Characteristics of Sugarcane Bagasse Fiber (*Saccharum officinale*) Reinforced Polypropylene Composites

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Abstract

This research investigates utilization of bagasse to make biocomposite with thermoplastic polypropylene by adding maleated acid anhydride (MAPP) as a compatibilizer and several fiber treatments. The blending was performed on Laboplastomil mixer, and sample was prepared by hot press. The effects of fiber loading (20, 30, 40 and 50% by weight), washing with 2% detergent, 5% alkali treatment and MAPP percentage (1, 3, 5, and 7.5% by weight), on the mechanical and physical properties were evaluated. The effect of elevated temperature on biocomposite was revealed by using thermogravimetric analysis under inert condition. Good compatibility and stress transfer was performed on 40% bagasse with alkali treatment by adding 5% MAPP; and the results on mechanical properties was better than detergent treatment. It showed that the mechanical properties for flexural strength, flexural modulus, tensile strength and tensile modulus increase by about 56.64%, 151.78%, 0.07% and 133.33%, respectively than pure matrix. Maleated acid treatment also shows the tendency to decrease water absorption. Alkali treatment also revealed to increase the thermal stability of composite, while the presence of MAPP decreased the thermal stability of composite. But it does not affect when processing is conducted below 225°C.

Key words: bagasse, flexural strength, MAPP, polypropylene, thermal analysis water absorption

Introduction

Natural fibers filled thermoplastic matrix (polypropylene) have becoming an interesting subject for many researchers at least for the last decade (Doan *et al.* 2006, Malkapuram *et al.* 2008, Mohanty *et al.* 2004a,b). This is due to the increasing environmental awareness, the continuously rising of crude oil prices and the growing global waste problems (Bismarck *et al.* 2005). Start from the tremendous final goals, exploration to add value of some by products from agricultural area is being concerned for many researchers. Bagasse fibers are produced as a residue of the sugarcane milling process on sugar industry or bio-ethanol plant. The main utilization of bagasse is for combustion in the sugar cane industry; however their calorific value is relatively low in comparison with other fuels (Vazquez *et al.* 1999). So, it will not give beneficial enough. Others utilization of this material are for animal feeding, pulp and paper industries and recently several researchers conducted to convert this lignocellulosic material such as bagasse for second generation of bio-fuel as a bio-ethanol. Linear with the high demand and increasing of population growth, bagasse as by products will be a promising material despite of to solve the problem from these wastes.

From sugarcane milling process, it is produced 90% of bagasse while around 5% is for sugar and the rest is molasses and amount of water (Fitria *et al.* 2007). In 2007, Indonesian national production was 2,587,600 ton (BPS 2009), so as an estimation waste could be reach at the amount of 2.3 ton. With this huge production, this study will be conducted to explore of using bagasse as reinforcement in matrix polypropylene for automotive parts. The background of this research is environmental awareness, such as that is mentioned in European Union's Directive on end-life vehicles/ELVs, no later than 1 January 2015, for all end-of life vehicles, the reuse and recovery shall be increased to a minimum of 95 % by an average weight per vehicle and year. Within the same time limit, the re-use and recycling shall be increased to a minimum of 85 % by an average weight per vehicle and year (Anonim 2000), for this purpose finding a new materials for automotive parts that renewable and biodegradable at the end-life will be promising at the future. The biocomposites that produced with natural fibers is light weight due to the lower density of natural fibers compare with inorganic filler; bagasse has density 0.55-1.40 g cm^{-3} (Vazquez *et al.* 1999). Using light weight material for automotives could reduce the total weight of vehicles. According to Wijaya (2004), without plastics, weight of a car will be more than 200-300 kg, and it could reduce the consumption of fuel up to 0.5 l 100^{-1} km (0.005 l km^{-1}). Assume that a car has 150,000 km old so the saving on fuel reach 750 l car^{-1}. Reducing fuel consumption means reducing the emission to environmental. Not only reduce the mass of the component but also lower the energy needed for production by 80% (Malkapuram *et al.* 2008). Other advantages of using natural fiber are higher sound attenuation, high stiffness and non abrasive.

On automotive components, the big three plastics type that total of this plastic is 66% from the total plastic on automotive are polypropylene (32%), polyurethane (17%) and polyvinyl chloride (16%). Finally, a lot of researcher concern to polypropylene for their research.

The drawbacks for substitution of inorganic filler with natural fiber are low compatibility between natural fiber and polymer matrix, low impact strength, wide variation in strength values depend on location and age, swelling and higher water absorption, also limiting process manufacturing below 200°C (Mohanty *et al.* 2001).

