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Mechanical and Microstructural Properties of Hybrid Bio-Composites using Microwaved Coconut Fibre and Rice Husk

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Mechanical and Microstructural Properties of Hybrid Bio-Composites using Microwaved Coconut Fibre and Rice Husk

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Abstract. The outstanding mechanical and environmental qualities of hybrid bio-composites have made them popular. The drying procedure to remove the moisture before manufacture, on the other hand, can take a long period. By modifying the macromolecular structure considerably faster, hybrid bio-composites with additional physical treatment utilising microwave energy could improve their mechanical capabilities. Fillers of 80:15:5, 90:5:5, and 98:1:1 coconut fibre and rice husk were combined with poly-lactic acid (PLA) utilising melt-mixing and hot press techniques. The fillers were dried in a conventional oven at 60°C for 24 hours and in a microwave oven at 2.45 GHz for 3 minutes. When tensile strength was tested, it was discovered that oven-treated fibres with a 98:1:1 composition had a higher tensile strength (63 MPa) than microwave-treated fibres (58 MPa). Microwave-treated fibres, on the other hand, had a higher flexural strength (69 MPa) than those treated in a normal oven (60 MPa). Furthermore, when compared to plain PLA, microwave energy enhanced the toughness of the bio-composites by at least 4%. For the 80:15:5 composition, microwave-treated fibres had a lower water absorptivity (2%) than conventionally treated fibres, which had a water absorptivity of 5%. SEM images confirmed the presence of agglutination and voids with higher fibre content, resulting in poor adhesion and low tensile and flexural strength.

1. Introduction

A composite is a mixture of two or more different materials having different mechanical and chemical properties. Due to the rising use of single-use non-biodegradable plastics and their overwhelming quantity ending up in landfills in recent years, much research and studies on bio-composite materials have been conducted. This pollution is harmful to the environment, and it raises questions about the need for and alternatives to single-use plastics. Furthermore, the production and use of agricultural waste is a source of concern. There is no denying that when agricultural wastes are used properly and creatively, they may help to contribute to a circular economy.

Lignocellulosic Thermoplastic Composites is one of the research fields (LTCs). LTCs are made from agricultural waste and thermoplastic resin [1]. These fibre polymers are biodegradable, recyclable, and have high specific stiffness and strength [2]. Cotton [3],[4], rice husk [5],[6], sisal [7],[8], wood flour [9],[10], coconut coir [11],[12], coffee husk [13],[14], plantain [15],[16], and other agricultural residues have all been studied and researched in various ways. The goal of bio-composites research and development is to improve mechanical properties [17], increase heat resistance [18] and so on. Bio-composites are typically made using traditional methods, such as a conventional furnace for

drying. Depending on the volume of production, this procedure might take a long time, and the drying process alone can take up to 24 hours or longer to eliminate the moisture from the fibres.

Microwaves, which are electromagnetic waves, are found between infrared and radio waves in the electromagnetic spectrum, and their reaction is usually absorption, reflection, or transmission [2]. The microwave frequency range is between 108 and 1012 Hz. Aside from communication and radar, microwave frequencies are also used in medical, scientific, and industrial applications [2]. Microwaves are safe and capable of being employed as a medium in any connected research because they are used in everyday life. Furthermore, the traditional method of heating concentrates on the material's outside or surface, which is incompatible with microwave radiation and time-consuming. After being absorbed by the material, the microwave energy is transformed into heat, causing the material to undergo an even heating process, both internally and externally, at a faster pace and for a shorter time. As a result, the purpose of this experiment is to look into the mechanical and microstructural features of a rice husk and coconut fibre hybrid bio-composite that has been treated with microwave energy. To determine the qualities of the hybrid bio-composite, tensile, flexural, hardness and water absorptivity tests are performed.

2. Methodology

2.1Materials

Nature Works LLC (USA) provided a PLA pellet with grade 3052D, which has a mass flow index (MFI) of 14g/10 min and a density of 1240 kg/m3 or 1.24 g/cm3. As a matrix, this PLA pallet was used, with coconut fibre and rice husk from the local market serving as fillers. After washing with distilled water to eliminate contaminants, the coconut fibre and rice husk were sun-dried for 5 hours. To eliminate moisture from the PLA pallet, it was placed in a 40°C oven for 24 hours.

