

Effect of fiber loading on mechanical and water absorption capacity of Polylactic acid/Polyhydroxybutyrate-co-hydroxyhexanoate/Kenaf composite

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Abstract. Natural fibers have been found to be excellent reinforcing materials for preparing polymer matrix based composites. Environmental concerns also have resulted in replacing petrochemically derived polymer with the biodegradable renewable resource. In this study, mechanical properties and water absorption of polylactic acid (PLA)/ polyhydroxybutyrate co-hydroxyhexanoate (PHBHH) matrix with blending ratio 75:25 and its composites reinforced with fibers of kenaf have been studied. The composites, with different fiber loading (10, 20, 30 and 40 wt%) were prepared by melt blending techniques using an internal mixer at 50 rpm and 165°C for 8 minutes followed by a hot compression moulding technique. The results showed that tensile strength and modulus increased with increasing of fiber content. The water absorption behavior of biocomposites as a function of days was also investigated. It was found that the water absorption amount of biocomposites increased with increasing of fiber content and exposure time. It can be summarized that the recent developments in PLA/PHBHH/Kenaf show highly promising perspectives for the replacement of traditional petrochemical based polymers in applications such automotive to encourage materials degradation.

1. Introduction

Increasing concerns about the potentially damaging effects of nondegradable plastics on the environment, fast depletion of traditional sources of fossil fuels and the increasing cost of petrochemical feedstocks has raised new challenges in polymer research. It is now widely accepted that the use of long-lasting polymers of short lived applications is not sustainable. Among the vast number of biopolymers studied, two of the most promising are polyhydroxybutyrate-co-hydroxyhexanoate (PHBHH) and polylactic acid (PLA). Both of them are biodegradable, sustainable and biocompatible polyesters that can be produced from renewable resources. They are used in consumer products by several industrial sectors and have comparable thermal and mechanical properties to those of some conventional polymers. PLA is semi-crystalline polyester derived from lactic acid, obtained entirely from renewable resources such as corn, sugar beet and wheat. It is an aliphatic thermoplastic polyester that boasts a high modulus, high strength and good clarity. The glass



transition temperature of PLA is in the range of 55 °C – 60 °C while the melt temperature is in the range of 145 °C -160 °C. It has the great mechanical strength and easy to process. PLA is now produced on a large scale and used in various applications including packaging, agriculture and textiles. Even though PLA is compatible with many current processing techniques, the fact that it has a high glass transition temperature leads to brittleness in the final products [1]. In order to resist this problem, many researchers have examined a few ways in modifying PLA, such as copolymerization or composite formation [2]. The combination of PLA with other polymers may be the suitable methodology since this process allows the incorporation of new materials with enhanced properties. This technique also less expensive compared to chemical modifications or syntheses of tailor-made macromolecules.

PHB discovered by Lemoigne in 1925, is the most representative members of the PHA family. However, its high crystallinity and large spherulites turn the material to be stiff and rigid. PHB is only suitable for processing in a narrow temperature window due to unstable thermal during conventional melt processing. To overcome these limitations, some PHB copolymers have been biosynthesized by a variety of microorganisms. PHBHH was synthesized from oils and fats using *Aeromonas* sp. OL338 and *Aeromonas* sp. FA440. The shortcomings of conventional PHAs such as PHB and PHBV can be resisted by this new co-polyester [3]. PHBHH is more suitable for making films and other soft articles [4]. This is due to the unique characteristic of PHBHH which is substantially more flexible and ductile. The randomly distributed monomer units (hydroxyhexanoate) are excluded from (hydroxybutyrate) crystalline lattice, forming a short chain branch. This branch acts as a molecular defect hence disrupts the excessive regularity of the polymer chain. Then, the melt temperature (T_m) and crystallinity also will be reduced. The selection of individual components and their proportions must be carefully considered in a composite system since it will give effect on the physical, mechanical and thermal properties of the final materials.

Natural fiber, such as kenaf fiber can be incorporated into the polymer to achieve the desired properties and better texture in the resulting biocomposites. It is a basic philosophy to create new products that are eco-friendly and sustainable. Kenaf fiber has attracted much attention for this purpose because it offers both ecological and economic advantages. Kenaf fiber was used as reinforcement in the polymer composites because of disintegration and hydrophilic properties. Kenaf fiber also suitable as a filler in composite materials because it is not abrasive during processing, biodegradable, has low density and specific mechanical properties. Kenaf can grow under a wide range of climate conditions to a height of more than 5-6 m in 6-8 months [5].

