

## The Biodegradation and the Rheological Properties of Polypropylene/Hyperbranched Polyester Blends for Industrial Applications

AL-MUTAIRI Nabeel Hasan <sup>1, a</sup>, AL-ZUBIEDY Ali <sup>2, b</sup>, AL-ZUHAIRI Ali J. <sup>3, c</sup>  
and IDZIKOWSKI Adam <sup>4, d</sup>

<sup>1,2</sup>Polymer and Petrochemical Industries Department, University of Babylon, Hilla, Iraq

<sup>3</sup>Department of Energy, College of Engineering, University of Babylon, Al-Musayyab, Iraq

<sup>4</sup>Department of Production Engineering and Safety, Faculty of Management, Czestochowa University of Technology

<sup>a</sup> nabeeleng90@gmail.com, <sup>b</sup> mat.ali.alzubiedy@uobabylon.edu.iq, <sup>c</sup> alijassim33@yahoo.com, <sup>d</sup> adam.idzikowski@pcz.pl

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**Abstract.** In this research, four novel types of hyperbranched polyester polymers (HBPs) were used and blended with polypropylene polymer PP, virgin VPP and recycled rPP, using a twin-screw extrusion machine. Hyperbranched polyester was added in different weight ratios (5%, 10%, and 20%). The Fourier transform spectroscopy FTIR, the water contact angle CA, the biodegradation in soil, and the rheological properties (melt flow rate MFR) of the prepared blends were investigated. The results showed that the contact angle of VPP and rPP blends has improved with the addition of HBPs, and in addition, the biodegradation results in soil showed that weight loss increased as the amount of HBPs increased in VPP and rPP blends. The rheological properties, melt flow rate MFR, showed that the addition of HBPs increased the MFR for both PP blends, VPP and rPP. In addition, it was found that the blends' viscosity was decreased and their shear rate was increased. This is an indication that the HBPs work as a processing aid additive by increasing the shear thinning behavior.

### Introduction

New materials have made significant contributions to economic and technical advancement throughout the past century. There are numerous metals, alloys, composites, ceramics, and polymers in the list of materials. Out of these, polymers have significantly influenced this process of development and have emerged as a significant class of engineering materials [1]. High molecular mass compounds and numerous repeating units make up the structure of polymers. Today, polymer materials are so crucial to human life that every aspect of it depends on them [2].

Polymers have a wide range of qualities that make them the perfect material for a wide range of uses, from polyethylene in sandwich and garbage bags to poly (p-phenylene terephthal amide) in bulletproof vests [3]. Since they combine flexibility, toughness, excellent barrier properties, ease of manufacturing, and good chemical resistance, polyolefins have drawn a lot of attention recently. This makes them excellent materials for various packaging applications, particularly in food packaging. Examples of this include polypropylene (PP) and polyethylene (PE), which are widely used in commodities and packaging applications [4],[5]. Due to their low cost, lightweight, high mechanical strength, water resistance, and strong barrier properties, PP is increasingly used in the plastic packaging industry [6].

It has been estimated that the volume of plastic packaging will expand by two and three times, respectively, in 2030 and 2050, compared to the current level of global plastic manufacturing [7].

Because of their superior physical and mechanical qualities, non-biodegradable polymers such as polyethylene (PE), polypropylene (PP), ethylene vinyl alcohol, poly (ethylene terephthalate), polystyrene, expanded polystyrene, polyamides, polyurethane, and poly (vinyl chloride) have previously dominated the packaging industry. These polymers typically remain stable in the environment for a very long time and are a major waste management challenge since they are inert and resistant to microbial attacks [8].

Eco-friendly polymeric materials should be used for potential short-lifespan applications to prevent the environmental and ecological damage caused by post-consumer plastic trash. For applications requiring disposable materials, such as packaging, consumer goods, and hygiene items, biodegradable polymeric materials make good choices. However, due to their comparatively expensive cost and inadequate mechanical and thermomechanical properties in comparison to some non-biodegradable commodity polymers, the use of biodegradable polymers is restricted for several applications [9].

