Manufacturing Engineered Wood Panels from Rice Husk Flake Reinforced with Glass and Carbon Fibers Using Epoxy Adhesive

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Rice husk flake (RHF) engineered wood panels were manufactured from unmodified rice husk flake in its original form and epoxy adhesive. One of the main objectives for using the original form of RHF was to minimize the manufacturing cost and processing time for treating the flake. Two types of short randomized reinforcements namely carbon fiber (CF) and glass fiber (GF) were employed to enhance the mechanical properties. The optimal compression pressure for the manufacturing process was 120 kg_f/cm^2. The short randomized CF and GF reinforcements were employed and investigated. An outstanding enhancement in mechanical performances was observed. At the given short fiber content, the CF showed superiority as reinforcement to the GF. The adhesive exhaustion was evidenced when the reinforcement loading exceed 40 wt%. In the combined CF/GF short fibers at constant 40 wt% loading, the mechanical performance was reduced by increasing the GF portion.

Keywords: Rice husk flake; Engineered wood; Mechanical properties; Carbon and glass fiber reinforcement

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INTRODUCTION

The development of green technologies is an important current issue for many industries with regard to environmental friendliness, resource sustainability, and human health. The utilization of wastes, especially agro-industrial byproducts, by conversion into high performance material products is an interesting challenge. Moreover, preserving the forest by reducing the harvest of trees is a benefit for all.

Lignocellulosic agricultural waste such as bagasse, rice husk flake, and palm oil fiber are abundant; they are the most valuable resources for innovating high value added products. Engineered wood and wood substituted products are in high demand for construction materials. Medium density fiberboard (MDF) is a traditional engineered wood produced made from lignocellulose fiber incorporated with adhesive and/or reinforced materials using hot-pressed processing. They are typically manufactured with a density between 450 and 800 kg/m^3 (Li *et al*. 2009; Yousefi 2009; Ali *et al.* 2014). The properties of MDF depend on its fibers and adhesive bonding. The adhesives are necessary to ensure effective bonding between the fibers. The most commonly used resins for MDF products are based on formaldehyde. They include urea-formaldehyde (UF), phenol-formaldehyde (PF), melamine-formaldehyde (MF), and melamine-urea-formaldehyde (MUF). The major drawbacks of using those adhesives are: (*i*) they contain harmful volatile organic compounds (VOCs), (*ii*) the limited supply of petroleum feedstocks for producing formaldehyde, and (*iii*) low resistance to moisture and insects (Li *et al.* 2009;

Nasir *et al.* 2013). Concerning VOCs, the European, North American, and some Asian countries have imposed banning regulations. Alternative adhesives such as epoxy-based or bio-based adhesives have been investigated. Not only are the epoxy adhesives considerably less toxic, but epoxy adhesive would also be beneficial for its resistance to moisture and insects (Meekum and Wangkheeree 2016; 2017a). Thermoset epoxy resin is not yet popularly used as adhesive in engineered wood. However, it is used in high performance composite manufacturing. It has excellent properties including good adhesion to many substances, high mechanical properties, and good resistance to moisture and chemical attack.

Rice husk (RH) has not yet been wildly used as a raw material for manufacturing particleboard or other engineered woods. Conventionally, it has been consumed as the household energy biomass feedstock, as it is an abundant byproduct of the rice milling. It represents 22 wt% of rice production (Ciannamea *et al.* 2017) and is vastly abundant in Asian countries. The unique characteristics of RH in comparison with other agricultural waste are its high silica contents (87 to97 wt% SiO_2), high porosity, light weight, and very high external surface area (Soltani *et al.* 2015).

For research and innovation aspects, using lignocellulose fibers as reinforcement for thermoplastic or thermoset polymer matrices involves a number of treatment steps. Common surface modifications of natural fibers include (*i*) chemical treatment (alkalization, acetylation, silane, *etc*.), (*ii*) physical-chemical treatment (solvent extraction), (*iii*) physical method (use of different rays or plasma, steam explosion, UV bombardment), and (*iv*) mechanical method by rolling and swaging (Satyanarayana *et al.* 2009; Jawaid and Abdul Khalil 2011; Gurunathan *et al.* 2015). These treatments are used to enhance the compatibility and adhesion between the lignocellulose fibers and polymer matrix. Compatibilizers or coupling agents such as silanes, maleated polypropylene (MAPP), and titanates are used to improve fiber/matrix interfacial adhesion (Jawaid and Abdul Khalil 2011).

The mechanical properties of rice husk fiber reinforced polyester composites have been reported (Wayan Surata and Krissanti 2014). The mechanical properties of rice husk fiber composites are improved by increasing the fiber fraction and fiber alkalization treatment. Rice husk/polyurethane foam material shows interesting sound absorption and thermal insulation applications (Buratti *et al.* 2018). Stronger natural fibers such as jute and wheat husk have been hybridized RH with to fabricate panel boards (Mavani *et al.* 2007). However, the best tensile strength is found only for the jute composite. Rice husk mixed with wood flour has been used for producing particleboard; the best mechanical and physical properties are found with 25% rice husk content and 9% adhesive content (César *et al.* 2017). Engineered fibers such as glass and carbon fibers improve the mechanical properties of the engineered wood (Gujjala *et al.* 2014). There is marginal improvement in mechanical properties of the jute/glass fibers/epoxy board. To achieve superior properties in a composite, the interfacial adhesion of fiber/matrix is improved, as measured by the mean interfacial shear strength (IFSS). Chemical and/or physical surface treatments of natural fibers improve the IFSS, but they are costly. Omitting these processing steps without diminishing the mechanical properties would be preferred, especially in engineered wood.

There are several reports on the effect of natural fiber surface modification/treatment on the final properties of biocomposite products. Silane-treated pineapple leaf fiber and kenaf show superior tensile properties and IFSS (Asim *et al.*

2016). The "layer by layer" (LBL) deposition of polyelectrolytes with combined branched polyethyleneimine and polyacrylic acid is an attractive, functional, and sustainable solution for the production of mechanically strong particleboards based on rice husk fiber (Battegazzore *et al.* 2017). Finally, the performance of the composite materials depend on fiber/adhesive contents, hybridized fibers ratio, fiber/matrix interfacial bonding, and the fiber structures. Weaving patterns have significant contributions to the composite products. In mechanical studies on the effect of weaving patterns *vs*. random orientations of banana, kenaf, and banana/kenaf fibers using polyester as adhesive, the tensile properties are improved with plain type compared with twill type (Alavudeen *et al.* 2015). Moreover, NaOH and sodium lauryl sulfate (SLS) treatments provide additional improvements in mechanical strength through enhanced interfacial bonding. The influence of jute and banana fibers forms on the hybridized woven fabric has a significant effect on mechanical behavior of its composite using polyester matrix (Rajesh and Pitchaimani 2017).

In this publication, the prime objectives were to minimize the processing cost and time on the fiber treatment, especially for the size reduction step, and to reduce environmental hazards from the chemical treatment of the rice husk flake. The untreated and original form of rice husk flake (RHF) was used as the main raw material for manufacturing the RHF engineered wood panels. Homemade and room temperature curing DGEBA-based epoxy resin with an amine hardener were used as adhesives. Randomized short unidirectional (UD) engineered fibers, carbon fiber (CF), and glass fiber (GF) were employed as reinforcements to enhance the mechanical properties of the RHF engineered wood.