There are three different mechanisms to figure about moisture diffusion in polymeric composites [6]. The first involves the diffusion of water molecules inside the micro gaps between polymer chains. The second one involves capillary transport into the gaps and flaws at the interfaces between the fiber and the matrix. It is caused by poor wetting and impregnation during the initial manufacturing stage. The third involves transport of microcracks in the matrix arising from the swelling of fibers, especially in the case of natural fiber composites. In this work, PLA/PHBHH/Kenaf composites were prepared via direct melt compounding. The mechanical and morphological properties of the composites were evaluated as a function of fiber loadings and water absorption rate. It can be hypothesized that a composite of the PLA, which is naturally hard and brittle, with PHBHH would effectively balance the shortcomings of the individual polymers. PHBHH is preferred rather than other PHAs conventional polymer such PHB or PHBV because this conventional polymers are thermally unstable during conventional melt processing and only suitable for narrow temperature processing. There is very little research has been reported regarding PLA/PHBHH and no systematic research published concerning the impact of natural fiber on these composites.

2. Experimental

2.1 Materials

A commercial linear and semicrystalline PLA (Ingeo™, 3052D) was provided in pellet form by NatureWorks LLC, USA. According to the supplier, PLA has a melt flow rate about 14 g/10 min and a density of 1.24 g/cm³. The bacterial polyester, PHBHH, Aonilex was supplied by the Kaneka Corporation (Osaka, Japan). Mechanically retted Kenaf fiber (KF) in lengths 1.0 mm was supplied by Kenaf Fiber Industries Sdn. Bhd. (KFI), Malaysia.

2.2 Composite Processing

Before manufacturing the composite samples, the two biodegradable polymers were dried at 50°C overnight. Kenaf fibers were dried in a circulating air oven at 80°C for 12 h and were kept in desiccators prior to processing. PLA/PHB-HH/Kenaf composites were prepared by direct melt compounding using a twin roll counter rotating mixer (RTOI-55/20, POTOP, Guangzhou) at polymer blending ratio of 75/25 of PLA/PHBHH. The barrel temperature was set at 165°C and the screw speed was 50 rpm. The total mixing time was 8 minutes. It is started by adding PLA and PHBHH together and mixed for 3 minutes at 10 rpm then kenaf was gradually added and mixed for another 5 minutes at 50 rpm before being discharged. The mixtures produced were then compression molded in a hydraulic compression press model Kao Tieh Gotech Testing Machine Inc. (Taipei, Taiwan) for 5 minutes at 165°C with a pressure of 150 kg/cm². Prior to testing, the moulded specimens were kept in desiccators and conditioned at room temperature for at least 24 h.

Table 1. Designation and Composition of Materials

Designation	Materials	PLA (wt %)	PHBHH (wt %)	Kenaf (wt %)
PLA	PLA	100	0	0
PLA/KF10	PLA + Kenaf Fiber	90	0	10
PLA/KF20	PLA + Kenaf Fiber	80	0	20
PLA/KF30	PLA + Kenaf Fiber	70	0	30
PLA/KF40	PLA + Kenaf Fiber	60	0	40
PLA/PHBHH	PLA + PHBHH	75	25	0
PLA/PHBHH/KF10	PLA + PHBHH + Kenaf Fiber	67.5	22.5	10
PLA/PHBHH/KF20	PLA + PHBHH + Kenaf Fiber	60	20	20
PLA/PHBHH/KF30	PLA + PHBHH + Kenaf Fiber	52.5	17.5	30
PLA/PHBHH/KF40	PLA + PHBHH + Kenaf Fiber	45	15	40
PHBHH	PHBHH	0	100	0
PHBHH/KF10	PHBHH + Kenaf Fiber	0	90	10
PHBHH/KF20	PHBHH + Kenaf Fiber	0	80	20
PHBHH/KF30	PHBHH + Kenaf Fiber	0	70	30
PHBHH/KF40	PHBHH + Kenaf Fiber	0	60	40

2.3 Characterization techniques

2.3.1 Tensile and Flexural Tests

Tensile and flexural tests were carried out in accordance with ASTM D 638 and ASTM D 790 using a universal testing machine model Instron 3366 equipped with control system series IX. A crosshead speed of 5mm/min was used for both tests. 5 specimens were tested for every series of composites. All tests were done at room temperature.

