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Mechanical Properties of Coconut Shell Reinforced Unsaturated Polyester Composites with the Presence of Titanium Dioxide as Additional Filler

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Abstract. Unsaturated polyester (UPE) composites filled with coconut shell (CS) powder was prepared by compression molding technique. To improve the mechanical and physical properties of CS-UPE composites, titanium dioxide (TiO₂) filler was added. CS also undergo two types of chemical treatment which were alkaline (NaOH) and silane treatment. Fourier transform infrared (FTIR) was used to investigate the changes of fibers chemical constituents after the two chemical treatments have been done. CS-TiO₂-UPE composites was characterized with physical testing (density, water absorption and thickness swelling), mechanical properties (tensile and flexural test) and scanning electron microscopy (SEM). Overall, the result shows that, the presence of CS and TiO₂ filler in UPE composites demonstrated the highest density. For water absorption and thickness swelling study, it was observed that the treated CS-UPE composites showed lower water absorption properties in comparison to those of untreated CS-UPE composites. The presence of TiO₂ filler showed an increased in those water absorption properties but still low compared to CS filled UPE. Both chemical treatment of CS caused a significant increase in the tensile and flexural properties. The presence of TiO₂ filler demonstrated a significant improvement in modulus properties but at the expense of strength properties. SEM investigation has shown that the surface modification of CS has better fiber-matrix interaction. Thus, chemical treatments on the CS improve fiber/matrix adhesion of UPE composites.

1. Introduction

Composite materials are one of the most influence materials used in industry applications such as automotive industry, building and construction industry and packaging industry besides metals, ceramics and polymers. Composites are known as the combination of two or more types of materials to give balance and at the same time enhance the properties such as physical and mechanical properties of the materials.



Biocomposite is a types of materials which involve the combination of reinforcement and matrix which one or both are from biological origins [1]. For examples, fibers such as rice straw, kenaf, banana, pineapple, oil palm, jute and sisal reinforced to polymer matrix. The use of biocomposites in industries are quite popular among the manufacturers as the reinforcement/fiber is easy to find, can be renewable, eco-friendly and save the operating cost. In this study, biocomposite will be fabricated by using a combination of coconut shell (CS) as reinforcement, titanium dioxide (TiO_2) as the additional filler and unsaturated polyester (UPE) as the polymer matrix.

Coconut (*Cocos nucifera*) are came from the *Arecaceae* of family (palm family) which is known as one and the only species of genus *Cocos* [2]. In making the biocomposite materials, there are several parts of coconut that can be used as reinforcement such as shell and coir. Coconut shell is known as one of the most important reinforcement produced in tropical countries such as Malaysia, Indonesia, Thailand, Sri Lanka and India and it is widely used in making biocomposite. This is because of high specific strength and modulus properties [3,4]. According to [5] CS reinforced composites showed 80% better elongation at break and 20% better Charpy impact strength than soft wood composites [6,7].

Due to hard-wearing quality and high hardness which is not fragile like glass fiber, good acoustic resistance, moth-proof, not toxic, resistant to microbial and fungi degradation, and not easily combustible, the coconut particles are remarkable interest in the automotive industry [8,9]. Coconut shells reinforced matrix composites are classified in structural biocomposite which can be defined as one that need pressure to carry load in use because of its high compressive strength can be used in broad range applications of load bearing [10]. Titanium dioxide (TiO_2) is one of the additional filler besides calcium carbonate (CaCO_3) and aluminum oxide (AlO_3).

TiO_2 has been used since decades due to the speciality which can increase the stiffness and the modulus of the composite. TiO_2 will be added into the combination of CS and UPE as an additional filler. UPE resins are used widely in the composites industry besides phenolic resins and epoxy resins since UPE are not only good in mechanical properties, they are also low cost and easily use [11]. Combinations of the UPE with natural fillers, such as coconut shells, are believed to improve the physical and mechanical properties of the composites.