Therefore, these restrictions can be circumvented by creating biodegradable polymer blends with suitable characteristics. Improving adhesion between blended components, lowering interfacial tension, and producing limiting inclusion phase size are the three key issues with polymer melt blending [10].

Additionally, when processing polymers, manufacturers want to enhance flow rate, lower energy costs, and limit the likelihood of extrudate abnormalities. The upper limiting production rate is greatly increased by using polymer processing aids (PPAs) to effectively remove processing abnormalities that lead to issues with products [11]. A number of chemicals, including polymers, have been used as processing aids in an attempt to minimize surface roughness in polyolefin processing. Hyperbranched polymers (HBP) have received significant interest as PPAs, owing to their unique dendrimer like branched structure with lack of chain entanglements and the resulting dramatically reduced melt viscosity [12].

In this study, four novel hyperbranched polyester polymers HBPs were blended in different weight percentages (5%, 10%, and 20%) with both VPP and rPP. The blends were prepared using a twin-screw extruder. The effect of HBPs on the biodegradation and rheological properties were investigated.

## Materials and Methods

**Materials.** VPP and rPP were purchased from sabic (KSA), and used as the major phase for the preparation of polymeric blends. A novel Hyperbranched polyester polymers HBPs (HBP-TA, HBP-AD, HBP-MA, and HBP-PA) were synthesized by polycondensation polymerization in our laboratory (polymer department/Babylon University) according our previous works [13] and [14]. Table 1 shows the properties of VPP and rPP.

*Table 1. Properties of the used PP*

Properties	Value		Testing Standard
	VPP	rPP	
Tensile strength (MPa)	20-35	20-30	ASTM D-638
Modulus of Elasticity (GPa)	0.13	0.12	
MFR (g/10min)	5-10	15-25	ASTM D-1238

**The preparation of PP/HBPs blends.** The HBPs and PP pellets (VPP and rPP) were mixed at various weight ratios, as shown in Table 2.

**Table 2.** The percentages of polymeric blends

VPP/HBPs (%)	rPP/HBPs (%)
80/20	80/20
90/10	90/10
95/5	95/5
100/0	100/0

The two polymers, PP and HBPs, were first mechanically mixed in the dry state for 10 minutes before being processed into blends using a twin-screw extruder. With a screw speed of 25 rpm and temperatures of 165–175 °C for VPP and 155–165 °C for rPP, the blends were extruded. A plastic sheet is created by passing the molten material that exits the extruder between two moving rollers. These sheets were cut in accordance with ASTM standards to provide various samples for various tests.

**Characterization.** FTIR were used identify the changes in chemical structure by the change in band values. Instrument, type IR Affinity-1 range (400-4000 cm<sup>-1</sup>) was used for this purpose.

MFR was used to measure the effect of HBPs on the melt flow rate of PP (VPP and rPP) using Shi Jia Zhuang MFI tester based on ASTM-D1238. The samples tested at 230 °C and 2.16 Kg load with 8mm die. The melt flow rate, the viscosity ( $\eta$ ) and shear rate ( $\dot{\gamma}_w$ ) were evaluated according to the equations below [15].

$$\text{MFR} = t_{\text{ref}} * w / t \dots \dots \dots \text{Eq (1)}$$

$$\eta = (4.98 * 10^4 * \rho * L) / \text{MFR} \dots \dots \dots \text{Eq (2)}$$

$$\dot{\gamma}_w = (1840 / \rho) * \text{MFR} \dots \dots \dots \text{Eq (3)}$$

Where: -  $t_{\text{ref}}$  = 600 sec,  $t$  = time (sec),  $w$  = average weight of of sample (g),  $\dot{\gamma}_w$  = Shear rate at the wall (s<sup>-1</sup>),  $\rho$  = Density (kg /m<sup>3</sup>).