Preliminary research reported by Fitria *et al.* (2007) revealed that chemical compositions of the bagasse i.e. cellulose, hemicellulose, lignin, extractive and ash content are 38.10%, 23.08%, 27.46%, 4.06% and 1.90%, respectively. Total holocelullose up to 60% lead to a better mechanical properties.

In this study, bagasse was used for reinforcement in matrix polypropylene by treatment with detergent, alkali and variation of MAPP to increase the compatibility with polymer matrix. Mechanical and physical properties were studied, and the thermal analysis for treated and untreated fiber was revealed.

Materials and Methods

Fiber treatments

Detergent

Dust fibers were soaked in 2% detergent solution, the amount of bagasse that added was 100 g l^{-1} solution, heated on 70°C, stirred for 1 h. Afterwards fibers were washed with water and oven dried at

100°C until a constant weight was achieved.

Alkali

Fibers from detergent treatment were placed in glass ware with 5% solution of NaOH and continuously stirred for 1 h at 80°C. The amount of bagasse that added was 70 g l^{-1} solution. Afterwards fibers were washed with water and oven dried at 100°C until a constant weight was achieved.

Coupling Agent

MAPP concentration was varied at 1, 3, and 7,5% of weight of composites.

Compounding

PP and bagasse fibers were mixed in Laboplastomill type 30R150 for 10 minutes at 180°C, 60 rpm. The amount of fibers loading were 20, 30, 40 and 50% by weight. MAPP was added during mixing with varied concetration at 1, 3, 5, and 7.5% by weight. After this step, the compound was compressed at specimen bar (150 x 105 x 3) mm^3 at 180°C, 5 minutes needed for melting process and then compound pressed at 100 kgf cm-^{2} for 3 minutes. Finally, the pressure was increased untill 150 kgf cm^{-2} for 2 minutes. Then, sample was cooled on cold press.

Testing

Flexural tests at least 4 replications were perfomed with Universal Testing Machine (UTM) SHIMADZU, according to ASTM D790-92 standard, the specimen was (11 x 3 x 80) mm^3, span was 50 mm, crosshead speed was 2 mm min^{-1} (Rodriguez *et al.* 2006). Tensile tests of at least 4 specimens of each sample were perfomed with UTM at crosshead speed of 2mm min^{-1} according to test specimen Baillif *et al.* 2009, (10 x 3 x 50) mm^3, span was 30 mm. Water absorption specimen was prepared at size of (20 x 20) mm^2. The final conditioned weight (Mo) was determined. Sample were immersed in distilled water for 24 h, dried and finally, the immersed weight (M1) was determined. Percentaged of water absorbed was calculated as follows: WA = (M1 – Mo)/Mo x 100%. Thermal stability analysis, thermogravimetric (TGA/DTG), was carried out by Seiko Instruments Inc, at rate of 10°C $minute^{-1}$ from 27-550°C under inert condition (nitrogen).

Results and Discussion

Effect of Fiber Loading

It is evident from Fig. 1 that fibers loading enhance the mechanical properties (flexural strength and flexural modulus) of composites, significantly. However, the tensile strength decreases with the adding of natural fiber. Incompatibility between polypropylene and natural fiber lead to decrease of tensile strength. Natural fiber is hydrophilic in the nature, because it contains of a lot hydroxyl groups. On the other hand, polymer matrix is hydrophobic. The flexural strength of composites is significantly higher than the corresponding tensile strength. In this study, 40% fiber content give the best flexural strength and at higher (50%) fiber content the mechanical properties decrease. This could be answered by phenomena fiber to fiber interactions that initial stress concentration and lead to a cracking; moreover it may cause adispersion problem. Malkapuram *et al.* (2008) stated that addition of wood dust to PP significantly decreases the tensile strength. That means bagasse fiber act as filler not as reinforce due to the lack of interface interaction with the matrix. Bagasse at 40% will be used for the next study.

