Mechanical and Thermal Properties of Kenaf Fiber Reinforced Polypropylene/Magnesium Hydroxide Composites

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ABSTRACT

This paper presents a study of the mechanical and thermal properties of kenaf fiber (KF) reinforced polypropylene (PP)/magnesium hydroxide (MH) composites. Pure PP samples show low tensile, flexural and flame retardant properties. It was found that KF and MH filler insertion improved the properties of PP composites. The incremental addition of KF fiber between 0 and 20 weight percent in composites results in higher tensile modulus and decomposed mass loss at onset temperature, but lower tensile strength, elongation, flexural strength and onset temperature. Addition of 25 wt% KF produces slightly higher flexural strength. Increasing the volume of MH filler in the composites caused lower strength, tensile modulus and elongation, but higher onset temperature and 2nd peak temperature in thermogravimetric analysis (TGA) testing. Increasing the KF content in the PP matrix resulted in lower mass residue. Increasing the KF content in composites containing MH increased the mass residue at the end of the testing.

Keywords: Kenaf fiber; Polypropylene; Magnesium Hydroxide; Biocomposites; Mechanical properties; Thermogravimetric analysis

NOMENCLATURE

KF	Kenaf fiber
PP	Polypropylene
MH	Magnesium hydroxide
TGA	Thermogravimetric analysis
RPM	Revolutions per minute

INTRODUCTION

In recent years, environmental issues have become important in every technological sector, especially in advanced materials. As a result, natural fibers have found increasing use for the reinforcement of polymer composites in order to achieve the desired properties [1-11]. Recently, natural fiber reinforced polymer composites have been extended to advanced applications, such as in automotive, aircraft, medical and food industries [12,13]. Many types of natural fiber have also been used to reinforce PP polymer, e.g. flax fiber [14-16], hemp fiber [17-21] and jute fiber [22-25].

KF/PP composites have demonstrated superior flexural properties compared to other natural fiber/PP composites [26]. High cellulose content and good orientation of microfibrils in KF are the main reasons for KF/PP composites demonstrate high flexural modulus. Additionally, an increase in crystallization temperature is observed in KF/PP composites. This is related to the high crystalline content of KF. KF/PP composites also showed higher storage modulus, (E') as a function of temperature due to the high energy absorption ability of KF [27]. However, the introduction of KF in PP matrix causes a reduction in the peak thermal degradation temperature [28]. This is attributed to the lower thermal stability of KF compared to the PP matrix [29]. The more rapid degradation of KF accelerates the thermal degradation of the composite structure.

To enhance the fire retardancy of natural fiber reinforced polymer composites, flame retardant filler are commonly added. Flame retardants disrupt the burning process by forming a multi-cellular foam char on the polymer surface or emitting water vapor to dilute the fuel concentration [30]. Magnesium hydroxide (MH) flame retardant is one of the most

popular flame retardants used in composites due to its high decomposition temperature and its smoke suppressing ability [31]. The alkaline nature of MH neutralizes acidic gases (NO_x, SO_2, and CO_2) generated during the burning process [32]. Therefore, MH is considered as an environmentally friendly flame retardant. Sain *et al.* [33] conducted a study of the effect of MH on properties of rice husk/PP composites and sawdust/PP composites. A loading of 25 wt% of MH in the composites resulted in 50% reduction in flammability. Stark *et al.* [34} found similar improvement in their studies in which MH demonstrated the best fire retardancy among five flame retardants in wood/PP composites. Another study showed that the addition of MH to oil palm empty fruit bunch fiber/PP composites improved the thermal stability based on TGA decomposition temperatures [35] Sisal fiber/MH/PP composites showed higher char residue compared to the sisal fiber/PP composites through the formation of magnesium oxide on the composite surface at high temperature, which protected it from further burning. In addition, water vapour, a side product of magnesium hydroxide decomposition, diluted the concentration of fuel, slowing down the burning process [36].

From the above review, it is evident that there is no previous work conducted on the mechanical and thermal properties of KF reinforced PP composites containing MH flame retardant filler. Therefore, the aim of this work is to study the effects of MH inclusion on mechanical and thermal properties of KF reinforced PP composites.

