



## Influence of calcium carbonate on mechanical properties of Kenaf/epoxy composites

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### ABSTRACT

The aim of this paper is to study the influence of calcium carbonate ( $\text{CaCO}_3$ ) particle content on mechanical properties of kenaf/epoxy composites. The composite was prepared by hand lay-up process. Different particle loadings were prepared; namely, 2.5%, 5%, 7.5%, 10%, 20% weight percent. Tensile, impact and density of the developed  $\text{CaCO}_3$ /kenaf/epoxy hybrid composites were evaluated and compared. The analysis of the result revealed that incorporation of  $\text{CaCO}_3$  particle into the kenaf/epoxy composites decreased their tensile strength. Impact strength increased by addition of 2.5%  $\text{CaCO}_3$  content.  $\text{CaCO}_3$  particle loading decreased density of the composite.

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## 1. Introduction

Today the amount of waste is increasing day by day as the world's population increases. Realising this, recycling was introduced as a way to solve the problem. The process of recycling natural resources can be used to protect the environment such as forests that provide oxygen for humans in life. Recycling also reduces the amount of waste production and can help solve the problem of garbage piling up. Recycling also reduces costs as manufactured products from raw materials to finished products get more expensive as time passes [1].

Applications of natural fibre-reinforced composites are growing rapidly in various engineering fields. Various types of natural fibre reinforced polymer composites have become important in various applications where they have been found in the construction, sports, aerospace, defence and other industries [2].

The addition of particles in polymer plastic matrix composites aims to reduce production costs. However, in recent years, particles as fillers are used to increase polymer stiffness [3]. Mineral fillers used in polymers are usually talc and calcium carbonate [4]. Calcium carbonate ( $\text{CaCO}_3$ ) is a low cost filler widely used in rubber, plastics, paints and other industrial applications. In general, the shape, size and content of calcium carbonate can affect the properties of composites. Calcium carbonate as a filler has a much smaller size than wood fibre, therefore calcium carbonate particles can be easily filled into the polymer matrix than wood fibre [5].

The kenaf plant is a plant of the Hibiscus genus that has good potential as a raw material for use in composite products [6]. Kenaf fibre (*Hibiscus Cannabinus* L.) is a type of natural fibre that offers many advantages and high potential as a reinforcement in composite materials, especially polymer composites. The use of kenaf fibre composites as reinforcement is becoming one of the interesting areas of research. Conventionally,

synthetic fibres such as carbon, glass and aramid fibres are usually used in the production of polymer composites. But kenaf fibres have comparable special properties and relatively low processing costs which are advantageous over synthetic fibres [7].

This research will discuss the development of hybrid-epoxy composite materials reinforced with kenaf fibre and CaCO<sub>3</sub> powder with varied compositions. Hybrid composites created to maintain an epoxy load at 80% by weight in all composites. The variation in the composition of CaCO<sub>3</sub> particles and kenaf fibre in a hybrid composite will produce varying composite mechanical strengths to support its use.

## 2. Methodology of Research

### 2.1 Materials

Epoxy is used as the matrix in the composite. Epoxy resin Eposchon 1011 A was obtained from Justus Kimia Raya Inc, Indonesia. Epoxy has a density of 1.01 g/cm<sup>3</sup> at 25°C and a viscosity of 0.5 to 1.0 poise at 25°C. This study used Eposchon 1011 B as a catalyst. Kenaf fibre (shown in Fig. 1) was obtained from BSIP-TAS Malang, Indonesia. Kenaf fibres were soaked in 6% NaOH solution for three hours in a container at room temperature. The fibres were washed thoroughly with aquadest to remove excess NaOH, then dried at room temperature for 72 hours, then dried in an oven at 100°C for 2 hours. Kenaf fibre was then cut into 30 mm. The calcium carbonate (CaCO<sub>3</sub>) powder used as composite reinforcement has particles with a size of 12 µm.



