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The Influence of Wood Flour on Properties of Polypropylene/Wood-Flour Composites

تأثير مسحوق الخشب على خواص المؤتلفات من البولي بروبلين / مسوق الخشب

M.A.H. EL-Meniawi

KEYWORDS:
Wood plastic composite (WPCs), wood flour (WF), thermal, mechanical properties, immersion test, the weight gain and microstructure.

الملخص العربي:- البلاستيك الخشبي المركب يتكون من البولي بروبلين المعاد تدويره ومسحوق الخشب. في هذه الدراسة تم تجهيز العينات من خلال الحقن في القالب مع تركيبات مختلفة من محتوى مسحوق الخشب (10%، 20% ، 30% ، 40%) وبإضافة (Maleic Anhydride Polypropylene) كعامل ربط لزيادة الربط بين المكونات . ترتكز هذه الدراسة على تأثير إضافة محتوى مسحوق الخشب بنسب مختلفة على الخواص الفيزيائية والحرارية والميكانيكية وقابلية المادة المتراكبة لامتصاص المحلول المغمور فيه. هذه الدراسة تناقش سلوك البلاستيك الخشبي المركب في امتصاصه لكلوريد الصوديوم بأيجاد وزن العينة المغمورة فيه وفحصها مجهريا باستخدام الميكروسكوب الالكتروني الماسح لمعرفة تأثير امتصاص المحلول .أظهرت نتائج الدراسة إن أعلى قوة شد وأعلى صلادة كانت عند إضافة نسبة 30% من مسحوق الخشب للمادة المتراكبة . لعب مسحوق الخشب كعامل مهم في زيادة الاستقرار الحراري للمادة عند درجات حرارة مرتفعة . وأخيرا أظهر اختبار الغمر في محلول ملحي بعد 60 يوم إن زيادة محتوى المحلول في العينات التي بها نسبة 40% مسحوق خشب كانت تزداد أكثر وذلك بسبب طبيعة مادة الخشب لامتصاص المحلول .

***Abstract*— In this study, wood plastic composites (WPCs) were made with wood flour and polypropylene matrix (PP). Samples of WPCs were prepared through injection mould with different contents of wood flour (WF) (10%wt, 20%wt, 30% wt , 40%wt). Malic anhydride (MAPP) was added as coupling agent to increase the interaction between the components. Physical, thermal, mechanical properties and immersion test for different contents were investigated. This study discusses the sodium chloride absorption behavior of wood-plastic composite (WPCs). The weight gain of the test sample was determined and microstructures of the composites were examined by SEM analysis to understand the mechanisms for the wood–plastic interaction which affected the solution absorption. The results showed that the addition of 30%WF to WPCpp increases the hardness and tensile strength and then decrease. Wood flour improved the thermal stability of the PP matrix. The specimens with 40% wood flour had much higher solution content than all the other test specimens after immersed up to 60 days in solution.**

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1. INTRODUCTION

Wood plastic composite is the most promising sector in the field of both composite and plastic industries. A new material has emerged, which is a combination of a thermoplastic component and a wood based component, known as wood-plastic composites (WPCs) [1, 2]. The industrial use of wood-plastic composite is growing since several years around the world. These composites are prevalent in outdoor decking applications and concern thermoplastic polymers reinforced by wood fibers or flour. The most widespread polymer matrices are polypropylene, polyethylene and polyvinyl chloride [3, 2]. Polypropylene possesses outstanding properties such as low density, good flex life, sterilizability, good surface hardness, very good abrasion resistance, and excellent electrical properties [4]. However, the main purpose for the addition of cellulose-based fillers to Polypropylene is to reduce the cost per unit volume and to improve stiffness [5]. Low-price cellulose-based fibers, such as wood flour, wood fibers, and cellulose fibers, have high stiffness and low density and are recyclable and nonabrasive [6 -9].In the WPC production the wood content can be increased by 70 wt.%. For this reason, WPCs have potential to take up water under humid conditions due to the presence of numerous hydroxyls. Dimensional changes due to moisture exchange in wood can lead to defects,

