International Journal of Nanoelectronics and Materials
Volume 14, No. 3, July 2021 [247-258]

Composite of *Hibiscus Tiliaceus* Stem Fiber/Polyester Modified with Carbon Powder: Synthesis and Characterization of Tensile Strength, Flexural Strength and Morphology Properties

Nasmi Herlina Sari[1*], Suteja Suteja[1], Muhammad Nurhayadi Samudra[1], Himawan Sutanto[2]

[1] Mechanical Engineering Department, Faculty of Engineering, University of Mataram, Mataram, West Nusa Tenggara, Indonesia.
[2]Development Economics Study Program, Faculty of Economics and Business, University of Mataram, Mataram, West Nusa Tenggara, Indonesia.

Received 1 March 2021, Revised 29 March 2021, Accepted 27 April 2021

ABSTRACT

A successful synthesis of composite particles of hibiscus tiliaceus bark fiber (HBF) and carbon powder from coconut shell (CP) by hot press technique has been reported. The microstructure of samples is investigated using an SEM analysis to check the morphology of the composite. Ultimate tensile and flexural strength of CP/HBF/polyester composites are examined. The percentages of CP were defined as 15, 20, and 30 (% by volume) and the HBF percentages were 5 and 10 (% by volume). The results showed that the tensile strength, modulus of elasticity, and bending strength of the composites increased significantly close to 110% with the increase in the amount of CP in 5% HBF/polyester composites. Conversely, the mechanical properties are low when the powder content of hibiscus skin is 10% due to the presence of pores at the interface between the CP-HBF-polyester, the weak adhesion of the interface, and the HBF pulling out. The results imply that these composites can be an alternative building construction material.

Keywords: *Hibiscus Tiliaceus* bark powder, coconut shell carbon powder, composites, bending strength, tensile strength, scanning-electron microscopy (SEM)

1. INTRODUCTION

The use of natural fibers as the composite filler has become a concern and interest for researchers and industrialists in solving environmental problems. Various types of natural fibers, such as pandanwangi fiber, corn husk fiber, sugar palm fiber, etc. [1-5], are known to be unique and superior to the synthetic fibers used as composite fillers. They have advantages such as low cost, less CO_2 emissions, cheap, and easy to manufacture. Among the various natural fibers, the bark fiber of *Hibiscus Tiliaceus* is one of the dominant natural bark fibers growing in Indonesia.

Hibiscus Tiliaceus (HT) trees can be found in various areas such as on the coast, mountains, and roadsides. It has become of interest for researchers and industrialists to investigate and develop a wider utilization of the stem fiber of *Hibiscus Tiliaceus* bark. To the best of our knowledge, there are very few works reporting on HT stem fibers and their composites. Sari and Yesung [6] have investigated the characteristics of the thermal and tensile properties of *Hibiscus Tiliaceus* bark (HTB) fiber before and after being treated in 8% KOH (potassium hydroxide) solution for two hours at room temperature. They found that the tensile strength increased to 5144.9 MPa (700% increment) with an elongation value of 4.75 mm. The surface morphology of HT fibers became clearer, coarser, and fibrils after KOH treatment. Further, Surata et al. [7] have investigated mechanical properties of fibers from HTB. They reported that fibers from HTB had the average tensile strength and modulus of young of single HTB of 44.604 MPa and 365.864 MPa,

* Corresponding Author: n.herlinasari@unram.ac.id

respectively. The known failure strain is 11.6%, thus they concluded that HTB is capable of replacing synthetic ropes for marine applications. Miriam et al. [8] have utilized chlorophyll derivatives extracted from the HT plant as an ingredient for the manufacture of multiwalled carbon nanotubes. They found that the nanotubes were easily grafted in the presence of chlorophyll derivatives for gas sensing, solar energy, or heavy metal remediation applications. Furthermore, several other researchers have investigated the properties of HT stem fiber-reinforced composites. Purnowidodo et al. [9] have investigated the influence of the orientation of the hibiscus fibers and the number of layers of the fibrous skin fibers on the static strength and fatigue life of FMLs. The tensile strength of FMLs decreases with increasing the number of layers of HT fiber. The tensile strength of FMLs with a fiber orientation of $0^{o}/90^{o}$ is higher than that of -45^{o}/+ 45^{o}. They concluded that FMLs with more layers of hibiscus fibers had a shorter fatigue life with increased notch displacement in FMLs. Then, Pandiatmi et al. [10] have investigated the flexural strength of polyester/HTB composites by adding rice husks as a filler using the response surface method. They reported that the best conditions were obtained from composites with a fiber volume fraction of 49.06% and a filler of 5.39% with a bending strength value of 99.71 N / mm^2.