EXPERIMENTAL

Materials

The materials used for manufacturing the rice husk flake (RHF) engineered wood panel were categorized into three categories; (*i*) in-house epoxy-based adhesive, (*ii*) untreated rice husk flake, and (*iii*) engineered fiber reinforcements. The in-house and room temperature cure epoxy resin was prepared by blending of the commercially available DGEBA, Novolac and aliphatic epoxy resins, namely, YD 127, YD 515, YDPN 631, and RD 108, respectively. The blending composition of the resin is given in Table 1. Those commercial resins were supplied from Aditya Birla Chemicals (Thailand) Ltd., Rayong, Thailand. The amine-based hardener was formulated from triethylenetetramine (TETA) and isophorone diamine (IPDA) at the weight ratio shown in Table 1. TETA and IPDA were available from Vista Co., Ltd., Bangkok, Thailand and BASF (Thai) Ltd., Bangkok, Thailand, respectively. The calculated **"phr"** between epoxy resin and amines hardener for the in-house epoxy formulation adhesive was 13. The epoxidized silane, Silquest®A187, at 3 phr with respect to 100 g of mixed resin was added as coupling agent. Silquest®A187 was purchased from Optimal Tech Co. Ltd. (Thailand), Bangkok, Thailand. The rice husk flake was obtained from a local rice mill, Nakhon Ratchasima, Thailand. The solid and dust impurity on the RHF was removed by sieving, mesh size#4, and then gently air blown. It was used as the original form, with no chemical treatment and size reduction. In the manufacturing of the RHF engineered wood panel, the residual moisture on RHF was eliminated by vacuum drying at 105°C for 4 h. CF and GF, both

UD and woven forms, were employed as engineered fibers reinforcement for the RHF engineered wood panel. The discontinuous UD, CF, and GF were obtained from the woven waste available at windsurf manufacturer (Cobra International Co., Ltd, Chonburi, Thailand). The woven waste was chopped into short UD form having the approx. fiber length of 2 to 8 mm by a high speed cutting machine (FBC-20, Chareon Tut Co., Ltd., Samutprakarn, Thailand). Figure 1 shows the optical and SEM photographs of the original rice husk flake form, CF and GF reinforcements, respectively. The chopped CF and GF had the average diameters, measured by SEM image metering system, at 8.80 and 6.50 μm, respectively (Meekum and Wangkheeree 2016). The plain weave CF and GF fabrics having the aerial density of 100 g/m^2 and 160 g/m^2 were employed, respectively. They were kindly supplied from Cobra International Co., Ltd. The woven with dimension of 20 x 20 cm^2 was scissor cut prior to use as laminated reinforcement. All of the engineered fibers, regardless to forms and types, were used without surface treatment and sizing removal.

Table 1. Formulation of in-house Epoxy Adhesive Formulation

Resin		**Hardener**	
Reagents	**Weight(g)**	**Reagents**	**Weight(g)**
YD127	100	TETA	100
YD515	100	IPDA	10
RD108	20		
YDPN631	5		
phr = 13			

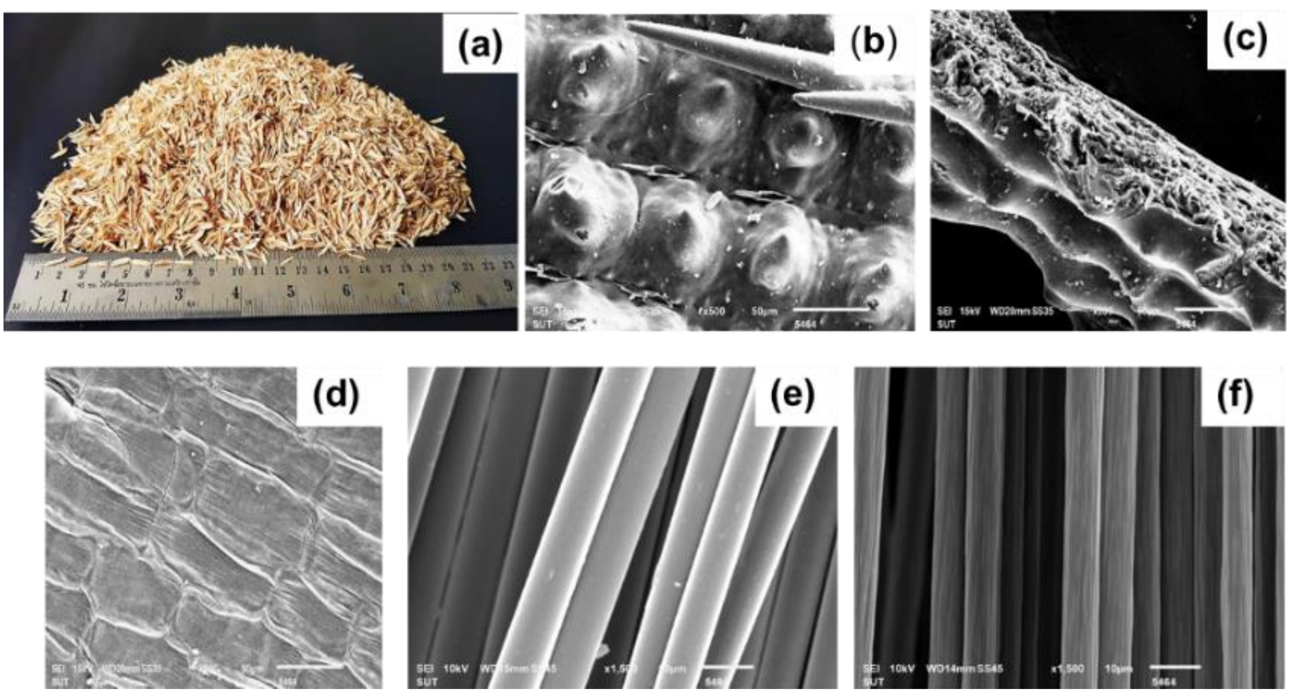

Fig. 1. (**a**) Optical photo of the original rice husk flake form (RHF) and SEM images of; (**b**) Outer Husk or Lemma (**c**) Husk cross section and (**d**) Inside Husk or Palea, (**e**) glass fiber(GF) and (**f**) carbon fiber(CF).

