EVALUATION OF POLYPROPYLENE/SAW DUST COMPOSITES PREPARED WITH MALEALATED POLYPROPYLENE (MAPP) PRODUCED BY REACTIVE EXTRUSION[1]

Ernani Trombetta[2], Thais Flores-Sahagun [3] and Kestur G. Satyanarayana [4]

ABSTRACT

Considering the importance of the environment and fuel economy, the Brazilian automotive industry has focused on the development of lightweight materials based on renewable resources. Replacement of PP- talc composite by PP- saw dust composite is a promising possibility. This paper presents the preparation of maleated polypropylene (MAPP) through reactive extrusion using different amounts of peroxides and maleic anhydride as well its characterization. Using 20% of saw dust coated with different amounts of prepared MAPP, several composites were prepared including one with commercial MAPP. Injection molded samples of all these composites along with that without the compatiibilizer were characterized for mechanical properties. These studies indicated fulfilling the proposed objectives: (i) finding the optimal reactive extrusion conditions to prepare MAPP samples; (ii) preparation of PP/saw dust composites with and without MAPP coating; (iii) to arrive at optimized composite to get the best performance through their characterization for various properties of all the produced composites.

Key words: Composites, Saw dust, Maleated Polypropylene, Reactive Extrusion

RESUMO

Considerando a importância do meio ambiente e da economia de combustíveis a indústria automotiva brasileira tem focado no desenvolvimento de materiais baseados em recursos renováveis. A substituição de compósitos PP/talco por compósitos PP/serragem é uma possibilidade promissora. Este artigo mostra a preparação e caracterização de polipropileno maleatado (PPMA) através da extrusão reativa usando diferentes quantidades de peróxido e anidrido maleico. Usando 20% de serragem revestida com diferentes quantidades de PPMA, vários compósitos foram preparados, inclusive um usando PPMA comercial. Corpos de prova injetados de todos os compósitos com e sem compatibilizante foram caracterizados quanto às suas propriedades mecânicas. Este estudo teve como objetivos: i) encontrar as condições ótimas de extrusão reativa para a preparação de amostras de PPMA; (ii) preparar compósitos PP/serragem com e sem revestimento de PPMA; (iii) encontrar um compósito de melhor desempenho através da caracterização de várias propriedades de todos os compósitos preparados.

Palavras chaves:compósitos, serragem, polipropileno maleatado, extrusão reativa.

1. INTRODUCTION

In recent years, there has been more stress on two important aspects of societal importance. They are (i) utilization of renewable resources to increase their utilization without allowing them go waste and (ii) the environment safety whereby any product including waste should be biodegradable without adding to environmental pollution. In addition, light weight materials thus produced should be beneficial to many sectors particularly to automotive sector due to increasing stress on energy efficiency and environment safety. These aspects have found good grounds in the case of composite materials, which are given major thrust particularly in European countries where the laws have been passed for the use of 95% of recyclable materials in all new automotives in order to achieve the "end of life" required by 2015. This has led to greater impetus for the development of commercially viable composites, particularly of biodegradable type. It is also well known that throughout the world wood based fiberboards are used extensively in both the building and furniture industries. Since these use toxic chemicals such as formaldehyde, phenols and isocynates, there is increasing interest in developing new type of materials without the use of such chemicals. This has led to the development of saw dust (wood fibers) incorporated polypropylene (PP) composites. Accordingly, a number of reports are available now on the preparation and properties of PP-saw dust composites (1-4). Further, improvement of the interaction, adhesion and stress transfer between the polymer matrix and the lignocellulosic fiber demand proper selection of coupling agents and other additives (5-8). Besides, the use of maleated polyolefins as compatibilizers/coupling agents in systems of lignocellulosic-polyolefin is well established (8-10).

Recognizing the above facts, the automotive industries in Brazil have been increasingly applying the technologies that employ polymers reinforced with lignocellulsoic fibers. Further, knowing that the selection of type of fiber to be used depends on the properties of the reinforcement and its availability saw dust (wood fiber) as reinforcement in PP matrix is being explored in the state of Parana, Brazil. It is also well known that in such composites, improvement of fiber / matrix adhesion is obtained generally using maleated polypropylene (MAPP), which is currently not produced in Brazil. Accordingly, the aim of this work is: (i) indigenous preparation and characterization of MAPP by reactive extrusion with different amounts of peroxides (dicumil peroxide and benzoyl peroxide) in the presence of maleic anhydride (ii) to evaluate mechanical properties and morphology of PP-20 wt. % saw dust composites, containing 10 wt.% both prepared and commercial MAPP, produced by extrusion followed by injection molding processes and (iii) finally, the best composite found by this characterization would be chosen to replace currently used PP-talc composite automotive component.