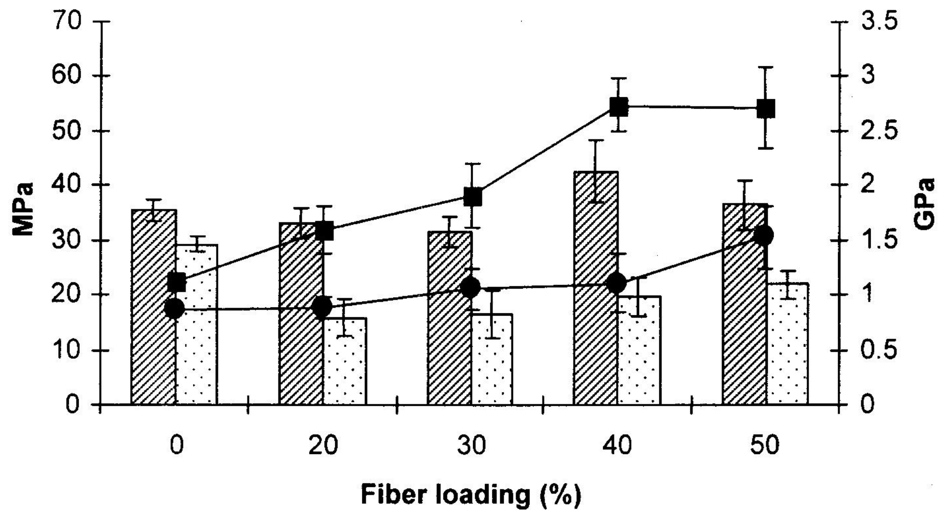

Figure 1 Effect of fiber loading on the mechanical properties of the composites.

Effect of Fiber Treatment

It is evident from Fig. 2 that several treatments to bagasse fiber affect the mechanical properties. Treatment with 5% alkali and 5% MAPP could increase the flexural strength, flexural modulus, tensile strength and tensile modulus by about 56.64%, 151.78%, 0.07%, 133.33%, respectively. The alkali treatment could increase the percentage cellulose content due to the dissolving of lignin and extractive compound. MAPP as coupling agent make the cellulose compatible with the formation of ester bonds between natural fiber and the polymer matrix (Mutje *et al.* 2005), another researcher (Sain *et al.* 2005) illustrate the formation of hydrogen bonding. Fig. 3a is illustrating MAPP function that causes the increasing of mechanical properties. Good interaction between natural fiber and polymer lead to a better stress transfer when the composites conducted with force. That means the bagasse fiber act as reinforce not as filler anymore.

Alkali treatment without the presence of coupling agent decreases the flexural strength and tensile strength, by 16.67% and 33.46%, respectively. This could be explained that alkali treatment has two effects on the fiber: first, it increases surface roughness resulting in better mechanical interlocking. Second, it increases the amount of cellulose exposed on the fiber surface (Li *et al.* 2007). It means that the increasing cellulose expose lead the fiber to be more hydrophilic due to the expose of hydroxyl group. Finally, the interaction with the polymer matrix decrease (Santos *et al.* 2008).

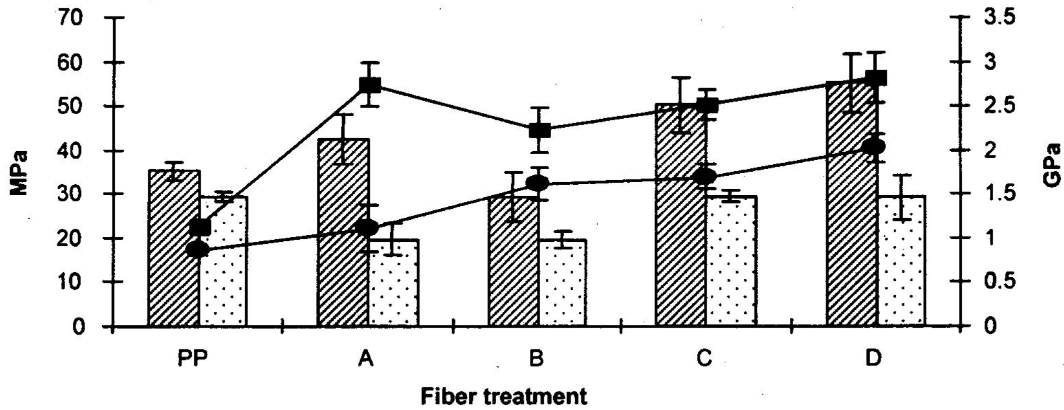

Figure 2 Effect of fiber treatment on the mechanical properties of the composites at 40% by weight fiber loading. Notes : PP: Pure Polypropylene A: 2% Detergent, B: 5% Alkali, C: 2% Detergent+5%MAPP, D: 5%Alkali+5%MAPP.

Flexural strength (MPa) Tensile strength (MPa)
Flexural modulus (GPa) Tensile modulus (GPa)