Manufacturing of the Rice Husk Flake (RHF) Engineered Woods

The RHF engineered wood panel samples were manufactured by compression molding. A two plate mold with the cavity dimension of 20 x 20 cm^2 and depth of 0.50 cm was employed, resulting in a calculated mold volume of 200 cm^3. A total weight of 220 g molding ingredients was prepared by vigorously mixing 163 g of the rice husk with/without engineered UD fiber(s) and 67 g of mixed epoxy adhesive in a high speed mixer for 2 min. The weight ratio of ingredients for manufacturing the RHF engineered wood in this work is presented in Table 2. This ingredient is adequate to 35 part of adhesive with corresponding to 100 part of rice husk flake plus fiber(s). The final calculated density of the obtained RHF wood was 1.10 g/cm^3. In the consolidated molding pressure was manually adjusted *via* the hydraulic valve to give the read-out pressure at 60, 90, 120 and 140 kg_f/cm^2, respectively. The hot compression machine, GT-7014-A30 (GoTech Testing Machine Inc., Taiwan) was employed. The compression temperature was set at 120 °C. The press/decompress/press molding cycle at 180, 30, and 120 seconds was adopted. The cured RHF wood panel was cooled down, demolded, and annealed at room temperature overnight. The standard size specimens were saw cut and edge polished. All test specimens were post cured at 60 °C for 12 h before testing. In the manufacturing of the RHF wood reinforced with short single fiber or hybridized fibers, the UD, CF, and/or GF at 10, 20, 30 and 40 wt% ratios, with respect to rice husk, were carefully weighed. The rice husk/fiber(s)/epoxy adhesive mixing and compression molding procedures were repeatedly performed as described before. Figure 2 shows the schematic drawing of the RHF engineered wood panels without and with short fiber reinforcement, respectively.

Table 2. Typical Rice Husk and Adhesive Ingredient for Manufacturing the RHF Wood

RHF/Fibers (g)	Resin (g)	Hardener (g)	Density(g/cm^3)
163.0	50.5	6.5	1.1

Fig. 2. Schematic drawing of the RHF engineered wood specimen; (**a**) unreinforced and (**b**) RHF/short UD Fiber(s), respectively

Standard Testing

The mechanical properties including three-point flexural bending, tensile, and Izod impact strengths(both notched and unnotched modes) testing were measured in accordance with ASTM D790-10 (2010), ASTM D638 (2014), and ASTM D256-10e1 (2010), respectively. The universal testing machine (Instron Model 5565, Norwood MA, USA) with load cell of 5 kN was employed. The flexural span length at 80 mm and crosshead speed of 15 mm/min were assigned. For the tensile testing, the strain rate at 15 mm/min by mean of the crosshead speed was also electronically controlled. The pendulum impact testing machine (Instron Ceast Model 9050) equipped with striking impactors were employed. The impactor having the striking energy of 2.16 and 11.0 Joules were used for notched and unnotched impact strength measurements, respectively.

The heat distortion temperature (HDT) at standard load of 1820 kPa were performed in accordance with ASTM D648-07 (2007). The durability of the RHF wood samples by mean of % water absorption (%WA_i), % thickness swelling (%TS_i) and dimension stability after removing of the moisture or % thickness swelling after vacuum drying (%$TS_{dried,i}$) during 1 and 7 day water submersion periods, were measured in accordance with ASTM D570-98(2010)e1 (2010). The morphological observation on RHF raw materials and also RHF wood test samples were conducted using scanning electron microscope (SEM) at the accelerating voltage of 15 keV (JSM 6400, JEOL Ltd., Tokyo, Japan).

RESULTS AND DISCUSSION

Effect of Manufacturing Compression Pressure

The effects of compression or consolidative pressures on the manufacturing of RHF engineered wood panel were evaluated. The read-out hydraulic pressures on the hot press machine were varied from 60, 90, 120, and 140 kg_f/cm^2, respectively. The density of the wood panel was equally controlled at 1.1 g/cm^3. Table 3 summarizes mechanical properties results by mean of Izod impact strengths and flexural properties obtained from the RHF panels manufactured at the experimented pressures. In notched impact strength, the test outcomes revealed that the strength, as expected, gradually increased with increasing the compression pressure. The upper limit optimal pressure was found at 120 kg_f/cm^2. There was no further improvement when the pressure exceeded 140 kg_f/cm^2. A similar trend was observed on the unnotched test results. Increasing the molding pressure resulted in the close compaction and hence good surface adhesion of rice husk flake/epoxy adhesive. Accordingly, superior in the impact strengths were obtained at high molding pressure. At the assigned density, the excessive consolidate pressure, at 140 kg_f/cm^2, cannot further enforce the close packing and also surface-to-surface adhesion of the husk/adhesive. Therefore, there was no improvement of the impact strength beyond excessive compression molding.

The effects of compression pressure on the flexural properties, in terms of strength, modulus, and max deformation at break, and shown in Table 3. All flexural parameters were gradually increased with increasing the pressure from 60 to 120 kg_f/cm^2. There was no significant improvement on the flexural characteristics when the pressure exceeded 140 kg_f/cm^2. The good compaction of the RHF wood and surface-to-surface adhesion at high consolidate pressure was repeated for these flexural results.

Table 3. Effect of the Impact Strengths and Flexural Properties of the 1.1 g/cm^3 RHF Panels on the Compression Molding Pressure

Pressure (kg_f/cm^2)	Thickness (mm)	Impact strength (kJ/m^2)		Flexural properties		
		Notched	Unnotched	Strength (MPa)	Modulus @ 0.5% (GPa)	Max. Def.[a] (mm)
60	6.55 ± 0.04	2.02 ± 0.08	1.95 ± 0.04	17.18 ± 0.75	1.899 ± 0.093	2.39 ± 0.05
90	6.22 ± 0.10	2.28 ± 0.10	2.21 ± 0.05	21.20 ± 1.12	2.488 ± 0.182	2.28 ± 0.03
120	5.96 ± 0.11	2.34 ± 0.09	2.19 ± 0.06	28.57 ± 0.95	3.082 ± 0.098	1.91 ± 0.14
140	5.69 ± 0.07	2.50 ± 0.11	2.65 ± 0.09	26.20 ± 0.84	3.163 ± 0.083	1.87 ± 0.03

[a]Maximum deformation at break

Table 4 reports the tensile properties and HDT of the RHF wood panel at the given experimental compression pressures. Predictably, the tensile strength and modulus were in a positive trend with respect to the molding pressure. However, the strain at break was almost unchanged regardless of the pressure. The tensile testing results unambiguously reinforced that good husk/epoxy compaction and also adhesion control the mechanical properties of the RHF engineered wood panel. Furthermore, there is no exception for the effect of molding pressure on the service temperature, by means of HDT, of the RHF wood. The measured HDT was gradually increased with increasing pressure. However, the increase was marginal. The HDT of the RHF panel or other composite material was mainly controlled by the thermal property of adhesive or matrix. In this work, the HDT of the room temperature cured epoxy adhesive used was approximately 60 °C (Meekum and Wangkheeree 2017b). The contribution of the reinforcement onto the HDT of composite material was minimal, especially for the high adhesive volume fraction. Therefore, the HDT of the manufactured RHF wood panel shows a marginal increase with the compression molding pressure.