2.2 Treatment of composites

Using a regular oven and a microwave oven, coconut fibre and rice husk were subjected to two different physical treatments. Coconut fibre and rice husk were cooked at 60°C for 24 hours to eliminate moisture for the standard oven. We used a microwave with a power range of 100W to 1000W and a frequency of 2.45 GHz. While treating both coconut fibre and rice husk, the microwave was set at 700 W for 3 minutes at a steady frequency. The grinding technique was then applied to all conventional and microwave-treated composites. The grinder utilised was an AIRMAC Universal High-Speed Grinder (MDY-200) at a speed of 25000 rpm. Both treated composites were then sieved to achieve a particle size of fewer than 250 μm and firmly stored in airtight plastics.

2.3 Sample Preparation

PLA was dried at 40 degrees Celsius for 24 hours before being used in the hot press to avoid hydrolysis. PLA pallets were then dispersed in a dog-bone and rectangle mould that was readily available and conformed to ASTM D638 Type IV and ASTM D790, respectively, with a thickness of 3 mm, as illustrated in Figure 1. After that, the PLA was pre-heated in a hot press machine at 185°C for 6 minutes to melt it, and then pressed for another 5 minutes. After that, the mould was placed for a 5-minute cold press.

Figure 1. Dog-bone shaped mould according to ASTM D638

An electronic balance was used to weigh the PLA, sieved coconut fibre (CF), and rice husk (RH) after microwave and oven treatment. The weighted amounts of CF and RH are calculated using the ratio provided in Table 1. It is then combined before being placed in a melt mixing machine at 180 °C for 10 minutes with a rotor speed of 50 rpm for blending purposes. A crusher is used to reduce the grain's size. The little grains are then equally placed in the same dog-bone and rectangle shape mould as the clean PLA. It's then melted for 6 minutes at 180 °C before being pressed for 5 minutes. The pictures of the composite samples are shown in Figure 2.

Table 1. Composition of bio-composites

Composition PLA:CF: RH	**No of Samples** Microwave	Oven
80:15:05	5	5
90:05:05	5	5
98:01:01	5	5

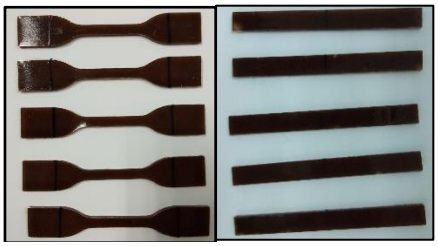

Figure 2. Dog-bone and rectangular shape of composites

2.4 Tensile Test

According to ASTM D638-04, a tensile test was performed using a SHIMADZU Universal Testing Machine (UTM) (PFG5K) with a maximum load of 5kN. A 5 mm/min crosshead speed was employed [19]. Five specimens were used to create an average measurement of tensile strength.

2.5 Flexural Test

Flexural testing is performed in accordance with ASTM D790 on the same machine as tensile testing (SHIMADZU -PFG5K). This 3 point bending test was performed at a crosshead speed of 2 mm/min with a span length of 60 mm between supports. An average of five samples was obtained.

2.6 Hardness Test

Vickers machine HV-1 (HUATEC, Beijing) is used to evaluate hardness in accordance with E384 standards. The goal of this test is to determine the composites' hardness after natural fibres have been added to PLA. In addition, each composite sample's hardness was averaged from three independent readings. Because the sample is a composite, a pressure of 100 kgf was applied for 10 seconds.