2. MATERIALS AND METHODS

2.1. Materials
The polypropylene used is the commercial PP H503 (hereafter called as `PP`) manufactured by Brasken and kindly supplied by SASIL distributor. This possesses melt flow index of 3g/10min. The saw dust utilized is (60 mesh size) from Pinus Eliotti and was supplied by the Centeri Company. Maleic anhydride (MH-99% purity) in the solid form was kindly supplied by Carbomafra Specialties Chemical Company. For convenience, it was grounded into powder form. 99.9% purity dicumyl peroxide

(DCP) and 75% purity benzoyl peroxide (BP) were obtained from Avec Rubber and Pharmachemical Company respectively.

2.2. Methods

2.2.1. Characterization of saw dust and PP:

The chemical composition of the saw dust used was determined according to the TAPPI standards. Its size distribution was determined using a sieve. The morphology of saw dust used was carried out using JEOL scanning electron microscope [SEM: Model JSM 6360 LV] at 15keV after giving them a gold coating. Density of both the saw dust and the matrix PP were evaluated, according to ASTM standard D618-96.

2.2.2. Manufacture Malealated Polypropylene

Maleated polypropylene (MAPP) samples were initially prepared with 0.5% or 1% (p/p) of maleic anhydride with or without any one of the two peroxides (DPC or BP) mentioned. Their concentration was also of 0.5% and 1% (p/p), as shown in **Table 1**. A mono screw extruder (Make/Model: EMT25) of L/D=30 was used with rotation speed of 25 to 50 rpm with a view to get good quality of the resulting material. A temperature profile of 4 heating zones was used with minimum of 154°C and maximum of 195°C for processing the material. During the reactive extrusion process, it was observed that as the material came out of the nozzle chemical reactions continued to take place. In order to guarantee the proper interaction of PP with maleic anhydride, the same material was reprocessed two times with a view to increase the resident time in the extruder to ensure completion of the reaction. The commercial MAPP was named `F1`.

Table 1. Designation of manufactured PPMAs with their composition

Samples	PP(%)	MA (%)	DCP(%)	BP(%)
F2	99.5	0.5		
F3	99.0	1.0		
F4	99.0	0.5	0.5	
F5	98.5	1.0	0.5	
F6	98.5	0.5	1.0	
F7	98.0	1.0	1.0	
F8.1	99.0	0.5		0.5
F9	98.5	1.0		0.5
F10	98.5	0.5		1.0
F11	98.0	1.0		1.0

2.2.3. Characterization of Products prepared.

All the ten manufactured MAPP samples were characterized by Fourier Transformed Spectroscopy (FTIR) in a Ponte Golden Gate equipment (Model: 370-DTGS) with mono reflection crystal of diamond produced by Nicolet Avatar. Melt flow index (MFI) was determined at 230ºC with a 2.16kg load, using the MI-3P, EQUO31 DSM instrument. Density of composites was determined according to ASTM D792-00 standards.

2.2.4. Coating of saw dust (Wood Fibers)

Firstly, the saw dust was dried in an oven at 60ºC for 24 hours before the coating with the PP and the MAPP. The variable in coating process was the quantity of

MAPP to be used (20%, 10% and 5.4%). With each one of these compositions, the commercial MAPP (designated as F1) and the MAPP that was manufactured in the laboratory (designated as F2R2) were used. **Table 2** presents various composites prepared with their compositions. The maximum amount of fibers used was 40%, which neither hampered the mixing of materials nor the extrusion of the components.

Table 2. Composition of 3 levels of fiber coating concerning MAPP amounts.

% Materials ->	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100
F2R2 E5 / F1E1	Fiber (40%)								MAPP (5,4%)	PP (54,6%)										

% Materials ->	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100
F2R2E8 / F1 E3	Fiber (40%)								MAPP (10%)		PP (50%)									

% Materials ->	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100
F2R2E7 / F1 E2	Fiber (40%)								MAPP (20%)				PP (40%)							

One problem encountered during the extrusion during the coating of saw dust was due to the large quantity of fiber present in the screw thread of the extruder. Rotations of 15 to 21 rpm were used along with constant temperature (168ºC) in all the 4 zones. Some samples of MAPP were reprocessed to verify whether the residence time was sufficient and/or whether reagents the mixing could be improved. Various composites were prepared with the extruder using the coated saw dust and by adding equal amounts of PP (50/50). Designations of various composites prepared with their compositions are presented in **Tables 3**. It was ensured that all the composites should contain the same amount of saw dust (20% pp) with a view to evaluate the coating performance with distinct quantities of MAPP. A composite (designated as FOC1) with 20% of fiber and 80% of PP, but without any MAPP was also extruded, with a view to compare the effect of compatibilizer (MAPP) on the properties of composites.

Table 3. Designation of composites in relation of the type and quantity of PPMA used.

