STUDY ON TENSILE PROPERTIES OF SALAK FIBER  
(SALACCA-ZALACCA) REINFORCED CACO\textsubscript{3}/EPOXY RESIN HYBRID COMPOSITES

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Abstract

In this study, the matrix modification of Salak Fiber (Salacca-Zalacca) (SF) reinforced polymer composites with micro-particles is an effective way to improve its matrix properties. After micro-particle modification, understanding mechanical properties is important in structural applications, and improvement of such properties can lead to the usage in the wider fields. The aim of this work is to investigate experimentally tensile properties of CaCO\textsubscript{3} modified epoxy/SF micro-composites. For this, various amounts of CaCO\textsubscript{3} micro-reinforcements of 0, 2, 4, 6, and 8 vol% were added into the epoxy matrix, and the micro-reinforced epoxy resin was used to impregnate SF by press moulding. The prepared fiber reinforced micro-composites were subjected to tensile test. As a result of all experiments, the addition of 4vol% CaCO\textsubscript{3} micro-reinforced SF/epoxy resin hybrid composites yield the optimum on tensile properties. The optimum of CaCO\textsubscript{3}-epoxy matrix was reinforced with various SF content of 0, 10, 20, 30, and 40 vol%. The tensile strength of CaCO\textsubscript{3}-epoxy/SF increases with increasing of SF content (from 0 vol% to 40% of SF). The strength of 134.49 Mpa was obtained at composite with 40 vol% of SF.

Keywords: Epoxy resin, Calcium Carbonate (CaCO\textsubscript{3}), Salak fiber, tensile properties

1. INTRODUCTION

A composite materials are generally engineered materials made from two or more constituents with different physical or chemical properties into one system called matrix or binder, and gives unique properties of material, which may be different in composition, and characteristics [1]. The continuous phase is known as “matrix”, and the discrete phase is known as “reinforcement”. Composite material mainly consists of reinforcements, and the matrix. The matrix is the binding material which is used to hold the reinforcement together in solid form. Whereas, the reinforcements are the materials which are used to carry the load along the length of the fiber, provide strength, and stiffness in one direction [2]. In recent years polymers have generated wide interest in various engineering fields, including tribological applications, in view of their good strength, and low density as compared to monolithic metal alloys. Because of lightweight they are the most suitable materials for weight sensitive uses, but their high cost sometimes becomes the limiting factor for commercial applications [3].

SF is a fruit plant that spread in almost all local in Indonesia. Besides relying on fruit, profits potentially can also be a breakdown from another part of this crop include seeds, skins, and frond. Frond of Salak is just solid waste in agricultural, and has a great chance as
natural fiber resources. Some researchers classify six basic types of natural fibers as follows: bast fibers, leaf fibers, seed fibers, core fibers, grass and reed fibers and all other types (wood, and roots). Refer to a basic type, frond of Salak is a branch of leaf fiber, its form approaches wood. It has a good character like solid, stiff, strong, and suitable for engineering market or automotive industry [4]. SF is not strong enough as a reinforcement. So, it is required to propose hybrid reinforcement. The addition of various types of nano-fillers have been implemented extensively to reinforce fiber such as nano-clays, and metal oxides, because of their unique mechanical properties in improving the performance of fibers, and matrix. Among these, CaCO$_3$ nano-reinforcements have been substantially preferred to modify micro-composites due to their availability in readily usable form, and inexpensive [5]. The reinforcement of polymer using nano-CaCO$_3$ as filler in polymer production of modern plastics. The use of inorganic fillers that has been a common practice in the plastic industry to improve mechanical properties of thermosetting resins such as heat distortion temperature, hardness, toughness, stiffness, and mold shrinkage [6].

