax. The loss of these components causes morphological changes in the fibers, including increased surface roughness and the formation of internal voids, which also result in a reduction in fiber diameter. During the composite manufacturing process, these voids are not fully filled by the resin [19, 20]. As demonstrated in Table 2, there is a discrepancy between UPLF and APLF with regard to an increase in void content. The addition of filler results in a reduction of void content. However, the addition of 0.1 MWCNT filler has a large void content, which may be caused by the filler not completely covering all the voids in the fibre.

The E/APLF/0.5MWCNT hybrid composite has the lowest porosity compared to the E/APLF/0.1MWCNT, E/APLF/0.3MWCNT and E/APLF/1MWCNT hybrid composites. The porosity of the E/APLF/0.5MWCNT hybrid composite is 1.28%. This is likely due to the content of 0.5 MWCNT %wt evenly dispersed in the E/APLF composite so that the voids produced are small.

## 3.3 Tensile Properties

### Tensile Strength

The tensile strength of the hybrid composites can be seen in Figure 6. Epoxy has a tensile strength 49.89 ± 6.29 MPa. Then, the next composite where epoxy is given reinforcement from pineapple leaf. First, pineapple leaf that have not been alkalized and those that have been alkalized. The E/UPLF composite has a strength 78.05 ± 10.23 MPa, and the E/APLF composite increases by 93.71 ± 6.65 MPa.

The more robust E/APLF composite is due to the alkalization of pineapple leaf, which generate fiber of pineapple leaves causes the removal of impurities, changes in the crystalline structure of cellulose, increased absorbency, reduced fiber diameter, and increased fiber surface area, enabling the fibers to interact fully with the matrix. This has also been reported by Gomes *et al.* [21] that alkalization treatment can result in a reduction in fiber diameter. This increasing tensile strength is likely due to the alkali treatment of pineapple fiber (APLF) which removes hemicellulose, lignin and other impurities in pineapple leaf fiber. Therefore, fiber hardness rises, resulting in stronger bond between fiber and matrix [20, 21]. The hybrid composite with 0.1 %wt MWCNT (E/APLF/0.1 MWCNT) addition experienced an increase of tensile strength by 15.71% to 108.43 ± 8.69 MPa. `On the other hands, addition of 0.3 %wt MWCNT increases the tensile strength by 21.21% to 113.73 ± 5.01 MPa. In the last sample, the highest increase in tensile strength is obtained after the addition of 0.5 %wt CNT with an increase of tensile strength of 26.70% (from 93.71 ± 6.65 MPa to 118.73 ± 7.68 MPa).

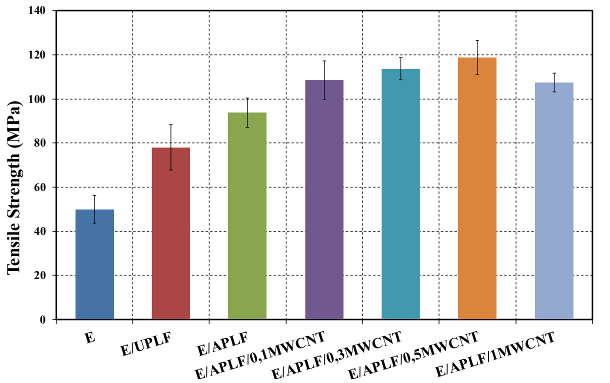


**Figure 6** Tensile strength epoxy/pineapple leaf fiber/MWCNT hybrid composites

Increasing tensile strength due to the MWCNT addition can increase the mechanical properties of E/APLF/MWCNT hybrid composites compared to without the addition of MWCNT. This phenomenon may happen due to the increase in the interfacial bonding between the epoxy matrix and the MWCNT, causing strong covalent connections between those substances. This also improves not only tensile strength, but also whole mechanical performance of the composite [14,38]. Previous researchers [10,24,43] reported that the addition of nanofillers (MMT, OMMT and CNT) can increase the tensile strength of epoxy/pineapple leaf fiber composites. The decrease in tensile strength also occurs in hybrid composites with the addition of MWCNT 1%wt by 12.79% due to the MWCNT agglomeration during vacuum infusion process. Moreover, stress concentration occurs on the certain part of hybrid composites, causing initial defects. The presence of voids in the vacuum infusion process can reduce tensile strength.