Composites	Type/quantity of MAPP	PP(% p/p)	MAPP(% p/p)	Fibers (% p/p)
FOC1	Non-treatement	80	---	20
F2R2E5C1	2.7% MAPP	77.3	2.7	20
F1E1C1	2.7% Comercial MAPP	77.3	2.7	20
F2R2E8C1	5% MAPP	75	5	20
F1E3C3	5% Comercial MAPP	75	5	20
F2R2E7C1	10% MAPP	70	10	20
F1E2C2	10% Comercial MAPP	70	10	20

2.2.5. Injection of the specimens.
Composite specimens were then obtained according to ASTM standard using an injection molding machine (Model: MIR). This machine has an injection pressure of 100 ton and a cylinder diameter of 45 mm with maximum injection stroke of 150 mm. This machine has five heating zones as follows: zone 1 for feeding, zones 2, 3 and 4 for the cannon blower and zone 5 for the nozzle. The composites were injected with

the following temperature profile: 220ºC in zone 1, 190ºC in zones 2, 3 and 4 and 210ºC in zone 5. Obtained specimen samples for the mechanical tests are shown in **Figure 1**.

Figure 1. Injection molded test specimens

2.2.6. Mechanical Characterization:

Ten samples for each composition were tested for all the tests described below.

The tensile tests were carried out according to ASTM standard 638M using an Emic DL2000 Machine, (Cell Model: Trd23), with an extensometer (Model: TR15) at a test speed of 5 mm/min. The deformation values were measured through extensometers of 80.0mm.

Using the same machine except for the flexural test device, the flexural tests were also carried out according to ASTM standard D790 with the flexure of specimens at a 13.4 mm/min speed.

The Izod impact testing was carried out according to ASTM standard D256 using an impact machine (Make: EMIC, Model: AIC 1). The test specimens of length of 64 mm, width of 12.7 and thickness of 3.2 mm without any notch were used keeping in view the low strength of the materials. All the samples were maintained for 48 hr at the test conditions of room temperature of 23ºC, with 50% relative humidity. Type A test method was carried out with pendulum of 2.7 J at 150° inclination in relation to CdP in vertical, where type C fracture was obtained, with complete break of samples. It may be noted that the necessary energy to disrupt the sample is determined through the loss of kinetic energy of the pendulum (11).

2.2.7. Water absorption

Water absorption tests were carried out for all the composites prepared in this study according to ASTM standard D570-95 using the injected specimens in the form of a bar similar to those used for impact testing (Size:64x12.3 x 3,1 mm). The initial mass of the composite samples was determined at different times starting after 24hs and for 1, 2 and 3 weeks.

3. RESULTS AND DISCUSSIONS
3.1. Characterization of Saw dust

The chemical composition of the saw dust used shows about 50% cellulose, about 30% lignin, 9.7% hemicelluloses and 10 % of other substances with about 9.15% moisture as its contents. The size distribution of the saw dust showed that 39.3% of the fibers were in the range of 40 to 60 mesh, 27.2% below 40 mesh and 33.5% higher than 60 mesh. The density of saw dust determined was 0.374 g/cm^{3}.

Morphology of the fiber used is shown in **Figure 2**. It can be seen that the fibers have different sizes and shapes (**Figure 2a**), while its surface is similar to that

observed in any of the lignocellulosic fibers (**Figure 2b**) showing longitudinal channel type structure with surface defects. **Figure 2c** is the cross section of a fiber showing
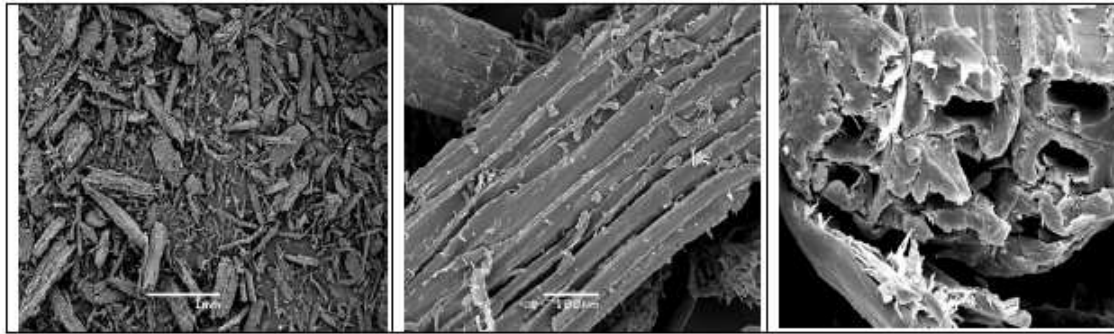

Figure 2: Morphology of saw-dust: (a) distribution of different size of fiber (x25); (b) Surface structure of the fiber (x200); (c) Cross section of a fiber showing cellular structure (x1000).

usual cellular structure revealing thick cell walls and large irregular shaped lumen, not so commonly observed in other lignocellulosic fibers (12).