Although there are many obvious benefits, the research on composites enhanced with CaCO$_3$ micro/nano-fillers is still very limited. Previous investigations have been mainly focused on the mechanical performance of thermoset resins after the addition of CaCO$_3$ particles with various sizes, modified epoxy resin with micro/nano-sized CaCO$_3$, and reported the ratio of tensile test had greatly improved. According to previous research studied on epoxy/calcium carbonate nanocomposites, the sample was prepared by mixing 0, 2, 4, 6, 8, and 10 wt% of nano-CaCO$_3$ respectively. From experimental of CaCO$_3$/epoxy resin can be concluded that the incorporation of nano-CaCO$_3$ improves the rigidity, maximum use temperature, and toughness of epoxy resins. The maximum improvement on mechanical properties was observed at a filler loading 4 wt% of CaCO$_3$ in the epoxy matrix [6]. Effect of nano-CaCO$_3$ particles on mechanical properties of epoxy resin cast and its composites, for the obtaining nano-CaCO$_3$/epoxy cast shows a good dispersion with 4 wt% treaded nano-CaCO$_3$ particles as observed by transmission electron microscopy (TEM). It can be concluded that even small contents (2-6 wt%) of nano-CaCO$_3$ in the epoxy cast can increase the thermal stability and mechanical properties of the nano-CaCO$_3$/epoxy cast [7]. Revealed that the flexural modulus for glass fiber reinforced epoxy composites increases about 24% with the addition of 8 wt% of CaCO$_3$ nano-reinforcement within the matrix [8]. Emphasized remarkable improvement of 13.5%, 6.1%, 42.5% and 106.3% in compressive strength, elastic modulus, displacement and the total fracture work of epoxy resin filled with 4 wt% of CaCO$_3$ nano-reinforcement with respect to neat epoxy [9]. Reinforced epoxy resin with CaCO$_3$ nano-reinforcement and demonstrated that the impact strength of the nanocomposite increases with an increasing CaCO$_3$ nano-reinforcement content up to 6 wt% [10].

Up to the author’s knowledge, there are no previous reports addressing the effectiveness of CaCO$_3$ micro-reinforcements (CCM) on low velocity impact performances of SF/epoxy micro-composites. In this study, firstly effect of the CCM addition on tensile test of epoxy resin composites was investigated. To give full play of micro-particles, CCM particles were pretreated by manually technique, and then they were mixed with epoxy to prepare epoxy cast, and Epoxy matrix/SF hybrid composites, respectively. As a result, the ternary micro-composites represented an obvious improvement on mechanical performance.
2. MATERIALS AND METHODS

2.1 Materials

Commercially available matrix system consists of epoxy resin, and corresponding epoxy hardener. The epoxy resin, and hardener were supplied by Wisma Justus Jl. Danau Sunter Utara No. 27-28 Blok O3, Jakarta 14350-Indonesia. This epoxy system was preferred due to its low viscosity, which makes it suitable for vacuum chamber degassing method processing.

The calcium carbonate (CaCO$_3$) micro-reinforcements (particle size 14 $\mu$m, purity 98.5%) were purchased from Alfa Kimia, Jl. C Simanjuntak No.12, Terban, Gondokusuman, Kota Yogyakarta, Daerah Istimewa Yogyakarta 55223.

Salak fiber (SF) is a kind of the natural fiber, it was front obtained from Salak tree (salacca-zalacca) agriculture in Sleman, Yogyakarta, Indonesian.

2.2 Manufacturing Method

The Salak tree is a long, hard, shiny plant fiber that can be spun into coarse, strong threads. The Salak stalks are cut off close to the ground 1000 mm from the base point. The stalks are soaked in fest water for 3 weeks. This process softens the tissues, permits the fibers to be separated, and the each Salak stalk was broken down into a single fiber. Single fiber fibrillation was done through several stages of work includes the selection for best fiber. The fibers are stripped from the nerve fibers cut to length 200 mm strands, and it is Aqaedest cleaning, then alkaline treatment with 2% NaOH for 6 hours at room temperature. Than lead it’s to hung up or spread on thatched roofs for drying in natural sunlight at least 4 days to remove moisture content.

Figure 1 Extraction Salak fiber

Table 1 The mechanical properties of SF

<table>
<thead>
<tr>
<th>Item</th>
<th>Unit</th>
<th>Untreated</th>
<th>Treated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>mm</td>
<td>350</td>
<td>300</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>MPa</td>
<td>160</td>
<td>271</td>
</tr>
<tr>
<td>Density</td>
<td>g/cm$^3$</td>
<td>0.64</td>
<td>1.12</td>
</tr>
</tbody>
</table>

2.3 Preparation of CaCO$_3$/Epoxy Resin Micro-Composites

The samples prepared by mixing (0, 2, 4, 6, and 8 vol%) of micro-CaCO$_3$ particle, they were designated as (C-0, C-2, C-4, C-6, and C-8). The epoxy resin was mixed with varying
amount of CaCO$_3$ micro-particle. The mixing of CaCO$_3$-epoxy was done by manually technique for 10 minutes approximately. After thorough mixing, used Vacuum Chamber to remove bubble, air, and oxygen form mixture of resin for 30 minutes. After degassing, mixture was poured into rectangular steel mould, and curing was done in the room temperature for 12 hours. The samples were then taken out from the mould after hardening. The samples of CaCO$_3$/epoxy composites were done mechanical properties by tensile test to find out the best composition. It was found that the composition of 4vol% CaCO$_3$-epoxy showed the optimum tensile strength.