Figure 1. Kenaf fibre

### 2.2. Manufacture of composite samples

The CaCO<sub>3</sub>/kenaf/epoxy hybrid composite was fabricated by hand lay-up method. In the first stage, CaCO<sub>3</sub> particles are added to the epoxy with a variable loading of 2.5% by weight, 5% by weight, 7.5% by weight, 10% by weight and 20% by weight

as displayed in Table 1. Stirring from these samples was carried out to obtain a homogeneous mixture of CaCO<sub>3</sub> particles and epoxy without hardener. The magnetic stirrer was set to spin at 1000 rpm for 10 minutes. After it was evenly dispersed, the epoxy/CaCO<sub>3</sub> solution was added with hardener in a ratio of 2:1.



Figure 2. Composites Moulding

Table 1. Combination of CaCO<sub>3</sub>/kenaf/epoxy hybrid composite samples prepared.

Epoxy (wt%)	Kenaf (wt%)	CaCO <sub>3</sub> (wt%)
100	0	0
80	20	0
80	17,5	2,5
80	15	5
80	12,5	7,5
80	10	10
80	0	20

After this step, the Epoxy/CaCO<sub>3</sub> mixture is poured evenly into the mould to form a single layer, then the kenaf fibre is placed evenly and pressed gently using a spatula in order to minimise the formation of voids. The same process is repeated until the mould is filled in all parts. Then pressing is done with a glass plate placed on the mould as a cover. After this step, the curing process was carried out for 24 hours in a room and then the sample was removed from the mould. The composite mold used is made of MDF board (shown in Fig. 2).

### 2.3. Tensile tests

Hybrid composite specimens for tensile test were prepared. Hybrid composite specimens with different CaCO<sub>3</sub> additions were tensile tested using WDW-50E UTM at crosshead speed of 2 mm/min using samples according ASTM D368 (as shown in Fig. 3). The test

results from the repetition of each composition variation were recorded as the average.



**Figure 3.** CaCO<sub>3</sub>/kenaf/epoxy composite specimen of tensile test

#### 2.4. Impact tests

Impact tests were carried out using a HUNG TA Universal Impact Tester. The impact strength was calculated by dividing the absorbed energy by the initial cross-sectional area behind the notch (100 mm<sup>2</sup>). All measurements were carried out in three-fold.

#### 2.5 Density tests

The density of the prepared samples was measured according to ASTM D 792-91 using a water immersion technique based on the Archimedes principle. In this method, the weights of samples in air and in distilled water are taken separately. Using the measured weight of the sample and the specific gravity of the water, the density of the sample is measured according to equation (1). where  $\rho_e$  is the experimental density,  $\rho_w$  is the density of water,  $W_a$  is the weight of the sample in air, and  $W_w$  is the weight of the sample in water.

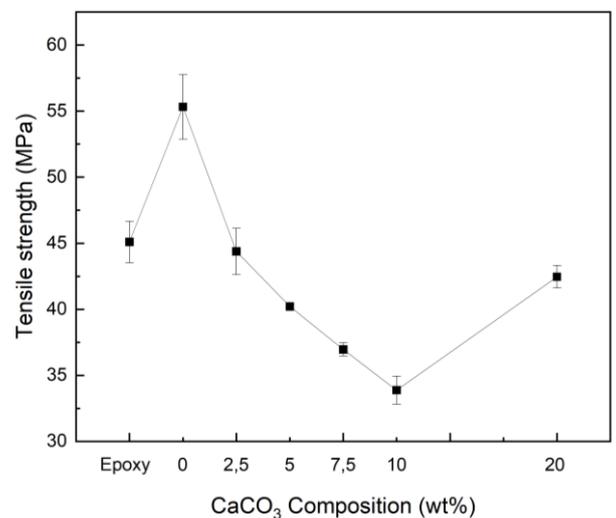
$$\rho_e = \frac{W_a}{W_a - W_w} \times \rho_w \quad (1)$$