3.2. Characterization of Prepared MAPP

The coating of the fibers with MAPP and use of minimum amount of PP are important steps as these will improve fiber dispersion in the PP matrix when the composites are prepared, while maximizing the amount of the compatibilizer (MAPP) in contact with the wood fiber. These steps would enhance fibers/matrix interfacial adhesion (13). Also, structure and properties such as fracture behavior of PP blends and its composites are reported to be influenced by the processing methods including compatibilizers used, molecular weight and fiber (reinforcement) size (14-16). Therefore, preliminary reactive extrusions were carried out with distinct conditions (using varying amounts and types of peroxide and/or maleic anhydride).

**Figure 3** shows FTIR spectrum of these samples and also, of the original PP.
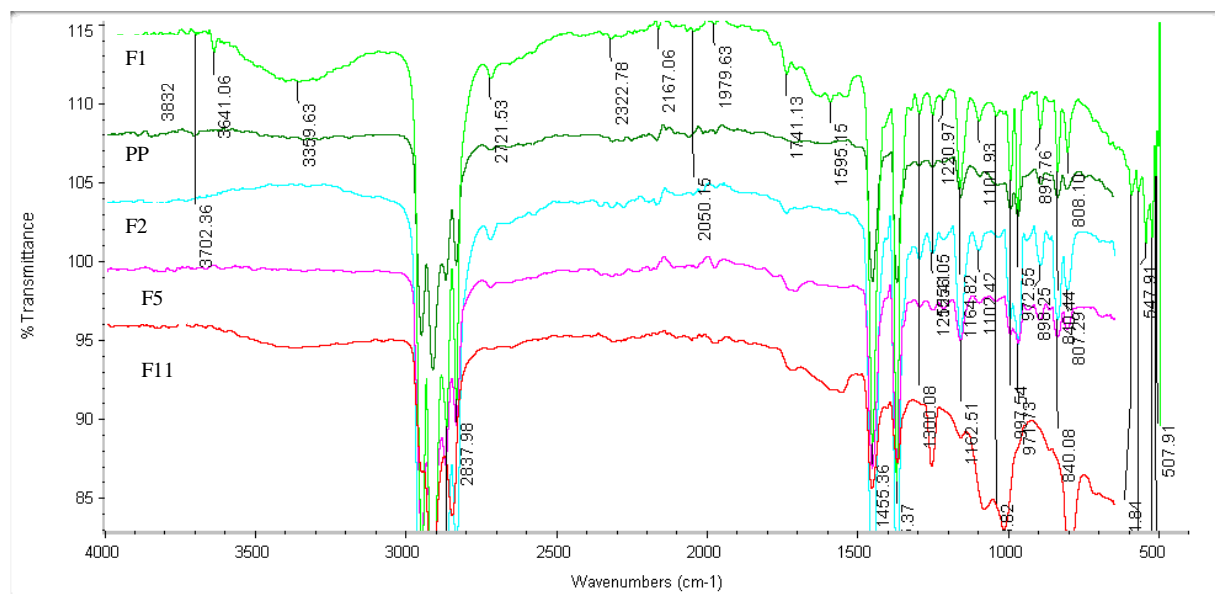
Figure 3: FTIR of manufactured PPMA, PP and Commercial PPMA.

It can be seen that although all MAPP samples (F1 to F11) were characterized by FTIR, only some samples (F1, F2, F5 and F11) showed characteristic bands of

anhydride (asymmetric and symmetric stretching of C=O at the range of 1770-1750cm $^{-1}$). Samples F3 toF9 and F10 do not present the C=O band suggesting that the reactive extrusion reduced only the molecular weight of the original PP samples (17). All MAPP and PP samples showed PP characteristic bands with symmetric vibration of H atoms of CH_2 at 2.853 ±5 cm $^{-1}$, asymmetric and symmetric deformations of C-H of CH_3 at 1450 ± 20cm $^{-1}$ and 1380-1370 cm $^{-1}$, respectively. From a comparison of the prepared MAPP samples and the commercial MAPP sample (F1), the composition of F2 was selected to coat the saw dust and to be used as the PP/saw dust compatibilizer, as it was prepared without any peroxide. Lesser use of reagents is better since it not only brings down the cost, but also marginally affects the decrease in molecular weight. The MFI all samples is shown in **Figure 4**. It can be seen that F2, F5 and F11 present a melt flow index of 5.6g/10min, 27.8 g/10min and 5.2 g/10min respectively. Nevertheless, since F2 still presented a reactive nature during extrusion, it was reprocessed once (F2R) and the melt flow index of this sample was found to be 6.7 g/10min, although the material was still reactive. The reprocessing of F2 reduced the molecular weight of the polymer probably because there was non reacted maleic anhydride in F2 and also due to the scission of chains generated during the reprocessing itself. So, F2R was reprocessed again (F2R2) and this was the compatibilizer used in this study. The MFI of F1 was 54 g/10min, which may be efficient for the dispersion of fibers although, the low molecular weight may contribute in lowering the mechanical properties.