Furthermore, investigations related to expanding the properties of composite materials by modifying other fillers such as carbon powders have also attracted the attention of researchers due to their properties of being cheap, strong and harmless to health [11-14]. Ojha et al. [11] have studied the mechanical properties of polymer composites with carbon powder fillers. They stated that the mechanical properties increased with increasing carbonization temperature of the wood apple shell carbon black and the volume fraction of carbon powder. Dias et al. [12] have combined recycled carbon microfibers at a mass fraction of 0-10% with epoxy polymers. They found that the tensile modulus was increased by 36.6% with the addition of recycled carbon microfibers. The tensile strength (compressive) was reported at 27% (19%) when the carbon microfibers mass fraction was 5%, and the flexural strength increased by 28.6% when the waste carbon powder was 10 %wt. Kumar et al. [13] investigated the thermomechanical and electrical properties of carbon powder-filled composites prepared from human hair. They reported that carbon cloth reinforced polymer composites and carbon-carbon (CC) composites had tensile and bending properties reaching 25% - 73% when 30% wt of human hair-derived carbon powder was added to the composites. The storage modulus (E') and the loss modulus (E'') of the composites increased by 132% and 104%, respectively. Moreover, the electrical conductivity and thermal stability of the composites were substantially increased by 233% and are suitable for structural applications at high temperatures. Furthermore, Mostafa et al. [14] have investigated the effect of adding carbon (AC) particles on the mechanical properties of bulk polyester and hemp fiber/polyester composites with variations in the volume fraction of activated carbon 1, 3, 5, and 10 wt%. They reported that the tensile and flexural strength of the polyester and hemp/polyester decreased as the AC particles increased. However, the tensile and bending modulus increased by up to 3% by weight, but the impact strength decreased. However, adding 3 wt% AC to the hemp/polyester composite resulted in impact strength of ~ 4.3 to ~ 6.4 kJ/m^2, as well as increased stiffness of the composite. Sari et al. [15] investigated the mechanical strength of a corn husk fiber composite filled with carbon powder from coconut shell. They reported that composites filled with 10% carbon powder (CSP) had higher flexural strength than 5% CSP composites. From previous studies, it is shown that the study related to the mechanical properties of the composite stalk bark fiber with coconut shell carbon fiber has not been investigated and needs to be developed so that new composite materials can be obtained with better properties and an extension of the composite function for the desired application.

Therefore, this work aims to investigate and provide a detailed understanding of the mechanical properties of HT stem powder/polyester composite with coconut shell carbon powder. The analysis of mechanical properties includes tensile strength, modulus of elasticity, elongation, and bending properties. Observation of the fracture surface has also been carried out using SEM.

2. MATERIAL AND METHODS

2.1 Materials

The stem bark of *Hibiscus Tiliceus* (HTB) is taken from the trunk of a small *Hibiscus Tiliceus* tree (see Figures 1(a) and 1(b)). They are washed clean to remove dirt on the surface of the stems, then dried under the hot sun (see Figure 1(c)); intended to facilitate the extraction of fibers from the skin of the hibiscus (Figure 1(d)).

Figure 1 The process of preparation of *Hibiscus Tiliaceus* bark powder: (a) *Hibiscus Tiliaceus* stem, (b) *Hibiscus Tiliaceus* bark, (c) drying hibiscus bark, (d) fibers after drying, (e) pieces of fibers, and (f) the powder of *Hibiscus Tiliaceus* bark fiber (HBF)

The fibers are cut into small pieces 1 cm in size (see Figure 1 (d)) and then milled using a grinding machine to obtain a powder with a size of 300 mesh (see Figure 1€). Coconut shell carbon (CP) is obtained from PT. Pelapak Megah Abadi Kimia, Tangerang Indonesia with a size of 1000 mesh (see Figure 2).

Figure 2 Carbon powder from coconut shell

Polyester resin has been used as a matrix in the manufacture of these composites, with specifications as proposed by Sari, et al. [2019], namely the tensile strength, elongation, and flexural strength of 8.8 kg/mm^2, 2.3%, 2.5 kg/mm^2 respectively.

2.2 Composite manufacturing

Hibiscus bark powder (HBF), carbon powder (CP) and polyester resin were weighed using a digital analytical balance according to a predetermined composition, as shown in Table 1. HBF, CP, and polyester resin were mixed evenly in a container then poured into a mold made of the steel plate and closed with an emphasis of 5 MPa at a temperature of 105^{o}C for 5 minutes and left for 4 hours, and then removed from the mold.

Table 1 Composition of HBF and CP used in polyester composites

No.	Volume fraction		
	CP (%)	**HBF (%)**	**Polyester (%)**
1	15	5	80
2	20	5	75
3	30	5	65
4	15	10	75
5	20	10	70
6	30	10	60

1.3 Characterization of composites

1.3.1 Mechanical tests

The composite specimens for the tensile strength and bending strength tests refer to the international standards ASTM D 3039 and ASTM D790, respectively. The shape and size of the test specimens are shown in Figures 3(a) and 3(b). Tensile and bending strength tests were carried out using the RTG-1310 tensilon testing machine with a load capacity of 20 KN, at a speed of 5 mm/min and a load cell of 5 kN at room temperature 31 °C.