such as warping, checking or splitting, which may compromise the performance of the WPC. The application of wood fillers is limited mainly because of the changes in geometry due to moisture absorption and swelling. When the hygroscopicity of the wood fibers is decreased by thermal-treatment method, the WPCs can be used in severity conditions. In addition, destruction of hydroxyl groups in hemicelluloses leads to lower hydrophilic and polarity, low polarity of wood flour cause better compatibility with non-polar polymers such as polypropylene. This study will focus on wood flour reinforced polypropylene composites [10, 11]. Some advantages of WPCs compared with glass fiber reinforced or mineral filled thermoplastics are less environmental impact (e.g. lower embedded energy, smaller carbon footprint, and better recyclability), a less abrasive processing, lower price, increased cooling rate (leading to a decrease in product cycle time in injection molding) . The wood component used in conventional WPCs often originates from planer shavings or sawdust. The producers of WPCs normally use commercial wood flour, which has a broad size distribution, and consequently makes it more difficult to predict the properties of the WPC products. Typical particle sizes used in WPCs are 10-80 mesh (Clemons 2002). In a comparative study on the effects of particle have concluded that it is the particle shape, not the size that has the greatest influence on strength and stiffness. In the present investigation, the conceptual idea was to use residuals from the production of modified wood or fibers, such as sawdust, shavings or boards rejected because of cracks or discoloration. This will mean that no additional wood resources were used and the waste products were turned into value added products. An increase in the resistance of the wood component to moisture and fungal decay could enable a significantly higher weight-% wood in WPCs for outdoor use, which could result in a lower overall cost of the composite because of less use of the generally more expensive thermoplastic matrix.

2. EXPERIMENTAL WORK

2.1. Preparation of the Experiment material:

2.1.1. Wood Flour Sieving

The wood flour utilized is formed from sawdust with fine particle sizes. This type of Wood flour generally is a by-product of wood sawing which ranges from 20 to 5000µm .The common adopted sizes of wood utilized for the production of WPC range from 50 to 700 Micrometers; better properties are obtained when the size approaches the 700 Micrometers. As a result, two sieves were decided to be used with sizes of 500 Micrometers (0.5mm) and 1180 Micrometers (1.18mm) as they gave higher flexural strength and modulus , it was decided to use various mixtures of these two sizes during main experiments; as it was suspected that a mixture of two.

2.1.2. Wood Flour Drying

Wood flour contains molecules of water inside it and it can't be removed by drying only, so we used sodium hydroxide to dry the Wood Flour. First we bring ban contains 20 liters of water, we add 50 gm. of sodium hydroxide to the water then we add wood flour and are left for two hours. Then we removed the wood flour from the water & wash it with water to get rid of the sodium hydroxide and are exposed to the sunlight. Second we put it in the dryer to dry it from any moisture. The dryer used was set at 115º C to avoid wood flour burning. The meshed wood flour is left for 4 hours in the dryer to get rid of the moisture. It was assured that the moisture was totally eliminated through a test that was done. The test consisted of taking samples of the 2 wood waste types; sizes of up to 0.5mm and 1.18mm, utilized within experiments and weighs it. Then, it was left in the dyer for2 hours then weighed. Each hour after the second hour, it was weighed. At the 5th and 6th hour the weight was not changed for the two types (see table 1). Therefore, it was concluded that 4 hours was sufficient to dry the meshed wood waste. Table 1 contains the weights of a sample with a size up to 1.18 mm with the corresponding hours.

TABLE 1. Wood flour drying:

Time	Hours in furnace	Weight (grams)	% Water lost (drying)
9:00 AM	0	25.6	0
11:00 AM	2	24.7	3.51
12:00 PM	3	24.1	5.85
1:00 PM	4	23.8	7.02
2:00 PM	5	23.8	7.02
3:00 PM	6	23.8	7.02

2.1.3. Material Weight

The total weight of injected material was (600gm), we weighted the additives according to considered proportions as indicated at table 2.

TABLE 2. (PP) + (wood flour) + (Malic anhydride (3%)

PP	Wood flour	Malic anhydride (3%)
87% (522gm)	10% (60gm)	18gm
77% (462gm)	20% (120gm)	18gm
67% (402gm)	30% (180gm)	18gm
57% (342gm)	40% (240gm)	18gm

2.1.4. Injection

Before feeding the extruder (single screw extruder), the plastic is mixed; using a mixer, with (wood flour and calcium carbonate) and Malik Anhydride. This mix is composed of shredded plastic waste, Malik anhydride is used as a mineral additive; to enhance mechanical properties, with percentages varying from 0 to 3% of the total weight. The mix is then being fed into the hopper of the extruder and the process starts. Setting the five heaters at 160º C for the first one, 170º C for the second, 190ºC; for the third, 220ºC; for the forth and 240ºC; for the last one, injecting this mixture in the mold. The samples obtained within about 17 min for a 600 gm. used. The temperatures' settings were dependent mainly on the plastic utilized as it has major effects on the process and therefore the final product obtained.

