

Mechanical properties of kenaf fibre reinforced floreon biocomposites with magnesium hydroxide filler

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ABSTRACT

This paper presents a study of the mechanical properties of Kenaf fibre (KF) reinforced floreon (FLO)/ magnesium hydroxide (MH) bio-composites. The mixing of all materials was done by using a 21 mm lab twin screw extruder followed by hot pressing. The composite sheet was then cut into specimens for testing purposes. The scanning electron microscopy (SEM) was used to study the cross-section of the interface. In this regard, insufficient resin for fibre wetting, hydrolytic degradation on the biopolymer and poor interfacial bonding were attributed to the low strength profile. Yet, further addition of KF increased the tensile strength and flexural to 18.91 MPa and 73.09MPa, respectively. Nevertheless, inserting KF and MH filler were found to have a positive outcome on the flexural modulus by especially 10KF5MH and 10KF10MH for 3.02GPa and 3.17GPa, respectively. Insertion of KF and MH showed the deterioration of impact strength. However, addition of KF increased the impact strength to 16.82 J/m². FLO is a hydrophobic biopolymer, and showed only 0.49% of the total water absorption in 14 days. Meanwhile, for the first 24 hours, the rates of water absorption were very high for all bio-composites. Hence, it is worth mentioning that the high contents of KF in bio-composites were found to have higher saturation period and higher total amount of water absorption while the MH caused shorter saturation period but lower total amount of water absorption. However, incompatibility of the interface bonding had increased the water absorption of KF/FLO/MH composites. 5KF5MH and 10KF5MH recorded water absorption at 10.65% and 13.33%. On the other hand, 10KF10MH was saturated at day 6 with 6.59 % of water absorption. Although 10KF5MH specimen did not have the best performance in mechanical properties, higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in the advanced sector, especially a hazardous environment.

Keywords: Kenaf fibre; Floreon; Biocomposites; Mechanical properties; Magnesium hydroxide.

INTRODUCTION

Floreon (FLO) is an advanced novel biopolymer, which was recently invented in November 2013 [1]. The FLO is mainly composed of standard polylactic acid (PLA); hence, it is equipped with biodegradable properties. Furthermore, 160 °C of processing temperature provides a lower production energy and low chance for fibre thermal degradation [2-5]. These features have garnered much interest from the public. Other than being fully biodegradable and easily decomposed into soil, the FLO can undergo mechanical recycling by using lower energy. It is done to reproduce biopolymer that can reduce land waste problems. In this regard, only half of the production energy is needed [6]. The FLO has also been proven to have better toughness, strength and durability, as compared to conventional polymer [7]. In the meantime, biopolymer could lose its strength with ultraviolet (UV) degradation. FLO has a good UV stability and has hence been promoted for the use in lithographic printing. In this light, natural fibres are commonly selected as composite reinforcement to enhance performance of materials [8-17]. Solving environmental issues and better tool life are the main reasons being selected [18, 19]. Besides, producing composites with lower density and cheaper cost is expected [20]. However, unfortunately, many factors have caused fibres to differ from each other. Inherently, inconsistencies of the component composition are the main reasons for the wide range of properties [21].

A previous study found that kenaf fibre (KF) reinforced PLA biocomposites have better tensile properties compared to pure PLA matrix [22]. This showed that the KF has a good interfacial bonding with PLA matrix. Another study conducted some experiments towards KF reinforced PLA biocomposites on basic mechanical properties (flexural properties, impact resistance and compression properties) as a potential material for construction material [23]. The results obtained indicated that the properties of KF reinforced PLA biocomposites are superior to most types of traditional building composites. However, higher water absorption behaviour is expected due to the hydrophilic nature of KF. Apart from that, voids presented between KF and PLA had increased the water uptake behaviour. These voids were caused by the poor interfacial bonding between the KF and PLA [24]. To reduce these voids, alkalization treatment by using sodium hydroxide on KF has improved the mechanical properties compared to untreated short KF. The 6% concentration of sodium hydroxide was found to be optimum in terms of cleaning the fibre surfaces [25, 26]. On the other hand, insertion of flame retardant filler into KF reinforced PLA biocomposites has deteriorated the tensile strength [27]. It is believed that the flame retardant has caused microstructural defects in the matrix. Besides that, a degradation of polymer is always accompanied by a reduction of its molecular weight while magnesium hydroxide (MH) is reported to be a cursor on controlling the polymer degradation rate [28], where the insertion of MH into PLA matrix has increased the rate of degradation due to the lower molecular weight found in MH contented composites. Thus, it is concluded that the higher MH concentration in PLA matrix has caused a higher rate of PLA degradation. In addition, lower strength properties are expected for low molecular weight polymer [28]. From the reviews above, it is evident that there is no previous work conducted on the mechanical properties of KF reinforced FLO composite, particularly by using MH as flame retardant filler. Therefore, the aim of the present work is to study the effects of MH inclusion on the mechanical properties of KF reinforced FLO composites.