2.4 Preparation of Composite

SF were cut in the length of 200 mm, and made into layer. Fabrication of different composite sheets manufactured by hand lay-up method. Epoxy resin, and hardener were mixed in ratio 1:1 by weight as recommended. The rectangular steel mould with size dimension (200 × 180 × 32)mm was used to prepare the composites. Five different types of SF content (0, 10, 20, 30, and 40 vol%) has been fabricated with the matrix of 4vol% CaCO$_3$. Each SF composite has different percent volume has been designated as (C00, C10, C20, C30, and C40) respectively. Release agent wax was used to facilitate the easy to removal of composite from the mould. In the composite prepare curing was done under the 5 MPa for 12 hours. After 12 hours the composite was cut according to ASTM stranded for tensile test. The nomenclature is shown in Table 2.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Composition units in (vol%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C00</td>
<td>00 SF + 96 E + 4 CaCO$_3$</td>
</tr>
<tr>
<td>C10</td>
<td>10 SF + 86 E + 4 CaCO$_3$</td>
</tr>
<tr>
<td>C20</td>
<td>20 SF + 76 E + 4 CaCO$_3$</td>
</tr>
<tr>
<td>C30</td>
<td>30 SF + 66 E + 4 CaCO$_3$</td>
</tr>
<tr>
<td>C40</td>
<td>40 SF + 56 E + 4 CaCO$_3$</td>
</tr>
</tbody>
</table>

2.5 Mechanical Testing
2.5.1 Tensile Test

Tensile test was carried out with the five separate specimens for test, and the average value was report. Tensile testing was performing on the SERVOPULSER machine in Materials Laboratory Department of Mechanical, and Industrial Engineering, Universitas Gadjah Mada, Yogyakarta, Indonesia. The uniaxial tensile test was carried out using the machine with a load capacity of 2000 kgf, and 0.5mm/min cross-head speed. The tensile test was performed according to ASTM D638-14 standard, specimens dimension was (165 × 190 × 3.2) mm with gauge length 50 mm [11]. The shape of tensile testing was double-dumbbell as shown in Figure 2. According to this Figure it should be noted that the fracture tips are progressively non-uniform with increasing amount of SF in the composite. In particular, loosen SF can be observed in association with longitudinal rupture due to crack propagation at the SF/matrix interface.
3. RESULTS AND DISCUSSION

3.1 Tensile properties of micro-composite

For tensile strength evaluation, there were two types of composites i.e., the firstly CaCO$_3$/epoxy resin, and SF reinforced CaCO$_3$/epoxy resin hybrid composites.

The typical of stress-strain curves for CaCO$_3$/epoxy, and SF/CaCO$_3$-epoxy micro-particle are given in Figure 3. The results of modulus of elasticity, the ultimate tensile strength, and yield strength of CaCO$_3$/epoxy are shown in the Table 3. The initial relatively steep part of the curve represents elastic behavior, and the slope of the curve defines the elastic modulus. Using a sharp pencil, and a ruler, by carefully draw a straight line through as much of the straight portion of the curve as possible, extending it from the bottom of the chart. This straight line is referred to as the modulus-line, from which the modulus of elasticity was calculated for the material tested. The curves of CaCO$_3$ micro-reinforced epoxy resin micro-composites (without Salak fiber) have been shown in Figure 3a. As seen in this Figure, the addition of CaCO$_3$ micro-reinforcements increases the tensile strength of CaCO$_3$/epoxy resin, its maximum at 4 vol% CaCO$_3$ content as 42.35 MPa with 18% increase compared to neat epoxy resin. In addition, due to the physical interactions between the epoxy resin, and CaCO$_3$ micro-reinforcements, the tensile strain at fracture, and Young's modulus also increase with increasing of CaCO$_3$ micro-particle content. Tensile strain, and Young’s modulus of the prepared micro-composites are calculated as 10% at 207 MPa for 4vol% of micro-reinforcement increments respectively, compared to neat epoxy resin. Further, for the Figure 3b demonstrates stress-strain curves for CaCO$_3$-epoxy/SF micro-composites. While the trend is similar for that of the tensile strength epoxy resin composites, the addition of 4vol% CaCO$_3$ micro-reinforcements constant within SF/epoxy composites that results in a drastic increase in the tensile properties. The drastic increase in tensile strength of 10vol% SF hybrid composites is found as 16%, while the composites containing 40vol% SF exhibit tensile strength of 134.49MPa, which is almost double increase compared to the control samples. The maximum tensile stress value of the SF/CaCO$_3$-epoxy hybrid composites was 134.49MPa for 40vol% SF loading. When SF reinforced composites are subjected to load, the fibers act as carrier of load, and stress is transferred from the matrix along the SFs leading to effective, and uniform stress distribution, which results in a hybrid composite having good mechanical properties [12].
Based on the results, the average values by tensile properties are listed in Table 4 for the different volume fraction of SFs reinforced CaCO$_3$/epoxy hybrid composites.