Table 4. Summarized of the Tensile Properties and HDT Results Obtained from Various Consolidated Pressure Processing

Pressure (kg_f/cm^2)	Tensile properties			HDT (°C)
	Ultimate Strength (MPa)	Modulus (GPa)	Strain @ break (%)	
60	7.66 ± 1.22	1.117 ± 0.069	0.84 ± 0.18	55.2 ± 0.7
90	11.79 ± 2.06	1.566 ± 0.065	1.08 ± 0.18	55.7 ± 1.0
120	11.47 ± 1.09	1.545 ± 0.067	0.99 ± 0.17	56.1 ± 0.1
140	14.68 ± 0.99	1.686 ± 0.032	1.06 ± 0.08	57.1 ± 0.3

The durability values, as measured by $\%WA_i$, $\%TS_i$ and $\%TS_{dried,\,i}$ of the 1.1 g/cm^3 RHF/epoxy wood panels with respect to the compression pressure after prolonged submersion in water for 1 and 7 day(s), are reported in Table 5. Both $\%WA_1$ and $\%WA_7$ decreased with elevating molding pressue, which indicates better resistance to water absorption at high manufacturing pressure. For thickness swelling upon water absorption, the test results revealed that $\%TS_1$ and $\%TS_7$ are not dependent on the molding pressure. The obtained % expansions are almost unchanged with increasing pressure. Upon removing the absorbed moisture by vacuum drying at 105 °C for 4 h, the negative $\%TS_{dried,1}$, with a value close to zero, was observed. Similarly, the very low value of $\%TS_{dried,\,7}$, mostly in positive value, was typically obtained. Ignoring the negative or positive sign, the degree of expansion/contraction of the RHF engineered wood panel did not exceed 1%. The RHF wood panel using epoxy adhesive had good dimensional stability over the vigorous moist atmosphere. The contraction of the wood, negative value of $\%TS_{dried,\,i}$, was caused during the vacuum drying process. At the given drying temperature, 105 °C, the epoxy matrix was softening. Under the compaction vacuum force, the air spaces inside the RHF wood, mainly caused from incomplete folded of the curvature rice hush and the porous structure of the husk, was evacuated and then retracted. Accordingly, the thickness of the vacuum dried sample was further compacted. Consequently, the thickness of the vacuum dried sample was marginally smaller than the original one. Hence, the negative $\%TS_{dried,\,i}$ would be recoded.

Table 5. Summarize the Effect of Molding Pressure on the Durability Properties

Pressure (kg_f/cm^2)	Durability Properties					
	1 Day			7 Days		
	WA_1 (%)	TS_1 (%)	$TS_{dried,1}$ (%)	WA_7 (%)	TS_7 (%)	$TS_{dried,7}$ (%)
60	23.75 ± 1.41	6.47 ± 0.88	-0.99 ± 0.17	35.61 ± 0.14	8.20 ± 0.22	0.48 ± 0.32
90	22.62 ± 1.43	6.82 ± 0.68	-0.79 ± 0.39	28.68 ± 0.30	9.32 ± 0.52	0.60 ± 0.09
120	18.14 ± 2.57	6.83 ± 0.79	-0.74 ± 0.37	26.31 ± 0.65	8.68 ± 0.12	0.77 ± 0.32
140	14.48 ± 1.48	7.17 ± 0.20	-0.51 ± 1.04	24.26 ± 0.79	7.74 ± 0.45	-0.05 ± 0.05

The closer compaction of husk/epoxy and hence better in surface-to-surface adhesion at higher compression pressure was justified for the superior mechanical properties. A visual investigation of the RHF engineered wood panel at the given manufacturing pressures is required. Figure 3(a) to 3(d) are the SEM photographs of the RHF samples at the assigned manufacturing pressures. SEM photos were taken at the cross section of the broken piece of notched impact samples. They were saw cut and surface polished by fine gain sand paper before undergoing SEM investigation. Figures 3(e) to (h) are the SEM photos of the surface at the machine direction, perpendicular direction to the compression pressure, of the RHF wood manufactured at the given molding pressure. Figures 3(a) to 3(d) show that close packing of the husk/epoxy was observed at high molding pressures, above 120 kg_f/cm^2. At pressure below 90 kg_f/cm^2, the inter spaces between the rice husk flaks were clearly apparent. These SEM observations emphasize that good surface-to-surface adhesion *via* epoxy adhesive of rice husks, or fiber compaction, is achieved by using the high compression pressure, above 120 kg_f/cm^2. Consequently, excellent mechanical properties and/or durability were obtained. At manufacturing pressures of 90 kg_f/cm^2 and below, the compaction of the husk/epoxy/husk was insufficient, resultng in inferior mechanical properties and moisture resistance. Figures 3(e) to (h) show that all of the surfaces of rice husk flake were completely coated with epoxy adhesive, regardless of the molding pressure. Evidence of epoxy adhesive bleeding due to the excessively loading was apparent when operation the molding pressure above 120 kg_f/cm^2. Marginal excessive epoxy adhesive loading occurred at high compression pressure. Obeying the composite rule of mixture, the mechanical properties of the RHF wood panel was enhanced by the stronger fiber fraction. Excessive loading of weaker matrix or adhesive decreases the mechanical performance of the composite. As evidence of excessive epoxy loading was found from the SEM investigation, further fine tuning in adhesive loading at high compression molding pressure could be used to obtain better mechanical superiority of the RHF wood panel.

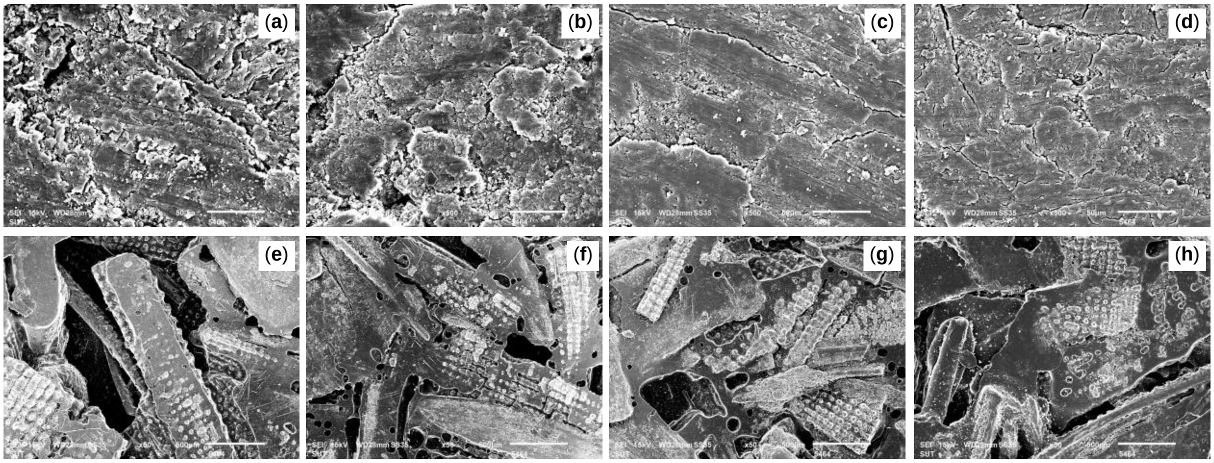

Fig. 3. SEM photographs of the polished cross section specimens at (**a**) 60, (**b**) 90 (**c**) 120, (**d**) 140 kg_f/cm^2 at 500x and specimens surface at (**e**) 60 (**f**) 90 (**g**) 120 (**h**) 140 kg_f/cm^2 at 50x

The optimal compression pressure for the manufacturing process of the 1.1 g/cm^3 RHF engineered wood panel using 35 phr of epoxy adhesive was 120 kg_f/cm^2. The good compaction of the husk/epoxy/hush and superior in surface-to-surface adhesion of the husk flakes at this optimized consolidate pressure were the explanation. Subsequently, superior performance properties were achieved. Excessive pressure is unnecessarily for the achievement of close packing. In addition, shortening the machine lifetime and speeding up the mold wearing would be experienced in the case of prolonged operation at high pressure. This would be an economic disadvantage.