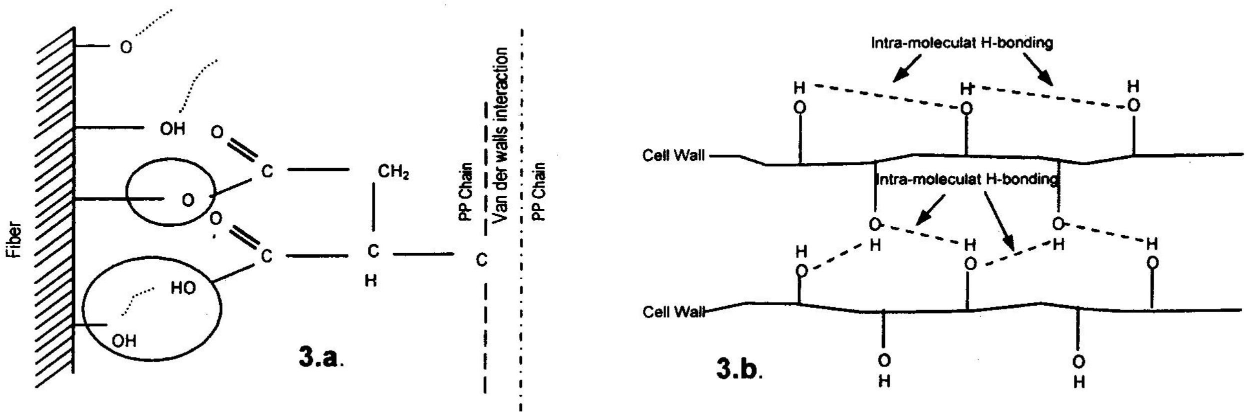

Figure 3a Schematic of the interaction between fiber, coupling agent and polymer (esterification, hydrogen bonding and van der walls interaction, Mutje *et al.* (2005) and Sain *et al.* (2005)), 3b Schematic hydrogen bonds between cellulose (Mohanty *et al.* 2001).

Besides that, Mohanty *et al.* (2004a) explain that the increases of hydroxyl group lead to agglomeration cause between cellulose form intermolecular hydrogen bonds inside macromolecules itself and intermolecular hydrogen bonds among other cellulose macromolecules as well as with hydroxyl groups (water) from the air. Fig. 3b illustrates the phenomena. Detail explanation gave by Numez *et al.* (2006) agglomeration creating regions of stress concentration that require less energy to initiate or propagate a crack. Mishra *et al.* (2007), state that at higher alkali concentration, excess delignification of natural fiber occurs resulting in a weaker or damaged fiber. In this study, 5% alkali treatment with presence of 5% MAPP had better mechanical properties than that detergent do.

Effect of MAPP Concentration in Detergent Treatment

Detergent treatment functionalize as a surfactant, it is hope to decrease natural fat from natural fiber. The lower amount of alkali concentration in detergent is to prevent the excess delignification or even damaged the fiber during treatment. From Fig. 4, it is evident that treatment with 2% detergent and 5% MAPP increase the flexural strength, tensile strength and tensile modulus by about 18.28%, 50.08%, 53.15%, respectively. On the contrary, the flexural modulus decreases. Some researchers (Pracella *et al.* 2006, Doan *et al.* 2006, Rana *et al.* 1998) concluded that the compatibilizer had no effect on flexural modulus or tensile modulus, however, the effect of the fiber content was obvious (See Fig. 1).

The optimum concentration of MAPP that give the best performance was at 5% by weight. It is evident that higher MAPP concentrations decrease the mechanical performance. According to Mohanty *et al.* (2004a) and Santos *et al.* (2008) this is may be MAPP has a low molecular weight if in compare with pure PP which seems to be responsible for plasticizing effect. In this study, we did not check the melt flow rate (MFR). At the higher concentration of MAPP, this could be attributed to the migration of too much compatibilizer around the fibers, causing self-entanglement among the compatibilizer rather than the polymer matrixes, resulting in slippage. It is believed that an excess of MAPP could increase water affinity, since it is more polar than the PP matrix, this is very important since water absorption usually affects mechanical properties and dimensional stability of composites (Nachtigall *et al.* 2007).

Effect of MAPP concentration in alkali treatment

From Fig. 5, it is very clear that bagasse fibers are act as reinforce at PP matrix due the present of MAPP. As clear as discussion above alkali treatment could expose the cellulose into fiber surface and then the present of MAPP form an ester bonds and hydrogen bonds like illustrate in Fig. 3a. It was also suggested that the removal of the intracrystalline and intercrystalline lignin and other waxy substances, by alkali, substantially increased the mechanical and chemical bonding (Fung *et al.* 2002). The best mechanical properties are at MAPP concentration of 5%. At higher concentration MAPP gave the same phenomena with treatment with detergent.

Effect of MAPP to polypropylene

In the earlier study, the best MAPP concentration for each treatment is 5% by weight, and it is excess at 7.5% by weight. Hence, it is necessary to check the effect of MAPP to polymer matrix to know how significant it is. From Fig. 6 it is evident that MAPP present at polymer matrix decrease the mechanical properties resulted in a decrease in crystallites of materials (Ley *et al.* 2007). The decrease of mechanical properties from our research maximum at 11.93% at 5% MAPP, It is better than result from Yuan *et al.* (2008), the mechanical properties decrease until 36.06% at 3% MAPP by weight. It is depend to the specification of polymer that we used in study.