The wettability of PP (VPP, rPP) and its blends with HBPs at different wt. % percentages have been investigated using the device, SL 200C - Optical Dynamic I Static Interfacial Tensiometer & Contact Angle Meter, which using circle fitting method of water on samples surface. The tested data have been collected at two intervals after 3 sec and 60 sec. The aim of this test is to study the effect of the HBP on the wettability of polypropylene PP.

The biodegradation of the blends in soil were measured according to ASTM-D 5988, by burying in soil with pH 7.56 at a distance 10 cm from the surface for 90 days. The weight loss of samples was calculated 10 days according to the equation below [16].

$$\% \text{ weight loss} = [(W_b - W_a) / W_b] * 100 \dots \dots \dots \text{Eq (4)}$$

Where: -  $W_b$  and  $W_a$  are initial mass before and after degradation in the soil respectively.

**Results and Discussion**

**Characterization of PP and PP/HBP Blends – FTIR analysis result.** FTIR spectroscopy was used to analyze the effect of the prepared HBP addition on the chemical structure of VPP and rPP. Figure 1 and 2 shows the bands values and the changes in these values with the addition of HBP. Table 3 and 4 lists the most important bands of PP and its blends with HBP as derived from Figure 1 and 2 and compare them with the bands mentioned in [17, 18]. The following bands were identified for VPP and rPP: 2954.95 cm<sup>-1</sup> and 2885.51 cm<sup>-1</sup> for –CH<sub>3</sub> asymmetric stretching vibration and –CH<sub>3</sub> symmetrical stretching vibration, bands at 995.27 cm<sup>-1</sup>, 972.12 cm<sup>-1</sup> and 1165 cm<sup>-1</sup> related to –CH<sub>3</sub> rocking vibration. The peaks at 1458.18 cm<sup>-1</sup>, 2839.22 cm<sup>-1</sup> and 2924.09 cm<sup>-1</sup> are related to –CH<sub>2</sub> symmetric bending, –CH<sub>2</sub> symmetric stretching and –CH<sub>2</sub> asymmetric stretching. The peak at 1373.32 cm<sup>-1</sup> is attributed to –CH<sub>3</sub> symmetric bending vibration mode. The peak at 840.96 cm<sup>-1</sup> for C–CH<sub>3</sub> stretching vibration and peak at 810.10 cm<sup>-1</sup> is for CH<sub>2</sub> rock, C–C stretch, C–CH stretch. The addition of the prepared HBP to vPP and rPP had some shifting on the bands of PP; also, the bands of HBP appears in the IR spectrum. From these results, it can be

concluded that the interaction between the two polymers, PP and HBP, is physical and not chemical.

**MFR Results.** The rheological properties (MFR, viscosity, and shear rate) of PP and its blends with HBP have been characterized using MFR. The MFR of PP and PP/HBP blends shown by Figure 3. From figure 3A for VPP and VPP/HBP blends, it's found that the MFR of all blends has been increased with the increase of HBP content. It's shown that the VPP has MFR of 10 (g/10 min), as the HBP blended with VPP the MFR was increased by 2.6, 6.23, and 4.14 g/10 min for 5%, 10%, and 20% of HBP-TA samples and increased by 2.8, 28.5, and 15.7 g/10 min for 5%, 10%, and 20% of HBP-AD samples respectively. While, it increased by 5.7, 12.1, and 6.1 g/10 min for 5%, 10%, and 20% of HBP-MA samples and increased by 7.8, 11.24, and 9.6 g/10 min for 5%, 10%, and 20% of HBP-PA samples respectively.