METHODS AND MATERIALS

Materials

The polymer used in this work was Floreon (FLO) resin Grade 100, which was supplied by The University of Sheffield. The short kenaf fibre (KF) with the average length of 8-15mm was used as the reinforcement fibre and was supplied by Tazdiq Engineering (002203302-V). Meanwhile, non-toxic flame retardant to enhance the fire barrier properties came from the 95% purity of Acros Organics Magnesium hydroxide (MH) which was purchased from Fisher Scientific UK Ltd, while the sodium hydroxide (NaOH) used in the alkali treatment was contributed by APC Pure, UK.

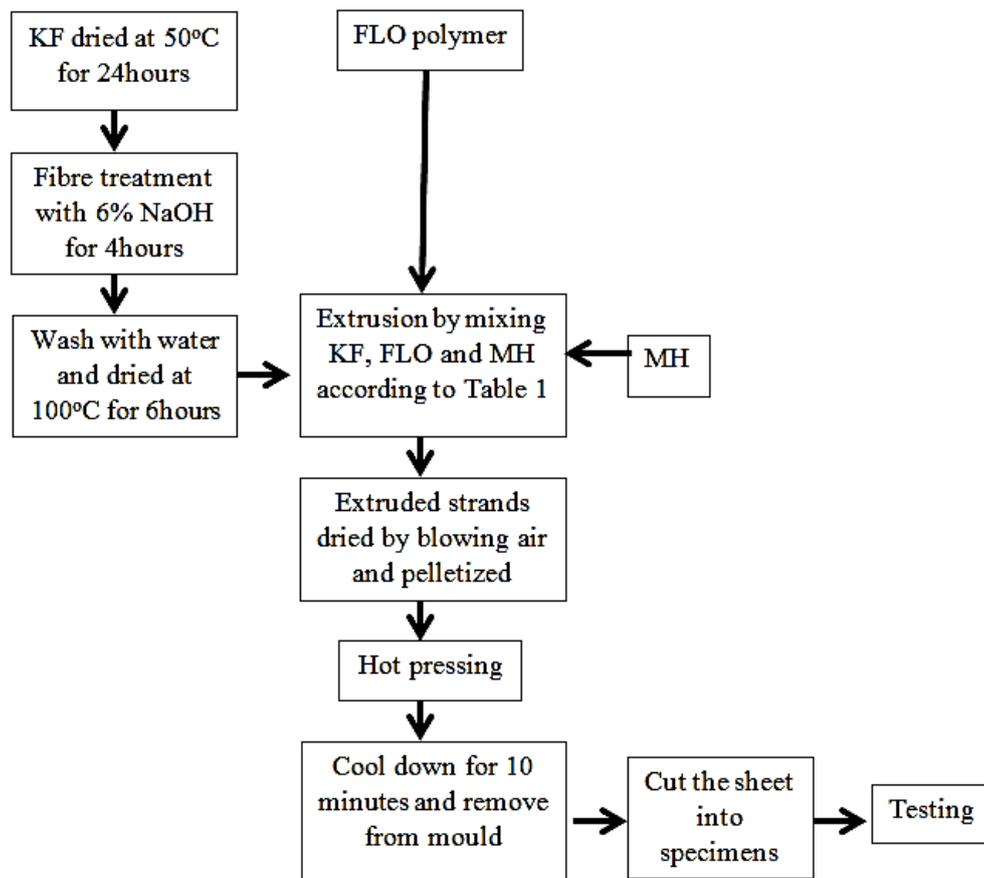


Figure 1. Process flow chart.

Processing of Composites

Table 1 lists the different ratios of FLO composite samples, which were prepared using a 21 mm lab twin screw extruder. Here, KFs were dried for 24 hours at 50 °C before being treated with 6 % NaOH, for 4 hours. Then, it was washed and rinsed by using water and dried for 6 hours at 100°C. The working temperature for the extrusion during compounding was set at 180 °C at the die head and was increased to 186 °C at the feed section with 50RPM. The process method was adapted from a previous study [29]. The extruded strands were then dried by blowing air before being pelletised. The pellets were then carried to a hot press machine in The University of Sheffield Csic laboratory for sheet fabrication. The setting of the hot press machine was set to five bar of pressing

pressure for 10 minutes, before going through 10 minutes of pre-heating. The mould was then allowed to cool for 10 minutes in room temperature. The process flow is shown in Figure 1.

Table 1. Combination formula of KF/FLO/MH composites.