EXPERIMENTAL

Materials

The PP used was Propelinas 600G (homopolymer), which was purchased from Polypropylene (M) Sdn. Bhd. with a melt flow index of 12.00 g/10min. KF was obtained from from Malaysia. Fiber lengths as reported by the supplier ranged from 8-15mm. MH of 95% purity was added to improve the flame retardancy of the composites. Both of the KF and MH were supplied by Tazdiq Engineering, Serdang, Malaysia. A maleated polypropylene (MAPP) coupling agent (E-43) was used to compatibilize the components in the within the composite structure. It has a density of 0.930 g/cm^3, a molecular weight of 9100 g/mol and was supplied by Suka Chemicals (M) Sdn. Bhd.

Composite Preparation

The composites consisted of PP, KF and the flame retardant MH. The MAPP coupling agent was added at 3 wt% to each composite to improve the surface interaction. Pure PP was used as a control sample. Sixteen different compositions were studied- these are set forth are shown in *Table I*. All of the materials were dried at temperature of 50^{o}C for 24 hours. Melt mixing was performed using a HaakeRheocord at a temperature of 170 ^{o}C at 50 revolutions per minute (RPM) for a duration of 15 minutes. PP and KF were melt blended with MAPP and MH using the same internal mixer. The PP was inserted and melted for 5 min, then MAPP and MH fibers were added to the melted PP matrix and mixed for another 10 min. The mixture was then taken out of the mixer while it was still hot, placed in a 1 mm x 3 mm square mold and compressed using a Scientific Laboratory Hydraulic Press Type LP-S-80. Pre-heating and final compression were each accomplished at 200^{o}C for 5 minutes. The compressed sheets were cooled for 5 minutes at 50^{o}C.

Characterization Techniques

Tensile tests were carried out according to ASTM D638, using an Instron 5kN machine located in Universiti Putra Malaysia (UPM) [37]. Dumbbell specimens 63.5 mm x 9.5 mm x 1 mm in size with 3.1 mm x 9.5 mm neck sections were cut out from the molded sheets using a die. A cross-head speed of 1 mm/min was used to strain the samples. The tests were performed at 25^{o}C. Flexural testing was also conducted in the Instron 5kN machine using the ASTM D790 testing standard [38]. The dimensions of the specimens were 60 mm x 10 mm x 3 mm, including overhanging by at least 10% of the support span. In this process, 10 samples were tested in each testing and averages are reported.

TGA testing was conducted under a nitrogen atmosphere in order to determine the thermal stability of the composites. Samples weighing 5g were tested. The samples were heated from ambient temperature to 600^{o}C with a heating rate of 20^{o}C/min. The results were then analyzed by computer software to extract the onset temperature, the 2nd peak, mass loss and mass residue.

TABLE I. Specimens' composition and thermogravimetric analysis (TGA) data.

Specimens	KF Fiber (%)	MH Filler (%)	Onset Temperature (^{o}C)	Mass Loss (%)	2nd peak (^{o}C)	Mass Residual (%)
Pure PP	-	-	383	99.79	-	0.21
10KF	10	-	318.8	8.497	441.6	2.324
15KF	15	-	318.3	9.664	442.6	2.911
20KF	20	-	313.3	10.03	442	2.819
25KF	25	-	322.9	24.09	395.2	0.648
10KF10MH	10	10	317.6	8.115	460.2	8.118
15KF10MH	15	10	310	10.42	460	8.279
20KF10MH	20	10	313.5	11.95	461	9.019
25KF10MH	25	10	306.2	15	461.5	9.407
10KF15MH	10	15	315.7	8.922	463.1	10.45
15KF15MH	15	15	305.1	11.96	461.3	11.12
20KF15MH	20	15	308.2	11.7	460.4	12.71
25KF15MH	25	15	301.3	17.91	458.3	13.81
10KF20MH	10	20	331.6	7.81	464	13.8
15KF20MH	15	20	328.8	9.866	461.7	14.56
20KF20MH	20	20	309.8	14.8	460.5	14.75
25KF20MH	25	20	295.9	17.03	457.8	16.31

RESULTS AND DISCUSSION

Tensile Properties

Figure 1 and *Figure 2* show the tensile strength and tensile modulus for KF reinforced PP composites with MH fillers. The pure PP resin acts as a control sample. It was found that all composite samples have higher tensile properties than pure PP, except elongation at break. This is because the KF and MH filler act as the load bearing components in the composites. The 10KF sample had the highest tensile strength of 39.80 MPa and tensile modulus of 1143 MPa. However, the tensile strength of the composites dropped by 30.90 % from sample 10KF to sample 25KF as the contents of KF and MH increased.