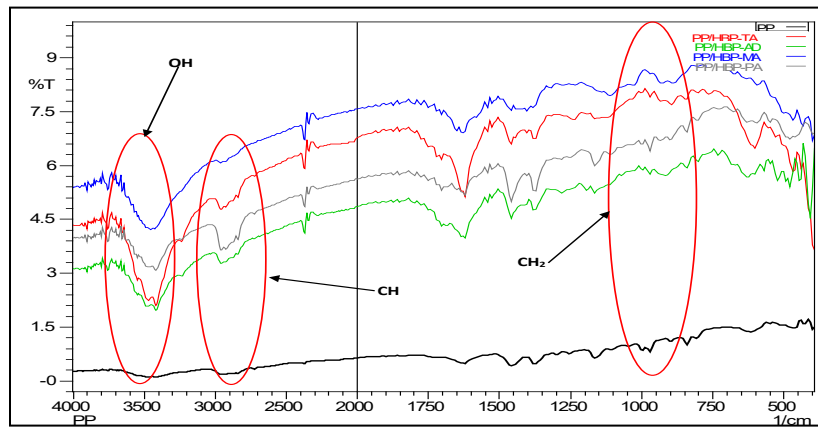
However, for rPP and rPP/HBP blends shown in Figure 3B, it was found that the MFR improved by the addition of HBP. The MFR of rPP is 26.7 g/10min, as the HBP added the MFR improved by 6.2, 20.8, and 10.3 g/10min for 5%, 10%, and 20% of HBP-TA samples and improved by 37, 65.45, and 56.8 g/10min for 5%, 10%, and 20% of HBP-AD samples respectively. While, it improved by 15.4, 48.7 and 34.9 g/10min for 5%, 10%, and 20% of HBP-MA samples and improved by 21.7, 31.2 and 10.1 g/10min for 5%, 10%, and 20% of HBP-PA samples respectively.

From the above results, it's found that HBP addition improve the MFR for rPP better that VPP due to the less entanglements and shorter chains of rPP. In addition, it is found that 10% of HBP has the highest MFR value for VPP and rPP blends due to the better compatibility and distribution of HBP within PP matrix as indicated by SEM. These results attributed to the fact that hyperbranched polymers HBP have relatively lower viscosity than linear polymers PP, which mean that these additives act as viscosity reducers (processing aids additives). So that the presence of HBP in the molten PP generate a rolling effect which facilitates sliding of melt on the cylinder wall of MFI tester, serve as ball bearings, and reducing the interlayer interaction.

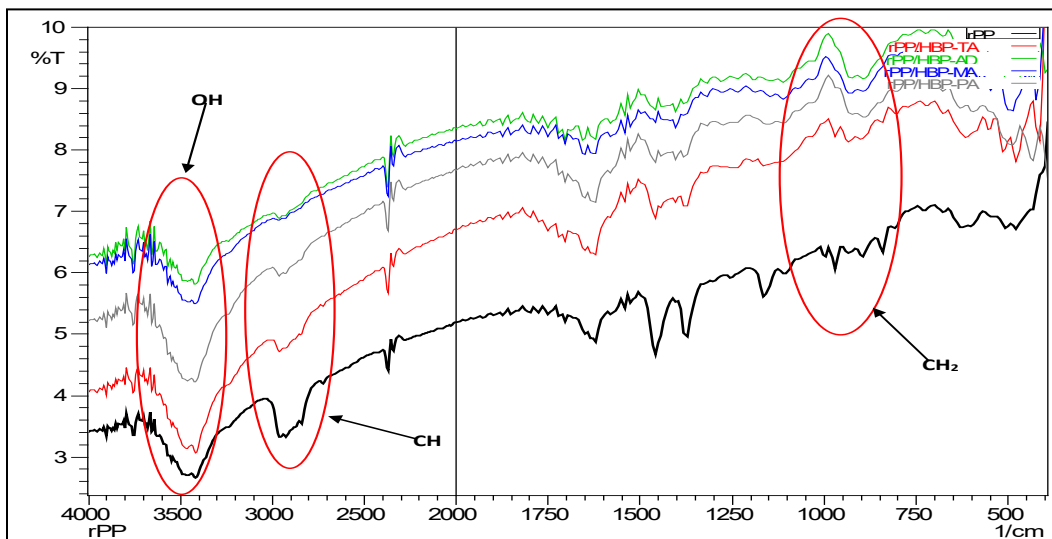
According to the results above, the viscosity, shear rate and the power law index (n) of VPP, rPP, and their blends were calculated easily from equations 2 and 3 to give the data in table 5.