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>C-0</th>
<th>C-2</th>
<th>C-4</th>
<th>C-6</th>
<th>C-8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>MPa</td>
<td>35.94</td>
<td>36.41</td>
<td>42.35</td>
<td>37.92</td>
<td>25.19</td>
</tr>
<tr>
<td>Yield Strength</td>
<td>MPa</td>
<td>5.62</td>
<td>6.17</td>
<td>9.17</td>
<td>7.60</td>
<td>4.93</td>
</tr>
<tr>
<td>Strain</td>
<td>(mm/mm)</td>
<td>0.048</td>
<td>0.050</td>
<td>0.060</td>
<td>0.056</td>
<td>0.44</td>
</tr>
<tr>
<td>Tensile Modulus</td>
<td>(GPa)</td>
<td>0.122</td>
<td>0.125</td>
<td>0.156</td>
<td>0.140</td>
<td>0.109</td>
</tr>
<tr>
<td>Real Density</td>
<td>g/cm$^3$</td>
<td>0.88</td>
<td>0.90</td>
<td>0.92</td>
<td>0.91</td>
<td>0.79</td>
</tr>
<tr>
<td>Void Fraction</td>
<td>%</td>
<td>2.09</td>
<td>3.19</td>
<td>2.69</td>
<td>3.05</td>
<td>4.64</td>
</tr>
</tbody>
</table>

Table 4 Tensile test results of SF reinforced CaCO$_3$/epoxy hybrid composites.

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>C00</th>
<th>C10</th>
<th>C20</th>
<th>C30</th>
<th>C40</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>MPa</td>
<td>42.35</td>
<td>57.78</td>
<td>69.84</td>
<td>104.65</td>
<td>134.49</td>
</tr>
<tr>
<td>Strain</td>
<td>mm/mm</td>
<td>0.200</td>
<td>0.108</td>
<td>0.112</td>
<td>0.114</td>
<td>0.116</td>
</tr>
<tr>
<td>Tensile Modulus</td>
<td>GPa</td>
<td>0.215</td>
<td>0.547</td>
<td>0.627</td>
<td>0.956</td>
<td>1.175</td>
</tr>
<tr>
<td>Real Density</td>
<td>g/cm$^3$</td>
<td>0.92</td>
<td>0.99</td>
<td>1.03</td>
<td>1.06</td>
<td>1.08</td>
</tr>
<tr>
<td>Void Fraction</td>
<td>%</td>
<td>2.69</td>
<td>4.01</td>
<td>3.96</td>
<td>3.88</td>
<td>3.29</td>
</tr>
</tbody>
</table>

Figure 3 Tensile stress-strain curves of micro-composites prepared with different micro-particle addition: a) effect of CaCO$_3$/epoxy resin micro-composites, b) SF/CaCO$_3$-epoxy resin laminated hybrid composites.

The average values of the tensile strength, and elastic modulus are listed in Table 3, and Table 4 for the matrix composites are plotted in Figure 4 as a function of the volume fraction of SF, and CaCO$_3$. In this figure it is important to notice that the introduction of SF increases the strength of the epoxy matrix. In fact, values of strength above 134.49 MPa were
found for 40% of fiber volume fraction, while the elastic modulus that characterizes the stiffness of the composite reached a value above 1.175 GPa. This indicates that the SF acts as an effective reinforcing phase for hybrid composites.

Figure 4 Effects of precipitate micro-CaCO$_3$ content on tensile strength of SF/CaCO$_3$-epoxy composites: a) CaCO$_3$/epoxy composite, b) SF/CaCO$_3$-epoxy hybrid composite.

4. CONCLUSIONS

In this study, epoxy matrix modification has been performed by adding a different ratio of CaCO$_3$ micro-reinforcements to reveal their effect on tensile properties of micro-composites. Experimental results have shown that the mechanical performances of micro-composites were remarkably enhanced with 4vol% mico-CaCO$_3$ particle adding. Mechanical performance enhancements were associated with the toughening mechanism. From the experimental tensile strength of SF reinforced CaCO$_3$/epoxy hybrid composites containing 4vol% CaCO$_3$ micro-particles was maximum at 134.49 MPa with 40vol% SF, and young's modulus was 1.175 GPa respectively.

The results of present work have indicated that 4vol% CaCO$_3$ micro-particles added micro-composites are a potential candidate as structure in SF reinforcements due to its appropriate properties.

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REFERENCES