2.3.2 Water Absorption Test

Water absorption tests were carried out according to ASTM D-570 specifications. Tensile specimens were cut from the compression-molded plates and used for the measurements of water absorption. After drying at 50°C for 24 hr to a constant weight to a precision of 0.001 g, the weight of the specimens before water immersion (W_d) were measured with a balance. The specimens were immersed in water at room temperature for 50 days. The weights of the specimens were measured at regular intervals and the amount of water absorption (M_t) was calculated according to Eq. (1):

$$M_t(\%) = \frac{W_w - W_d}{W_d} \times 100 \quad (1)$$

where W_d and W_w denote the weight of the dry material (the initial weight of materials before water immersion) and the weight of materials after water immersion, respectively. The surfaces of these specimens were thoroughly dried with tissue papers, and they were weighed immediately to determine the weight of the specimens (W_w). The specimens were immersed until they were saturated.

2.3.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was employed in order to observe the morphology of the sample's fracture surface from composites broken with a field-emission Scanning Electron microscope model Zeiss Supra 35VP. All samples were first coated with a thin layer of gold using a VG Microtech-Polaron Sputter Coater and analyzed.

3. Result and Discussion

Good strength properties of biocomposite materials can be produced when the fiber is uniformly dispersed and distributed in the matrix. PLA/PHBHH/Kenaf biocomposite tensile test sample were successfully prepared by melt blending techniques and then compression molded. The surfaces of all samples were smooth and showed brown in colour which indicating the uniform dispersion of kenaf within blends. Fiber loading and water absorption effect on mechanical properties were determined. The morphology of the fracture surface also was examined by scanning electron microscope.

3.1 Effect of Fiber Loading on Mechanical Properties

3.1.1 Tensile Properties. Figure 1 shows the effect of fiber loading on the tensile strength of PLA/PHBHH/kenaf fiber composites. In this study, PLA and PHBHH were used as the reference sample.

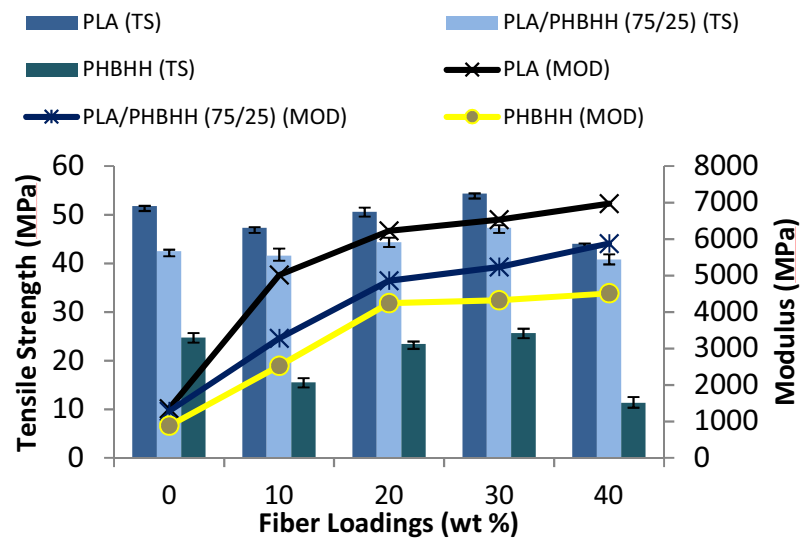


Figure 1. Tensile strength and tensile modulus of composites at different fiber loadings.

The neat PLA present the highest tensile strength results among all composites. Generally neat PLA show better tensile strength than composite PLA and these findings are in agreement with previous study [7]. In the figure, PLA/PHBHH/Kenaf composite present the tensile strength results with 47.28 MPa at 30 wt% of fiber loading. The tensile strength increased from 41.54 MPa, 44.35 MPa and 47.28 MPa with the increased of fiber loading and decreased thereafter. The strength increases until reaching the maximum filler loading at 30 wt% and decreased as more fiber was incorporated into the matrix. Suddenly dropped of strength can be seen at 30 wt% to 40 wt% of fiber loading from 47.28 MPa to 40.78 MPa.