Figure 4 depicts all the combinations with MAH, peroxides and PP during the reactive extrusion along with their measured MFI.
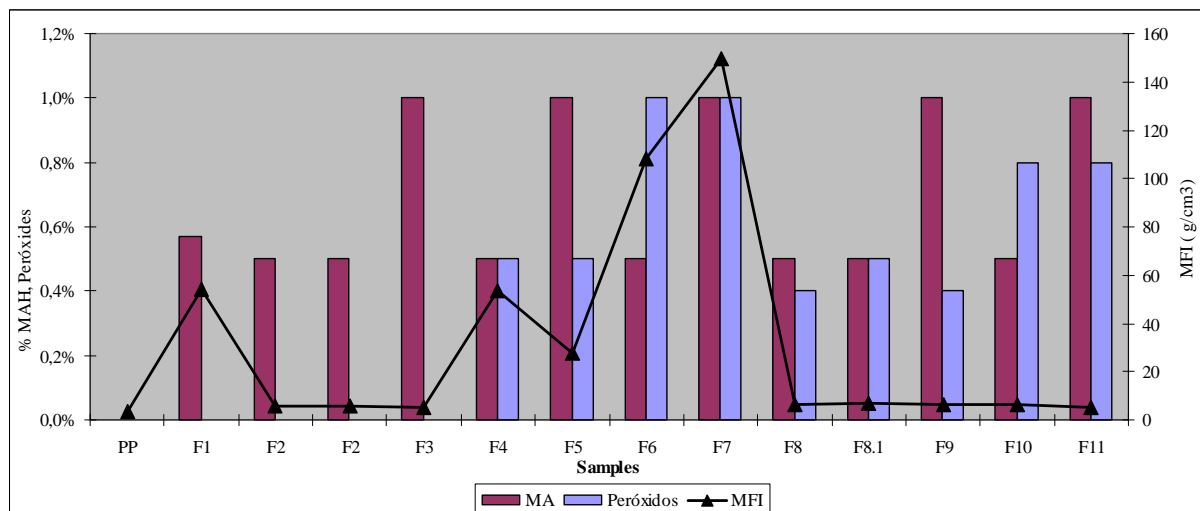
Figure 4: MFI and MAH and peroxides quantities

The density values of polypropylene and the saw dust used were found to be 0.905 g/cm^3 and 0.374 g/cm^3 respectively. The theoretical densities of the prepared composites were calculated using the rule of mixtures (Đ $_{composite}$ = 0.20 x Đ $_{saw dust}$ + % PPMA x Đ $_{PPMA}$ + %PP x Đ $_{PP}$) and compared to their experimental densities.

The theoretical density of composites calculated was found to be 0.798 g/cm^3. It can be seen from **Figure 5** that the theoretical density is lower than the experimental ones, which suggests that the amount of saw dust in the composite was less than20%. It can also be observed from that the composites prepared with the

commercial MAPP showed lower density value than those prepared with MAPP obtained through reactive extrusion (F2).
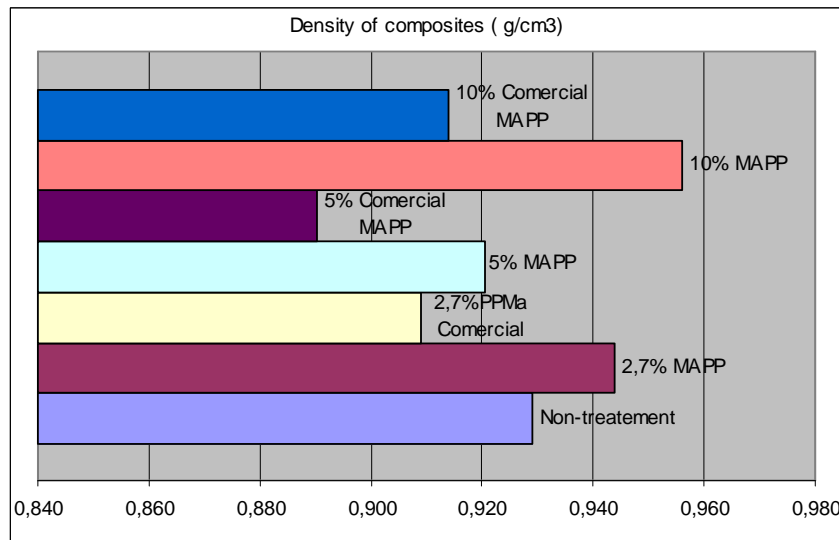

Figure 5. Density of Composites for different quantities of MAPP

This may be understood as due to the lower molecular weight of F1, which helped in the fiber dispersion in the PP matrix and therefore, the composites presented densities closer to the theoretical value, when compared to that of prepared MAPP based composites.