### 3. Result and Discussion

#### 3.1. Tensile strength

The tensile strengths are shown in Fig. 2. Based on the graph in Fig. 4 shows the effect of CaCO<sub>3</sub> powder composition the tensile strength of hybrid composite, where the more CaCO<sub>3</sub> powder increases the tensile strength decreases. The decreased tensile strength is due to poor interfacial bonding between the reinforcement and the matrix. Poor interfacial bonding eventually leads to the formation of small voids and this reduces the effectiveness of the filler in

the composite. The highest tensile strength is found in the kenaf/epoxy composite, which is 55.32 MPa. While the lowest tensile strength is in the 10 wt% CaCO<sub>3</sub>/kenaf/epoxy hybrid composite, which has a tensile strength of 33.88 MPa. The presence of few fibres can result in low tensile strength because fibres play a role in transferring loads in the composite. In addition, the low tensile strength of the 10 wt% CaCO<sub>3</sub>/kenaf/epoxy hybrid composite material is also due to the presence of powders with an increasing composition where increasing the number of particles also increases the possibility of agglomeration which then creates a stress concentration region that requires less energy to prolong the propagation of cracks [8].

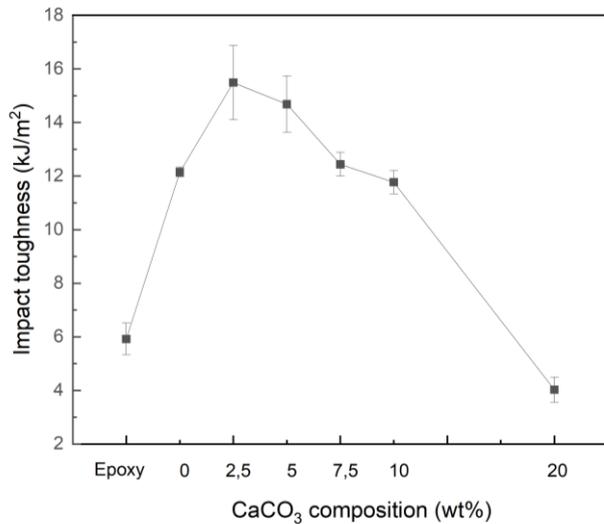


**Figure 4.** Tensile strength of CaCO<sub>3</sub>/kenaf/epoxy composites

The increase in tensile strength occurred in the CaCO<sub>3</sub>/Epoxy composite. The increase in tensile strength is due to the homogeneous dispersion of CaCO<sub>3</sub> powder which results in good interfacial interaction with the matrix. In addition, uniform powder dispersion can have an impact on reducing the interparticle distance in the matrix which then results in increased composite resistance to indentation [9]

#### 3.2. Impact strength

Fig. 5 shows that the addition of powder has the effect of increasing the impact strength. The impact strength increases until the composition of 2.5 wt% CaCO<sub>3</sub>/kenaf/epoxy hybrid composite with an impact strength of 15.49 kJ/m<sup>2</sup>. After that, the 5 wt% CaCO<sub>3</sub>/kenaf/epoxy hybrid composite experienced a gradual decrease in impact strength until the CaCO<sub>3</sub>/Epoxy composite.



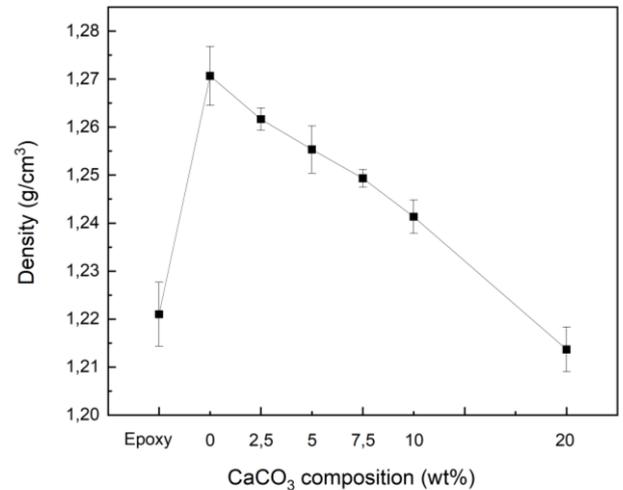
**Figure 5.** Impact strength of CaCO<sub>3</sub>/kenaf/epoxy composites