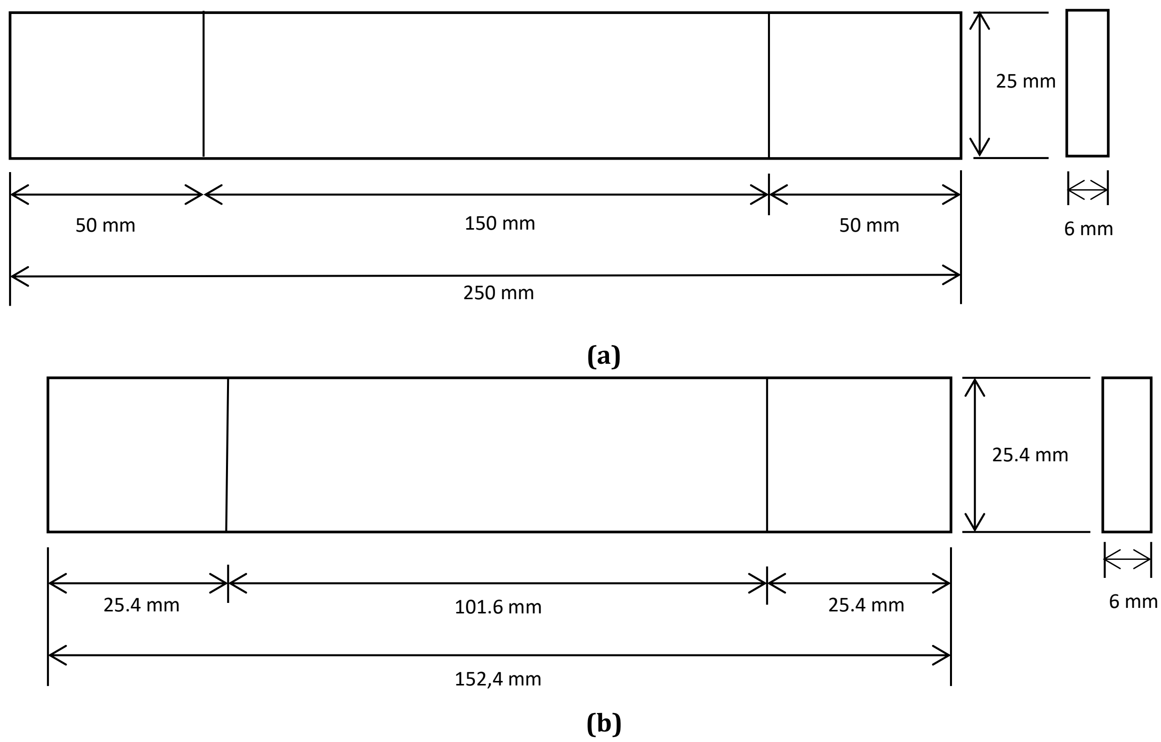

Figure 3 Specimens' test: (a) tensile and, (b) bending of the composite studied

1.3.2 Scanning electronic microscopy (SEM)

The fracture morphology of the composite specimen surface was observed using the SEM type (FEI inspect S50). The machine was operated with a 10 kV and 18 mA. Samples with a diameter of 1 cm and a thickness of 0.6 cm were sprayed with gold for 5 minutes to obtain a thickness of 10 nm at a pressure of 0.1 torr.

3. RESULTS AND DISCUSSIONS

3.1 Tensile strength analysis

Figure 4 presents the tensile strength of the studied HBF/CT/polyester composites. From this figure, it is found that the average tensile strength of the 5% HBF/polyester composite with variations in CP is 15%, 20%, and 30% of 36.06 MPa, 42.188 MPa, and 54.978 MPa, respectively. In the 10% HBF/polyester composite with the same variation of CP, the composite tensile strength was obtained at 31.329 MPa, 36.638 MPa, and 43.188 MPa, respectively. Overall, the tensile strength of 5% HBF/polyester composites and 10% HBF/polyester composites increased when the volume fraction of carbon powder increased. The tensile strength value of the composite 5% HBF was higher than the 10% HBF composite. The tensile strength of 5% HBF composites is high because the interface adhesion is stronger, so that it is strong enough to transfer tensile stresses than other composites; as evidenced by SEM images (Figure 8). On the other hand, when the HBF increases from 5% to 10% (volume fraction), dispersion, accumulation of HBF occurs, and the resin cannot wet the powder because the resin does not enter between the two adjacent fibers and pulls out, as a result, the tensile strength of the composite is small. Besides, the presence of some voids at the interface between the HBF-CP, HBF pull out, and the unevenness of the particle shape may also be suspected to be the cause of the decrease in the tensile strength value of the composite which results in the stress concentration in the resin body so that the interface adhesion may be too weak to transfer the tensile stress; as evidenced by SEM images (Figure 9).