3.3. Mechanical Properties
3.3.1. Tensile and Flexural Properties:

The tensile test results of prepared composites containing 20% of saw dust are presented in **Figure 6**.
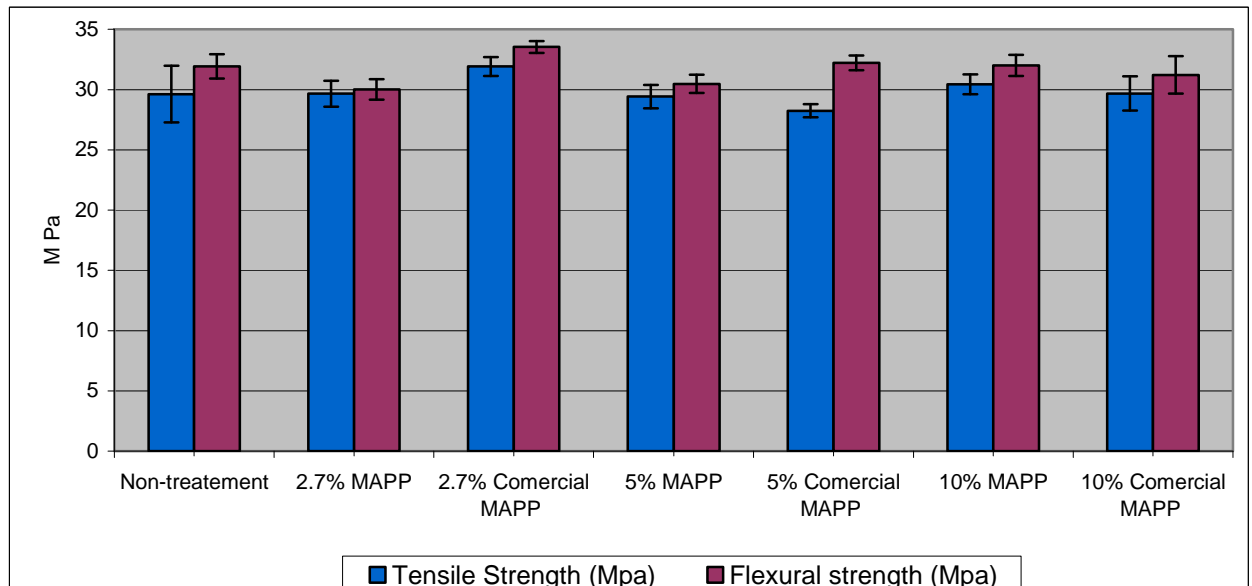
Figure 6. Tensile and Flexural strength of composites

A small increase in both tensile and flexural strengths can be observed for composites with 2.7% of commercial MAPP. This suggests that this material may have better dispersion of saw dust than the composites containing 5% and 10% of

the same material, which showed lower strength values. In view of this, the theoretical and experimental density values are compared. Comparing the tensile strength of samples prepared with F2R2 with the one prepared with F1, it can be concluded that the prepared compatibilizer show better properties because, when the amount of F2R2 is increased, the tensile strength increases. Unfortunately, all injected specimens presented voids in the fractured area, which reduced their true area of stress, contributing to a lower tensile strength than the real one. The performance of the compatibilizer has not been well understood. In fact, it appears that 20% of saw dust may not be enough to change the tensile strength values. It suggests that it may be better to study the compression molded composites containing 30 and 35% saw dust and compare their properties with those of samples without any MAPP. In the case of flexural properties, the composites containing higher amounts of prepared MAPP presented higher flexural strength than those prepared with F1.These injected samples did not present voids and therefore, these results are more reliable although the densities values showed that the amount of saw dust in these composites seemed to be less than 20%.