According to the investigation, at 10 wt% of kenaf loading, the tensile strength of the materials were not improved indicating that there was poor dispersion and fiber distribution which lead to poor adhesion between the fiber and the matrix. This is due to the fact that the polymer resin unable to transmit and distribute the applied stress to the kenaf fibers resulting in lower strength. The fiber distribution and dispersion started to improve at 20 wt% to 30 wt% of fiber loading thus gives better strength compared to 10 wt% of fiber loading. Similar findings have been reported who incorporated 10–30 wt % kenaf fibre into a PLA matrix [8]. They reported that the tensile strength of the composite increased with an increase in the amount of the fibers in the composite. However, at 40 wt% of fiber loading, the tensile strength of the materials decreased due to insufficient wetting of fiber by matrix. Other than that, this might also be caused by fiber-fiber interaction and agglomerates of fiber in the matrix since there are no polymer chains between the fiber layers. The presence of fiber agglomerates would promote weak points within the composite which would greatly affect its mechanical properties. The addition of kenaf will increase the modulus, but the higher fiber content will not improve the modulus in composites. Other factors that may contribute to the enhancement of the strength and stiffness of the biocomposite materials are porosity of the composites and chemistry of the natural fiber's surfaces [9]. The porosity arises due to inclusion of air during processing, limited wettability of fibers, lumens and other hollow features within fibers/fiber bundles (which may become closed during processing at high pressure) and due to the low ability of fiber to compact. Porosity in natural fiber composites has been shown to increase with fiber content, more rapidly once the geometrical compaction limit has been exceeded, depending on fiber type and orientation of fiber; flax/PP composites were found to have porosity increasing from 4 to 8 volume% as fiber content increased from 56 to 72 m% [10]. As mentioned earlier, its inclusion in models has been shown to give an improved prediction of strength and stiffness. Overall, the results obtained suggest an almost linear increase in the modulus with the addition of kenaf fiber. This may be due to the kenaf fibers

imparting an overall increase in stiffness to the composite as the kenaf content is increased. As the fiber content in the composite increases, it restricts the mobility of the matrix, consequently the composite become stiffer and the tensile modulus increase. This kind of trend in the Young's modulus with increased fiber content in the composite was also observed in the case of polypropylene (PP)/wood fiber composites where the stiffness of the composite were found to be dependent on the fiber content and the homogeneity of the fiber dispersion [11].

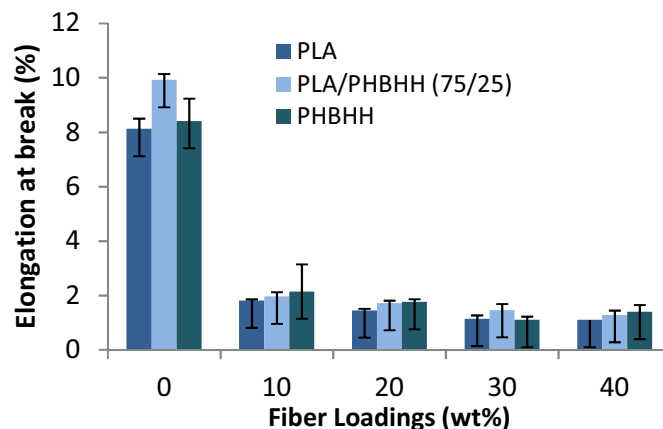


Figure 2. Elongation at break of composites at different fiber loadings.

The effect of various kenaf loadings on the elongation at break of the composite materials is shown in Figure 2. In general, the elongation at break properties of the composites decreased when 10 wt % of kenaf fiber was added and the value was gradually reduced with more addition of kenaf fiber and this trend has been expected. A reduction in strain at break with increasing fiber content in a polymer matrix is common in biocomposites [12]. PLA/PHBHH can elongate more, due to its elasticity. The blend is naturally brittle; adding more fibers in the composite should lower the strain at break and create stress-concentrated areas resulting in lower strength. It shows that the presence of kenaf fibers does not contribute to the elasticity or the final composite flexibility. The observed decrease in flexibility of the composites is likely to be related to the high stiffness of the composites. As we increased the fiber content, the composites became more rigid and the fiber restricted the stretching of composites. Therefore, the elongation at break decreased due to lower deformation behavior of the composites. There also reported that a rigid interface between the fibers and the matrix material reduces the deformability of the composites [13].