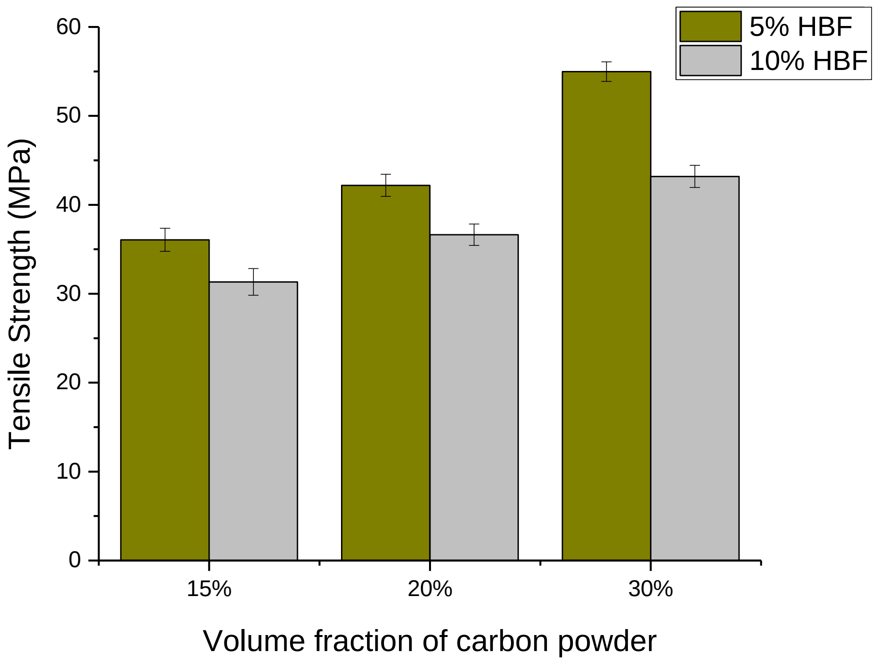

Figure 4 Effect of volume fraction of carbon powder on tensile strength of HBF/polyester composite

Figure 5 shows that when the amount of carbon powder increases, the strain value of the 5% HBF composite increases significantly. The highest elongation was obtained in composites with 30% carbon powder content of 3.22 ± 0.19 (%). It may be assumed that the increase in carbon content has filled the blank of the composite and caused the ductility of the composite to increase so that the % elongation of the composite is high; consequently, the higher the elongation value. Meanwhile, the 10% HBF/polyester composite decreased with increasing carbon powder from 15% to 30% carbon in the composite by 3.105%, 2.829%, and 2.576%, respectively. Elongation at break of the composite was found to be reduced with increasing content of carbon powder with hibiscus powder (10%) because the addition of carbon powder reduced mobility and increased the brittleness of the composite so that the % elongation of the composite was low compared to 5% HBF composites.

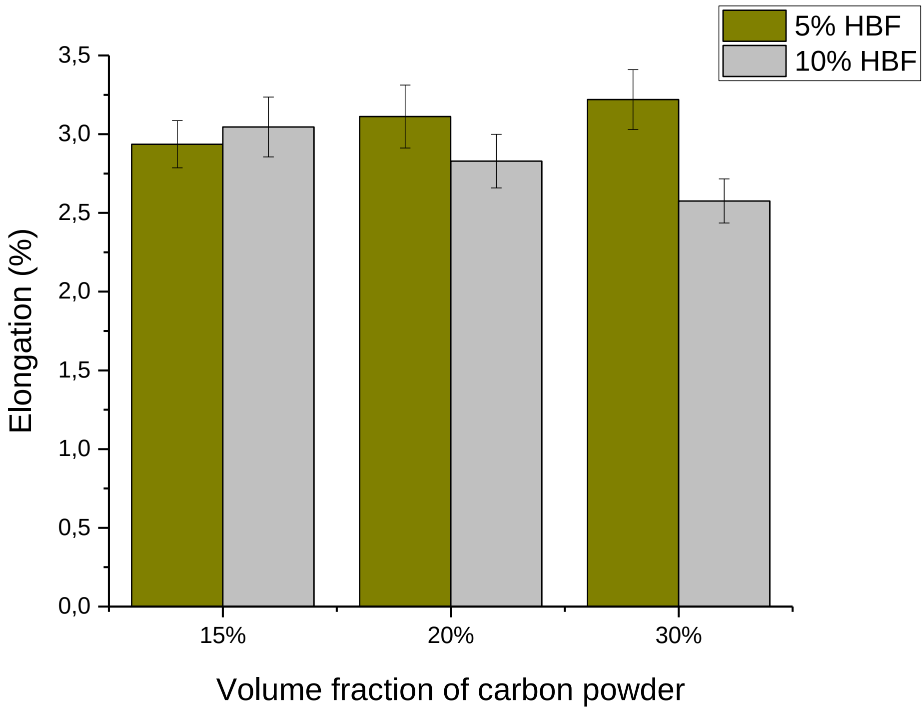

Figure 5 Elongation of HBF/CP/polyester composites

Figure 6 shows the modulus of elasticity of the developed composite. It is known that the tensile elastic modulus of the composites tended to improve with increasing CP in the 5% HBF composite. The highest value of the modulus of elasticity of composite was obtained by the composite 5% HBF/30% CP of 1707.4 MPa followed by the composites value of 5% HBF/15% CP and 5% HBF/20% CP of 1228,202 MPa and 1360,026 MPa, respectively. This increase in the modulus of elasticity is associated with the stiffness of carbon powder. This increase in the modulus of elasticity is associated with the stiffness of the carbon powder. For 10% HBF composites (see sample 10% HBF/15% CP, 10% HBF/20% CP, and 10% HBF/30% CP); in the same carbon volume fraction; was found to have lower elastic modulus values compared to 5% HBF composites. It may be assumed that the presence of 10% HBF in the composite has reduced the stiffness of the carbon powder so that the modulus of elasticity of the composite is small.