Effect of Short UD Fibers Reinforcements on RHF Engineered Wood Panel

The 1.1 g/cm^3 rice husk flake/epoxy wood panels reinforced with short UD, CF, and GF were manufactured at the compression molding pressure of 120 kg_f/cm^2. The CF and GF contents were varied from 10, 20, 30, and 40 wt% with respect to the husk. Table 6 summarizes the impact strength and flexural parameters of the short UD reinforced wood samples at the given CF and GF loading. For the notched impact strength, the strength was proportionally increased with increasing fiber content. The same trend was observed for the unnotched strength. Comparing the unreinforced and reinforced woods, there was an approximately two-fold increase in the impact strength, especially for the CF reinforcement, obtained when only 10% wt of the fiber was loaded. As expected, the short CF showed reinforcing superiority to the GF. However, increasing the fiber loading from 30% to 40%, the impact strength declined. The adhesive exhausting effect was suspected for this phenomena and was investigated by SEM.

Table 6. Izod Impact Strengths and Flexural Properties of the RHF Wood Panel Reinforced with Short UD Fibers

Sample	Fiber Content (%.wt)	Impact strengths (kJ/m²)		Flexural properties		
		Notched	Unnotched	Strength (MPa)	Modulus @ 0.5% (GPa)	Max. Def.[a] (mm)
X0	0	2.34 ± 0.09	2.19 ± 0.06	28.57 ± 0.95	3.082 ± 0.098	1.91 ± 0.14
CF	10	4.91 ± 0.56	6.19 ± 0.84	44.71 ± 1.09	3.831 ± 0.173	2.81 ± 0.21
	20	8.56 ± 0.47	9.61 ± 0.84	60.61 ± 1.03	5.662 ± 0.132	3.29 ± 0.65
	30	12.34 ± 0.56	17.34 ± 0.59	63.43 ± 4.45	6.621 ± 0.290	3.74 ± 0.13
	40	11.95 ± 0.12	16.05 ± 0.88	57.24 ± 2.52	6.361 ± 0.198	3.42 ± 0.13
GF	10	3.90 ± 0.26	4.12 ± 0.75	30.66 ± 1.42	3.185 ± 0.207	2.68 ± 0.15
	20	5.07 ± 0.25	5.56 ± 0.45	44.84 ± 1.47	3.434 ± 0.151	2.69 ± 0.10
	30	7.83 ± 0.28	8.50 ± 0.71	47.54 ± 2.11	4.277 ± 0.108	2.97 ± 0.23
	40	10.08 ± 0.54	12.00 ± 0.92	46.59 ± 0.88	4.794 ± 0.064	2.91 ± 0.08

[a] Maximum deformation at break

In the flexural testing results, the strength and modulus were proportionally elevated with increasing fiber loading, both for CF and GF. The improvement was typically observed at the fiber loading increasing from 0 to 20 wt%. With closer observation of the flexural strength and modulus of the reinforced RHF wood sample, the turning point for the flexural superiority was at 30% and 40% fiber loading. By further increasing the loading from 30% to 40%, the flexural strength declined. The high fiber loading caused epoxy adhesive exhaustion. The SEM investigation revealed that the marginal excessive epoxy adhesive loading, at 35 phr, was evidenced and justified at high molding pressure. However, on the rice husk hybridized with fine short UD CF and GF systems, the mixed hybrid fibers required more amount of adhesive for complete wetting of the fiber surfaces. The diameter of short UD, CF, and GF was smaller than rice husk flake. Hence, a considerable increasing in the specific surface area for adhesive wetting must be generated, especially at high fraction of the manmade fiber. Consequently, at the constant epoxy loading, the adhesive exhaustion phenomena would be experienced at high CF and GF dosing. According to the flexural and impact testing results, adhesive exhaustion likely occurred when the CF and GF loading exceeded 30% wt. Consequently, the diminished mechanical toughness properties of the RHF/CF and RHF/GF woods were manifested at the fiber content exceed 40 wt%. However, the decrease in the toughness properties at 40% loading of RHF/GF was less obvious than RHF/CF wood. It is due to the fact that the diameter of short GF used in this study was 8.80 μm, but the diameter of CF was 6.5 μm (Fig. 1) (Meekum and Wangkheeree 2016). At equal loading, CF had a higher specific surface area than GF, such that CF requires more surface wetting adhesive than GF. The adhesive exhaustion effect at 40% loading was less pronounced for the RHF/GF than the RHF/CF sample. The refinement of this hypothetical statement will be presented by SEM investigation later on. For the maximum deformation at break upon flexural bending testing, improvement was noticed only between unreinforced RHF wood and fiber-reinforced RHF material. However, the deformation at break of the RHF reinforced with CF and GF woods did not show an obvious dependence on the fiber contents. At the identical loading, the flexural properties of RHF reinforced with randomized short UD CF were superior to the GF reinforcement.

Table 7 demonstrates the tensile properties and HDT results of the RHF wood without and with short UD, CF, and GF, reinforcements. The strength of the panel reinforced with CF and GF was gradually improved with respect to the fiber loading. A similarly trend was found for the modulus and strain at break, especially for the RHF/GF wood. By further increasing the fiber content from 30% to 40%, the tensile characteristics declined. This reversing trend strengthen the adhesive exhaustion phenomena hypothesis at high CF and GF fraction. In comparison, at the given loading, there is the short UD CF reinforcement had more tensile properties enhancement than the short UD GF.

HDT measuring outcomes, at 1820 kPa, of the panels are reported in Table 7. The test data indicate that the significant elevate in HDT with the fiber loading was observed for both RHF/CF and RHF/GF woods. At 30% reinforcement loading, there was 15 to 20 ºC of HDT improvement in comparison with unreinforced sample. The HDT diminishing trend occurred when the loading increased from 30% to 40%. At the identical loading, short UD CF exhibited more HDT improvement than the GF.