2.1.5. Crusher

The injected samples are crushed in the crusher forming small particles with identical sizes to be fed into the injection machine. The shredding operation was important as it avoided bad distribution of the mix during experiment. As this process at first was done without shredding this resulted in several cases of non-homogenous final product. The main reason behind this that the extrudates have different sizes and the material's concentration within each extrudate wasn't distributed the same. Therefore, it was decided to use a crusher. After crushing the samples, we re-injected the small particles in the mold to obtain the final samples which have been tested.

2.2. Characterization and testing

2.2.1. Immersion Test

Wood plastic specimens ($30\times 15 \times 2mm^3$) with different amount of wood flour (10%, 20%, 30%, 40%) were immersed in 5% of Sodium Chloride (NaCl) solution at 25°C. The initial weight of the test specimens was determined after they were dried at 60◦C in oven for more than 75 hours. The specimens were periodically withdrawn from the water, wiped dry to remove water droplets, and then weighed using an analytical balance of up to 10^{-4} g accuracy to monitor the weight change during the solution absorption process. The moisture content W (t) absorbed by each specimen was then calculated as the weight gain percent relative to its initial weight (w_0) as follows :

$$W(t) = \left(\frac{w_t - w_0}{w_0}\right) \times 100 \quad (1)$$

Where w_t is the sample weight after time t. Specimens were immersed up to 60 days in 5%NaCl solution depending on their composition until complete saturation was reached.

2.2.2. Microstructure

The morphology of the samples were examined with a JEOL JSM 6510 v scanning electron microscope operating at 15 kV.

2.2.3. Thermal Test

Thermal behaviors of wood plastic composites (WPCs) were carried out using an equipment type (SETARAM labsys TG-DSC16) . Each composite was heated from room temperature to 600 °C with a heating rate of 10 °C /min under nitrogen atmosphere.

2.2.4. Tensile Test

Tensile tests are performed on Instron 8501 universal testing machine (Buckinghamshire, UK). These tests are performed according to the ASTM D638 type V [12]. The test specimens were cut into strips of 37 mm 89 mm length , 30 mm width and 3 mm thickness. The crosshead speed is set at 2 mm/min for the break property measurements. Five measurements are carried out for strength and elongation at break measurements.

2.2.5. Hardness Test

Shore D hardness measurements were carried out using hardness tester (OTTOWOLPERT – Werke GMBH , Germany).This test was performed on ten replicates to report the average value .

3. RESULTS AND DISCUSSION

3.1. Immersion Test

The NaCl solution uptake behavior of wood plastic test specimens is described in Fig.3.1. The curves generally display two phases: A high solution absorption rate up to half saturation followed by a slow uptake thereafter. The moisture content increases as the immersion time increases until equilibrium saturation is achieved. the composite sample having 10%w increase in again of weight about (0.055 %) in two days and the composite sample having 20%w increase in again of weight about (0.07 %) in two days , while the composite sample having 30%w and 40%w increase in again of weight about (0.138 % , 0.169 %) respectively in two days . It is interesting to observe that time to reach the saturation point was not the same for all the test specimens. As can be seen in Fig.3.1, the time of saturation of the specimens with 10% and 20% wood flour was actually lower than that of the specimens of 30% and 40% wood flour. The time of saturation of the specimens with 10% and 20% wood four was nearly after 28 days of immersion. While both specimens of 30% and 40% projected continuous water absorption beyond 28 days. As shown in Fig.3.2. it was clear that the specimens with 40% wood flour had much higher solution content than all the other test specimens. The increase of solution absorption by incorporation of wood particles to the plastic matrix is well known [11]. A wood cell wall consists of hydroscopic substances, like carbohydrates and lignin, which leads to solution uptake. The higher the wood proportion in the wood plastic, the more solution will be absorbed. Similar observations are made in literature on wood based polypropylene composites [13-15]. The water absorption and thickness swelling of composites decreased with increase of reinforcing filler. It is well established that the water absorption in natural fiber thermoplastic composites is mainly due to the presence of hydrogen bonding sites in the natural fibers. Cellulose and hemicelluloses are mostly responsible for the high water absorption of natural fibers, since they contain numerous accessible hydroxyl groups. The absorption of water by non-polar polymers, which contain fillers, depends on the nature of the fibers. For cellulose fibers, which are hydrophilic fibers, an increase in water sorption can be expected. Because polypropylene is hydrophobic and the wood flour is hydrophilic, the absorption of water depends solely on the fibers alone [16].Two mechanisms could be mainly responsible for the long-term water absorption of the composites. One of them is capillary transport of water into the gaps and flaws at the interface between fibers and polymer, and transport by microcracks in the matrix formed during the compounding process. Other possible mechanism may involve diffusion behavior (Fickian diffusion process) in the polymer matrix of wood plastic composites [17].