The CaCO<sub>3</sub>/Epoxy composite has the lowest impact strength, which is 4.03 kJ/m<sup>2</sup>. The decrease in the impact strength of the composite is due to poor compatibility between the reinforcement and the matrix where there is agglomeration formation. In addition, in the CaCO<sub>3</sub>/Epoxy composite, the absence of kenaf fibre in the composite makes it have a low impact strength. This is due to the reduced ability to absorb energy when receiving a load, where the function of fibre in the composite can help to absorb energy when the composite receives a load. A high fibre content in the composite will require more energy to weaken the matrix bond, in other words more energy will be absorbed by the fibres [10].

### 3.3 Density of Composite

Figure 6 shows that the increase in the composition of CaCO<sub>3</sub> powder in Kenaf/epoxy composites decreases the density value of the composite. It is clearly seen in the figure that the 2.5 wt% CaCO<sub>3</sub>/kenaf/epoxy composite has a lower density value when compared to the kenaf/epoxy composite with a density value of 1.262 g/cm<sup>3</sup>. The CaCO<sub>3</sub>/epoxy composite has the lowest density value, which is 1.214 g/cm<sup>3</sup>. The decrease in density value is due to the reduction in the composition of kenaf fibre as the CaCO<sub>3</sub> powder increases, which then in the reinforcement mixture in the composite is

dominated by CaCO<sub>3</sub> powder which has a smaller specific gravity than the specific gravity of kenaf fibre. So that the more the weight percent of CaCO<sub>3</sub> powder increases as the kenaf fibre decreases in the composition, the smaller the specific gravity of the CaCO<sub>3</sub>/kenaf/epoxy hybrid composite.

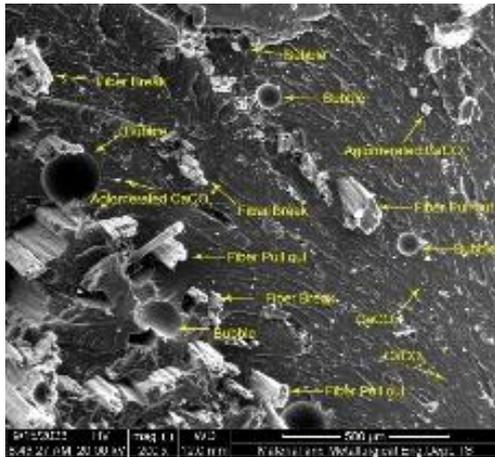


**Figure 6.** Density of CaCO<sub>3</sub>/kenaf/epoxy composites

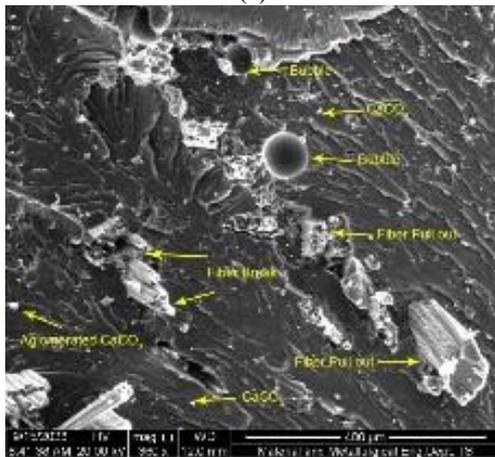
### 3.4 Morphological analysis of composites

The morphological properties of the tensile testing fracture surface of CaCO<sub>3</sub>/kenaf/epoxy hybrid composite materials were observed using scanning electron microscopy (SEM). The samples tested were CaCO<sub>3</sub>/kenaf/epoxy hybrid composite and CaCO<sub>3</sub>/epoxy composite. The scan results shown in Figure 7. explains that there is a bond between the surface of the matrix with kenaf fiber and calcium carbonate.