2. Experimental setup

2.1 Mechanical Testing

Tensile test was conducted towards the composite material samples to measure the length of the test specimens. The specimen was cut into suitable size according to ASTM D-638-I for tensile test. This experiment was conducted by using Instron universal testing machine with its cross of speed of 5 mm/min. From this testing, the volume fraction and the average value of the composite material samples was taken for analysis.

$$\text{Tensile strength} = \frac{\text{maximum load force (F)}}{\text{surface area (A)}} \quad (1)$$

$$\text{Tensile modulus} = \frac{\text{stress}}{\text{strain}} \quad (2)$$

Three-points bending flexural test was known as the test which conducts according to the ASTM D-790 standard to produce flexural strength and flexural modulus. The composite samples was tested at a cross head speed of 3 mm/min with the temperature of 25 ± 3 °C and the humidity of 50 %. Average value of the samples was recorded. The flexural strength in a three-point bending was calculated by using equation as follows.

$$\sigma_{\max} = \frac{3P_{\max}L}{bh^2} \quad (3)$$

Where, P_{\max} is the maximum load at failure (N), L is the span (mm), b and h is the width and thickness of the specimen (mm) respectively. The flexural modulus was calculated from the slope of the initial portion of the load-deflection curve by using equation as follows.

$$E = \frac{mL^3}{4bh^3} \quad (4)$$

Where, m is the initial slope of the load deflection curve for each stacking sequence, the samples was tested and average result was obtained.

2.2 Scanning Electron Microscopy

The morphology of the tensile fracture surface of the sample composites was carried out by using a scanning electron microscope (SEM) model Jeol JSM-IT100. SEM was used to analyse the interfacial adhesion between matrix and reinforcement of the damaged composites after conducted the tensile testing.



Figure 1. Samples for Scanning Electron Microscopy (SEM).

The composite sample was cut into a small pieces as shown in Figure 1 so that can be placed onto the pellet. SEM was operated at an acceleration voltage of 10 kV. To collect SEM images of the fracture surfaces of the tensile samples, a magnification ranged from $\times 25$ to $\times 350$ was used. The overall process of the preparation and characterization of CS-TiO₂-UPE composite.

3. Results

3.1 Mechanical Properties of CS-UPE Composites

3.1.1. Tensile and Flexural Strength

According to [12], the tensile of polymer composite increased with the addition of CS content. This is due to CS contain hard lignocellulose which give better strength to the composite [13]. Same goes to the flexural strength, which increasing with the addition of CS content.

With the same amount of CS content, the silane treated CS composite, UCS and alkaline treated CS composite, UCN show increase in tensile strength compared to untreated CS composite, UC. The reason is due to both of alkaline and silane treatments give better strength to the reinforcement. Alkaline treatment removed the impurities such as wax, oil, lignin and improves the rough surface. Meanwhile, silane treatment give smooth surface of the composite. For flexural strength, UCS exhibits lower flexural strength compared to UC while UCN exhibits higher flexural strength compared to UC.