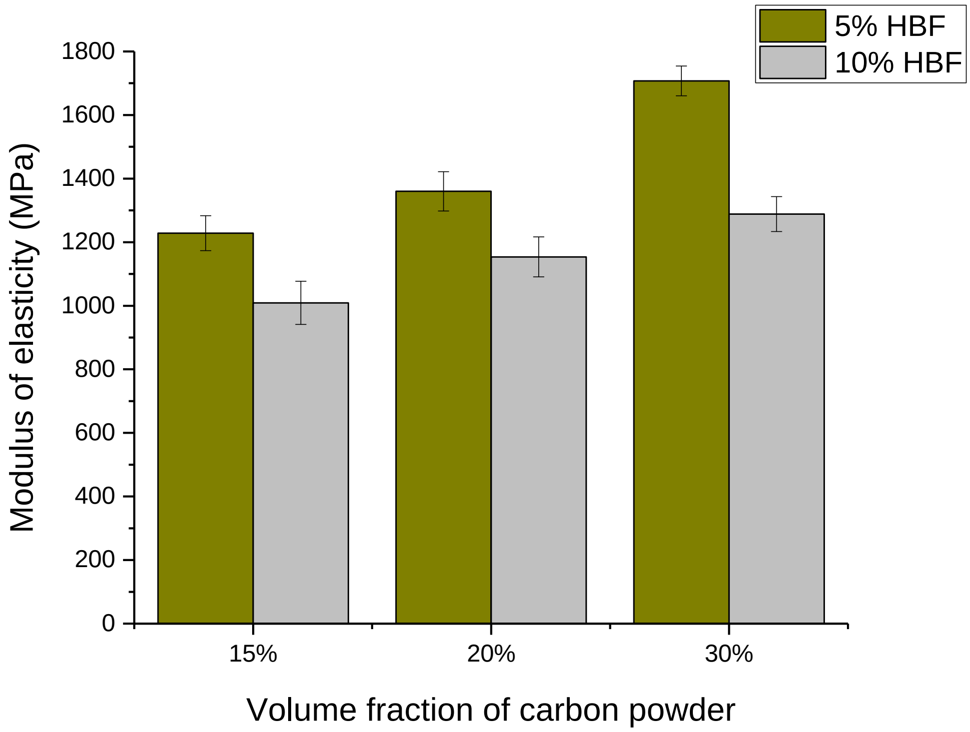

Figure 6 Modulus of elasticity of HBF/CP/polyester composites

3.2 Bending strength analysis

Figure 7 presents the bending strengths of the different composites studied. The bending strength of the composite increases with increasing CP contents. The highest bending strength of the composites was obtained from the 5% HBF/30% CP composite of 104,234 MPa. This increment occurs because the strong interfacial bond between the HBF-CP and matrix contributes to higher bending properties. However, the bending strength values decreased when HBF was 10% in polyester composites. It is suspected that there is an incompatibility of HBF (hydrophilic) and polyester (hydrophobic), which causes poor interface bonding so that bending and tensile strength of composite become small; which has been confirmed by SEM results. Besides, the lower value of the bending properties can also be attributed to powders interaction, cavity (voids), and powder disperses problems. Islam et al. (2017) [16] stated other factors that can affect the reduction in bending strength of composites are the size and non-uniform shape of powder; this factor may also answer the reason why the bending strength of an HBF 10% composite is lower than an HBF 5% composite.

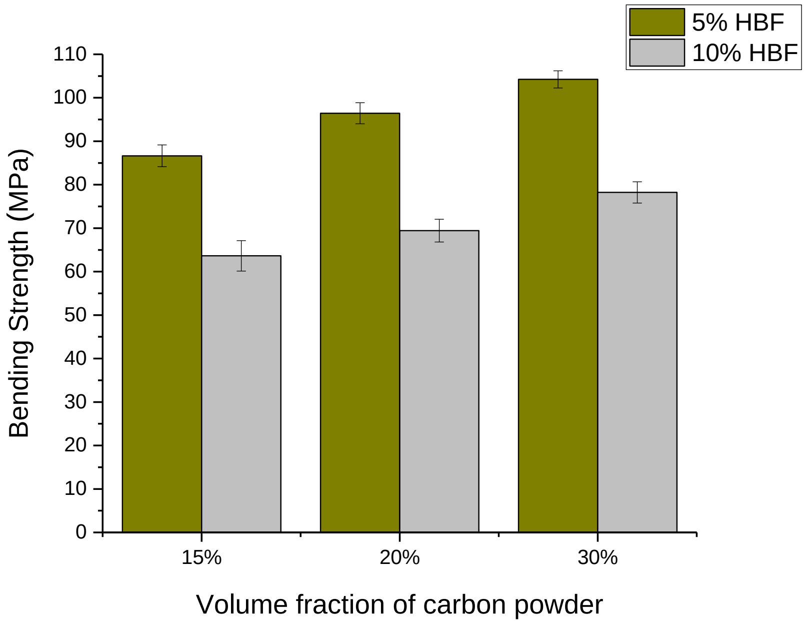

Figure 7 Effect of CP contents on the bending strength of HBF/CP/polyester composites