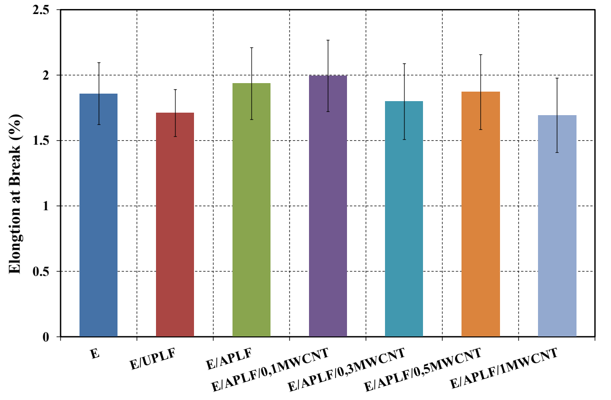


**Figure 7** Elongation at break of epoxy/pineapple leaf fiber/MWCNT hybrid composites

Furthermore, the composite tensile strains of E, E/UPLF, E/APLF, E/APLF/0.1MWCNT, E/APLF/0.3MWCNT, E/APLF/0.5MWCNT, and E/APLF/1MWCNT are shown in Figure 7. The the E/UPLF composite tensile strain is 1.71 ± 0.18% while the E/APLF composite tensile strain is 1.94 ± 0.28%. This shows that the use of pineapple leaf fiber after alkali as reinforcement can increase the tensile strain of the composite. The elongation at break value of the E/APLF/0.1MWCNT increases by 1.99% due to good interface bonding and no agglomeration during the vacuum infusion process. The amount of tensile strain on the E/APLF/0.1MWCNT hybrid composite indicates the ability of the hybrid composite to deform.

Other facts from Figure 7, it can be seen that the addition of MWCNT into the E/APLF composite tend to decrease its tensile strain. This reduction in tensile strain is possible due to the strong bond between the fibers and the matrix [22,32]. The phenomenon is likely caused by weak interfacial adhesion between matrix and pineapple fibers, which also happens in nano filler agglomeration [23]. An investigation in previous research found that as the carbon nanotube content was higher, the elongation at break decreased, and vice versa [24].

Figure 8 illustrates the tensile modulus of epoxy/pineapple leaf fiber/MWCNT hybrid composites, which shows the stiffness properties of the composite. If the elastic modulus is high, the material is more rigid. The elastic modulus obtained for the E/APLF composite (4019.03 ± 273.65 MPa) is lower than the E/UPLF composite (5139.76 ± 405.41MPa). This indicates that the E/APLF composite has a lower stiffness than the E/UPLF composite. This low stiffness is likely due to the alkaline effect of pineapple leaf fiber which removes hemicellulose, lignin, and other impurities on the fiber surface.