3.1.2 Flexural Properties. The effect of different kenaf loading on the flexural properties is shown in Table 2. Increasing the kenaf loading from 10 to 30 wt% resulted in an increase of the flexural strength of the composites. However, at 40 wt% kenaf loading, a reduction of about 16.32% in flexural strength was observed. This indicates that 30 wt% could be considered as the optimum fiber loading, and a further increase of kenaf loading would lead to inferior flexural strength. It is expected at 30% of fiber loading, good interfacial adhesion happened between kenaf and polymer matrix due to chemical interactions, such as hydrogen bonds. Hydrogen bonds are present in the composite between the hydroxyl groups in the kenaf fibers with the terminal hydroxyl groups [14] and the carbonyl groups of the ester groups of PLA [15]

Table 2. Flexural properties of composites at different fiber loadings

Composites	Flexural (MPa)					
	Strength			Modulus		
	PLA	PLA/PHBHH	PHBHH	PLA	PLA/PHBHH	PHBHH
0	115.80	85.45	38.32	3700	2700	1040
10	93.50	80.32	40.11	3967	2892	2243
20	96.17	81.23	41.63	4885	3445	2997
30	100.20	91.47	44.95	6021	4743	3493
40	88.46	78.63	25.80	6790	5110	3725

Insufficient wetting of the incorporated fiber by the polymer matrix with the presence of the fiber bundles and more fiber-fiber interactions had caused the decrease in flexural strength of 40 wt% of kenaf loading. According to Rowell definition, fiber bundles can be defined as a combination of individual fibers adhering together by a cementing material, usually lignin. The contact area between fibers and the matrix in fiber bundles is reduced by fiber-fiber adhesion resulting in poor stress transfer from the matrix phase to the dispersed fibers. In addition, inter-fibrillar voids also could be formed from the fiber agglomeration of entangled fibers. It will also contribute to the reduction of fiber-matrix interactions. All of these factors are detrimental to the mechanical performance of the composites, especially at high fiber loading. According to [16], the orientations of short fiber in a compression moulded specimen are usually in a combination of random-in-plane orientations of multi-layer composite laminates. Increasing the kenaf loading will increase the possibility of fibers in same lamina or different laminas orienting in a better direction to bear the bending load. However, there is an optimum fiber loading to be considered that will promote to fiber-fiber interactions or agglomeration. In this case, the fibers have the possibility of contacting each other either with fibers in the same laminate or fibers in neighbouring laminates leading to lower flexural properties.

The flexural modulus of the PLA/PHBHH/Kenaf composites shown to be linearly increased with increasing (0-40 wt%) kenaf content. High kenaf content combined with the stiffness of the fiber could explain the significantly enhanced flexural modulus of PLA/PHBHH/Kenaf composites. Kenaf might restrict the mobility of the polymer chain and thus hinder the ability of the polymer to deform. These observations are supported by [17], who concluded that good fiber dispersion in the composite system could be confirmed by observing the linear increase in the composite's modulus.

3.2 Effect of Water Absorption on Mechanical Properties

It was found that the water absorption amount of the composites increased with increasing the cellulose content. As it can be seen in the Figure 3, 10 days of soaking presented a rapid increase the level of water absorption then demonstrated a gradual increase in the water uptake, then the composites finally reached an equilibrium point, which is no more water uptake into biocomposites and the weight of biocomposites remains constant. As reported [18], the number of hydroxyl (OH) group in the composites increased when the cellulose content increased, resulting in increasing the water absorption due to these free OH groups come in contact with water and form hydrogen bonding, which results in weight gain in the biocomposites.