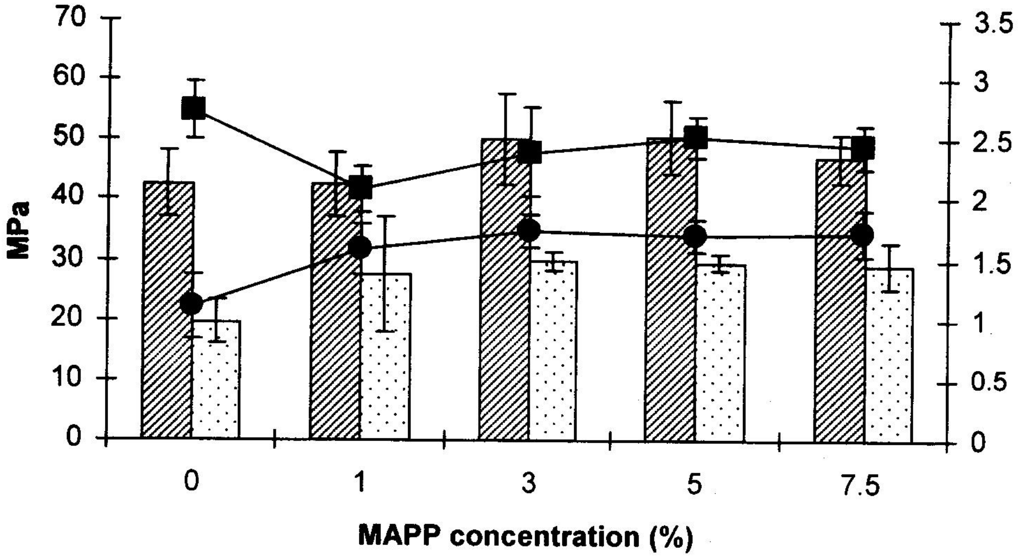

Figure 4 Effect of MAPP concentration on the mechanical properties of the composites at 40% by weight bagasse detergent treatment.

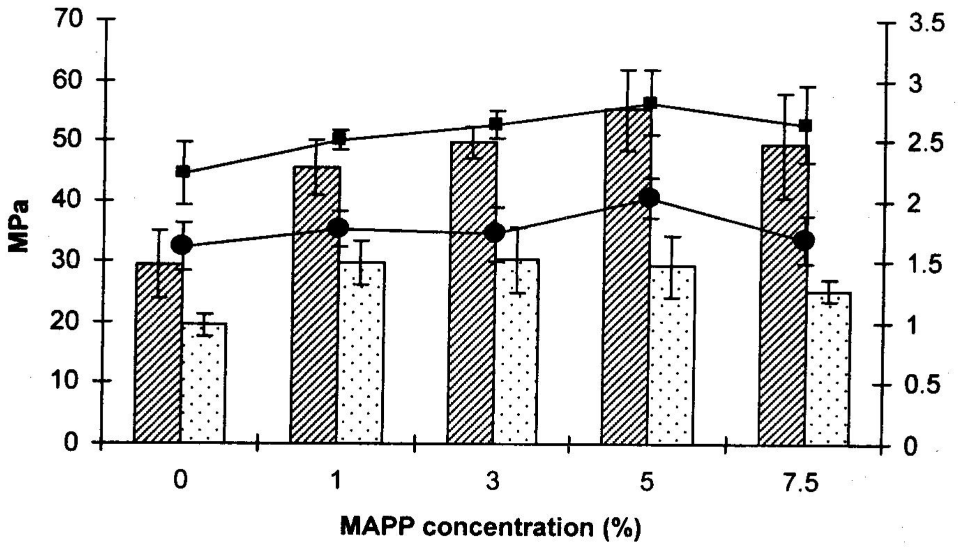

Figure 5 Effect of MAPP concentration on the mechanical properties of the composites at 40% by weight bagasse alkali treatment.

