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Effect of the Characteristics of Maleic Anhydride-Grafted Polypropylene (MAPP) Compatibilizer on the Properties of Highly Filled (85%) Kenaf-Polypropylene Composites

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Kenaf polypropylene composites with high fiber load of 85 wt% were produced using glycerine as a processing aid and maleic anhydride-grafted polypropylene (MAPP) as a compatibilizer/coupling agent. Commercially available MAPPs with different molecular weights and anhydride contents were used to determine what properties of the MAPPs were important to achieve high mechanical properties of these highly filled composites. A homo-polymer and a random-polymer, were compared as matrix polymers. Composites were produced using a high-shear kinetic mixer followed by compression molding at pressures ranging between 345 and 5520 kPa. The data suggests that adding MAPPs with a low molecular weight and high anhydride content at concentrations of 5 wt% resulted in composites with the highest mechanical properties. The authors suggest that a combination of a high surface area, low viscosity and high anhydride content of the MAPP are resulting in a good stress transfer between fibers and matrix polymer.

Keywords: *highly filled composites, compatibilizer, interface, kenaf, polypropylene, glycerine, fiberboard, composites.*

1. Introduction

Wood- and agro-based fiber-reinforced thermoplastics are increasingly used in the construction and automotive industries[1-13], with fiber content generally limited to about 60 wt%. In recent years, composite boards with a fiber content of up to 85 wt% have been developed using innovative processing techniques in combination with plasticizers[14-16]. Sanadi and Caulfield[14,15], developed a technique involving the use of glycerine in a high-shear compounding process blending 85 wt% kenaf fibers with polyolefins with no thermal degradation of the fiber. The composites were prepared by thermokinetic mixing of the fibers and the matrix polymers[17,18]. They have shown that linear polyolefin chains act as a binder and adhesive between the kenaf fibers. This is unlike traditional wood and natural fiber composites (WPCs), where the matrix is in a continuous phase surrounding the fibers or particles.

The polar nature of the lignocellulosic fibers combined with the non-polar characteristics of the polyolefins creates difficulties in compounding, often resulting in composites with poor fiber–matrix adhesion and therefore low stress transfer[3,7,19-24]. The proper selection of compatibilizers is necessary to improve interaction, adhesion and stress transfer between fibers and polymer. The use of maleic anhydride-grafted polyolefins (MAPP), as compatibilizers/coupling agents in lignocellulosic-polyolefin systems, is well established. MAPP is known to form a link between the fiber surface and the polymer matrix. The polypropylene (PP) segment of MAPP forms compatible blends with the bulk PP through co-crystallization, and the polar segment of MAPP, maleic anhydride, links to the fiber surface[16]. The concentration and the molecular weight of MAPP are likely to affect its adhesion to the fiber surface and thus the mechanical properties of the composite material. It was therefore important to conduct a study on how the amount of anhydride and its molecular weight are affecting the the mechanical properties of these highly filled boards, as it could be quite different from how these factors affect the properties of WPCs.

The present study reports on how the wt% of anhydride and its melt index (the latter gives an indication of molecular weight) affect the properties of PP boards with 85wt% kenaf fiber filling. Some processing conditions, such as the pressure applied during the hot pressing of the boards, and their effect of board properties have also been reported in this study. *As far as the authors are aware, all scientific works published on highly filled (85 wt%) lignocellulosic fiber PP composites have been published by the first author of this study , thus the prior works referred to in this paper are limited to these articles.*

2. Materials and Methods

Kenaf filaments, a few meters in length and harvested from mature plants, were obtained from the Kengro Corporation (Charleston, Mississippi, USA). The fibers were cut to about 2 cm in length using a plastic granulator. A PP

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homopolymer, Certene PHM-20AN (Muehlstein and Co., Inc, Wilton, Connecticut, USA) and a PP random copolymer P5M2Z-012 (Huntsman Corp, The Woodlands, Texas, USA) were used in this study. The polymer properties are summarized in Table 1 based on data from the manufacturers technical data sheets. The random copolymer (RPP) had a much lower melt flow index (higher molecular weight) than the homopolymer (HPP), indicating that it had a much higher molecular weight (MW). The RPP usually has a small percentage of ethylene units in the backbone of the polymer, resulting in higher toughness.