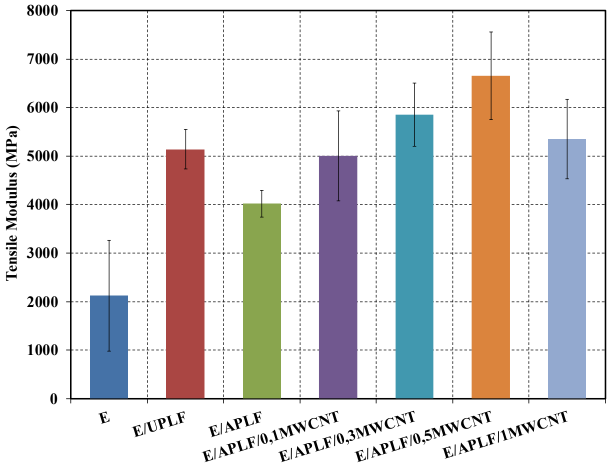


**Figure 8** Tensile modulus of epoxy/pineapple leaf fiber/MWCNT hybrid composites

The addition of MWCNT to E/ARF composites shows higher elastic modulus. This is likely due to the higher modulus elasticity of the MWCNT. Higher modulus elasticity in one material rises average composite modulus elasticity. Therefore, the material will obtain higher elastic modulus. Moreover, the fully dispersed MWCNT creates a robust composite, which means there is no MWCNT located outside the agglomeration area. The higher Young Modulus due to well distribution of MWCNT is also predicted in a previous research [25,41,43]. The highest elastic modulus is obtained in the E/APLF/0.5MWCNT hybrid composite which amounted to 6653.18 ± 904.88 MPa. The addition of 0.5 wt% MWCNT rises the tensile elastic modulus by 65.54%. The increase in tensile elastic modulus due to the addition of nanoparticles is also found in previous research [26] where the elastic modulus of the epoxy/kenaf composite was 3463.54 MPa then after the addition of CNF the tensile elastic modulus increased by 83.34%, the elastic modulus of the epoxy/kenaf/CNT composite became 6350.08 MPa. Another report on the results of nanofiller research is that the addition of nanofiller reinforcement can provide an increase in tensile modulus [27].

## Flexural Properties

Figure 9 shows the flexural strength of epoxy/pineapple leaf fiber/MWCNT hybrid composites. The E/UPLF hybrid composite has an increasing flexural strength of 98.71 ± 7.24 MPa, while E/APLF has a flexural strength value of 100.61 ± 1.60 MPa. This is the effect of the addition of natural fiber reinforcement without treatment and with treatment on E/UPLF and E/APLF hybrid composites. The addition of MWCNT also results in the flexural strength increase of the hybrid composites. The highest increase in flexural strength is found in the E/APLF/0.5MWCNT hybrid composite (126.58 ± 16.39 MPa).

MWCNT creates a noticeable change in the flexural strength. The increase in flexural strength is due to a high aspect ratio and crystallinity. These attributes trigger the interfacial bond between the matrix and pineapple leaf fiber, that allowed an effectife stress transfer between the leaf fiber and MWCNT [45]. Moreover, as crack initiation occurs, MWCNT serve as nanoscale bridges across microgaps, retarding crack propagation and significantly enhancing the flexural strength [46]. This phenomenon is also supported with the fully distributed MWCNT in the whole composite. Therefore, the flexural strength increases. The decrease in flexural strength also occurs in E/APLF/1MWCNT hybrid composites due to agglomeration and voids, resulting in a decrease in flexural strength of hybrid composites. The increase in flexural strength in composites due to the addition of nanometer reinforcement is also reported in previous studies [28,29,39]. In their research, they obtained an increase in the flexural strength value of the studied composites.