**Table 3.** IR transmission bands for VPP and its blends

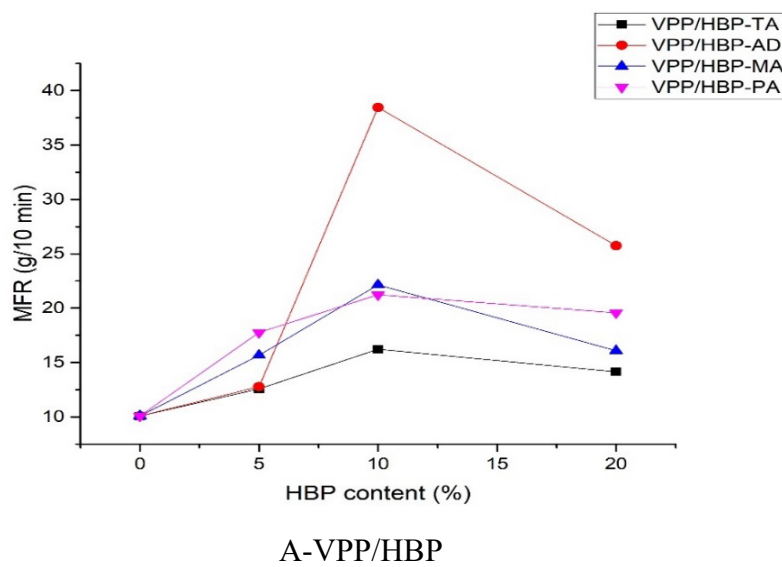
Bond type	PP[17], [18]standard	VPP	PP/HBP-TA	PP/HBP-AD	PP/HBP-MA	PP/HBP-PA
C-C stretch	808	810.1	802.39	802.39	802.39	802.39
C-H rocking	840	840.96	848.68	840.96	843.55	840.96
CH <sub>3</sub> rocking	973	972.12	972.12	972.12	972.12	972.12
C-C stretch						
CH <sub>3</sub> rocking	996	995.27	996.07	995.27	=	995.27
C-C stretch						
CH wagging	1166	1165	1165	1165	1141.86	1165
CH <sub>2</sub> rocking						
CH <sub>3</sub> sym.bend	1376	1373.32	1381.03	1381.03	1404.18	1373.32
	1456	1458.18	1458.18	1458.18	1458.18	1458.18
CH <sub>3</sub> stretch	2870	2885.51	2877.19	2885.51	2877.19	2877.79
CH <sub>3</sub> asym.stretch	2920	2924.09	2924.09	2931.8	2931.8	2924.09
CH <sub>3</sub> asym.stretch	2950	2954.95	2962.66	2954.95	2962.66	2954.95