The maleic anhydride polypropylene (MAPP) compatibilizers, Polybond PB3150, PB3200, PB3002 and PB3000 with different melt flow indices (MFIs) and anhydride contents were obtained from the SI Group (Schenectady, New York, USA). It is important to note that it is very difficult to control both the MW and the anhydride content separately[17], and no attempts were made to self-produce the compatibilizers for this work by the authors. The MAPP used in the study was provided by the manufacturer and used "as is" without any purification steps. Henceforth, the MAPPs will be referred to by number and their properties are shown in Table 2. *It is also important to note that the melt flow index gives an indication of the molecular weight in nonpolar polymers, with a high melt flow index suggesting a low molecular weight, and vice versa.* Glycerine (propane-1,2,3 triol) was obtained from Sigma-Aldrich (St Louis, Missouri, USA) with a purity of 99% and a boiling point of 290 ºC. The high-shear thermokinetic mixer used, henceforth referred to as the 'mixer', had a processing volume of one liter and came from Synergistics Industries Ltd. (Mississauga, Ontario, Canada).

The kenaf fibers were spray-coated with glycerine as described in detail in a previous work[15]. About 4.5 kg of kenaf fibers were placed in a rotating cylindrical drum with baffles on the inside surface. The sprayer was positioned in the middle of the drum and moved back and forth the length of the rotating drum to evenly distribute the glycerine. Glycerine was mixed with distilled water in a 1:20 volume ratio and the total amount of glycerine sprayed onto the fiber surface resulted in 2 wt% of fibers. The fibers were air-dried until the moisture content was stabilized at 8 wt% at ambient conditions.

Table 1. Mechanical properties of the polypropylenes used as matrix polymers.

PP polymer type	Homopolymer (HPP)	Random copolymer (RPP)
MFI (g/10min)	20	1.9
Specific gravity	0.905	0.894
Flexural modulus(GPa)	1.86	0.82
Izod notched (J/m)	32	70
Tensile strength yield (MPa)	37	28

The glycerine-coated kenaf fibers were placed in the mixer, which was run at about 2000 rpm to cut the fibers to a smaller length and to separate them from the agglomerates formed during the coating and drying process. The required amounts of PP and MAPP were calculated and added to the mixer. The mixer was run at 5000 rpm, corresponding to a blade velocity of 30 m/s at the tip of the blade. The material was automatically dumped into a collection vessel when the temperature reached 190 ºC (after about two to three minutes). The mixture was immediately compression-molded using a laboratory-scale hot press with an 18 x 18 cm mold. A calcium stearate-based release agent was applied to the plates, making it easier to remove the boards after pressing. The pressure was varied depending on the experiments, from 345 kPa to about 5540 kPa. The temperature of the hot press plates varied from about 180 ºC to 190 ºC. The time of hot pressing was defined as the time from when the applied pressure stabilized until the cooling cycle began. The operation time of the hot press was set to three minutes, followed by a cooling cycle, which involved running cold water through the plates. After four minutes of cooling, the cull plates were removed from the press and the sample board was detached from the plates. Three boards were prepared for all different conditions with a thickness of about 3 mm.

Specimens that were 15 cm long and 1.2 cm wide were cut and stored under controlled conditions (20% relative humidity and 32 °C) for 3 days before testing. At least five specimens were tested for each data point. The samples were tested in a universal testing machine and flexural tests were conducted using the ASTM 790-90 standard, with a crosshead speed of 1.7 mm/min. Izod impact strength tests as defined in ASTM D 256-90.

SEM was used to study the fracture surface of the composites. Samples were coated with a thin layer of gold using a sputter coater (Desk II, Denton Vacuum, USA). Electron micrographs were recorded using a scanning electron microscope (JSM 5200, Jeol, Japan) operated at 10-20 kV. The presented micrographs were selected carefully and regarded to be representative for the material.

3. Results and Discussion

Table 2 compares the mechanical properties of specimen containing four different types of coupling agent . The specimen with PB3000 had the highest modulus of rupture (MOR) and modulus of elasticity (MOE).The authors suggest that

Table 2. Mechanical properties of the kenaf polypropylene composites using four different MAPP coupling agent types (85 wt% kenaf, 10 wt% PP, 5 wt% MAPP pressed at 345 kPa).