Sample name	Floreon (FLO), wt%	Kenaf fibre (KF), wt%	Magnesium Hydroxide (MH), wt%
Pure FLO	100	-	-
5KF	95	5	-
10KF	90	10	-
5MH	95	-	5
10MH	90	-	10
5KF5MH	90	5	5
5KF10MH	85	5	10
10KF5MH	85	10	5
10KF10MH	80	10	10

Characterisation Techniques

Scanning electron microscopy (SEM) was used to study the morphology of the specimen where the cross-sections of flexural testing specimen represent the location of study. The accelerating voltage of 15kV helped to image the cross-sections. Tensile tests were carried out with ASTM D638-14 using the Instron 5kN machine located in Universiti Putra Malaysia (UPM) [30]. A one mm thick dumbbell specimen was cut from the moulded sheet while a cross-head speed of 1mm/min was used and the test was performed at 25 °C. Flexural testing was conducted using the same Instron 5kN machine in UPM according to the ASTM D790-10 testing standard [31]. The dimension of the specimen was 96 mm × 12.7 mm × 5mm, which included 10% of the support span for overhanging. The un-notched specimens were subjected to Charpy impact testing which adheres to ASTM D6110-10 [32]. The dimension of the test specimen was 127 mm × 12.7 mm × 5 mm. Each specimen was measured with a Vernier caliper before the impact testing was conducted where the accuracy of the reading was up to 0.1 mm. The specimen was then positioned horizontally on the supports of the impact testing machine. The pendulum was raised and secured in the release mechanism and the indicating board was reset. The pendulum was then released to provide impacts to the specimen; consequently, the indicated readings were recorded for impact strength calculation.

Meanwhile, to test water absorption, the test specimen was initially dried at 50 °C for 24 hours by following ASTM D570-98(2010)e1 [33]. The specimens were then placed so that they were entirely immersed in a container of distilled water which was prepared by the UPM chemical laboratory. At the end of the first, second and 24th hours, the specimens were removed from the water and all of their surfaces were wiped off with a dry cloth. Here, the weighing scale was nearest to 0.001g and one specimen was put back into the container before the next one was taken out and the process was continued until all the specimens had been processed. After that, these drying and weighing procedures were repeated for every 24 hours until day 7. Then, the repeating duration was changed to every week (7 days) until all specimens had been saturated. In this light, each specimen was considered as saturated when only less than 1 % of its total weight (5mg) increased. All in all, five specimens were tested for each group, and the average results were recorded and reported.

RESULTS AND DISCUSSION

SEM Morphology

The scanning electron micrographs for all samples, except the pure FLO polymer, are shown in Fig. 2(a-h). The chemical treatment of the fibre surface had created good interfacial bonding between the fibre and the matrix (figure 2a, b). Meanwhile, figure 2c and d show a coarser surface under SEM due to the insertion of the MH fillers, which is similar to what has been found in a previous study as a result of severe agglomerations and less uniform distribution of the MH filler [34]. On the other hand, figure 2e-h show the formation of voids between the fibre and the matrix. This high degree of pull-out indicates poor interfacial bonding [35]. In this light, the deterioration of mechanical properties was expected [36, 37].