As KF content increased, poorer compacting of the composite caused weaker bonding strength between the matrix and the fiber. Hence, a decline in the tensile strength results [39]. In addition, high fiber contents in the composites resulted in insufficient fiber wetting, negatively impacting the load transfer mechanism in the composite, leading to lower strength properties [40].

The MH flame retardant further decreased the tensile strength of the composites. The greatest drop (23.45 %) in tensile strength was found at 10% add level; the deterioration of strength was insignificant after further addition of the MH flame retardant filler (15 and 20 wt %). The deterioration of the mechanical properties of composites containing flame retardants was previously reported [41].

Figure 3 shows the elongation of KF reinforced PP composites containing MH fillers. The elongation at break was dramatically reduced when the KF and/or MH fillers were inserted. It was predicted that filler insertion would cause the samples to be more rigid. There is a slight non-linear decrease in the elongation when the MH content in composite is decreased a total of 0.089% between from 0MH and 20MH in samples containing 25% fiber. Similar results have been found in previous research [42, 43, 44]. On the other hand, the KF fiber addition made the composites more rigid and significantly reduced elongation to break. These results are similar to those obtained in previous studies on Alfa fiber reinforced PP composites and KF reinforced PP composites [45-47].

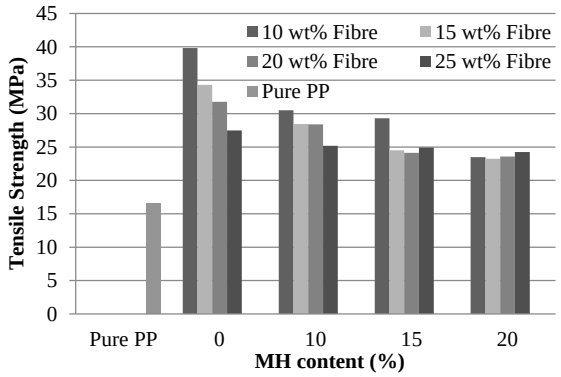


FIGURE 1. Tensile strength for KF reinforced PP composites with MH fillers.

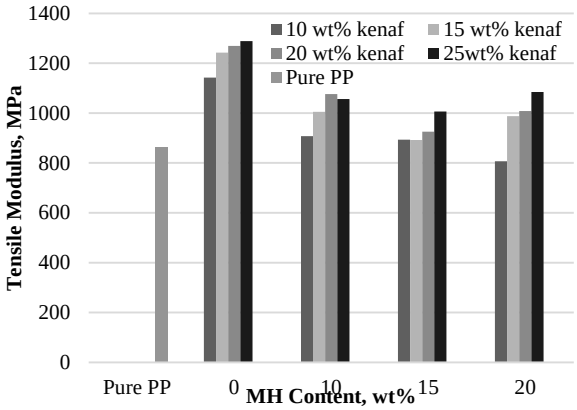


FIGURE 2. Tensile modulus for the KF reinforced PP composites with MH fillers.

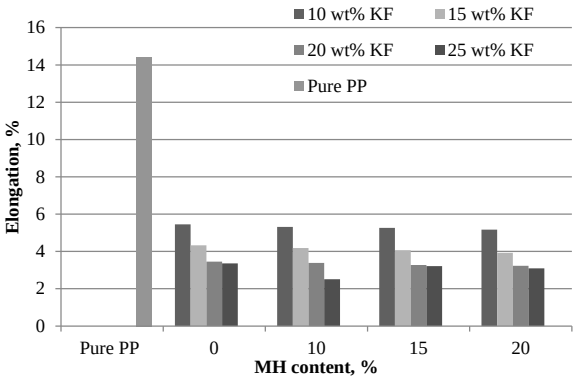


FIGURE 3. Elongation of KF reinforced PP composites with MH fillers.

Flexural Properties

Flexural strength and modulus of the KF reinforced PP composites with MH fillers are shown in *Figures 4 and 5* respectively. It was found that all composite samples have higher flexural moduli than pure PP. While the flexural strength dropped by 14.79 % from 10KF to 20KF, yet, the 25 wt% KF composite shows a slightly higher flexural strength. This is similar to what was observed in an earlier study [48]. All KF reinforced PP composites showed similar trends in flexural strength as for content increased from 0 to 20 wt. Percent. However, the 25KF20MH sample has a lower flexural strength than the 20KF20MH.

There are some factors that led to the observed reductions of the flexural strength. As the fiber content increased the number of fiber ends increased simultaneously. The ends act as a stress concentration spot and cracks began at the fiber ends, leading to the flexural strength reduction [49]. There is also some possible strength reduction due to damage to the fibers during processing at high temperatures [50]. The higher the content of the fiber in composites, the greater the damage to the composite properties.