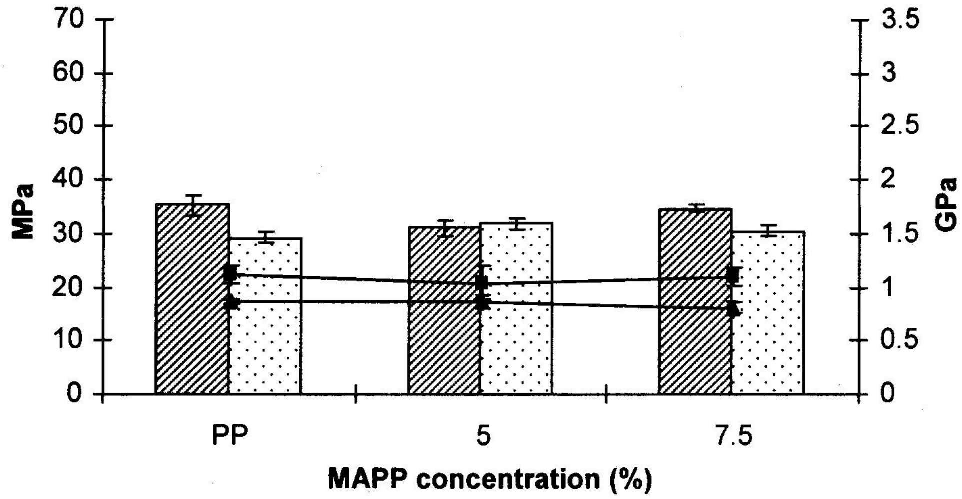

Figure 6 Effect of MAPP concentration to pure Polypropylene.

Flexural strength (MPa) Tensile strength (MPa)

Flexural modulus (GPa) Tensile modulus (GPa)

Water absorption

It is evident from Fig. 7 that MAPP treatment could decrease water absorption. This is because the hydrophilic –OH groups present in the surface of fiber react with the acid anhydride group present in MAPP to form ester linkages. Water absorption in alkali treatment, slightly increase as an evident that alkali treatment expose cellulose into fiber surface and expose the hydroxyl group and form hydrogen bonds with water. It is also evident that detergent treatment only view to degrade lignin, lignin is more hydrophobic than cellulose. No absorption occurs at PP matrix.

Thermal stability

Thermal gravimetric analysis (TGA) was conducted to study the high temperature decomposition characteristics of the composites as well as the constituent phase (Fung *et al.* 2002). The stability of the bagasse fibers under the high melt processing temperature is of major concern. If serious degradation of the fibers took place during melt processing of the composites, the mechanical reinforcement effects of bagasse fibers may be lost. In Fig. 8, we can see that under nitrogen condition pure PP start to decompose at about 370°C and the DTG curve have peaks at just below 450°C. However, during melt compounding, the PP melt will be in contact with trapped air, there is present of oxygen that leads rapidly decomposition. It was also observed that its thermal decomposition showed a narrow temperature range with degradation in a single mass loss step (Santos *et al.* 2008).

From Fig. 9, 10, 11, and 12, melting temperature of all composites from bagasse fibers decrease. It is different result for fiber reinforcement compare to other researchers (Mohanty *et al.* 2004b and Ichazo *et al.* 2001). They reports the increase of melting point, but they concluded that melting temperature practically does not change neither with the addition of natural fibers nor with the different treatments (See Table 1.)

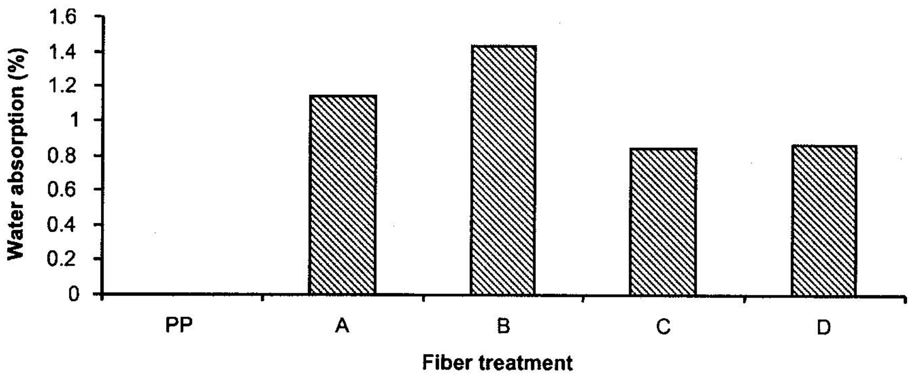

Figure 7 Effect of fiber treatment on water absorption composites. Notes: PP: Pure Polypropylene A: 2% Detergent, B: 5% Alkali, C: 2% Detergent+5%MAPP, D: 5%Alkali+5%MAPP.