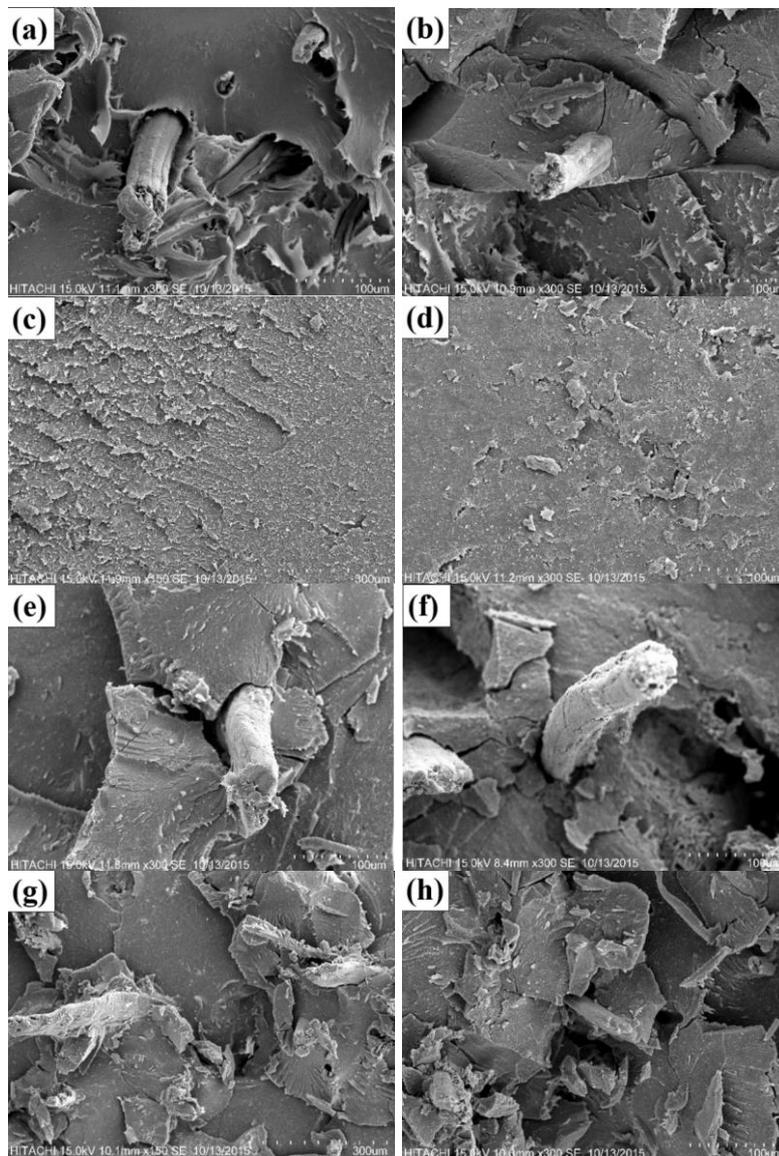


Figure 2. SEM micrographs of the (a) 5KF; (b) 10KF; (c) 5MH; (d) 10MH; (e) 5KF5MH; (f) 5KF10MH; (g) 10KF5MH; (h) 10KF10MH.

Tensile Properties of Composites

Figure 3 and figure 4 show the tensile strength and tensile modulus for KF reinforced FLO composites with MH fillers. The net FLO polymer presents the best tensile strength compared to the other samples and compared to other specimens. This is due to poor interfacial adhesion between fibre and matrix and the voids created [38]. The low fibre content had shown a lower load transfer ability, and then the stress was accumulated within the matrix, causing the composite to fail [39]. Therefore, insertion of 5 wt% of KF did not improve the tensile strength. However, the tensile strength had increased with the increase of fibre loading. The tensile strength had increased by 25.2 % from 15.1 MPa to 18.9 MPa for 5KF to 10KF because there are more reinforcement fibre content in 10KF to withstand more load before breaking compared to 5KF [40]. This suggests that the fibre dispersion and distribution had started to improve. Similar results indicated that the tensile strength had shown significant improvement for the fibre volume of more than 30 wt% [41]. Hence, Ibrahim *et al.* [42] concluded that 30 wt% of KF is the optimum loading for PLA biopolymer. Furthermore, the insertion of the MH showed a negative impact on the tensile properties, where the MH was found to be a cursor for controlling the polymer degradation rate and accelerated the rate of degradation of composite [28]. The highest drop (56.5 %) in tensile strength was found in 10KF10MH, but not in 10KF5MH. Besides that, high fibre and filler contents in the composites had caused insufficient resin to transfer the load applied [43]. A bad wetting affects the load transfer mechanism in the composite, leading to low strength properties. Insufficient resin was also found to face difficulties to maintain its structure. In this case, the sample would fail before it could reach the highest absorbable strength of the fibre. On the contrary, the highest modulus was achieved by 10KF as agreed by previous studies [38]. On the other hand, MH insertion reduced the modulus of composites, regardless of the KF contents in the composites. Other factors like porosity of composites and composite's crystallinity were also accounted to the variations of the stiffness and strength of the composite [42].