3.3 SEM Analysis

The fracture photos in the composite resin polyester/HBF/CP are shown in Figures 8 and 9. In Figure 8(a-c) it is found that there are voids in the 5% HBF/polyester composite. In Figure 8(a), it can be seen that the number of voids is found more with small sizes and the holes are deeper; this is why the interface bond is weak to transfer the tensile stress, resulting in low strength. However, the voids are getting fewer and wider but not deep (see Figures 8(b) and 8(c)) with an increase of 20% CP and 30% CP, and the interface is tighter; so that it is strong enough to transfer the tensile stress and produce higher strength. Furthermore, the SEM photo in Figures 9(a-c) shows the fracture of the 10% HBF/polyester composite. Figure 9(a) shows that there is a small amount of HBF pulling out, the cavities and interfaces of CP/HBF/polyester look quite tight; this is the reason why the tensile strength of the composites with a 15% carbon powder higher than the other composites studied. Furthermore, Figures 9(b) and 9(c) present an increasing number of HBF pullouts on the fracture surface of the composite; the interface bond between HBF-CP-polyester becomes weak; which may be due to the incompatibility between HBF (hydrophilic) and resin (hydrophobicity); which causes the mechanical properties of the composite to be reduced.

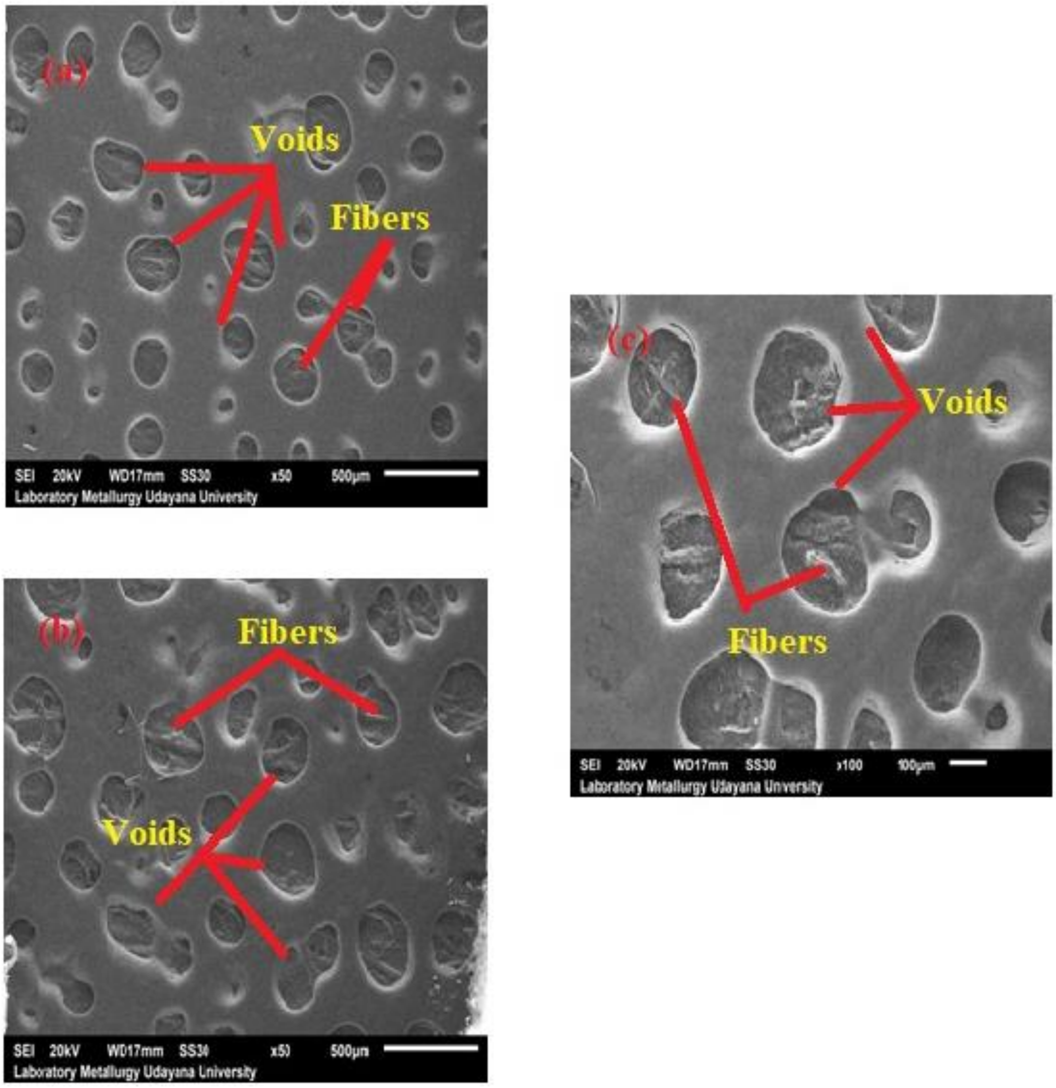

Figure 8 SEM photos of 5% HBF/polyester composites with the addition of carbon powder: (a) 15%, (b) 20%, and (c) 30% (volume fraction)