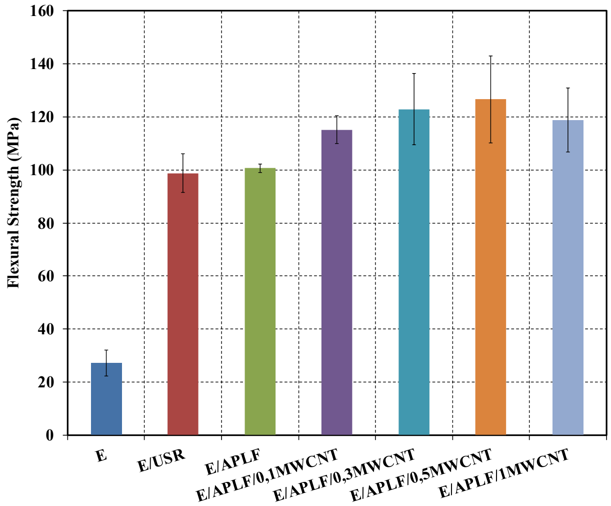


**Figure 9** Flexural strength of epoxy/pineapple leaf fiber/MWCNT hybrid composites

Figure 10 illustrates the flexural modulus of epoxy/pineapple leaf fiber/MWCNT hybrid composites. The addition of pineapple leaf fiber reinforcement results in a surge in the value of E/UPLF hybrid composites which is 2,491.55 ± 187.58 MPa, and E/APLF hybrid composites has a value of 2,566.50 ± 340.16 Mpa, compared to epoxy alone, which is 505.12 ± 151.95 MPa. This is the effect of adding natural fiber reinforcement without treatment and with treatment in the hybrid composite. The addition of MWCNT to the hybrid composites also increases the flexural modulus. The highest flexural modulus rise is found in the E/APLF/0.5MWCNT hybrid composite, (3,968.61 ± 471.07 MPa). The increase in flexural modulus of MWCNT is likely due to MWCNT that is evenly dispersed in the composite, which generates a better interface bond between the matrix, fiber and MWCNT[45]. The addition of MWCNT also increases the surface area of interfacial contact between the matrix and pineapple leaf fiber. The decrease in flexural modulus also occurs in E/APLF/1MWCNT hybrid composites due to agglomeration and the presence of voids, resulting in a decrease in flexural modulus in hybrid composites. The increase in flexural modulus in composites due to the addition of nanofiller reinforcement is also reported in previous studies [29,30,47]. In their research, they obtained an increase in flexural modulus value in the studied composites.