Table 7. Tensile Properties and HDT of RHF Engineered Wood Panel Reinforced with Randomized Short UD, CF, and GF

Sample	Fiber Content (%.wt)	Tensile properties			HDT(°C)
		Ultimate Strength (MPa)	Modulus (GPa)	Strain at break (%)	
X0	0	11.47 ± 1.09	1.545 ± 0.067	0.99 ± 0.17	56.1 ± 0.1
CF	10	15.86 ± 1.73	1.185 ± 0.049	2.33 ± 0.31	62.7 ± 0.7
	20	19.87 ± 1.77	1.309 ± 0.123	2.35 ± 0.35	73.1 ± 0.4
	30	26.59 ± 2.08	1.689 ± 0.074	2.59 ± 0.42	74.1 ± 0.4
	40	23.40 ± 0.68	1.481 ± 0.046	2.50 ± 0.11	74.0 ± 0.9
GF	10	16.92 ± 1.78	1.329 ± 0.203	1.97 ± 0.19	62.5 ± 0.6
	20	19.82 ± 2.73	1.631 ± 0.110	1.74 ± 0.21	71.9 ± 0.1
	30	25.18 ± 0.86	1.948 ± 0.115	1.94 ± 0.03	71.2 ± 0.1
	40	23.16 ± 1.38	1.865 ± 0.018	2.33 ± 0.32	70.8 ± 0.8

For the evaluation of dimensional stability upon prolonged water immersion for 1 and 7 days, or hereby called the durability testing, the %WA_i, %TS_i, and %$TS_{dried,\,i}$ test outcomes are summarized in Table 8. The %WA_1 and %WA_7 of the unreinforced RHF wood were higher than the reinforced samples, irrespective to the content. The %WA_1 and %WA_7 of both RHF/CF and RHF/GF samples decreased when the CF and GF contents were increased, especially at 0 to 30% loading. At the fiber content beyond 30%, % WA_i increased. Rice husk flake was found to be less hydrophilic than other cellulose fibers but it obviously was more hydrophilic than CF and GF. Therefore, reducing the husk portion would lower the %WA_i. However, for this engineered wood panel manufacturing process, the surface coating by epoxy adhesive is important for the durability of the wood. Moreover, cured thermoset epoxy adhesive has lower hydrophilicity than the thermoplastic formaldehyde-based adhesive. Therefore, high %WA_i was not observed for these RHF wood panels. Again, the upright trend on the % WA_i at 40% fiber portion was justified by the adhesive exhaustion. With sufficient adhesive coating on rice husk surface and also on the reinforcement fiber, the good adhesion and water protective coated surfaces would be established. Accordingly, the engineered RHF wood panel with high mechanical performances and low water absorption would be achieved. In contrast, with the adhesive exhaustion phenomena, the fading of those properties must be experienced. For the dimension stability testing under hazard environment, water immersion for 1 and 7 days, by means of the thickness swelling recorded by %TS_i and $TS_{i,\,dried}$ values are concluded in Table 8. Unreinforced RHF wood showed higher %TS_1 and %TS_7 than the reinforced specimen. Regardless of the immersion times, %TS_1 and %TS_7 of both RHF/CF and RHF/GF wood panels decreased with increased fiber loading. The $TS_{1,\ dried}$ and $TS_{7,\ dried}$ results were close to zero, regardless to the fibers loading and types. After removing the absorbed moisture by vacuum drying, the thickness of the samples was generally retracted to the original dimension.

Table 8. Durability Properties of Rice Husk Composite made from Various Content of Short Fibers Reinforcement

Sample	Fiber Content (wt%)	Durability Properties					
		1 Day			7 Days		
		WA_1 (%)	TS_1 (%)	$TS_{dried,\ 1}$ (%)	WA_7 (%)	TS_7 (%)	$TS_{dried,\ 7}$ (%)
X0	0	18.14 ± 2.57	6.83 ± 0.79	-0.74 ± 0.37	26.31 ± 0.65	8.68 ± 0.12	0.48 ± 0.32
CF	10	12.97 ± 0.63	5.18 ± 0.24	-1.35 ± 0.18	21.68 ± 0.68	7.04 ± 0.45	0.80 ± 0.54
	20	10.11 ± 0.39	6.85 ± 0.91	-1.37 ± 0.50	20.43 ± 0.26	6.67 ± 0.29	0.76 ± 0.14
	30	11.30 ± 2.35	3.75 ± 0.18	-1.52 ± 0.55	20.37 ± 1.36	5.85 ± 0.67	0.37 ± 0.04
	40	21.47 ± 0.39	0.75 ± 0.03	0.63 ± 0.48	30.75 ± 0.44	5.78 ± 0.60	-0.34 ± 0.21
GF	10	12.72 ± 0.08	6.21 ± 0.46	-0.90 ± 0.02	27.69 ± 1.13	7.40 ± 0.59	1.27 ± 0.27
	20	8.89 ± 0.48	5.30 ± 0.24	-0.92 ± 0.05	19.22 ± 1.52	6.37 ± 0.30	0.66 ± 0.41
	30	11.80 ± 0.68	5.12 ± 0.30	-1.68 ± 0.05	23.12 ± 1.70	5.82 ± 0.13	0.69 ± 0.21
	40	13.03 ± 1.20	5.04 ± 0.22	-0.45 ± 0.18	17.14 ± 0.56	5.80 ± 0.32	-0.68 ± 0.23

Figures 4(a) to 4(c) are the SEM photographs of the RHF/CF wood panels having the loading of 20, 30 and 40 wt%, respectively. Rich or excessive adhesive can be clearly seen on the samples having 20 and 30% CF contents. However, adhesive exhaustion, especially on the CF surface, is evidenced at 40% CF loading. These SEM investigations strengthened the hypothesis that additional adhesive dosing is required for sufficient fiber surface wetting when the RHF wood panel is manufactured by using a high fraction of fine short UD, CF, and GF reinforcements. This is due to the fact that the fiber diameter of short UD CF and GF are much smaller than rice husk flake. Accordingly, at the given weight, there was a large increase in the specific surface area of CF and GF over the husk flake. Consequently, there was excessive surface to be wetted and bonded by the epoxy adhesive. At the constant dosing of 35 phr of epoxy adhesive with respect to the total amount of rice husk and fiber, adhesive exhaustion was experienced at high CF and GF loading. Adhesive exhaustion occurred when the CF or GF loading exceeded 40 wt%, leading to diminished mechanical performance and durability.

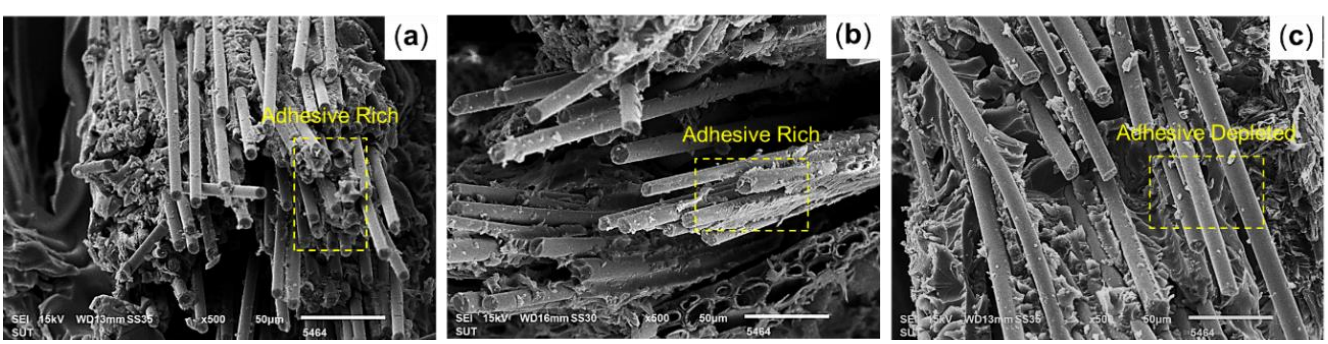

Fig. 4. SEM pictures of the RHF/CF wood panel with; (**a**) 20, (**b**) 30 and (**c**) 40 wt.% fiber contents