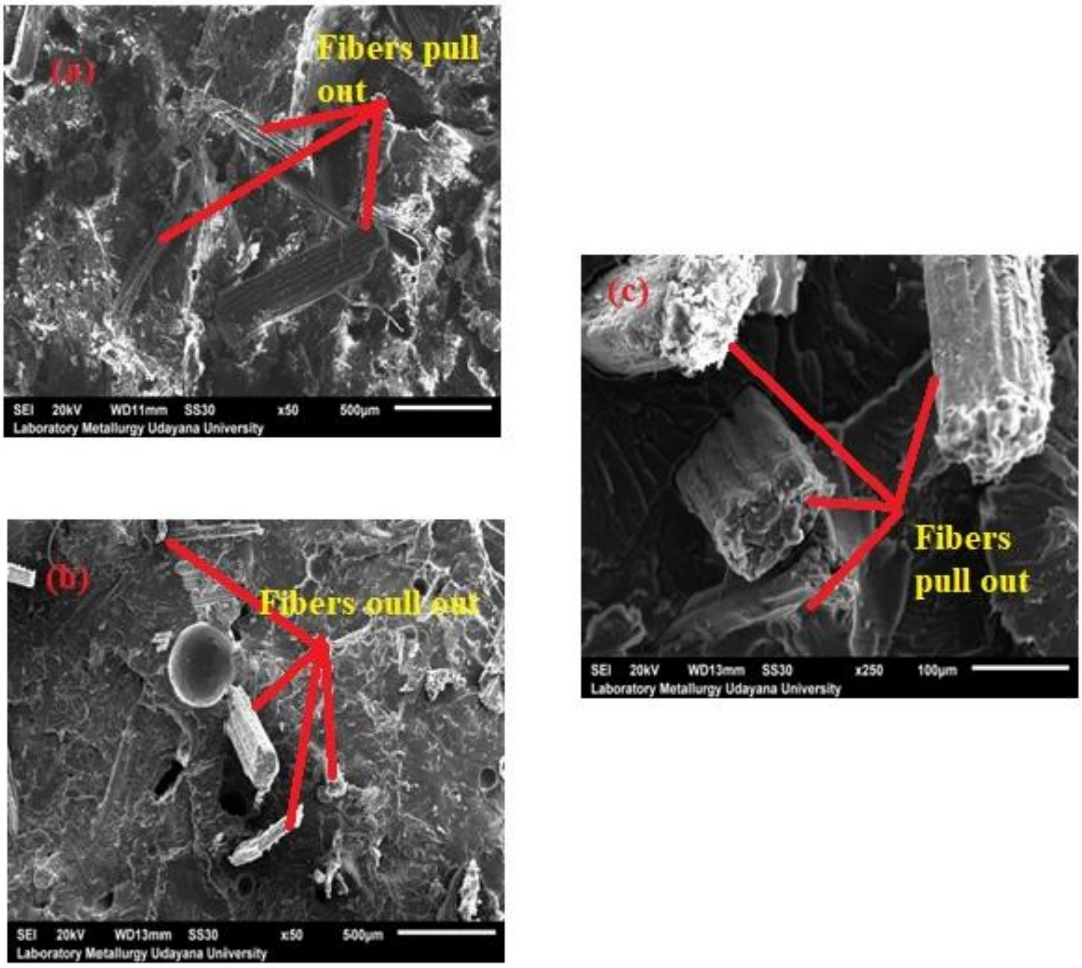

Figure 9 SEM photos of 10% HBF/polyester composites with the addition of carbon powder (CP): (a) 15%, (b) 20%, and (c) 30% (volume fraction)

4. CONCLUSIONS

The polyester/HBF composite was successfully made by incorporating carbon powder (CP) as a filler. It was revealed that the addition of CP increased the tensile and bending strength of the HBF/polyester composites, but when the HBF volume fraction in the composites increased, the mechanical properties of the composites became low due to the weak interface bond between HBF and polyester to transfer tensile stresses and HBF pullout. The highest tensile strength and bending strength were obtained from the 5% HBF/30% CP/polyester composite of 54,978 MPa and 104,234 MPa, respectively. While the highest tensile elasticity modulus was obtained from the 10% HBF/30% CP/polyester composite of 10.688 MPa.

Authors' statement

All authors have participated in conception and design, or analysis and interpretation of the data; drafting the article or revising it critically for important intellectual content; and approval of the final version.

Conflicts of interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

REFERENCES

[1]. Sari, N. H., Sanjay, M. R., Arpitha, G. R., Pruncu, C. I., Siengchin, S., Synthesis and properties of pandanwangi fiber reinforced polyethylene composites: evaluation of dicumyl peroxide (DCP) effect. Composites Communications **15**, (2019) 53-57.