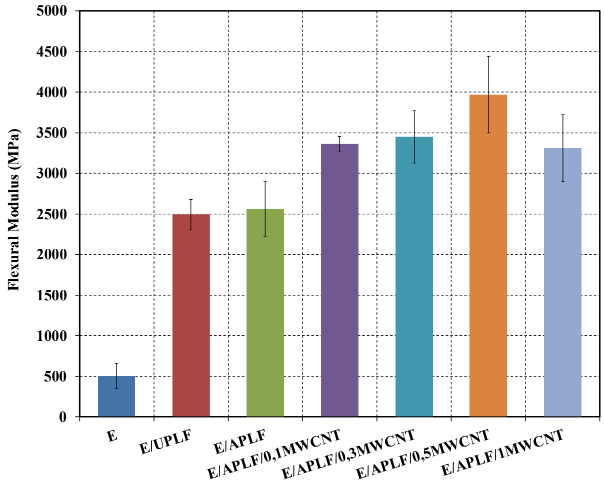


**Figure 10.** Flexural modulus of epoxy/pineapple leaf fiber/MWCNT hybrid composites

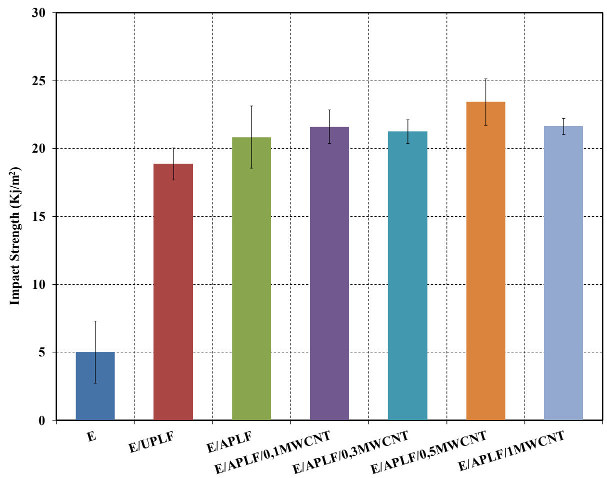


**Figure 11** Impact strength of epoxy/pineapple leaf fiber/MWCNT hybrid composites

## Impact Strength

Figure 11 shows impact toughness value on epoxy. The impact toughness of the epoxy is 5.01 kJ/m2. The addition of pineapple leaf fiber reinforcement results in a significant rise in hybrid composites, which is 18.86± 1.18 kJ/m2, while E/APLF hybrid composites have a value of 20.85± 2.29 kJ/m2. This is the effect of adding natural fiber reinforcement to the hybrid composite composition. The addition of CNF also increased the impact toughness of the hybrid composites. The increase in impact toughness occurred in hybrid composites E/APLF/0.1MWCNT, E/APLF/0.3MWCNT, E/APLF/1MWCNT with toughness values of 21.60 ± 1.23 MPa, 21.26 ± 0.86 MPa and 22.08 ± 1.12 MPa, respectively. The highest increase in impact toughness is found in the E/APLF/0.1MWCNT hybrid composite which is 23.43 ± 1.71 MPa.

Pineapple leaf fiber as a reinforcement increases the impact toughness of the composite. Even dispersion of nanofillers improves the interfacial bonding between the matrix and fibers. Energy is easily absorbed, and crack propagation is prevented, thereby enhancing impact toughness as reported in previous studies. Energy will be easily absorbed and crack propagation will be prevented so that the impact toughness increases as in previous research [29-31]. The increase occurs because MWCNT are evenly distributed during the mixing and vacuum infusion process. The high impact toughness is related to the nanofiller dispersion rate. The even dispersion of nanofillers promotes good interfacial bonding between matrix and fiber. The results of researcher [32] showed an increase in impact toughness at the addition of 0.5%wt CNC. Another study reported an increase in impact toughness of 49% in epoxy/polysulfone composites with the addition of 0.2%wt CNF [33,42].

## Morphology Studies

Figure 12(a) shows the FE-SEM image of the fracture surface of the E/UPLF composite, which shows fiber pull out, crack, void, and debonding. This indicates a weak interfacial adhesion between the non-alkali treated pineapple leaf fibers (UPLF) and the matrix. This weak interfacial bonding is most likely due to the hydrophilic nature of the pineapple leaf fiber which opposed to the hydrophobic nature of the matrix. This mismatch hinders the stress transfer efficiency between the fiber and the matrix, thus lowering the mechanical performance of the composite.

Meanwhile, Figure 12(b) shows the FE-SEM image of the fracture surface of the E/APLF composite, which shows fiber pull out, fiber bonding, and an indicated epoxy layer attached to the pineapple leaf fiber surface after alkali treatment (APLF). This alkali treatment is alleged to remove amorphous contents such as hemicellulose, lignin, and other impurities, which increase the roughness of the fiber surface. Therefore, the matrix adheres more easily and forms a better interfacial bond between the fiber and the matrix. This strong interfacial bond allows for more effective load transfer, thereby increasing the mechanical strength of the composite [18]. In addition, the addition of MWCNT to E/APLF composites also improves their mechanical properties, which is most likely due to the even distribution of MWCNT on the fracture surface of E/APLF composites. The study of alkali-treated coir/pineapple leaf fibers reinforced hybrid composites by Siakeng *et al*. [34,44] has shown the effect of alkalized fiber.

In Figure 12c, the fracture surface of the E/APLF/0.5MWCNT hybrid composite shows a rougher texture, which indicates plastic deformation during surface fracture. This deformation is closely related to the increased material toughness. The fracture surface shows the presence of a thin layer of matrix that is tightly attached and partially covers the surface of the pineapple leaf fiber, indicating a strong attachment between the matrix and the fiber. Observation through FE-SEM at high magnification revealed that the addition of MWCNT can act as an effective reinforcing agent. The absence of gaps around the fibers also indicates a good interaction between the two parties, which is caused by homogeneous dispersion of MWCNT in the matrix. This even distribution of MWCNT supports stress transfer efficiency and contributes to the mechanical properties improvement of the composite through the synergy between the matrix, natural fibers, and MWCNT. The optimal dispersion of MWCNT in the matrix systems is proven to improve the mechanical strength of the E/APLF/0.5MWCNT hybrid composites. This finding is in line with the increase in tensile strength shown in Figure 6. The addition of MWCNT can provide better stress transfer from the matrix to the fibers, thus minimizing stress concentration in the matrix. However, the addition of MWCNT beyond 0.5%wt actually decreases the mechanical strength due to the agglomeration formation in the epoxy matrix.