The agglomeration of the MH fillers could be another factor responsible for the reduction in flexural properties. These agglomerates were observed at short mixing times. This is a result of the hydrophilic behavior and resulting bonding between the MH fibers. The inconsistencies in material dispersion in the composite caused the agglomerates to act as concentration points. Carrot *et al.* [49] explained that in some areas, the large amount of agglomerated MH filler in an olefinic polymer was due to the low viscosity of the melt- the polymer matrix was unable to break the agglomeration of small particle fillers such as MH. *Figure 6* shows the dispersion of the MH (in black) in the polymer matrix (in white) by scanning electron micrograph (SEM) image. The agglomeration of MH is evident, even at low volume content.

The flexural modulus showed non-linear increases for the KF reinforced PP composites with no added MH until 20KF, suggesting that the optimum fiber volume is 20 wt% for composites without MH fillers. On the other hand, the highest flexural modulus was obtained with 15 wt% of MH. A slight reduction of modulus values is noted as MH content increases from 15 to 20 wt% in the composites. Ismail *et al.* [51] reported that the flexural modulus could be increased at flame retardant contents as high as high as 30 wt% with improved dispersion of the flame retardant in the composite.

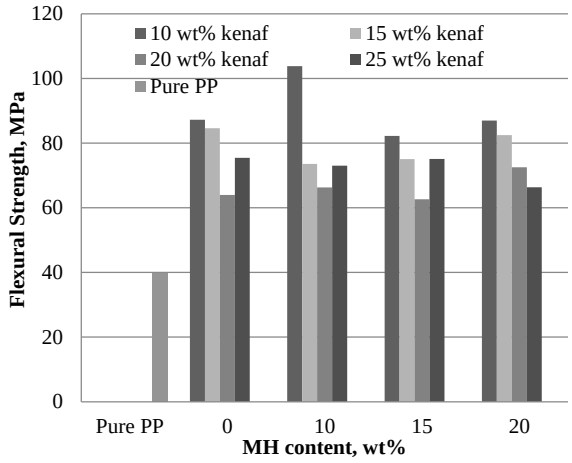


FIGURE 4. Flexural strength for KF reinforced PP composites with MH fillers.

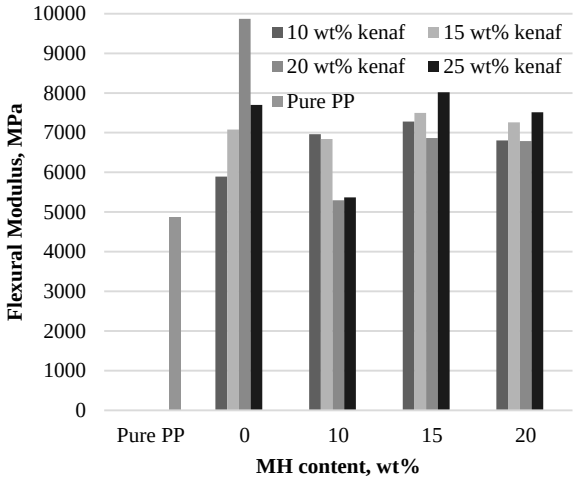


FIGURE 5. Flexural modulus for KF reinforced PP composites with MH fillers.

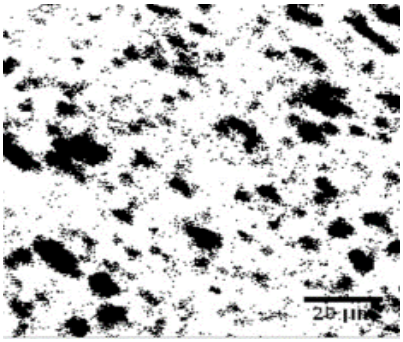


FIGURE 6. Scanning electron micrograph (SEM) image of dispersion of the MH (in black) in the polymer matrix (in white) [49].

Thermogravimetric Analysis (TGA)

Figure 7(a,b) shows the TGA for all sample composites. The testing was run under N_2 atmosphere. This parameter was used previously to study the effect of MH on thermal stability of polymer composites [43, 52, 53]. The thermal degradation process of the composites begins with moisture weight loss of the natural fiber at around 100^{o}C.