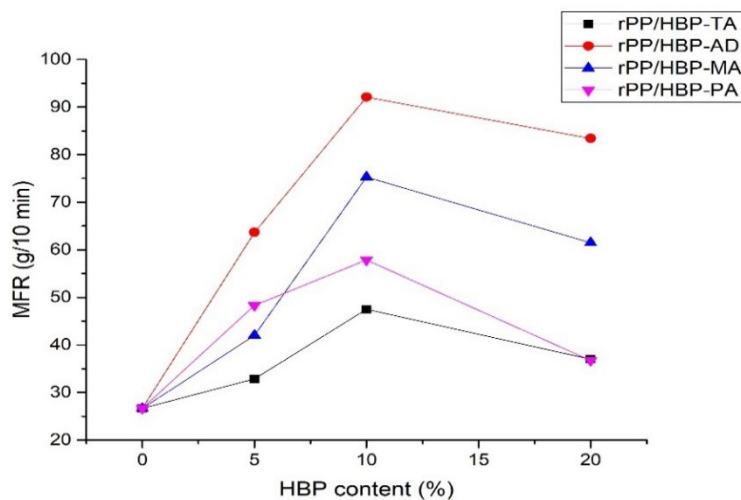


**Fig. 1.** IR spectrum for VPP and PP/HBP.



**Fig. 2.** IR spectrum of rPP and rPP/HBP.





B-rPP/HBP

**Fig. 3.** MFR of VPP, rPP, and their blends with HBP.

**Table 4.** IR transmission bands for rPP and its blends

Bond type	PP[17], [18]standard	rPP	PP/HBP-TA	PP/HBP-AD	PP/HBP-MA	PP/HBP-PA
C-C stretch	808	810.1	810.1	802.39	802.39	802.39
C-H rocking	840	840.96	840.96			
CH <sub>3</sub> rocking	973	972.12	972.12	972.12	972.12	972.12
C-C stretch						
CH <sub>3</sub> rocking	996	995.27	=	=	=	=
C-C stretch						
CH wagging	1166	1165	1165	1149.57	1188.15	1149.57
CH <sub>2</sub> rocking						
CH <sub>3</sub> sym.bend	1376	1373.32	1381.03	1381.03	1381.03	1381.03
	1456	1458.18	1458.18	1458.18	1458.18	1458.18
CH <sub>3</sub> stretch	2870	2877.79	2877.79	2877.79	2883.11	2889.37
CH <sub>3</sub> asym.stretch	2920	2924.09	2931.8	2924.09	2931.8	2924.09
CH <sub>3</sub> asym.stretch	2950	2954.95	2962.66	2962.66	2962.66	2962.66

**Table 5.** The calculated data from the MFR for VPP, rPP, and their blends

Samples	Viscosity (Pa.s)	Shear rate (S <sup>-1</sup> )	Power law index (n)
VPP	34903.5	21002.4	0.43
VPP/5%HBP-TA	28836.2	25421.4	0.392
VPP/10%HBP-TA	22382.1	32752	0.343
VPP/20%HBP-TA	26326.2	27845.1	0.374
VPP/5%HBP-AD	28205.97	21002.4	0.388
VPP/10%HBP-AD	9564.3	25989.4	0.195
VPP/20%HBP-AD	14281.42	76645.1	0.262
VPP/5%HBP-MA	22888.92	32026.7	0.348

VPP/10%HBP-MA	16896.91	43384.03	0.292
VPP/20%HBP-MA	23388.7	31342.4	0.352
VPP/5%HBP-PA	20471.84	35808.02	0.327
VPP/10%HBP-PA	17215.23	42581.83	0.295
VPP/20%HBP-PA	18097.1	40506.92	0.304
rPP	13141	55783.9	0.39
rPP/5%HBP-TA	10850.8	67557.8	0.366
rPP/10%HBP-TA	7713.2	95039.3	0.311
rPP/20%HBP-TA	10205.7	71828.2	0.355
rPP/5%HBP-AD	5619.28	130453.73	0.263
rPP/10%HBP-AD	3891.9	188356.12	0.211
rPP/20%HBP-AD	4432.6	165379.9	0.229
rPP/5%HBP-MA	8537.14	85866.7	0.327
rPP/10%HBP-MA	4808.84	152439.21	0.24
rPP/20%HBP-MA	6073.82	120691.13	0.275
rPP/5%HBP-PA	7364.21	99543.01	0.303
rPP/10%HBP-PA	6288.02	116579.7	0.28
rPP/20%HBP-PA	10242.1	71572.9	0.356

From Table 5, it is shown that the viscosity of all blends PP/HBP were decreased by the increase of HBP while the shear rate increased with HBP increasing, which proves that HBP addition increases the flow behavior for PP. The shear thinning increased with the increase of HBP content as indicated by table 5, the values of power law index decreased for all blends.

From these results, it can be concluded that the addition of HBP has improved the processability of PP and the energy consumption during the preparation of blends, these results are in agreement with Guzmán [19] and Mesias [20].