Figure 12d displays the FE-SEM image of the MWCNT which shows the evenly distributed MWCNT morphology structure on the natural fiber surface. This dispersion contributes to local reinforcement of the matrix around the fibers, creating a gradual modulus gradient along the fiber-matrix interface. This gradient allows for more efficient stress transfer, which ultimately improves the mechanical performance of the composite. Based on the morphological analysis of the FE-SEM images, it can be confirmed that the E/APLF/0.5MWCNT composite exhibits higher tensile strength than the E/UPLF composite, as described in the previous discussion. This result is also confirmed by some researchers that visible epoxy matrix and adhering particles on the fibers is a clear sign of strong interface bond of the leaf and matrix [35,36,40].

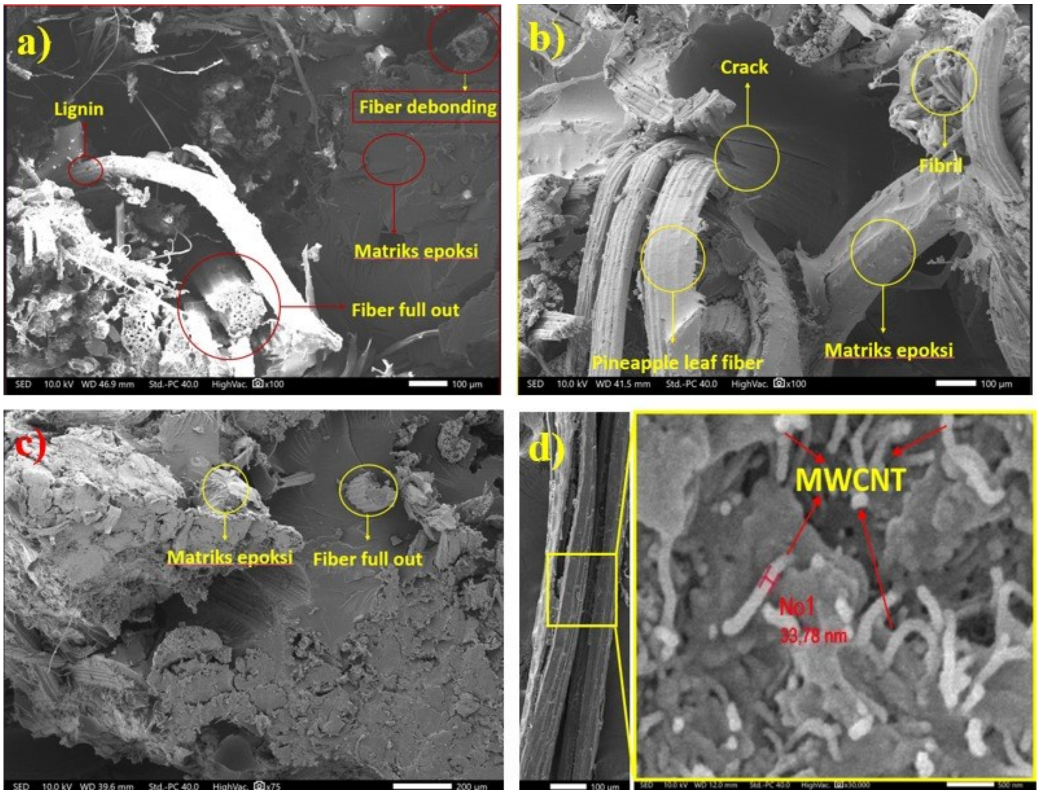


**Figure 12** FE-SEM images of tensile fractured surface (a) composite hybrid E/UPLF, (b) composite hybrid E/APLF, (c) composite hybrid E/APLF/0,5MWCNT, and (d) MWCNT

Energy dispersive x-ray specroscopes (EDS) observations were made as shown in Figure 13. Observations on E/UPLF hybrid composites, E/APLF hybrid composites, E/APLF/0.5MWCNT hybrid composites in order to analyze the elemental or chemical characteristics of the hybrid composite specimens. In Table 3. EDX analysis elemental composition in E/UPLF, E/APLF, and E/APLF/0.5MWCNT EDS testing was conducted to obtain the elements contained in the hybrid composites.