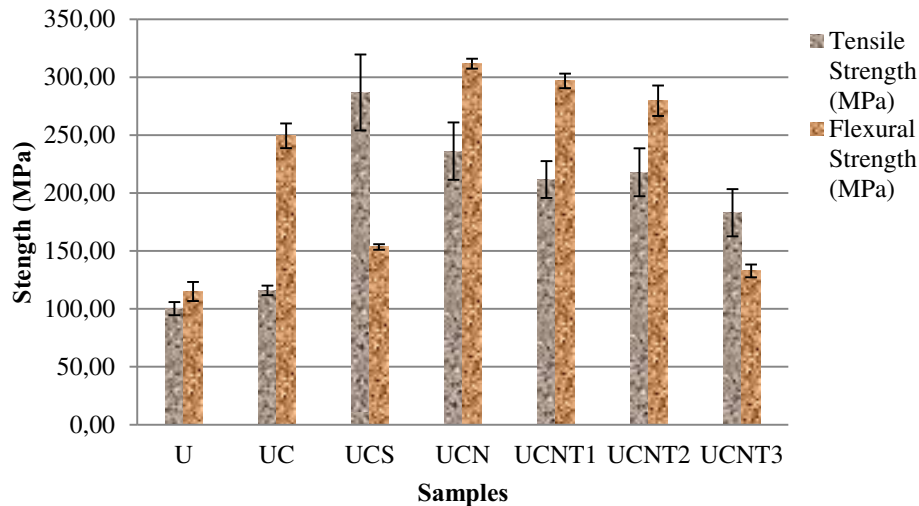


Figure 2. Tensile and flexural strength of UPE composites

With the same amount of CS content and the same treatment method which is alkaline treatment, the tensile strength of UCNT1, UCNT2 and UCNT3 shows different. UCNT2, which contain 1 wt.% TiO_2 filler become the highest tensile strength among the three followed by UCNT1, 0.5 wt.% TiO_2 and UCNT3, 1.5 wt.% TiO_2 respectively. This indicates that UCNT3 is the best percentage of filler used because the tensile strength decreased as more filler are added. Meanwhile, the flexural strength of UCNT1, UCNT2 and UCNT3 are decrease respectively when TiO_2 filler are added. UCNT1 which contain 0.5 wt.% of TiO_2 is the highest flexural strength, followed by UCNT2 which contain 1 wt.% TiO_2 and UCNT3 which contain 1.5 wt.% TiO_2 . This indicates that the flexural strength decreased as the filler are added.

3.1.2. Tensile and Flexural Modulus

Figure 3 shows the graph of tensile and flexural modulus of the 7 composite specimens. The tensile modulus of U, UC, UCS, UCN, UCNT1, UCNT2 and UCNT3 are 0.51 GPa, 0.60 GPa, 0.96 GPa, 0.77 GPa, 1.21 GPa, 1.25 GPa and 1.38 GPa respectively.

Based on the graph, UC exhibits higher tensile modulus compared to U. This is due to the addition of CS as the reinforcement in the UC composite compared to U which only contain neat UPE. However, with the same amount of CS content but with different types of treatments, both UCS and UCN which undergo silane treatment and alkaline treatment respectively show lowered tensile modulus compared to UC which contain untreated CS. By comparing the two composites with the treatment, UCS exhibits higher tensile modulus compared to UCN. The graph also indicates that the tensile modulus of CS composite become higher as more filler is added. The flexural modulus of U, UC, UCS, UCN, UCNT1, UCNT2 and UCNT3 are 0.54 GPa, 4.28 GPa, 1.41 GPa, 8.03 GPa, 6.09 GPa, 3.58 GPa and 1.45 GPa respectively. Based on the graph, flexural modulus become higher as the CS was added. This is due to UC exhibits higher flexural modulus compared to UPE. UCS shows lower flexural modulus compared to UC but UCN exhibits higher flexural modulus compared to UC. With the same amount of CS content and also same treatment to CS, the graph shows the tensile modulus of CS composite increase as more TiO_2 filler are added.