[2]. Sari, N. H., Wardana, I. N. G., Irawan, Y. S., Siswanto, E., Characterization of the chemical, physical, and mechanical properties of NaOH-treated natural cellulosic fibers from corn husks. Journal of Natural Fibers **15**, 4 (2018) 545-558.

[3]. Ilyas, R. A., Sapuan, S. M., Ibrahim, R., Abral, H., Ishak, M. R., Zainudin, E. S., et al. 2019a. Sugar palm (Arenga pinnata (Wurmb.) Merr) cellulosic fibre hierarchy: a comprehensive approach from macro to nano scale. Journal of Materials Research and Technology **8**, 3, 2753-2766.

[4]. Ilyas, R. A., Sapuan, S. M., Ibrahim, R., Abral, H., Ishak, M. R., Zainudin, E. S., et al., Effect of sugar palm nanofibrillated cellulose concentrations on morphological, mechanical and physical properties of biodegradable films based on agro-waste sugar palm (Arenga Pinnata). Journal of Materials Research and Technology **8**, 5 (2019) 4819-4830.

[5]. Sari, N. H., Pruncu, C. I., Sapuan, S. M., Ilyas, R. A., Catur, A. D., Suteja, S., et al. 2020. The effect of water immersion and fibre content on properties of corn husk fibres reinforced thermoset polyester composite. Polymer Testing 91, 106751

[6]. Sari, N. H., Padang, Y. A., The characterization tensile and thermal properties of hibiscus tiliaceus cellulose fibers. IOP Conf. Series: Materials Science and Engineering **539** (2019) 012031. doi:10.1088/1757-899X/539/1/012031.

[7]. Surata, I. W., Nindhia, T. G. T., Widagdo, D. M., Promoting natural fiber from bark of *Hibiscus tiliaceus* as rope to reduce marine pollution from microplastic fiber yield from synthetic rope. E3S Web of Conferences **158**, 00005 (2020). https://doi.org/10.1051/e3sconf/202015800005.

[8]. Miriam, M., Tostado-Plascencia, Sanchez-Tizapa, M., Zamudio-Ojeda, A., Synthesis and characterization of multiwalled carbon nanotubes functionalized with chlorophyll-derivatives compounds extracted from Hibiscus tiliaceus. Diamond & Related Materials **7193,** S0925-9635 (2018) 30285-1. doi:10.1016/j.diamond.2018.09.004.

[9]. Purnowidodo, A., Suteja., Darmadi, D. B., Anam, K., Tensile Strength and Fatigue Crack Growth Behaviour of Natural Fibre Metal Laminates. Journal of Engineering Science and Technology **15**, 4 (2020) 2809 – 2822. School of Engineering, Taylor's University.

[10]. Pandiatmi, P., Dyah, E., Sastriawan., Bending Strength Optimation of Polyester Composite Strengthened By Coast Cottonwood Bark Fiber with Rice Husk Filler By Using Response Surface Method. IOP Conf. Series: Materials Science and Engineering **494** (2019) 012061. doi:10.1088/1757-899X/494/1/012061.

[11]. Ojha, S., Acharya, S. K., Raghavendra, G., Mechanical Properties of Natural Carbon Black Reinforced Polymer Composites. Journal of Applied Polymer Science (2014) 1-7, DOI: 10.1002/APP.41211.

[12]. Dias, T. da C., Panzera, T. H., Freire, R. T. S., Garcia, C. T., Santos, J. C. D., Epoxy Polymers Reinforced With Carbon Powder Wastes. 4th Brazilian Conference on Composite Materials. (2018). https://doi.org/10.21452/bccm4.2018.14.04.

[13]. Kumar, R., Varshney, S., Kar, K. K., Dasgupta, K., Enhanced thermo-mechanical and electrical properties of carbon-carbon composites using human hair derived carbon powder as reinforcing filler, Advanced Powder Technology (2018), https://doi.org/10.1016/j.apt.2018.03.004.

[14]. Mostafa, N. H., Hunain, M. B., Jassim, A., Mechanical Properties Of The Jute Fibers-Activated Carbon Filled Reinforced Polyester Composites. Materials Research Express (2019). in press. https://doi.org/10.1088/2053-1591/ab5566.

[15]. Sari, N. H., Suteja, Suteja., Fudholi, A., Zamzuriadi, A., Sulistyowati, E. D., Pandiatmi, P., Sinarep, S., Zainuri, A., Morphology and mechanical properties of coconut shell powder-filled untreated cornhusk fibre-unsaturated polyester composites. Polymer **222**, 22 (2021) 123657. https://doi.org/10.1016/j.polymer.2021.123657.

[16]. Islam, M.T., Das, S.C., Saha, J., Paul, D., Islam, M.T., Rahman, M., Khan, M.A., Effect of coconut shell powder as filler on the mechanical properties of coir–polyester. composites, Chemical Material Engineering **5,** 4 (2017) 75–82.