The decrease in interfacial adhesion between kenaf fiber, CaCO<sub>3</sub> powder and epoxy matrix can be clearly seen from the SEM micrograph of the tensile fracture surface, as shown in Figure 7. In this image, there are many trapped bubbles and fiber pull out. It shows the lack of interfacial adhesivity in the CaCO<sub>3</sub>/kenaf/epoxy hybrid composite. The lack of adhesivity in the composite will reduce its tensile strength.



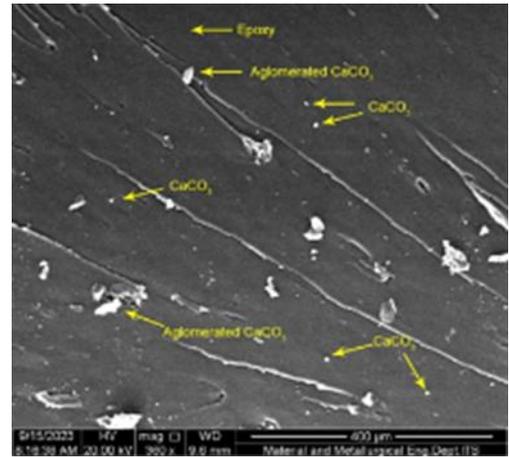
(a)



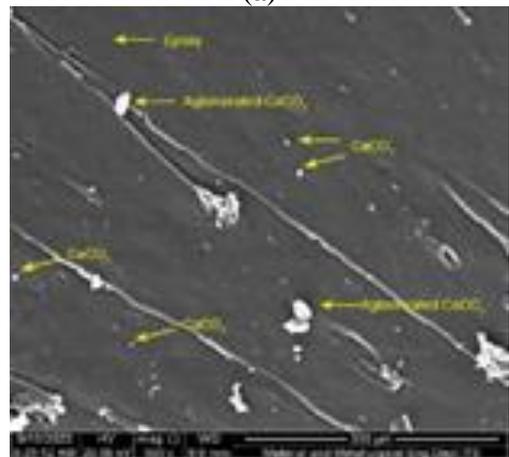
(b)

**Figure 7.** SEM images of tensile fractured 7,5% CaCO<sub>3</sub>/kenaf/epoxy hybrid composites

Figure 8 shows the SEM characterization results of the CaCO<sub>3</sub>/epoxy composite material. From the scanning image of the tensile fracture surface morphology, it can be seen that there is no kenaf fiber as reinforcement in the composite, so the visible fracture is a fracture in the matrix. Visually, it appears that the calcium carbonate (CaCO<sub>3</sub>) powder is evenly dispersed and there are no voids in the composite. However, there are several points where the CaCO<sub>3</sub> powder forms agglomerates. This initiates the crack in the composite because in agglomerated powder the bond between calcium carbonate powder and the matrix becomes weaker. So that when the CaCO<sub>3</sub>/epoxy composite receives a load, the stress is focused at the point where the agglomerated calcium carbonate powder is located.



(a)

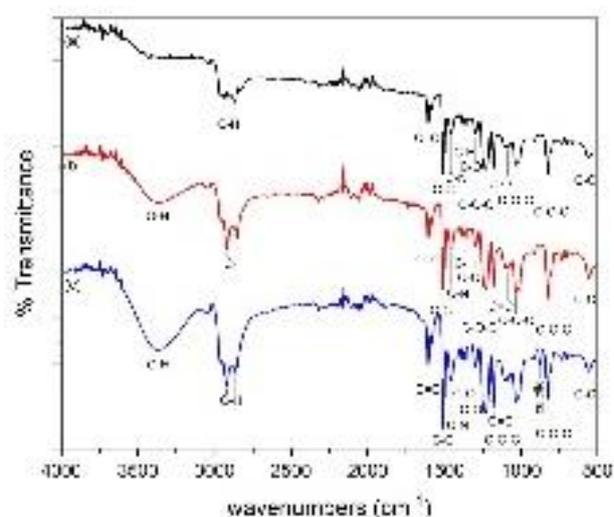


(b)