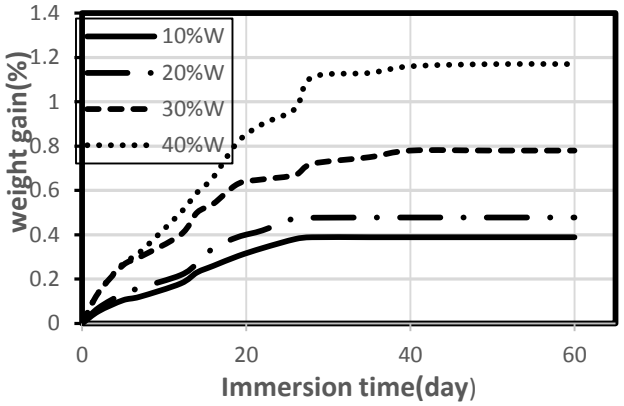

Fig.3.1. Weight change as a function of time for wood plastic test specimens containing different amounts of wood flour when exposed to 5% NaCl solution.

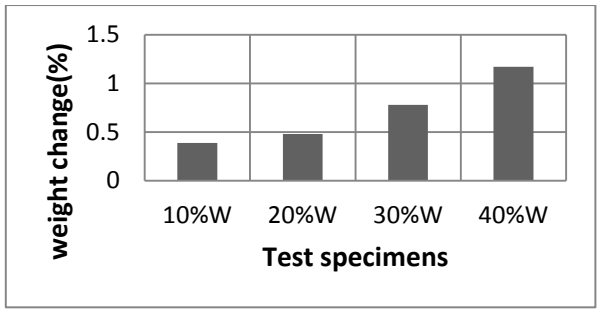

Fig.3.2. weight change (%) wood plastic test specimens over time during immersion test for 60 day

3.2. Structure and morphology (SEM)

The changes in surface morphology of the wood plastic specimens under SEM before and after immersion tests were shown in Fig.3.3. Before immersion test, the surfaces of the specimens were smooth, while after immersion test cracks can be found on the surfaces of the four specimens. Severe cracking was observed on the specimens surfaces of plastic wood with the highest wood/plastic ratio (i.e. 40%, Fig. 3.3(**i**)), and the cracking was less severe at the surface of the specimen with 10% wood flour Fig.3.3(**e**). The cracks can be induced by the expansion/contraction of wood particles due to solution absorption/desorption [15]. It was stated that the characteristics of WPCs mainly depend on the dispersion and adhesion of WF with the polymeric matrix [18]. Figure 3.3 describes the morphology of the WPCs with different WF contents. WF particles appear as white dots in the PP matrix. In all samples, wood flour exhibited a smooth surface with good interfacial adhesion in general. It is clear from Figs. 3a and 3b, where the WF percentage is lower, that the WF particles have a good dispersion and are uniformly distributed in the PP matrix with very no observed voids, while with the increase in WF content to 30 and 40 wt%, Figs. 3c and 3d, it could be observed that the matrix is not enough to encapsulate the solid particles of wood and large aggregates were noticed. The aggregate size increased substantially in these micrographs with the higher wood percentage even at 40 wt% of WF, composite showed a coating layer, evenly distributed on the entire surface which may be described as small colonies of WF.