RHF Wood Panel Reinforced with Hybridized Short CF/GF

Utilizing the individual short randomized UD fiber as the reinforcement for the RHF engineered wood panel unambiguously showed that, at the given fiber loading, the mechanical properties of RHF/CF wood were superior to the RHF/GF. For the engineering point of view, it will be fruitful to verify the the effect of hybridized CF/GF as reinforcement on the properties of RHF wood panel. The 1.1 g/mL of RHF wood panel was reinforced with 40 %.wt of CF/GF. The assigned CF/GF ratios were 40/0, 30/10, 20/20, 10/30 and 0/40, respectively. The epoxy adhesive loading was constantly controlled at 35 phr. The wood was compression molded at 120 kgf/cm^2, 120 ºC, and 180/30/120 seconds molding cycle. Table 9 summarizes the impact strengths, notched and unnotched, of the RHF/CF/GF woods panel samples. The impact strength of RHF/CF was superior to the RHF/GF panel. At the assigned CF/GF ratios, the impact strengths were gradually decreased with increasing GF ratio. A similar trend was observed for the flexural properties, as reported in Table 9. The flexural strength and modulus were progressively decreased with increasing GF fraction. These mechanical performances of the RHF reinforced with hybridized short CF/GE are obeyed the "composite rule of mixture", in which the strengths of the reinforced composite material are generally enhanced by the fractional loading of high performance reinforcement. Typically, CF has remarkable excellent mechanical properties over the GF. Accordingly, the mechanical performance of the RHF/CF/GF wood panel was enhanced by the CF fraction. In fact, this system is the hybridized CF/GF reinforcement. The "rule of hybrid mixture (RoHM)" to predict the elastic modulus was published by Mansor *et al*. (2013). At the iso-strain condition, the two single composite systems are defined as,

$$\varepsilon_{hy} = \varepsilon_{CF} = \varepsilon_{GF} \tag{1}$$

where ε_{hy}, ε_{CF}, and ε_{GF} are strains of the hybridized, CF reinforced, and GF reinforced, respectively. An assumed balanced condition among forces requires that,

$$E_{hy}\varepsilon_{hy} = E_{CF}.\varepsilon_{CF}.V_{CF} + E_{GF}.\varepsilon_{GF}.V_{GF} \tag{2}$$

where E_{hy}, E_{CF}, and E_{GF} are the elastic moldulus of the hybridized, CF, and GF reinforced composite, respectively. V_{CF} and V_{GF} are the volume fraction of CF and GF reinforcement, respectively. Applying the iso-stain condition given in Eq. 1 onto Eq. 2, the modulus of the hybridized composite can be resolved from the RoHM equation as shown in Eq. 3.

$$E_{hy} = E_{CF}.V_{CF} + E_{GF}.V_{GF} \tag{3}$$

By adopting the RoHM onto the RHF wood panel reinforced with hybridized CF/GF short fibers, the RoHM plot for the flexural modulus is illustrated in Fig. 5. It can be seen that the measured flexural moduli closedly laid along the predicted RoHM trend line. This RoHM result confims the previous statement that the mechanical performances of the RHF reinforced with hybridized short CF/GE obeyed the "composite rule of mixture (ROM)".

Table 9. Impact Strengths and Flexural Properties of the RHF Wood Reinforced with Hybridized CF/GF

Fiber Ratio (%)			Impact Strength (kJ/m^2)		Flexural Properties		
RHF	CF	GF	Notched	Unnotched	Strength (MPa)	Modulus (GPa)	Max. Def.[a] (mm)
60	40	0	11.95 ± 0.12	16.05 ± 0.88	57.24 ± 2.52	6.361 ± 0.198	3.42 ± 0.13
60	30	10	9.58 ± 0.39	14.38 ± 0.56	51.55 ± 1.06	6.240 ± 0.291	3.15 ± 0.13
60	20	20	9.01 ± 0.80	13.10 ± 0.41	46.79 ± 0.45	5.890 ± 0.077	3.11 ± 0.26
60	10	30	8.44 ± 0.78	11.37 ± 0.34	45.03 ± 0.37	5.405 ± 0.042	3.27 ± 0.17
60	0	40	8.31 ± 0.90	10.50 ± 0.50	46.59 ± 0.88	4.794 ± 0.064	2.91 ± 0.08

[a]Maximum deformation at break

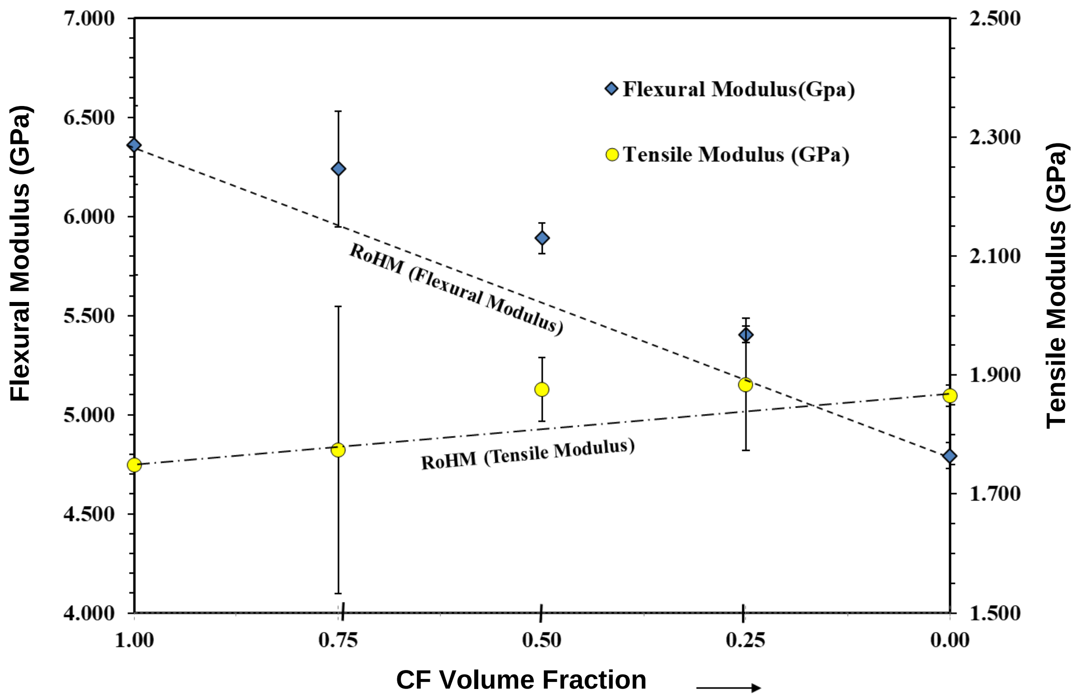

Fig. 5. The rule of hybrid mixtures (RoHM) plots of flexural and tensile modulus

Tensile properties were tested at a strain rate of 15 mm/min. The ultimate strength, modulus, and strain at break of the RHF wood reinforced with the hybridized CF/GF fibers samples are reported in Table 10. The ultimate strength of the wood samples was gradually lowered with increasing GF fraction, but the tensile modulus increased with higher GF loading. For the tensile strain at break, it slowly decreased with increased GF loading. Commonly, in tensile testing, instances of lowering in strength and strain at break but then elevating in modulus of material are interpreted as meaning that the material is transformed from a ductile to a brittle state. For the manufacturing of the RHF wood panel reinforced with hybridized short CF/GF, the outcome material would become more brittle under tensile loading with a high GF fraction, but highly ductile wood would be obtained with increasing the CF fraction. The RoHM plot between tensile modulus and CF volume fraction is also shown in Fig. 5. It is obviously to observe that the measured modulus is closely positioned onto the linear RoHM line. The outcome confirms that the RHF wood reinforced with short hybridized CF/GF fibers system obeyed the hybrid ROM of composite material.