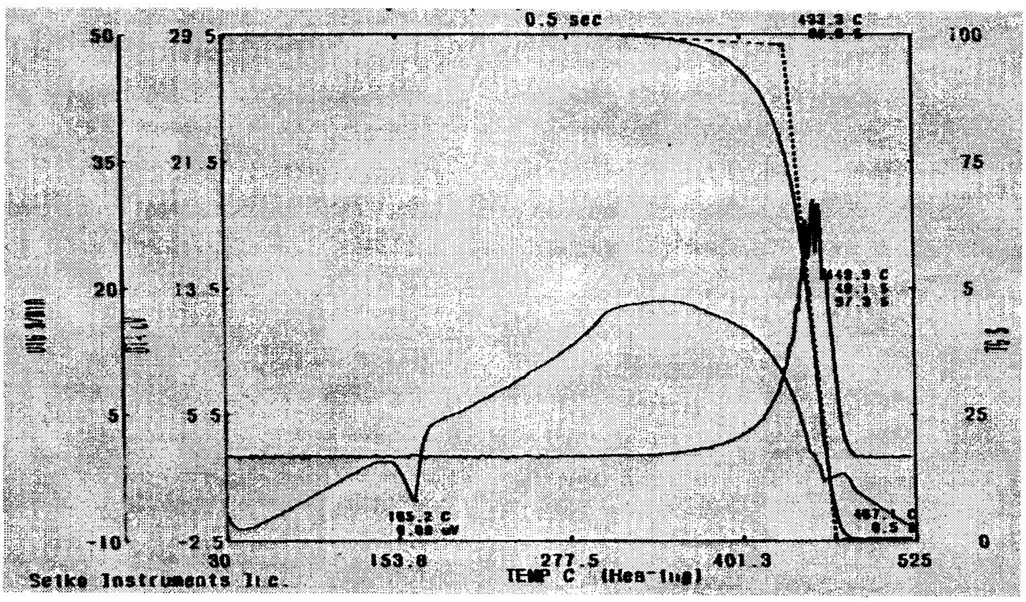

Figure 8 TGA/DTG curves for pure PP.

Table 1 Melting temperatures (Tm) of composites

Fiber treatments	Tm (°C)
PP	165.5
Detergent	161.8
Alkali Treatment	161.8
Detergent+MAPP 5%	161.8
Alkali+MAPP 5%	162.7

Further with Fig. 9-12, alkali treatment shows a peak at the beginning of heating different with the detergent one, this is correspond to the evaporation the amount of water in the sample, that means alkali treatment more absorb water, as we discuss in Fig. 7. It is also very clear shows that after elevated temperature around 320°C, the curve exhibits two decomposition steps. The first decomposition peak is around 310°C due to depolymerization of hemicellulose and glycosidic linkages of cellulose (Albano *et al.* 1999), the second decomposition peak at about 360°C is due to α-cellulose decomposition. Further elevation on temperature shows the peak decomposition of polymer PP. In alkali treatment the shoulder due to the hemicellulose decomposition is little disappear. This is because hemicelluloses are very sensitive with alkali treatment.

From Fig. 9 and 10 we can infer that alkali treatment increase thermal stability of composites. In detail, at detergent treatment the fiber starts to decompose at 241.5°C lower than 249.3°C for alkali treatment. The presence of MAPP at the composites, Fig. 9 and 10, Fig. 11 and 12, showed that the thermal stability of PP in the composites decreased, since it started its degradation earlier. Decomposition of the coupling agent attached to the degrading filler. Although, it is need more evident to proof it. Unfortunately, we didn't check the thermal stability of MAPP blend with pure PP. It will be our next research to reveal. The early decomposition of the matrix was observed at approximately the same temperature of the fibers, that is, above 225°C, indicating that processing below this temperatur does not affect the stability of the materials.