2.7 Water Absorptivity Test

Natural fibres like coconut fibre and rice husk are hydrophilic, whereas PLA is relatively hydrophobic [20]. As a result, the goal of this test is to determine the bio-composites' water absorptivity. This test was carried out in compliance with ASTM D570. The original weight of a rectangular shape sample measuring 125 mm x 12.7 mm x 3.2 mm was determined before it was placed inside a beaker of pure water. The specimen's final weight was determined after 24 hours. The water absorptivity (WA) is calculated as a percentage difference between the initial and final weight by using (1).

$$Water\ absorptivity\ (WA)(\%) = \frac{Final\ Weight\ (g)\ -\ Initial\ Weight\ (g)}{Initial\ Weight\ (g)} \times 100\% \quad (1)$$

2.8 Microstructural Analysis

After the tensile test, the fractured piece of the sample was examined with a scanning electron microscope (SEM) to assess the microstructure of the bio-epoxy composite samples. Palladium was then applied to the shattered section area to provide a better view of the microstructure. The SEM machine utilised was the JSM IT-100 (JEOL Ltd, Japan), which has a magnification range of up to 200 µm.

3. Results and Discussion

3.1 Tensile Properties

The overall average values of tensile strength, elongation at break, and Young's modulus of five samples are shown in Table 2. Figure 3 indicates that neat PLA has the highest tensile strength of all, while composites with a 98:1:1 mix have slightly lower tensile strength but are the closest to neat PLA. Furthermore, oven-treated composites with a 98:1:1 composition have a compressive strength of 63 MPa, which is 9% higher than microwave-treated composites. Higher radiation causes a decrease in crystallinity, which causes substantial damage to the microstructure and results in lower tensile strength, according to [2], which experimented with 750W microwaved sisal fibre.

Table 2. Results of Composites

Composition PLA:CF:RH	**Average 5 samples** Tensile Strength (MPa)	Elongation at break, %	Young Modulus, (GPa)
Neat PLA	74	3.86	2.24
MICROWAVE			
80:15:05	30	1.95	2.14
90:05:05	43	2.19	2.28
98:01:01	58	2.74	2.36
OVEN			
80:15:05	33	1.81	2.44
90:05:05	41	2.38	2.11
98:01:01	63	2.77	2.31

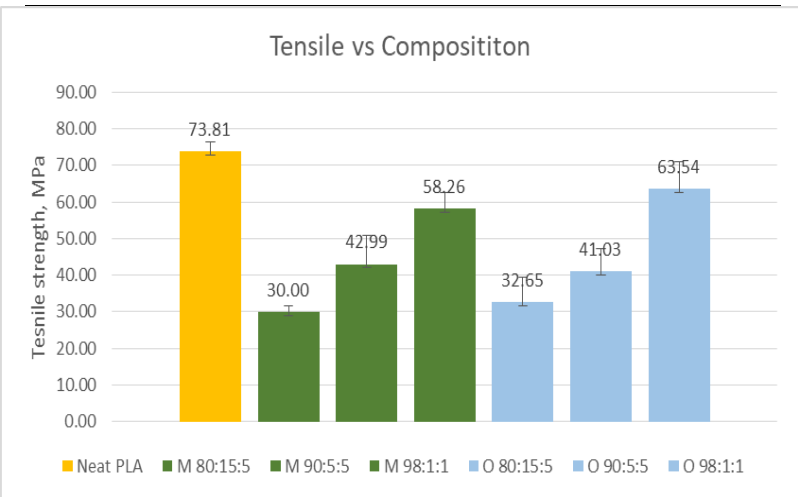

Figure 3. Tensile comparison according to composition

3.2 Flexural Properties

This test was used to assess the amount of stress caused by bending. Table 3 illustrates the average flexural strength for neat PLA and various composites based on five samples. In comparison to the oven-treated fibres, the hybrid bio-composite containing microwave-treated fibres has higher flexural strength, as shown in Table 3. Microwave-treated composite with a 98:1:1 ratio, for example, has an average value of 69 MPa, which is 21% lower than neat PLA but 15% higher than oven-treated with the same composition. In comparison to standard oven treatment, this data suggests that microwave treatment can enhance flexural strength. Figure 4 illustrates an increase in flexural strength utilising microwave radiation, as well as the toughness of the microwave-treated samples, which has been improved over the oven-treated samples. Furthermore, the modulus of elasticity of the hybrid bio-composite employing microwaved energy is 1.4-4.2 % greater than the oven-treated fibres for the three compositions, which is consistent with the rising flexural strength in microwaved fibre samples.