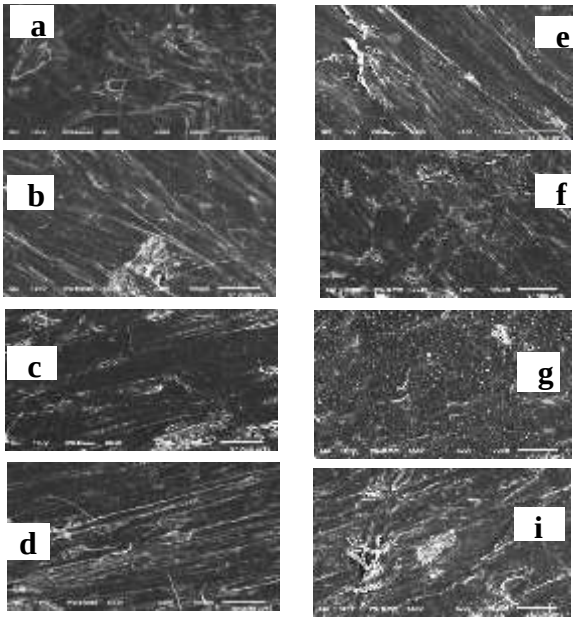

Fig.3.3. SEM images of wood plastic specimen with 10% wood flour before and after immersion test (a) 10%,(b) 20%, (c)30%, (d) 40% before immersion, (e)10%, (f) 20%, (g)30%, (i) 40% after 30 days of immersion

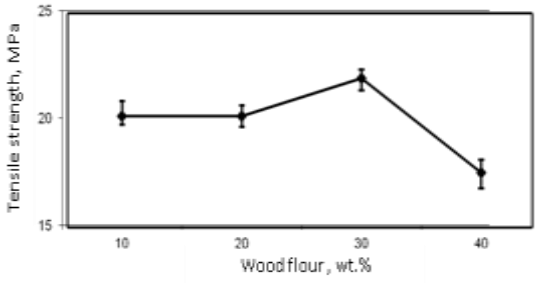

Fig.3.4. Effect of the wood flour content on the tensile strength of WPCs

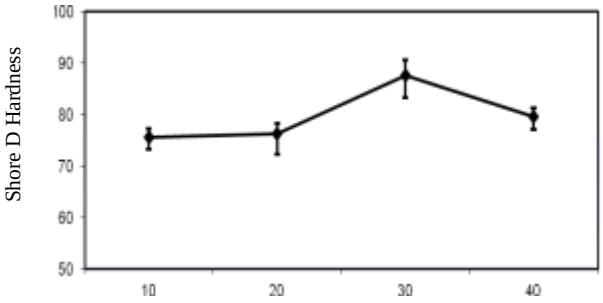

Fig. 3.5. :Effect of wood flour content on the hardness of WPCs

3.3. Mechanical properties

Figure 3.4 illustrates the effect of the wood flour (WF) content on the tensile properties. it can be noticed that when increasing the wood flour content there is a slight increase in tensile strength up to 30 wt%, then, the tensile strength decreases**.** The previous behavior was expected and is in agreement with several authors [19, 20]. From Fig. 3.3, it is clear that when the WF content increases up to 40 wt%; the particles are often not completely encapsulated by the PP

matrix resulting in poor flour–matrix adhesion. This poor adhesion promotes microcracks formation at the interface. Also, the increase in WF content renders the uniform stress transfer due to flour agglomeration within the matrix and optimal load transfer is not possible [21,20] . It was identified by Schwarzkopf et al [22], that decreasing quantity of the matrix polymer in the composites with high wood contents above 40% increases the likelihood of problems such as not fully encapsulated partials, water absorption, crack formation and biological attack.

Figure 3.5 indicates the effect of WF wt% content on the hardness of wood plastic composites. The hardness was found to increase with the increase in filler amount of up to 30 wt% of WF, whereas a decrease in hardness was observed in composites for filler loading amounts of 40 wt%. This behavior could be attributed to the high hardness of the WF filler compared to the soft PP matrix [23]. On the other hand, the decrease in hardness values after 30 wt% of WF could be due to the poor adhesion at the interfaces between particles and polymeric matrix of 40 wt% WF [18].