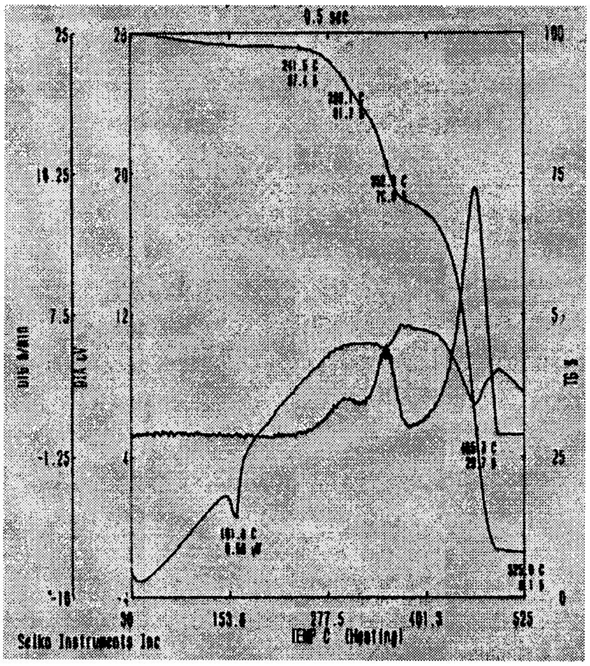

Figure 9 TGA/DTG curves for Detergent tratment 2%

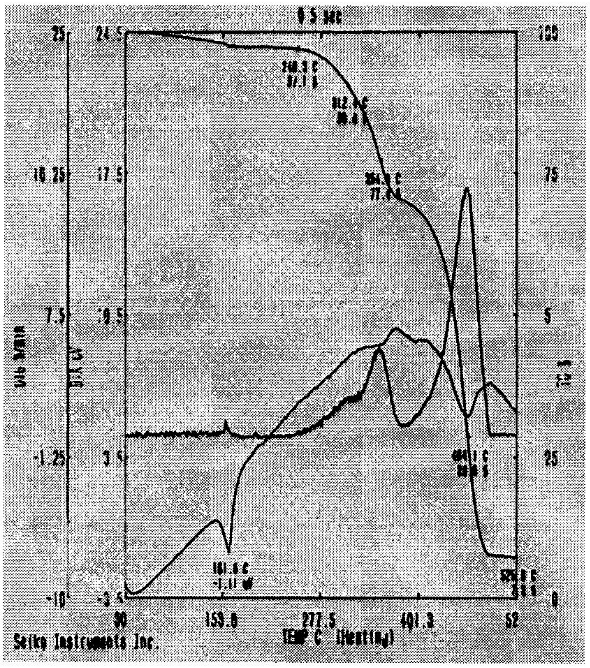

Figure 10 TGA/DTG curves for Alkali treatment 5%

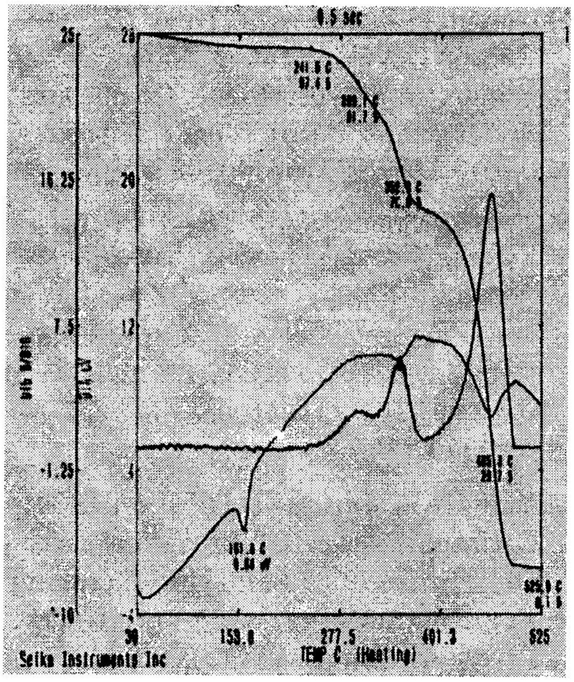

Figure 11 TGA/DTG curves for 2% Detergent + MAPP 5%

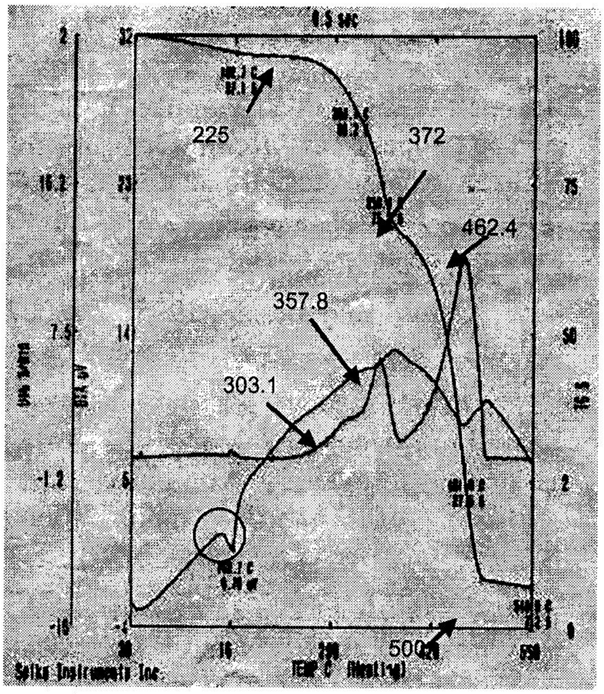

Figure 12 TGA/DTG curves for 5% Alkali + MAPP 5%

Conclusions

Bagasse fiber as a reinforcement of PP composite was investigated with several fiber treatments, it was revealed that 5% alkali and presence of 5% MAPP gave the best mechanical and physical properties. Alkali treatment increase the cellulose expose in the surface of fiber, and cause the decrease of mechanical properties without the present of coupling agent. At the same concentration MAPP added, alkali treatment shows better mechanical properties than detergent treatment. MAPP also show the tendency to decrease the water absorption. Alkali treatment also revealed to increase the thermal stability of composite, while the presence of MAPP decreases the thermal stability of composite. But it does not effected when processing is conducted below 225°C.

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