Table 3. Flexural strength of 5 samples

Composition PLA:CF:RH	**Average 5 samples** Flexural Strength, (MPa)	Modulus of Elasticity, (GPa)
Neat PLA	88	2.70
	MICROWAVE	
80:15:05	29	1.50
90:05:05	48	2.12
98:01:01	69	2.24
	OVEN	
80:15:05	28	1.47
90:05:05	45	2.09
98:01:01	60	2.15

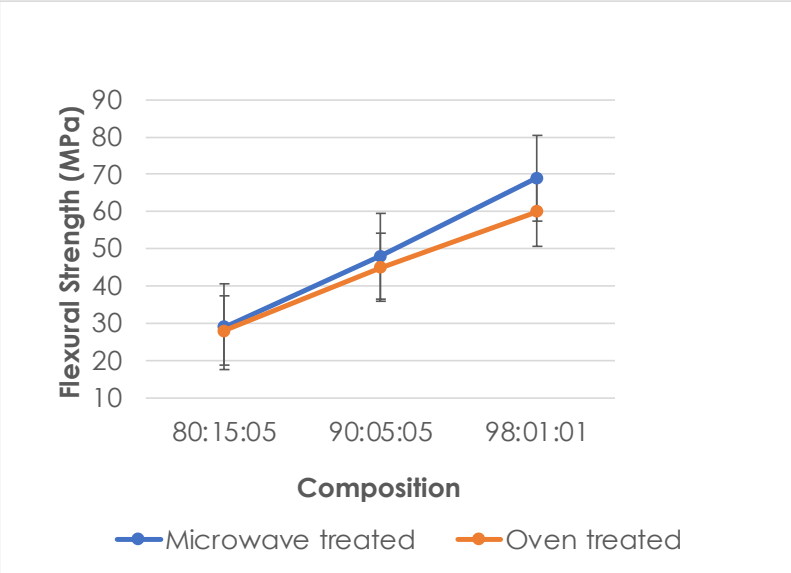

Figure 4. Flexural strength comparison for each composition

3.3 Hardness Test

Table 4 was used to record the results of the hardness test. The hardness of the composite reduced when the content of natural fibres, such as coconut fibre and rice husk, decreased, as shown in Figure 5. Tensile results on the brittleness of samples back up these findings. It reveals that the brittleness of the composites increased when more fibres were added as fillers. Furthermore, as demonstrated in Table 5, microwave-treated fibres in the hybrid bio-composite have a significantly different hardness value than neat PLA and traditional oven-treated fibres. This significant improvement implies that the hybrid bio-composites' microwave-treated fibres are more resistant to indentation when a load is applied. As a result, when parts are subjected to frictional stress, there will be reduced abrasion on the composite's surface.

Table 4. Hardness value for all compositions

Vicker's Test - Hardness (HV0.1)			**Composition PLA:CF:RH**		
Neat PLA	**Type**	**Sample**	**80:15:5**	**90:05:5**	**98:01:1**
18.10		**1**	21.40	19.70	19.70
18.30	**Microwave**	**2**	20.30	20.10	19.00
19.30		**3**	19.60	19.90	18.50
18.57	**Average**		20.43	19.90	19.07
		1	19.20	17.60	18.50
	Oven	**2**	19.70	19.30	18.90
		3	17.30	19.00	17.70
	Average		18.73	18.63	18.37