MAPP Type	Melt Flow of MAPP 230 ºC (10g/min)	Melt Flow of MAPP 190 ºC (10g/min)	Anhydride (%)	MOR (MPa)	MOE (GPa)	Deflection at max load (mm)
PB3002	7	---	0.2	17.48 ± 4.95	2.60 ± 0.58	3.04 ± 0.28
PB3150	50	---	0.5	22.43 ± 3.56	2.89 ± 0.34	3.42 ± 0.30
PB3200	250	110	1.0	19.64 ± 5.91	2.39 ± 0.75	3.71 ± 0.25
PB3000	1000	400	1.2	24.38 ± 2.38	3.50 ± 0.28	3.11 ± 0.36

this is due the coupling agent having the highest anhydride content and the lowest MW. The latter helps the polymer wet the fibers, resulting in a higher surface area of interaction and good MAPP–fiber interaction[25,26]. The combination of these two factors results in the best stress transfer, which in turn leads to the highest MOE and MOR. On the other hand, PB3002 and PB3150, with their lower anhydride contents and higher molecular weights (low melt flow), result in insufficient interaction with the fibers, which leads to a lower MOE and MOR. The mechanical properties of the composite are usually affected by the molecular weight and the anhydride content of the coupling agent. One could expect optimum mechanical properties of the composites using coupling agents with high molecular weight and high anhydride content. However, in case of MAPP the MA is grafted on the PP by a free radical mechanism and chain scission of the PP occurs[27-29]. Thus, it is not possible to achieve a high MA content combined with high MW in the manufacturing process of MAPP.

Usually a high molecular weight is beneficial to achieve good interphase strength and efficient stress transfer between matrix polymers and reinforcing fibers[9,21-23,25]. The higher fiber load could explain the fact that the molecular weight seems to be of lesser importance in the presented study. The key difference between traditional wood plastic composites with fiber loads of up to 60 wt% and highly filled composites with fiber contents of 85%wt and more, is that the surface area of the fibers is much greater due to the higher fiber load. In these types of composites, the linear polypropylene chains will most likely act more as a binder and adhesive between the fibers rather than a continuous matrix surrounding the fibers. This could explain why in the presented study a high MA content of the coupling agent is of greater importance than the molecular weight. The PB3000 has given the highest mechanical properties of the compsites and was therefore used in all the following experiments.

Table 3 compares the mechanical properties of the composites containing 0, 2.5, 5 and 10 wt% of MAPP. The data shows clearly that the amount of coupling agent is correlated to the mechanical properties of the composite and that properties change depending on the amount added to the mixture. The MOR and MOE increase with an increasing amount of coupling agent. There is a slight drop in strength when the amount of coupling agent reaches 10 wt%, which may be due to the higher amount of lower-molecular-weight MAPP as opposed to the PP matrix, reducing the physical entanglements that are necessary when making efficient composites. It can be concluded from the results that the best mechanical properties were obtained using 5 wt% MAPP.

The impact strength in relation to the MAPP concentration is shown in Table 3 for both notched and unnotched testing. The data for the notched impact strength indicates a maximum at about 2.5 to 5 wt%, decreasing when the latter rises further to 10 wt% and drops to 0 wt%. The unnotched data is of limited use as there is a high standard deviation, but at least does not contradict the data from the notched tests.

Figure 1 shows a scanning electron microscope image of the fractured surface obtained after the flexural test. The images show two samples: A and B) 0% coupling agent and C and D) 2.5 wt% MAPP. The fractured surface of the samples without coupling agent is much smoother than that containing 2.5 wt% MAPP. A rough and uneven surface is an indication of MAPP adhering to the fibers. It indicates good adhesion, while a smooth surface indicates no adhesion between the fiber and the matrix.

The type of polymer, HPP or RPP, and its molecular weight had a profound effect on the mechanical properties of the composites. Table 4 shows the difference in 85 wt% kenaf–PP composites pressed at 1380 kPa. As expected, due to the lower modulus of the RPP and the presence of ethylene units in the polymer backbone, the MOE of the composite is lower compared to the composite specimen containing HPP. However, the flexural MOR for the RPP composite was higher, which could indicate that the interphase region of the RPP allows better stress transfer, since the higher MW allows more chain entanglements between the MAPP and the polymer, resulting in a higher interphase strength. Furthermore, the elongation to the point of failure in RPPs containing composite is significantly higher compared to the HPP-based composites due to the presence of the flexible polyethylene (PE) in the backbone.