3.4. Thermal behavior

The thermogravimetry analysis curves of TGA of wood plastic composites content (10 , 20 , 30 and 40 wt%) WF are shown in Fig. 3.6. The main parameters of the thermal degradation process are given in table 3. In order to conveniently investigate the effect of WF content on the thermal properties of wood plastic composite (WPC) and additives were considered and integrated system. From Fig. 3.6 , thermal degradation of WPC 10% WF, the initial low temperature mass loss corresponds to loss of moisture and no degradation took place and WPC was considered as thermally stable at this stage . For the second stage rapid weight loss occurred in the temperature range 267 – 477 °C . The weight loss was about 4.7% at 267 °C , and main decomposition process at a high rate 98.2 % at 477 °C. After 477 °C the residue decomposed at a very slow rate and carbon – rich residual solid formed [24]. Thermal degradation of WPC 20% WF, the sample was thermally stable for temperatures below 267 °C. Decomposition started at about 267 °C and the weight loss was about 3.1% at this temperature . the ended Decomposition around 435 °C and the weight loss was about 99.3% . Thermal degradation of WPC 30% WF, was thermally stable for temperatures below 267 °C. Decomposition started at about 267 °C and the weight loss was about 5.2 % at this temperature . the ended Decomposition around 450 °C and the weight loss was about 95.5% . WPC 40% WF start degradation at temperature about 274 °C and below these temperatures seems was thermally stable , and the weight loss was about 4.7 % at 274 °C . The ended decomposition around 452 °C and the weight loss was about 95.7% . The result calculated by adding WF based on their percentages in the composite . Compared with different rates, WPC 20% WF was less weight loss at a temperature 267°C . WPC 40% WF was gave us the best result because of the beginning of degradation started at temperature 274 °C and its ability to withstand up to a temperature of 452°C and the remainder of it was 4.3 % at 600 °C. This indicating that wood flour improved the thermal stability of the PP matrix and this could be attributed to the high thermal stability of lignin in WF [25].

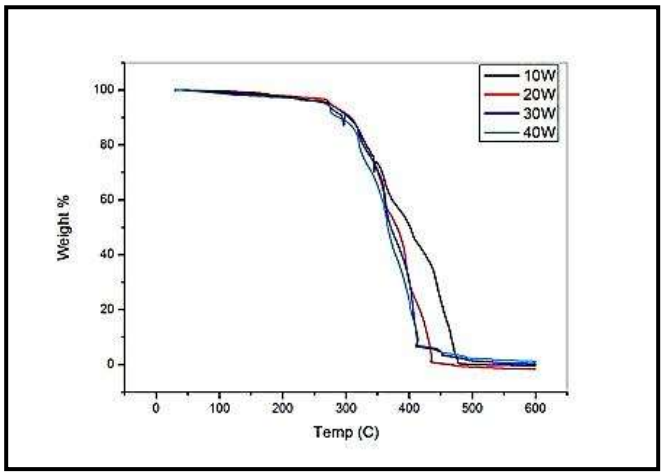

Fig. 3.6. TGA curve of WPCs

TABLE 3. THERMOGRAVIMETRIC ANALYSIS DATA:

Sample	T onset °C	Weight loss At T onset %	Tmax °C	Weight loss At T max %	Residue % At 600 °C
WPC-10%WF	267	4.7	477	98.2	1.2
WPC-20%WF	267	3.1	435	99.3	0.7
WPC-30%WF	267	5.2	450	95.5	4.5
WPC-40%WF	274	4.7	452	95.7	4.3

4. CONCLUSIONS

The influence of wood flour on mechanical property, thermal degradation, and sea water absorption of WPC were successfully examined.

1. Increasing the wood flour content from 20 to 40 wt% wood flour in composites increased the percentage change of weight. This increased of wood flour on the specimen surface, increased cracks after immersion test.
2. The specimen with 10 wt% wood flour had smaller changes in weight and less cracks on its surface. There is a slight increase in tensile strength up to 30 wt% of WF, then, the tensile strength decreases.
3. The hardness was found to increase with the increase in filler amount of up to 30 wt% of WF, whereas a decrease

in hardness was observed in composites for filler loading amounts of 40 wt%.

4. Thermal stability of WPCs was shown to be wood flour dependent. Wood flour improved the thermal stability of the PP matrix.
5. The time of saturation of the specimens with 10% and 20% wood flour was actually lower than that of the specimens of 30% and 40% wood flour.
6. The specimens with 40% wood flour had much higher solution content than all the other test specimens after immersed up to 60 days in solution.

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