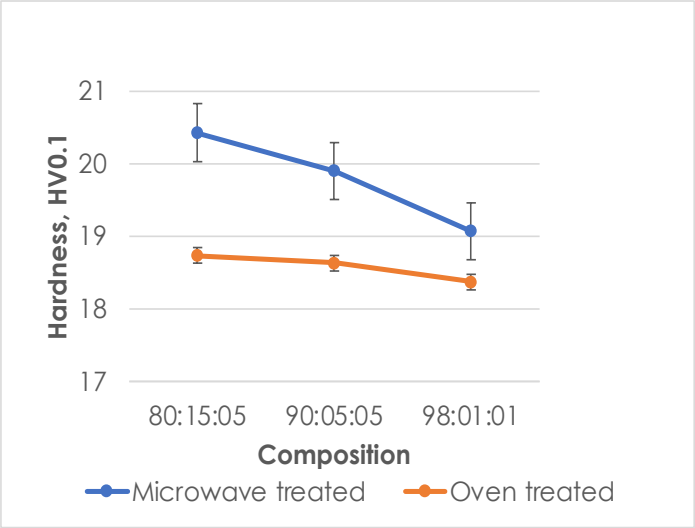

Figure 5. Hardness comparison for each composition

3.4Water Absorptivity

Natural fibre is hydrophilic, whereas PLA is hydrophobic. As a result, the water absorptivity (WA) value indicates the percentage of water absorbed by natural fibre when combined with PLA. The percentage value of WA after a 24-hour immersion is shown in Table 5. Because PLA has hydrophobic qualities, there are no weight differences between before and after immersion in distilled water. As a result, Farah et al. (2016) and Mokhena et al. (2018) found comparable results in their research [20],[21]. Table 5 demonstrates that the percentage of water absorbed is larger in all oven-treated composites, regardless of composition, than in microwave-treated composites, which show a small increase in final weight but have the least water absorptivity. Imoisili et al. (2018) observed that microwaves preserved the intrinsic hydrophobicity of materials in their plantain research [22]. As a result, the microwave-treated fibres in the hybrid bio-composite are appropriate and capable of being used to make disposable cutlery, disposable cups, and disposable plates as a replacement for single-use plastics.

Table 5. Water absorptivity result

Water Absorptivity		Initial Weight (g)	Final Weight (g)	Difference (g)	Average (g) (%)
Neat PLA	**1**	6.06	6.06	0.00	0.00 (0%)
	2	5.94	5.94	0.00	
	3	6.07	6.07	0.00	
80:15:05					
Oven	**1**	2.32	2.34	0.02	0.05 (5%)
	2	2.14	2.25	0.11	
	3	2.38	2.41	0.03	
Microwave	**1**	2.22	2.24	0.02	0.02 (2%)
	2	2.37	2.40	0.03	
	3	2.28	2.30	0.02	
90:05:05					
Oven	**1**	2.69	2.71	0.02	0.02 (2%)
	2	2.19	2.20	0.01	
	3	2.14	2.16	0.02	
Microwave	**1**	2.58	2.59	0.01	0.01 (1%)
	2	2.57	2.58	0.01	
	3	2.43	2.43	0.00	
98:01:01					
Oven	**1**	2.57	2.59	0.02	0.03 (3%)
	2	2.62	2.68	0.06	
	3	2.81	2.82	0.01	
Microwave	**1**	3.00	3.00	0.00	0.01 (1%)
	2	2.60	2.62	0.02	
	3	2.81	2.82	0.01	

3.5 *Microstructural properties*

The microstructure of neat PLA and all composites were evaluated using morphological observation on the cracked surface. To improve the photos and outcomes, all of the composites evaluated were first coated with palladium. Figure 6 indicates that neat PLA has a smooth surface with a slight indentation in the fractured surface, whereas (A) and (B), both of which contain 20% natural fibres, have severe agglutination on the PLA matrix regardless of treatment. Coconut fibre and rice husk, which are natural fillers, are readily visible. Furthermore, a big void was discovered in Figure 6 (C), which could be the source of low tensile strength for microwaves with a composition of 90:5:5. On the other hand, the identical composition treated with the oven shown in Figure 6 (D) has poor fibre dispersion and few voids. With only 2% of fibres, the microstructure of Figures 6 (E) and (F) resembled the picture of tidy PLA with few fibres. Both figures show a stacked-layer image with a uniform dispersion of fibres in the PLA matrix. Both figures show no evidence of fibre agglutination. This means that natural fibres were well-blended with PLA, yielding greater mechanical qualities than the other two compositions.