When the pressure was increased to 2760 kPa, all the properties—MOE, MOR and deflection at maximum load—were higher for the higher molecular weight polymer (RPP), shown in Table 4. This is the despite the higher flexural modulus and yield strength of the lower molecular weight HPP. Again, it appears that physical cross-links (entanglements) play an important role in determining flexural properties.

In the case of the impact properties, the presence of ethylene units in the polymer permits greater mobility and therefore an increase in the toughness of both the polymer and the kenaf composites.

The effect of the hot-pressing pressure on the composites' flexural and impact properties was evaluated using a blend of 85 wt% kenaf, 5 wt% MAPP and 10 wt% RPP. Table 5 shows the effect of pressure on flexural properties. The MOE kept increasing with higher pressures while the MOR was similar for both 2760 and 5520 kPa, the deflection at maximum load (DML) peaked at 2760 kPa. The properties of both notched

Table 3. Mechanical properties of the kenaf polypropylene composites at varying MAPP concentration (85 wt% kenaf, 10 wt% PP, 0-5 wt% MAPP, pressed at 1380 kPa).

MAPP (%)	MOR (MPa)	MOE (GPa)	Deflection at max load (mm)	Notched (J/m)	Unnotched (J/m)
0	4.24 ± 0.89	1.08 ± 0.41	2.18 ± 0.22	11.6 ± 3.9	17.5 ± 3.9
2.5	20.45 ± 2.72	2.77 ± 0.31	3.50 ± 0.32	27.7 ± 0.9	26.1 ± 8.1
5	25.38 ± 5.51	3.43 ± 0.67	4.04 ± 0.24	26.2 ± 2.5	33.5 ± 7.5
10	20.73 ± 5.07	3.41 ± 0.64	2.46 ± 0.29	18.6 ± 3.7	25.2 ± 10.8