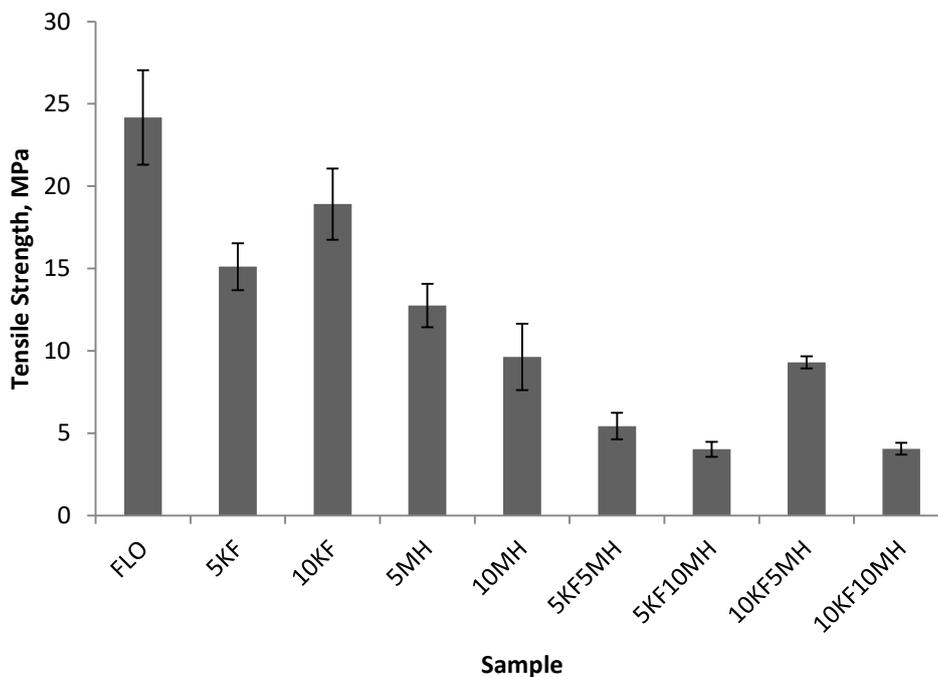


Figure 3. Tensile strength for the KF reinforced FLO composites with MH fillers.

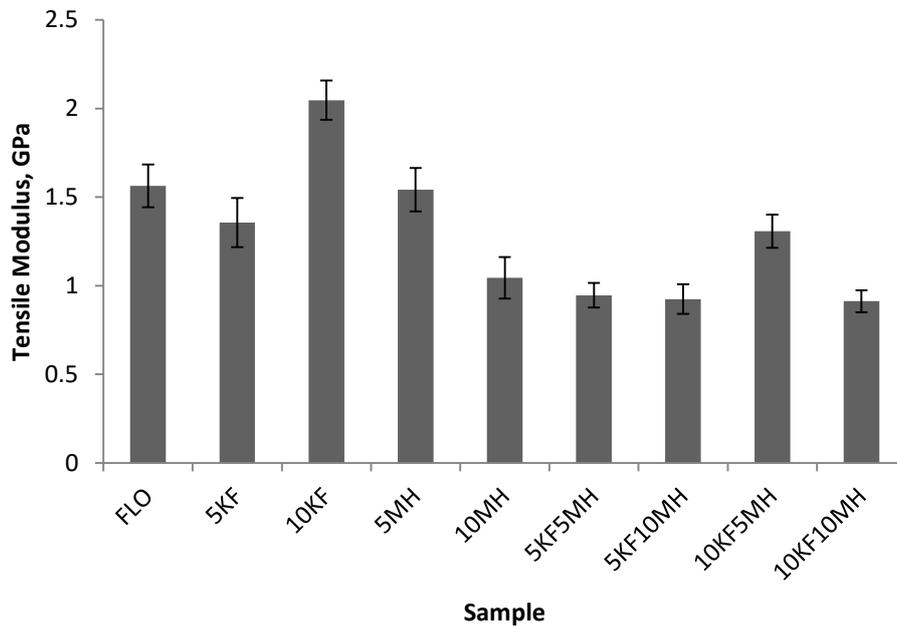


Figure 4. Tensile modulus for the KF reinforced FLO composites with MH fillers.

Flexural Properties of Composites

Figures 5 and 6 show the flexural strength and flexural modulus for the KF reinforced FLO composites with MH fillers, respectively. The flexural strength of the FLO was the highest amongst all composites and the reduction of flexural strength was recorded after the insertion of KF and MH fillers. Hydrolytic degradation, which is known as random hydrolysis on the PLA, had been agreed by a previous study as a reason for lower flexural strength [44]. Meanwhile, the shorter molecular chain of the FLO had decreased its ability to withstand the bending force [45, 46]. As the higher loading of the hydrophilic KF contains more hydroxyl groups to induce hydrolytic degradation, hence, further deterioration of the flexural strength was found. The flexural strength had reduced by 29.1 %, from pure FLO to 5KF. However, it had increased by 24.4% from 5KF to 10KF. The higher strength properties were contributed by the addition of KF. However, high temperature of the thermo-processing had induced some chain scissions to occur in the biopolymer. On the contrary, the degradation becomes faster as the MH concentrations increased [28]. Meanwhile, the flexural strength had reduced by 32.6 % and 54.1% for pure FLO to 5MH and to 10MH, respectively. Besides that, the low compatibility of the KF and MH fillers on the FLO was another factor for the reduction in flexural strength. This is because the load transfer within the composite was not working well under the low compatibility condition. A similar finding was reported by another study [47]. On the other hand, the flexural modulus of the composites was found to be affected by the KF and MH, where the insertion of KF and MH fillers will gradually increase its flexural modulus. The 10KF5MH and 10KF10MH showed positive results on flexural modulus where both had higher flexural modulus values of more than 3.00 GPa, indicating the rigidity. The differences between values of tensile modulus and flexural modulus were found to be identical in a past study, showing a difference in the trending of tensile and flexural modulus [38]. It was found that difference in the heterogeneous cross section

and shear deformation between the tensile and flexural test have created differences in the modulus value [48].