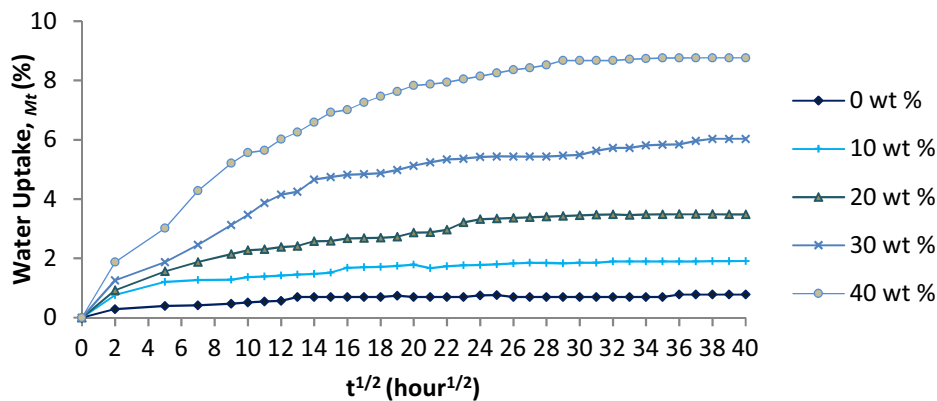


Figure 3. The percentage by weight of PLA/PHBHH/Kenaf composite after water absorption at different kenaf fiber loading

The higher the fiber loading is, the more hydrophilic sites exist in the composites and the higher water absorption of the composites. It is expected that the exposed fibers, which are located at or near the specimen's surface, absorb moisture faster than those in the interior parts (i.e., fibers that are entrapped or coated by the matrix). That is the reason behind the fast absorption of water in the composite with 40% KF since the composite has more fibers on the surface to absorb water faster. On the other hand, fibers in PLA/PHBHH/KF20 were mostly coated by the hydrophobic PLA and PHBHH matrix, which acts as a barrier for the diffusion of water, and, therefore, their absorption rates were slower.

3.2.1 Tensile Properties. The tensile properties of natural fiber composites decreased after water absorption because of the plasticizing effect of the water molecules, which weaken the interfacial bond strength between matrix and kenaf. When composites were exposed to moisture, the hydrophilic kenaf fiber starts swelling. As a result of fiber swelling, micro cracking of the brittle thermoplastic resin occurs. The cellulose content in kenaf fiber, then contributes to more water penetrating into the interface through microcracks induced by swelling of the fibers creating swelling stresses leading to composite failure. In other words, the higher hydrophilic character of fiber, which facilitates water absorption, promoting the degradation of the fiber-matrix interface, deleteriously affects the tensile strength of the composite. The lower tensile strength at higher fiber contents could be due to the lack of matrix that leads to inefficiency of stress transfer from the matrix to fibers and vice versa as reported [19].

Table 3. Tensile Properties of PLA/PHBHH/Kenaf after water absorption at different kenaf fiber loadings.

Composite	Tensile Strength (MPa)		Tensile Modulus (MPa)		Elongation at break (%)	
	Dry	Wet	Dry	Wet	Dry	Wet
0	42.45	41.32	1277	950	9.92	8.72
10	41.54	40.24	3277	1376	1.96	1.54
20	44.35	42.75	4852	2472	1.72	1.32
30	47.28	45.34	5237	2526	1.46	1.10
40	40.78	38.53	5876	2114	1.28	0.83

The decline in tensile modulus is expected because of the weakening of the fiber/matrix interface and the plasticization of the matrix and fiber. The normal trend for thermoplastic composites filled with solid fibers showed the tensile moduli of the composites increased as the fiber loading increased.

This is due to the restriction of the mobility and deformability of the matrix with the introduction of mechanical restraint.

3.2.2 Scanning Electron Microscopy (SEM) micrograph. A SEM micrograph of the fractured surface in Figure 4 (a) shows the fiber surface are clean, which indicated poor adhesion between the fibers and the PLA matrix. The composite specimen with 20 wt% of the filler consists of the considerable amount of pores. This porous structure leads to the poor tensile properties of the composites. It is rational to suggest that 20 wt% fiber-reinforced composite specimen were subjected to poor homogenous mixing as fiber fractures are evident. This phenomenon leads to a decrease in stress transfer capability of the matrix to the reinforcement. As a result, a decrease in tensile strength is observed. It also can be seen Figure 4 (b) evenly distribution of kenaf fibers in the 30 wt% PLA/PHBHH/Kenaf composite. The kenaf fibers were randomly distributed across a unit area and were separated from each other without the presence of clumps or agglomerates.