**Contact Angle Results.** The water contact angle gives an indication of the nature of the surface, whether it is hydrophilic or hydrophobic. The higher the contact angle the lower the wettability and the surface is hydrophobic and vice versa. In table 6 and 7, the water contact angle data for PP and PP/HBP blends are shown at different time intervals. For VPP and its blends with HBP, it is found that the contact angle decreased with the increase of HBP content from 0% to 20%, the contact angle after 3 sec for VPP decreased from 78.38° to 60.17° for HBP-TA and decreased to 62.1° for HBP-AD and decreased to 61.61° and 67.87° for HBP-MA and HBP-PA respectively after 60 sec the contact angle for VPP decreased from 78.38° to 69.4° and decreased further for the other blends, as shown in Table 6.

**Table 6.** Contact angle data for VPP, and their blends with HBP

Samples	Contact angle	
	After 3 sec	After 60 sec
VPP	78.38°	69.4°
VPP/5%HBP-TA	65.66°	54.8°
VPP/10%HBP-TA	63.18°	54.18°
VPP/20%HBP-TA	60.17°	49.95°
VPP/5%HBP-AD	62.24°	60.32°
VPP/10%HBP-AD	61.68°	57.07°
VPP/20%HBP-AD	62.1°	58.12°
VPP/5%HBP-MA	65.51°	59.06°

VPP/10%HBP-MA	63.36°	58.85°
VPP/20%HBP-MA	61.61°	57.32°
VPP/5%HBP-PA	65.95°	61.24°
VPP/10%HBP-PA	63.89°	61.43°
VPP/20%HBP-PA	67.87°	64.05°

**Table 7.** Contact angle data for rPP, and their blends with HBP

Samples	Contact angle	
	After 3 sec	After 60 sec
rPP	70.77°	66.5°
rPP/5%HBP-TA	65.02°	48.82°
rPP/10%HBP-TA	63.92°	42.57°
rPP/20%HBP-TA	58.23°	47.95°
rPP/5%HBP-AD	68.2°	59.25°
rPP/10%HBP-AD	65.89°	53.33°
rPP/20%HBP-AD	65.37°	58.53°
rPP/5%HBP-MA	65.41°	58.49°
rPP/10%HBP-MA	63.38°	55.57°
rPP/20%HBP-MA	64.1°	55.21°
rPP/5%HBP-PA	68.23°	59.61°
rPP/10%HBP-PA	67.3°	55.45°
rPP/20%HBP-PA	68.2°	59.85°

For rPP and rPP/HBP blends, it's found that the contact angle also decreased with HBP increasing. The contact angle after 3 sec for rPP decreased from 70.77° to 58.23° and 65.37° for HBP-TA and HBP-AD respectively and decreased to 64.1° and 68.2° for HBP-MA and HBP-PA respectively after 60 sec the contact angle for rPP decreased from 70.77° to 66.5° and decreased further for the other blends, as shown in Table 7.