It is presented in Table 3 that the elements in the fiber, namely carbon (C) and oxygen (O), are the main elements in the treated and untreated fibers. The C element in the untreated fiber has a value of 62.56% and decreased to 45.88% in the treated fiber.

The O element in the untreated fiber has a value of 36.4% and has increased to 54.12%. Similar results on EDX analysis showed that alkali-treated fibers reduced carbon content and increased oxygen compared to untreated fibers [37]. The test results in Table 3 also show that the fibers before alkali treatment in the E/UPLF hybrid composite still contain sodium (Na), but the fibers after alkali treatment in the E/APLF hybrid composite and E/APLF/0.5MWCNT hybrid composite do not contain the chemical element Na. Table 3 shows that the ratio of C elements decreased and O increased in the treated fibers compared to the untreated fibers indicating that the chemical treatment removed lignin from the fiber surface, similar to the research reported by previous researchers [37].

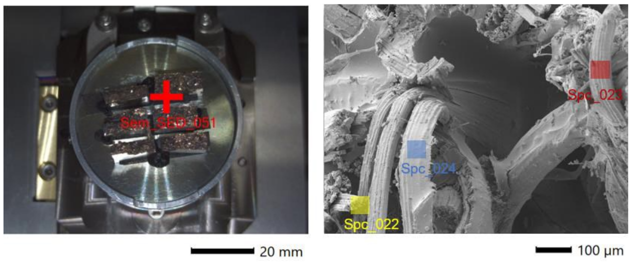


**Figure 13** EDX spectra of composite hybrid E/APLF

**Table 3** EDX analysis elemental composition in E/UPLF, E/APLF, and E/APLF/0,5MWCNT

| Area for EDX analysis | Chemical Substance (%wt) | | |
|---|---|---|---|
| | C | O | Na |
| Matrix of E/UPLF | 71,22 | 28,78 | - |
| Fiber of E/UPLF | 62,56 | 36,4 | 1,04 |
| Matrix of E/APLF | 61,07 | 38,93 | - |
| Fiber of E/APLF | 45,88 | 54,12 | - |
| Matrix of E/APLF/0,5 MWCNT | 82,36 | 17,64 | - |
| Fiber of E/APLF/0,5 MWCNT | 53,15 | 46,85 | - |

## 4.0 CONCLUSION

The investigation of the effect of alkali treatment of flax fiber and the addition of MWCNT 0.1, 0.3, 0.5 and 1 %wt on the mechanical properties and physical performance of hybrid composites by manufacturing through vacuum assisted resin infusion method has been successfully carried out. The results shows that fiber alkali treatment improves the mechanical properties of epoxy/pineapple leaf fiber hybrid composites through the removal of hemicellulose, lignin and other impurities, that creates better interfacial bonding between the matrix and fibers.

The addition of MWCNT to the epoxy/pineapple leaf fiber composite improves the mechanical properties. The strengthening mechanism of added MWCNT is by improving the contact surface area of reinforcement fibers to the epoxy matrix, resulted to better interfacial bonding between each other. The existence of a good interface bond between pineapple leaf fibers and the matrix is indicated by the visible epoxy matrix and MWCNT particles attached to the surface of pineapple leaf fibers as evidenced by the results of FE-SEM observations.

This work proofs that adding MWCNT as nanofiller to hybrid natural fiber composite is an effective way to improve its mechanical strength.

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## Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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