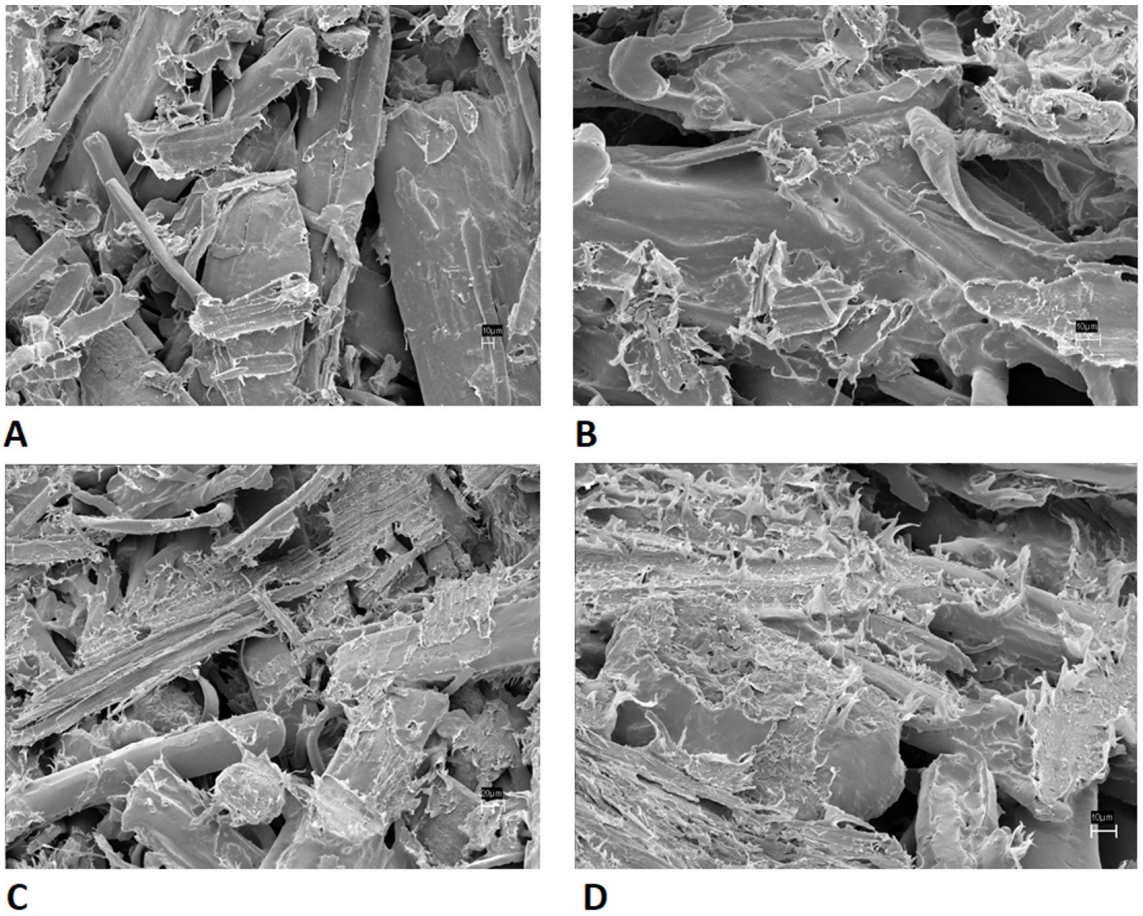

Figure 1. Scanning electron micrograph of: A and B) fractured sample surface with 0% MAPP. C and D) fractured sample surface with 2.5 wt%.

Table 4. Mechanical properties of composites using different matrix polymer types (10 wt% RPP or HPP, 85 wt% kenaf, 5 wt% MAPP) pressed at 1380 and 2760 kPa.

Type	Pressure (kPa)	MOR (MPa)	MOE (GPa)	Deflection at max load (mm)	Notched (J/m)	Unnotched (J/m)
HPP	1380	25.37 ± 5.51	3.43 ± 0.67	4.04 ± 0.24	---	---
HPP	2760	31.4 ± 12.1	4.32 ± 1.49	3.60 ± 0.30	26.09 ± 4.44	41.82 ± 22.13
RPP	1380	29.63 ± 3.97	2.97 ± 0.43	5.24 ± 1.14	---	---
RPP	2760	54.20 ± 3.50	5.40 ± 0.41	5.70 ± 0.36	36.04 ± 2.68	71.52 ± 30.65

Table 5. Effect of compaction pressure on mechanical properties of the kenaf polypropylene composites (85 wt% kenaf, 10 wt% RPP, 5wt% MAPP).

Pressure (kPa)	MOR (MPa)	MOE (GPa)	Deflection at max load (mm)	Notched Izod (J/m)	Unnotched Izod (J/m)
345	23.06	2.27 ± 0.05	4.63 ± 0.62	23.7 ± 7.5	26.2 ± 8.7
1380	26.15	3.13 ± 0.52	4.45 ± 0.42	26.0 ± 6.0	31.1 ± 8.6
2760	54.2	5.4 ± 0.41	5.70 ± 0.36	35.3 ± 4.4	56.2 ±13.5
5520	56.61	6.31 ± 0.16	5.18 ± 1.09	33.1 ± 3.8	54.0 ± 8.3

and unnotched samples pressed at 2760 and 5520 kPa were about the same, and much better than the properties of boards pressed at lower pressures. The hot-press pressure is important in determining the composite properties and should be carefully considered when making these highly filled composites.

4. Conclusion

The mechanical properties of kenaf polypropylene composites can be significantly improved by adding MAPP as a coupling agent. Low-molecular-weight MAPPs with a high anhydride content worked best, likely due to improved

wetting of the fibers resulting in a greater area of fiber-polymer surface interaction, which increases bond formation and improves the composite's mechanical properties. The optimum concentration of coupling agent is at about 5 wt%. MAPP has a significantly lower MW compared to the PP matrix polymer. Increasing its concentration may result in a reduction of physical entanglement and ultimately lead to the composite having poorer mechanical properties.

The use of RPP as a matrix gives the composite a lower MOE compared to using HPP, likely due to the lower modulus of the RPP and the presence of PE chains. However, the MOR and elongation to the point of failure are greater compared to when HPP is used as a matrix. The MOR is greater due to the higher MW of the RPP, resulting in better stress transfer between the polymer chains as a result of increased interaction and entanglement of the polymer chains. The higher elongation to the point of failure might be due to the presence of flexible ethylene units in the backbone of the RPP matrix; the ethylene units present in the RPP are also responsible for the greater mobility of the polymer, allowing chain movements to counteract mechanical impact. This gives the RPP and its composites a significantly higher impact strength compared to HPP. There is also a positive correlation between pressure and impact strength. This reaches an optimum at about 2706 kPa and shows no significant changes when the pressure increases further. Highly filled composites of this kind may be used in the furniture and automotive industries.

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