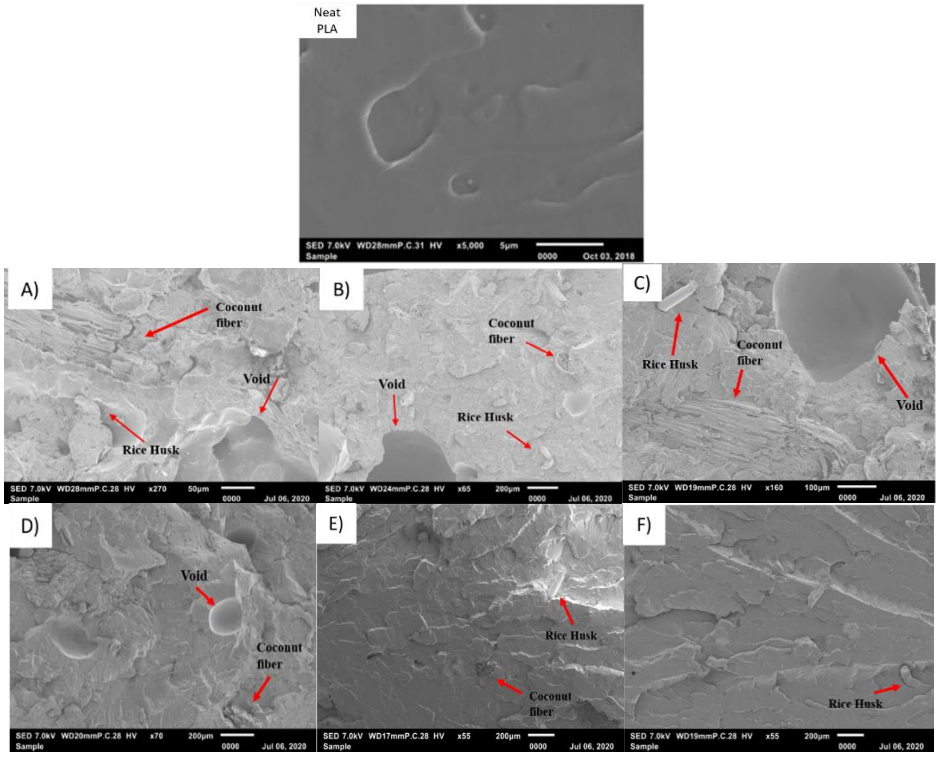

Figure 6. SEM Image of neat PLA, (A) M80:15:5 (B) O80:15:5 (C) M90:5:5 (D) O90:5:5 (E) M98:1:1 (F) O98:1:1

4. *Conclusion*

Firstly, the tensile and flexural strength of neat PLA was the highest among all with the strength of 74 MPa and 88 MPa. Next, microwave did the effect on the tensile strength of the hybrid bio-composite. From the result, it can be deduced that 2% wt of fibre for microwave-treated composite has a less tensile strength which was 58 MPa, reduced by 9% and 21% from oven treated and neat PLA, respectively. However, it differs for flexural strength. Microwave-treated composite has achieved higher flexural strength with the result of 69 MPa, improved by 12% from 60 MPa as achieved by oven-treated. Furthermore, adding fibres to both physical treatments lowered tensile and flexural

strength by up to 50%. SEM pictures confirmed that agglutination and cavities were discovered on a large percentage of fibres, independent of treatment, affecting the composite's strength indirectly. Furthermore, microwaves of 700 watts are thought to cause severe damage to fibre microstructure, resulting in poor adhesion and agglutination between PLA matrix and fibres. The addition of fibres enhanced the hardness of both microwave and oven-treated composites, resulting in a hardness of 20.43 HV0.1 for microwave and 18.73 HV0.1 for oven-treated composites. Microwave-treated fibres in the hybrid bio-composite, which are unaffected by fibre content, have a positive water absorptivity value of just 2%, compared to the oven, which recorded up to 5% absorption after 24 hours. This finding demonstrates that just 3 minutes of microwave heating changed the properties of fibres inside the composites. Thus, the mechanical properties of hybrid bio-composites are affected by microwave energy. The advantages of hybrid bio-composites using microwave treated fibres, such as enhanced flexural strength, hardness, and reduced water absorption could be considered a good alternative to replace single-use disposable cutlery, cups, and plates because PLA is completely biodegradable and harmless to the environment.

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