The HDT results tested at 1820 kPa are also reported in Table 10. There was also a significant influence of CF/GF ratios on the wood service temperatures. The measured HDT values show the down trend with increasing the GF fraction. As indicated earlier that, at the given reinforcement loading, CF had the better HDT enhancement than GF. Therefore, by increasing the GF fraction, or vice versa, the HDT of the fiber reinforced material must be decreased.

Table 10. Tensile Properties, HDT of RHF Reinforced with Hybridized CF/GF

Fiber Ratio (%)			Tensile Properties			HDT (°C)
RHF	CF	GF	Strength (MPa)	Modulus (GPa)	Strain @ break (%)	
60	40	0	28.17 ± 3.82	1.749 ± 0.320	2.67 ± 0.28	74.0 ± 0.8
60	30	10	26.34 ± 2.73	1.774 ± 0.242	2.66 ± 0.23	73.5 ± 0.4
60	20	20	25.83 ± 0.24	1.876 ± 0.054	2.55 ± 0.43	72.4 ± 0.6
60	10	30	25.45 ± 1.45	1.884 ± 0.111	2.42 ± 0.19	70.1 ± 0.7
60	0	40	23.16 ± 1.38	1.865 ± 0.018	2.33 ± 0.32	70.8 ± 0.8

Table 11 illustrates the durability testing results obtained from the RHF wood panel reinforced with hybridized CF/GR short fibers. The $\%WA_{1}$ and $\%WA_{7}$ values were decreased with increasing GF contents. However, the % thickness swelling after prolonged submersion in water for 1 and 7 day, $\%TS_1$ and $\%TS_7$, were marginally increased with increasing GF fraction. Upon the vacuum drying of the samples to remove the absorbed water, the thickness of the woods were generally retracted to the original dimension regardless to the CF/GF ratios. Thus, the minimal values of $\%TS_{dried,\ 1}$ and $\%TS_{dried,\ 7}$ were recorded. Their measured values mostly did not exceed 1%. Again, the negative values of the $\%TS_{dried}$ can be justified by a contraction effect of air pores and voids, caused mainly by the fact that the rice husk was porous in nature and there was incomplete folding of the curvature of rice husk flake, which was retracted under the force of vacuum. The higher %WA of RHF/CF over RHF/GF could be rationalized by the capillary action. As shown by SEM, the diameter of short UD CF used in this work was much smaller than the GF. The water diffusion along the incomplete CF/CF and CF/husk surface adherent by the capillary action was greater than in the GF reinforcing samples. Subsequently, %WA would be increased at high CF fraction. The lower degree in thickness swelling, $\%TS_i$, of RHF/CF than RHF/GF, was explained by higher strength of CF than GF. Under the hydrostatic pressure/load caused by water absorption, the RHF wood reinforced with CF would have more dimensional stability, less expansion, than the RHF/GF. Consequently, the dimensional stability of RHF/CF/GF was inferior, more swelling, by reducing the CF fraction.

Table 11. Durability Properties of Rice Husk Panel Derived from Hybridized Synthetic Fiber

Fiber Ratio (%)			Durability Testing					
			1 Day			7 Days		
RHF	CF	GF	WA_1 (%)	TS_1 (%)	$TS_{1,\,dried}$ (%)	WA_7 (%)	TS_7 (%)	$TS_{7,\,dried}$ (%)
60	40	0	21.47 ± 0.39	0.75 ± 0.03	0.63 ± 0.48	30.75 ± 0.44	5.78 ± 0.60	-0.34 ± 0.21
60	30	10	11.24 ± 0.43	4.68 ± 1.01	-2.49 ± 0.78	25.18 ± 1.68	5.29 ± 1.52	-1.72 ± 0.30
60	20	20	19.78 ± 0.63	5.20 ± 1.12	-0.64 ± 0.30	22.53 ± 1.03	5.19 ± 0.23	-1.26 ± 0.13
60	10	30	14.42 ± 0.82	5.06 ± 0.07	-0.22 ± 0.07	17.25 ± 1.87	5.59 ± 1.19	-0.60 ± 0.44
60	0	40	13.03 ± 1.20	5.04 ± 0.22	-0.45 ± 0.18	17.14 ± 0.56	5.80 ± 0.32	-0.68 ± 0.23

By adopting 40 wt% hybridized CF/GF short fibers as the reinforcement for the manufacturing of the RHF engineered wood panel, the mechanical performances were reduced by increasing the GF fraction. Commonly, GF has inferior mechanical capability to CF. Thus, the composite "rule of mixture" must be obeyed. For the dimension stability of the wood upon prolonging simulated water submersion, the experimental outcomes revealed that the $\%WA_i$ were increased with increasing CF fraction. The capillary action due to the smaller fiber diameter of CF were hypothesized for the experimental results. However, stronger strength of CF reinforcement showed the benefit in the dimensional stability by mean of the thickness swelling, $\%TS_i$.

CONCLUSIONS

In order to manufacture the engineered wood from the absolute untreated rice husk flake using epoxy adhesive and short CF/GF reinforcement, the main conclusions resulted from the experimental studies were;

1. The optimal compression molding pressure for the manufacturing process of the 1.1 g/cm^3 RHF engineered wood panel using 35 phr of epoxy adhesive was 120 kg_f/cm^2. The good compaction of the RHF and satisfactory in surface-to-surface adhesion of the rice husk flake at this consolidating pressure was justified based on the superior properties achieved. The application of excess compression pressure was unnecessary. The shortening of machine lifetime and increased wearing by continuously operating at high pressure was also economically justified.
2. The mechanical performances were improved by reinforcing the RHF/epoxy wood panel with short UD, CF, and GF. At the given short fiber loading, the CF showed superiority reinforcing capability to the GF. Adhesive exhaustion, especially on the surface of CF, occurred when the fiber reinforcement loading exceeded 40 wt%. Consequentliay, the mechanical performances were diminished.
3. At the given 40 wt% of hybridized CF/GF short fibers loading for manufacturing of the RHF engineered wood panel, the mechanical performances were reduced by increasing the GF portion. The inferior mechanical capability of GF to CF was explained by the theoretical composite "rule of hybrid mixture" (RoHM).

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