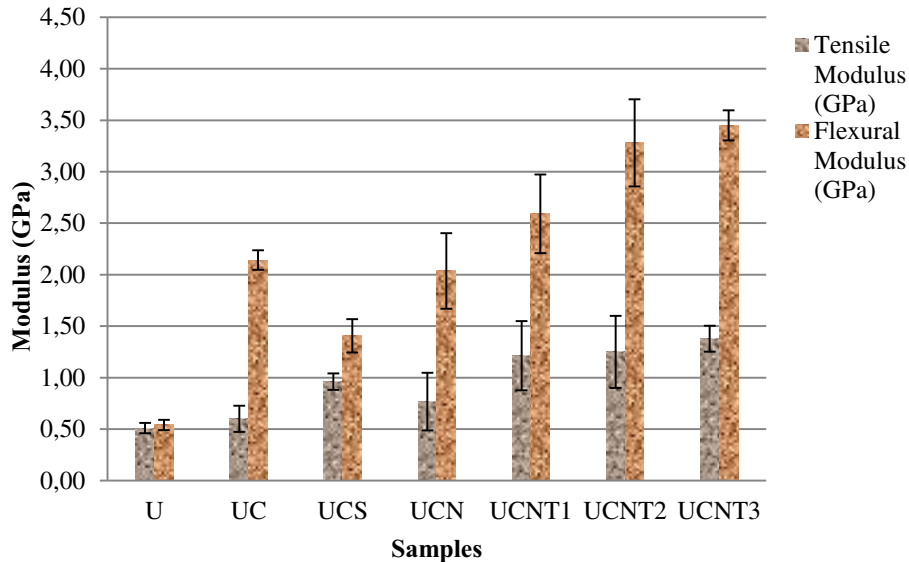


Figure 3. Tensile and flexural modulus of UPE composites.

3.2 Scanning Electron Microscopy

3.2.1. Morphological analysis of CS-TiO₂-UPE Biocomposites

The Scanning electron microscope was used to examine the tensile fracture surface of neat UPE, CS-UPE and CS-UPE-TiO₂ composites. The SEM micrograph of the tensile fracture surface of neat UPE is shown in Figure 4.7. The neat UPE composite has been represented as the control sample. The micrograph exhibits the homogenous surface and matrix tearing of UPE.

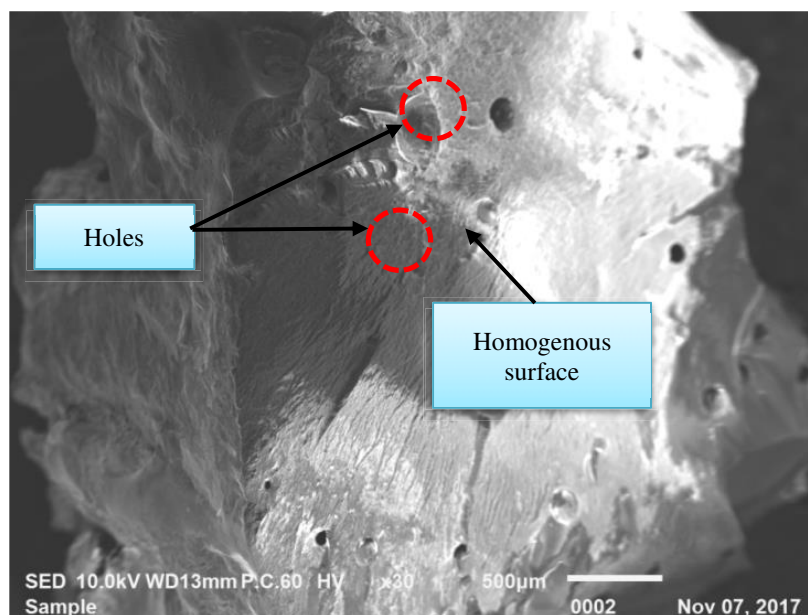


Figure 4. Scanning electron micrograph of tensile fracture surface of neat UPE composite.

As 30 wt.% CS is being added, there show the different between these two types of composites. SEM shows rough fracture surface and detachment of CS from matrix. This indicates the poor wetting and interfacial adhesion between CS and UPE matrix. The holes are the evidence weak bonding between CS and UPE matrix. The impurities observed on the surface of UC composite can act as crack initiators, thereby lessening the strength of the UPE matrix.

4. Conclusions

Mechanical properties consist of tensile and flexural strength and also tensile and flexural modulus. Based on the graph, tensile and flexural modulus increased as the CS was added and also after the chemical treatment. However, tensile and flexural modulus slowly down as TiO₂ filler addition increased. For tensile and flexural modulus, the modulus increased when CS content was added. Nevertheless, after the chemical treatment, the tensile and flexural modulus slightly decreased but increased back after the addition of TiO₂ filler. Morphology studies demonstrated the defects such as holes, crack and agglomerations which contributed to the reduction of mechanical properties such as tensile and flexural strength.

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