Figure 7 shows plots of flexural modulus values of different composites studied in this work. Contrary to the expectation that the modulus would not vary in all samples. Some variation was observed due to not so good dispersion of fiber, as already mentioned and shown in scanning electron fractographs (**Figure 8a&b**). Better
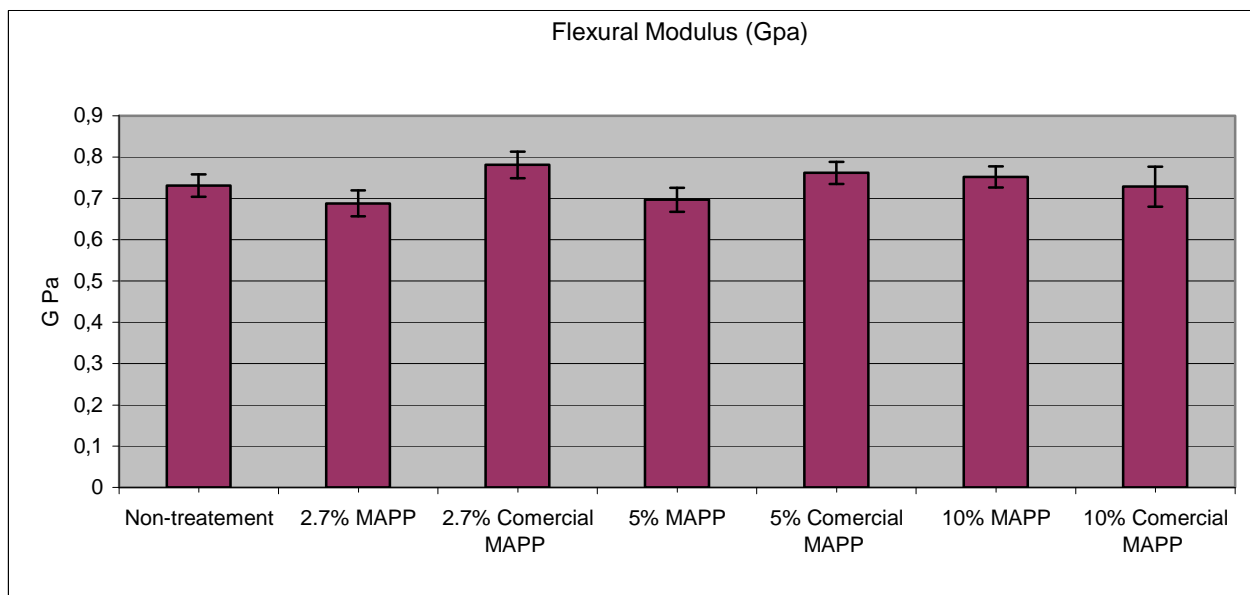
**Figure 7.** Flexural Modulus of composites
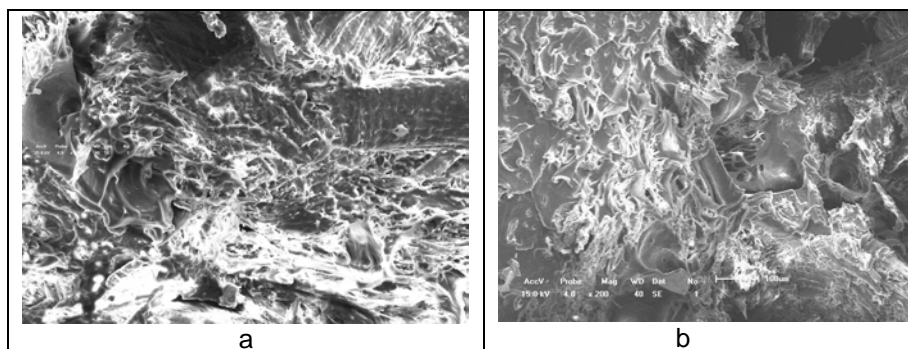
Figura 8. Fractographs of (a) F2R2E7C1 with prepared MAPP; (b) F1E2C2 with commercial MAPP (200x)

performance of commercial MAPP in the composites was probably because of its lower molecular weight, which helped better fiber dispersion as seen in **Figure 8b**.

3.3.2. Impact properties

Figure 9 shows the Izod impact strength values of all the composites studied in this work. It can be seen that the composites prepared using the prepared MAPP showed better impact strength values compared to that containing commercial MAPP, particularly the composite containing 5% of MAPP (showed the highest value).
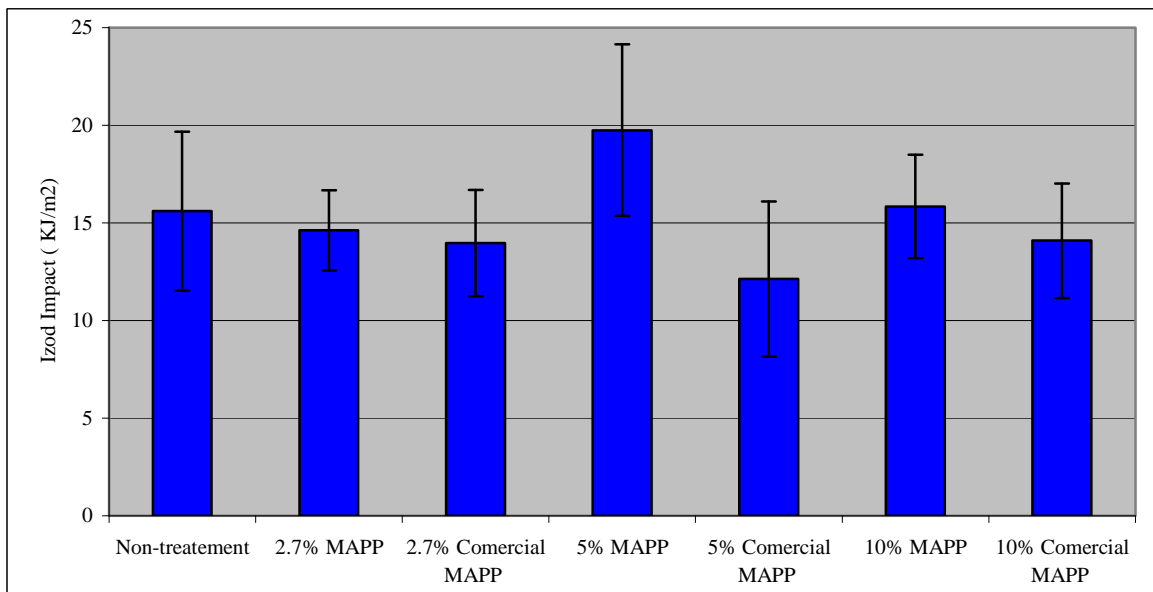
Figure 9. Izod impact strength for different PP-saw dust composites