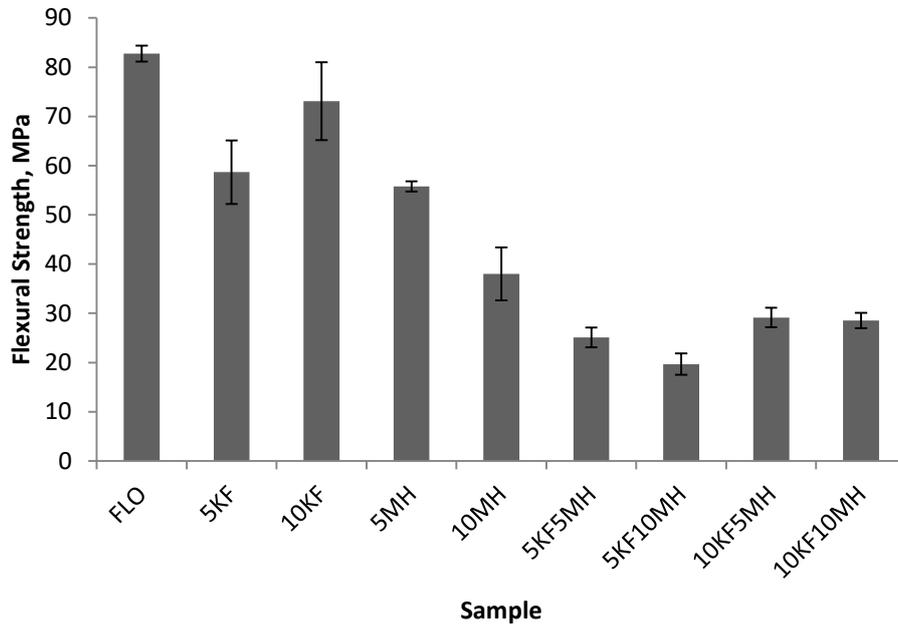


Figure 5. Flexural strength of the KF reinforced FLO composites with MH fillers.

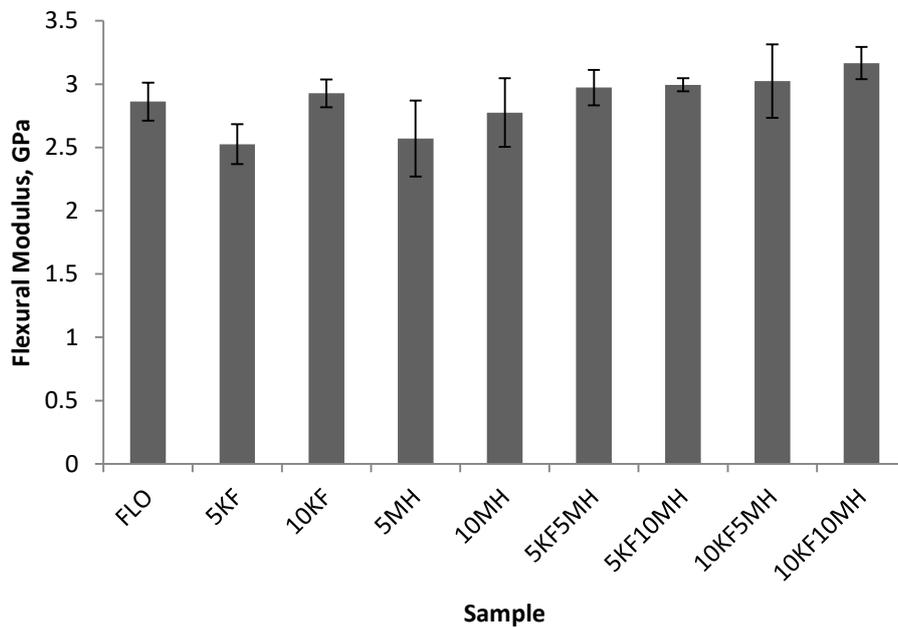


Figure 6. Flexural modulus of the KF reinforced FLO composites with MH fillers.

Impact Properties of Composites

Impact strength is a measurement of the capability of a material to absorb energy, which occurs when a sudden load is applied to the materials. Thus, un-notched Charpy impact testing was conducted and the results are shown in Figure 7. During the testing, all of composite samples were broken completely into two parts due to the brittle nature of the biopolymer [49]. In this light, the biopolymer is a significant factor for analysing the

impact strength. Brittle biopolymer has exhibited a low impact strength [50]. However, high impact strength of the FLO is expected and highlighted by the manufacturer [7].