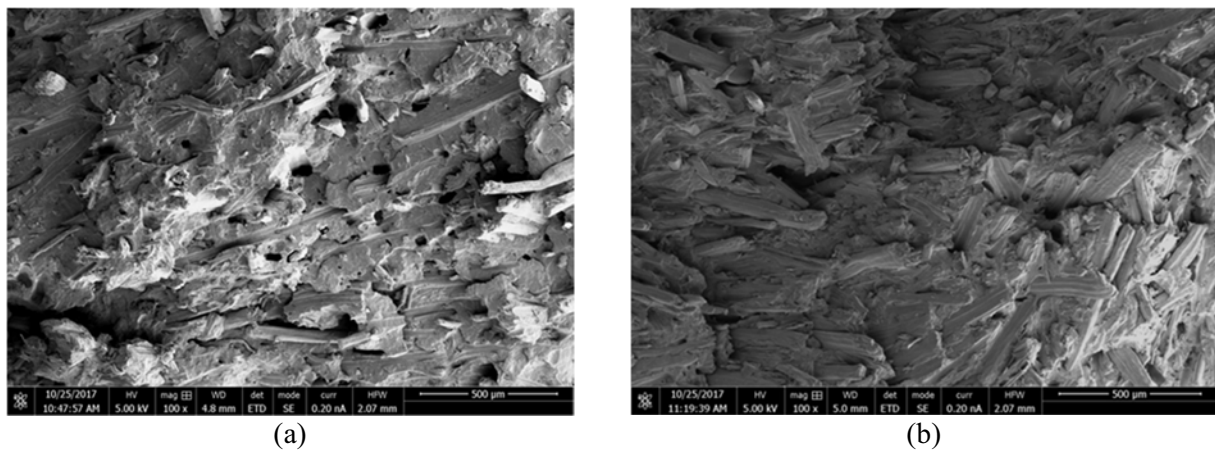


Figure 4. The FESEM micrograph image of the kenaf distribution of the (a) 20 wt% and (b) 30 wt% PLA/PHBHH/Kenaf composite.

The fractured surface of PLA/PHBHH/ KF (40 wt %) is shown in Figure 5 in which a pullout fiber can be seen clearly. Thus, the fracture surface of the composite appeared to be dominated by pull-out damage rather than fiber breakage. In fact, there was some fiber debonding observed. The strong interface between fiber and matrix results in composite with high flexural strength. The failure indicates the separation between fiber and matrix. When the content of reinforced fiber reached above the certain value; these excessive fibers affect the even combination between matrix and fiber.

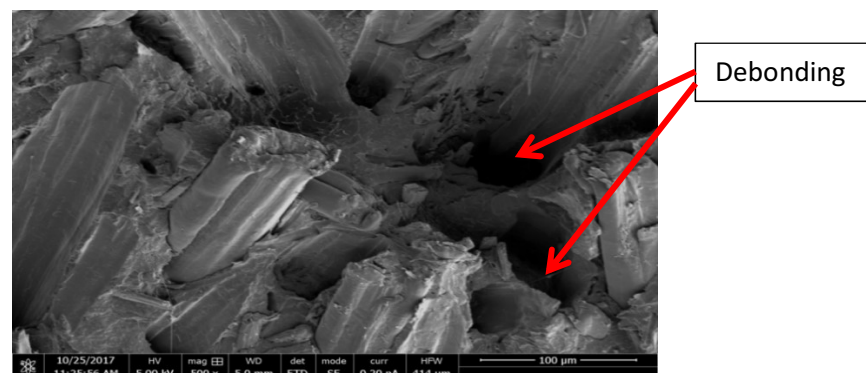


Figure 5. The FESEM micrograph image of the kenaf distribution on the fracture surface of 40 wt% PLA/PHBHH/Kenaf composite.

4. Conclusion

The effect of kenaf fiber loading and water absorption on the mechanical properties of PLA/PHBHH reinforced kenaf has been studied and concluded as follows. It shows that mechanical properties of composite increase when kenaf being incorporated into it. The reinforcement helps in stress transfer within the composite compared to the one without kenaf. However, the properties of the composite after water absorption are lower than dry composite due to weak interfacial bond between matrix and kenaf since plasticization effect occurs. Exposure to moisture results in significant drops in tensile properties due to the degradation of the fiber-matrix interface.

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