3.4. Water absorption

Figure 10 shows the plots of weight gain vs time of immersion of various composites prepared in this study. It can be seen that the water absorption increased continuously for the period of test. (24hrs to 3 weeks), some without reaching any plateau. Further, higher absorption was shown by the composites with 2.7% and 5%
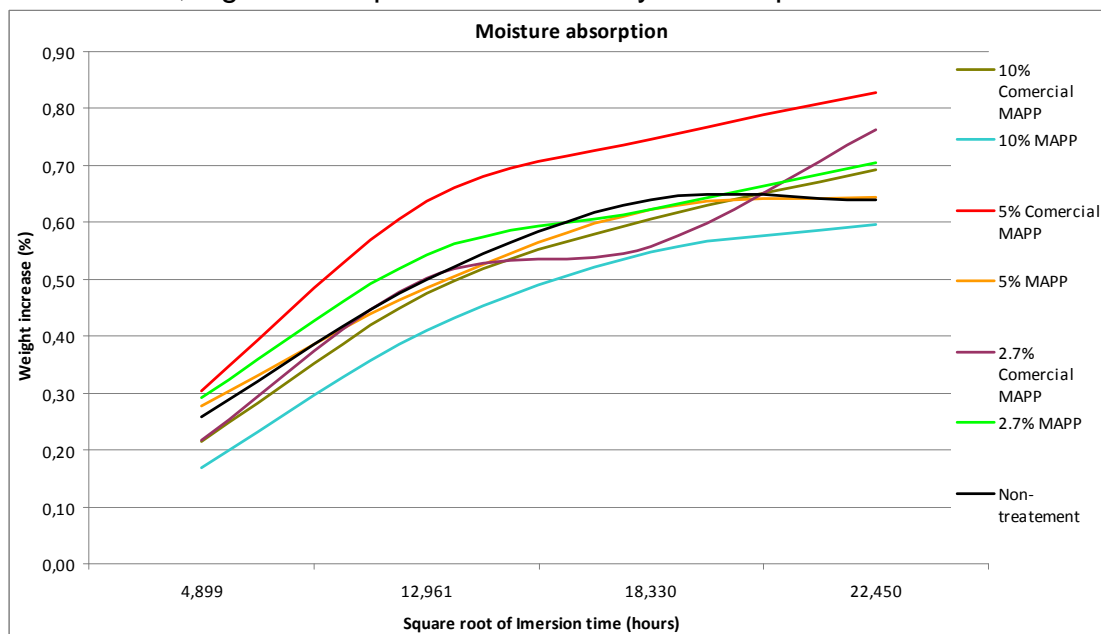
Figure 10. Water absorption curves for composites with different % MAPP

commercial MAPP, while the lowest value was shown by the composite prepared with 10% of MAPP. However, two composites [without MAPP (saw dust + PP) and the composite with 5% PPMA] showed stabilization after 3 weeks, while the composites with commercial MAPP did not stabilize at all. It was expected that the composites that presented a good fiber/matrix interfacial adhesion would present improved tensile strength and absorb less water. However, as fiber dispersion seems to have been poor, as discussed when theoretical and experimental densities were compared, and also, when observed that all mechanical properties specimen presented air voids, no absolute conclusion can be made at present in this respect.

4. CONCLUSIONS

1. The residence time of the materials in the extruder was found to be an important factor in the reactive extrusion of MAPP to achieve complete chemical reactions. The use of MAPP during the fiber coating step ensures the adherence of saw dust to the molecular chains of PP.
2. Temperature should be properly controlled while allowing good mixing to obtain homogeneously mixed materials to avoid any degradation of saw dust during mixing.
3. Coated saw dust composites exhibited a small improvement in tensile and impact strengths compared to those containing uncoated saw dust.
4. Although strength properties of composites prepared with prepared MAPP were at the same level or even better in some cases (impact) than those containing commercial MAPP, the reinforcing effect in the PP/saw dust composite compatibilized by MAPP might be possible with the incorporation of more than 20% saw dust into the matrix.
5. Use of MAPP reduced the water absorption in all the composites studied suggesting its efficiency in providing good fiber/matrix interface, which can assure a longer useful life for the product in practice.
6. Finally, despite obtaining of superior properties in composites prepared using the manufactured MAPP compared to those with commercial MAPP, it is necessary to carry out an economical assessment in order to compare the manufacturing costs with the acquired material cost vis- a-vis performance of the final composite.

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