On the other hand, the reinforcement fibre absorbed the energy for de-bonding and pull-out, which helped to enhance the impact strength of the composite. Therefore, a good interfacial bond between the fibre and the matrix is important [51]. Furthermore, although a low content of fibre in the reinforced composite has a lower impact strength than pure FLO, the increment of the KF loading in the composites (5KF to 10KF) had increased the impact strength by 49.62%, which has also been noted in a previous work [52]. As this study used short fibres as reinforcement, consequently, the high amount of fibre end acted as a stress concentration point, creating uneven energy transfer throughout the composite [53]. Besides that, the random distribution of the short KF, as can be seen from Fig. 1 (a-h), had caused lower impact energy values. On the contrary, the impact strength had reduced 5.8 %, 31.8 % and 11.4 % for 5MH to 10MH, 5KF5MH to 5KF10MH and 10KF5MH to 10KF10MH, respectively. Such reduction of the impact strength was expected with the insertion of MH to the KF reinforced FLO composites [54]. The weak bonding between KF-MH, KF-FLO and MH-FLO had reduced its impact strength.

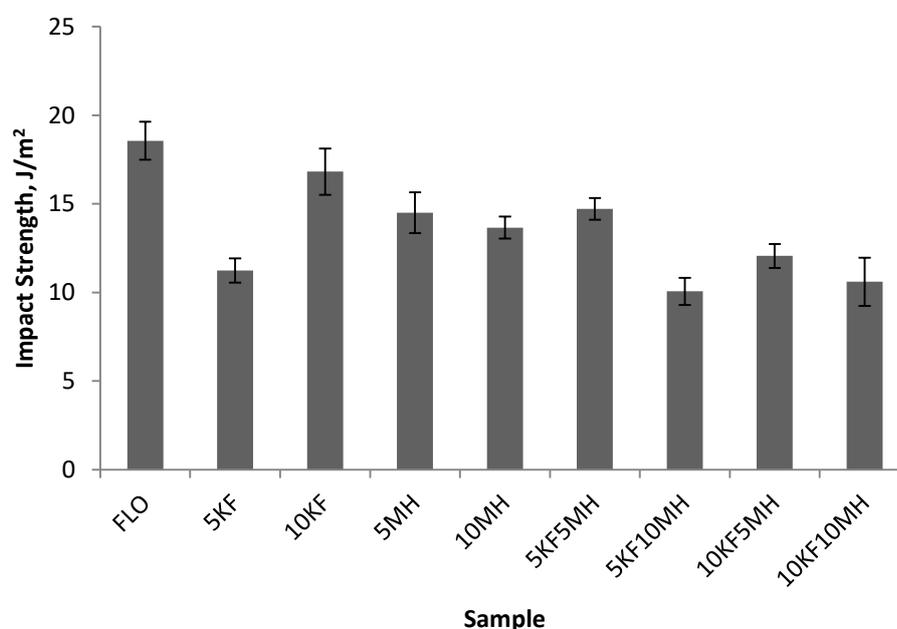


Figure 7. Impact strength of the KF reinforced FLO composites with MH fillers.

Water Absorption Behaviour of Composites

The effects of the KF content and the MH loading on the water absorption in 24 hours, 14 days and until saturation are shown in table 2. The rate of water absorption was very high for the first 24 hours and until it reached saturation. The water absorption of all composite samples was higher compared to the water absorption of FLO. This is because FLO is a hydrophobic biopolymer, which showed only 0.49% of the total water absorption in 14 days. Inherently, the insertion of the KF and MH had shifted the composite to become more hydrophilic in nature. Moreover, in the first 24 hours, none of the samples had reached the saturated water uptake. However, 5KF10MH, 10KF5MH and 10KF10MH had recorded 5.78 %, 3.52 % and 2.95 % of water absorption respectively. This high absorption rate was due to the high void content in the samples which was caused by the incompatibility of the interface bonding [20]. Several samples, including

FLO, had reached saturation after 24 hours. Furthermore, 5KF10MH and 10KF10MH were saturated at day 6 with 9.83 % and 6.59 % of water absorption respectively. In the meantime, 10MH and 10KF5MH were found to be saturated at day 21 with a great difference in total water absorption. These differences were mainly due to the insertion of hydrophilic KF in the 10KF5MH, which induced an extra 11.7 % of water absorption before saturation. The highly cellulose content of KF contents hydroxide group, which is hydrophilic in nature, had also caused the high water absorption of the specimen [55]. Annuar H. et al. also confirmed that the voids presented between KF and PLA had increased the water uptakes [24]. Consequently, 5KF and 10KF, which constituted the KF reinforced biocomposites, had shown a steady water absorption up to day 63. The composite with higher KF content took a longer time to reach saturation but had a higher total water absorption, as confirmed by Rashdi [56].