**Figure 8.** SEM images of tensile fractured CaCO<sub>3</sub>/epoxy composites

### 3.5 Fourier transform infrared spectroscopy (FTIR)

Figure 9 shows a comparison of the FTIR spectra of epoxy, kenaf/epoxy composite, and CaCO<sub>3</sub>/kenaf/epoxy hybrid composite samples, where the peaks in the spectra obtained appear at 500-4000 cm<sup>-1</sup>. The black-colored infrared wave absorption pattern shows that it belongs to epoxy. Epoxy has absorption spectra at wave numbers at 1608 cm<sup>-1</sup>, 1509 cm<sup>-1</sup>, 1036 cm<sup>-1</sup>, 915 cm<sup>-1</sup>, 831 cm<sup>-1</sup>, and 772 cm<sup>-1</sup> [11]. The epoxy spectra show the presence of absorption in the wave number region of 2929 cm<sup>-1</sup> which indicates the bonding of alkane groups (C-H) of CH<sub>2</sub> and CH aromatic and aliphatic, while the Aromatic functional group (C=C) is shown at a wave intensity of 1605 cm<sup>-1</sup>. In the absorption region 1507 cm<sup>-1</sup> is a benzene and C-C bond of aromatic, 1230 cm<sup>-1</sup> is a C-O-C bond of ether, 1082 cm<sup>-1</sup> is a C-O bond of aromatic, and 555 cm<sup>-1</sup> is a C-C bond of ether.



**Figure 9.** FTIR spectra of (a) Epoxy, (b) kenaf/epoxy composite, (c) CaCO<sub>3</sub>/kenaf/epoxy hybrid composite

Kenaf/epoxy composite shows the presence of Hydroxyl (O-H) functional groups in the 3360 cm<sup>-1</sup> absorption region. The presence of hydroxyl (O-H) groups indicates that there is a hydrophilic material, namely kenaf fiber where kenaf fiber in its components is composed of cellulose. Natural fibers tend to contain cellulose in which there are hydroxyl groups (O-H) that interact with water molecules through hydrogen bonds. Unlike glass fibers, where water adsorption only occurs on the surface, natural fibers can interact with water in all parts [12]. The absorption region with a wave of 2916 cm<sup>-1</sup> shows the type of Alkane bond (C-H) while at wave 1605 cm<sup>-1</sup> shows the Aromatic bond strain (C=C). and Ester (C-O) functional groups appear at a wave intensity of 1293 cm<sup>-1</sup> and the C-O-C absorption pattern for ether groups at 1030 cm<sup>-1</sup>.

The FTIR spectra of the CaCO<sub>3</sub>/kenaf/epoxy hybrid composite show the absorption pattern belonging to CaCO<sub>3</sub> at wave number 874 cm<sup>-1</sup> shown in Figure 7 with a blue color pattern. The absorption area of calcite is in accordance with the research of Wang et al. [13], explained that the characteristic peak of calcite (CaCO<sub>3</sub>) is at a wavelength of 877 cm<sup>-1</sup>. The absorption pattern of the methyl (C-H) functional group is shown at 1361 cm<sup>-1</sup>, while the absorption area with a wave of 3359 cm<sup>-1</sup> shows the hydroxyl group (O-H). Of the three absorption patterns, no new absorption patterns appeared other than the epoxy absorption pattern, kenaf fiber absorption pattern and CaCO<sub>3</sub> absorption pattern. From these conditions it

can be seen that there is no chemical reaction between epoxy, kenaf fiber and CaCO<sub>3</sub> in the CaCO<sub>3</sub>/kenaf/epoxy hybrid composite. So it shows that the bond formed between epoxy, kenaf fiber and CaCO<sub>3</sub> is a physical and mechanical bond.

#### 4. Conclusion

From this work, it can be concluded that the variation of CaCO<sub>3</sub> content resulted in different mechanical properties and density of the CaCO<sub>3</sub>/kenaf/epoxy hybrid composites. The tensile strength and density of the composites deteriorated with the increase of CaCO<sub>3</sub> loading. The impact strength of the composites showed an increasing trend up to the CaCO<sub>3</sub>/kenaf/epoxy hybrid composite of 2.5 wt%.

#### 5. Acknowledgement

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