It is seen that for all samples the component in a single stage at 383^{o}C. The insertion of fiber and filler split the thermal decomposition into two stages- the first at a lower temperature and the second at a higher temperature. All composite samples had higher mass residue than the pure PP polymer. However, higher mass loss was found as the fiber content in the composite was increased (*Table I*). This suggests that the cellulose and hemicellulose substances in the composites gradually increased, causing higher mass loss at the onset temperature as documented elsewhere [44, 54]. The 10KF sample has a mass loss of 8.49 percent. This increased to 9.664%, 10.03% and 24.09% for 15KF, 20KF and 25KF samples respectively. KF has lower thermal stability than the PP matrix, thus increasing fiber contents induced a lower onset temperature and higher mass lost at the onset temperature.

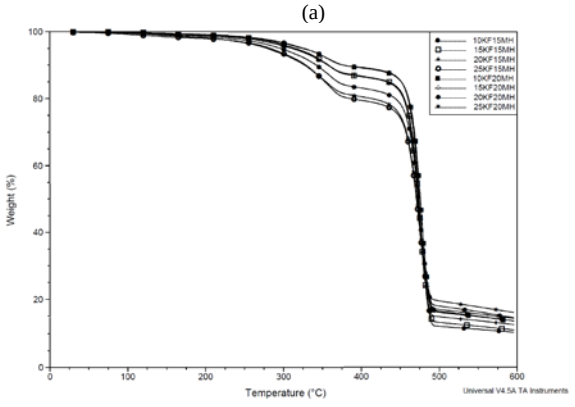
The inclusion of the flame retardant filler MH was meant to improve the thermal stability (higher onset temperature). However, the effectiveness of MH fillers in enhancing the thermal stability of composites depended on the KF loading. The KF threshold loadings were 20, 20 and 15 wt% for 10, 15 and 20 MH wt% loading respectively. At these fiber and filler loadings, the amount of MH filler was insufficient to counteract the flammability brought about by the presence of the by KF. Moreover, further addition of KF loading continued to reduce the thermal stability by decreasing the onset temperature. Therefore, a higher loading of MH filler will be considered in future development work.

The insertion of the MH filler slightly increased the 2nd peak temperature. However, it had no significant influence as the KF and MH contents were varied (*Table I*). This suggests that the 2nd degradation peak is a result of the decomposition of MH filler and PP matrix. A small portion of the MH had decomposed for 2nd stage mass loss while almost all PP matrices had decomposed via a complex radical chain mechanism [55].

The weight replacement of polymer using the MH reduces the amount of fuel available, reducing the mass loss at the end of the process. The mass residue increased with an increase in MH content in the composites. This enhanced the thermal stability of composite, causing it to have stronger flame retardant properties. The mass residue increased from 0.6484% to 16.31% as MH content increased from 0 to 20% from the 25KF sample composite to the 25KF20MH sample composite (*Table I*). Increasing the KF contents in the samples containing MH also increased the mass residue at the end of the test. This might be because the MH was protecting the lower thermal stability KF from being decomposed.

Finally, Shen *et al.* [55] found that decomposition temperature of MH was not significantly different when TGA was performed in in air and N_2 atmospheres. However, a reduced physical barrier effect of the inorganic filler was found under N_2 compared to air atmosphere. Therefore, further TGA investigations should be done under air atmosphere.

(a)



(b)

FIGURE **7**. TGA for (a) 10KF,15KF,20KF,25KF,10KF10MH, 15KF10MH, 20KF10MH ,25KF10MH (b) 10KF15MH, 15KF15MH, 20KF15MH ,25KF15MH, 10KF20MH, 15KF20MH, 20KF20MH ,25KF20MH.

CONCLUSION

The pure PP control sample shoed low tensile, flexural and flame retardant properties. It was evident that the insertions of KF and MH filler improved the properties of PP composites. However, the insertion of KF into the MH containing sample composites resulted in lower tensile strength. On the other hand, low interfacial strength, insufficient fiber wetting and a higher amount of stress concentration spots resulted in lower tensile and flexural properties. Additionally, increasing the content of KF resulted in lower thermal stability. The flame retardant MH filler did enhance the flame retardant properties of the composites, as evidenced by higher peak temperature and lower mass loss. However, the MH contents used in this study did not counteract the flammability brought on by the high volumes of KF added. Therefore, higher loading of MH filler will be considered in future development work. Lastly, a higher mass residue was observed at the end of TGA experiments conducted on the KF/PP/MH composites. This is because the MH was absorbing heat and protecting the KF fibers from decomposition.

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