It is also worth mentioning that the high content of MH fillers contributed to shorter saturation time and lower total water absorption. In this regard, 5MH was saturated at day 35 with 5.94 % water absorption while 10MH was saturated at day 21 with only 1.63 % of water absorption. This may be due to the hydroxyl group in the MH formulation which had strongly attracted water and caused faster rate of water absorption. However, the fine powder of the MH had attributed to the lower void content and lesser empty space for water absorption.

Table 2. Results of water absorption.

Specimen	Water absorption, %								
	1h	2h	24h (Day1)	48h (Day2)	72h (Day3)	96h (Day4)	120h (Day5)	144h (Day6)	168h (Day7)
FLO	0.01	0.03	0.21	0.32	0.34	0.36	0.39	0.40	0.49
5KF	0.07	0.14	0.59	1.02	1.33	1.49	1.82	1.98	2.20
10KF	0.21	0.27	0.81	1.30	1.68	1.94	2.45	2.71	2.93
5MH	0.10	0.17	0.55	1.06	1.40	1.65	2.14	2.38	2.73
10MH	0.10	0.12	0.31	0.52	0.71	0.82	1.06	1.17	1.35
5KF5MH	0.07	0.20	1.18	2.02	2.67	3.05	3.82	4.21	4.69
5KF10MH	1.30	1.71	5.78	7.58	7.71	8.24	9.30	9.83	-
10KF5MH	0.49	0.66	3.52	5.67	7.07	7.77	9.16	9.86	10.41
10KF10MH	0.41	0.55	2.95	4.73	5.83	6.02	6.40	6.59	-

Specimen	Water absorption, %							
	336h (Day14)	504h (Day21)	672h (Day28)	840h (Day35)	1008 (Day42)	1176h (Day49)	1344 (Day56)	1512h (Day63)
FLO	-	-	-	-	-	-	-	-
5KF	2.79	3.22	3.56	3.90	4.08	4.23	4.45	4.57
10KF	3.80	4.29	4.63	4.98	5.13	5.25	5.47	5.68
5MH	4.16	5.40	5.67	5.94	-	-	-	-
10MH	1.56	1.63	-	-	-	-	-	-
5KF5MH	6.68	8.31	9.28	10.25	10.49	10.65	-	-
5KF10MH	-	-	-	-	-	-	-	-
10KF5MH	12.46	13.33	-	-	-	-	-	-
10KF10MH	-	-	-	-	-	-	-	-

CONCLUSIONS

The scanning electron micrographs reviewed that the agglomeration of MH fillers and formation of voids were due to poor interfacial bonding on the cross-section of specimens. Meanwhile, insufficient resin for fibre wetting, polymer hydrolytic degradation and low compatibility between KF, MH and FLO were attributed to the low tensile properties and flexural strength. However, the tensile strength had increased with the increased fibre loading due to better fibre dispersion, while the insertion of KF and MH, especially 10KF5MH and 10KF10MH, had shown a positive observation on the flexural modulus, indicating good modulus on bending. In this light, a past study found similar differences between the values of tensile modulus and flexural modulus which indicates difference in the trending of tensile and flexural modulus. It was concluded that difference in the heterogeneous cross section and shear deformation between tensile and flexural test had created differences in the modulus value. On the other hand, the increased content of KF in composites caused an increment of impact strength, while the insertion of MH flame retardant fillers insertion caused a decreasing trend for impact strength and the impact strengths were reduced by 5.8 %, 31.8 % and 11.4 % for 5MH to 10MH, 5KF5MH to 5KF10MH and 10KF5MH to 10KF10MH, respectively. Besides that, the rates of water absorption for all samples were very high in the beginning but all had reached saturation by different rates. It is worth mentioning that the high content of the KF produced a higher total water absorption but slower saturated composite, while the insertion of MH caused faster saturation and lower total water absorption. However, incompatibility of the interface bonding increased the water absorption of KF/FLO/MH composites and even though 10KF5MH specimen does not have the best performance in mechanical properties, the higher flame retardancy shall provide KF reinforced FLO composite with MH filler for more applications in the advanced sector, especially in a hazardous environment. It was found that insertion of KF and MH filler had caused significant changes in composite properties. Therefore, it is important to take extra care in every process, especially specimen fabrication. In future development, different types of fibre pre-treatment and fillers could be applied on FLO polymer to investigate its properties for more advanced applications. In this light, nano-sized KF and MH fillers are highly recommended to be applied in FLO polymer as they can enhance the composite properties with lesser content volumes, compared to conventional KF and MH.

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