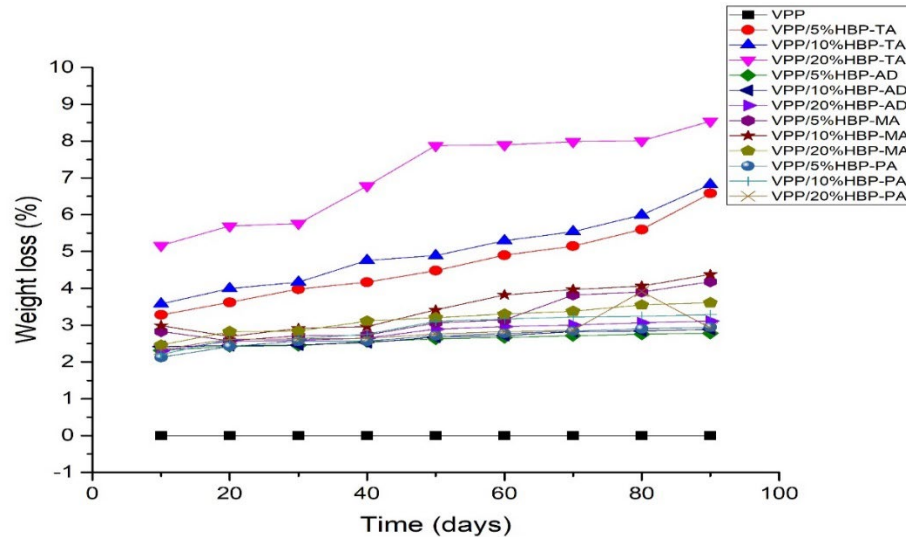
From the results, it's found that the addition of HBP to PP has improve its hydrophilicity (the surface wettability increased) by reducing the contact angle, this improvement is due to the presence of large number of hydroxyl group and carboxyl group in the prepared HBP (the presence of polar group can improve the wettability and hydrophilicity of the surface) these results are in agreement with Zhang [21] and Caicedo [22]. Also, it's found that the rPP/HBP blends have better wettability and lower contact angle than VPP/HBP, this due to the shorter the chains and the less entanglement in rPP than VPP. In addition, the blends with HBP-TA have the lowest contact angle, the best surface wettability and the best hydrophilicity, this is due to the higher content of hydroxyl and carboxyl group in HBP-TA when compared with the other types of the prepared HBPs.

**Biodegradation Results (Soil Burial).** Most types of polymers when they meet the required need and after the expiry of their use, they will accumulate in large quantities, which will have a dangerous impact on the environment as well as on the general appearance. In order to reduce the accumulation of these materials, these polymers are usually mixed with other types of materials that have the ability to decompose in order to improve the decomposition of these polymers and to reduce their danger to the environment and human life.

The biodegradation through the weight loss of PP and its blends with HBP in soil are shown in Figure 4 and 5. From the results, its note that both VPP and rPP did not show any weight loss during the 90-day examination period. These results indicate that this type of polymer does not have the ability to degrade in short periods, but needs very long periods, because the chemical structure of this type of polymer does not contain any groups that aid in degradation. For VPP/HBP

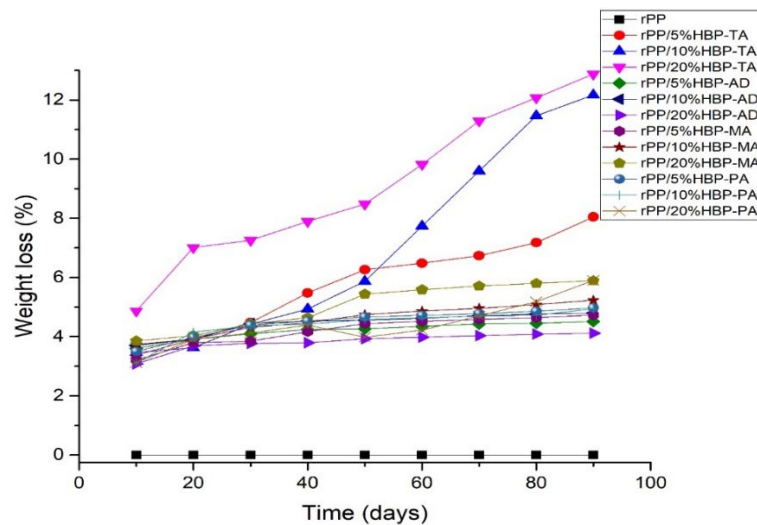


blends it is found that there is an improvement in the degradation of PP through the test period. It is shown from Figure 4 that the weight loss increases as both test period and the amount of HBP increase. It is found that the blend with HBP-TA increases the weight loss to 6.5%, 7.7%, and 8.5% as the HBP increase from 5% to 20%, and the blend with HBP-MA increases the weight loss to 3.5%, 4.2%, and 4% as the HBP increase from 5% to 20%. While for the blends with HBP-AD and HBP-PA it's found that the weight loss are in the range of 2.5% to 3.25%.



**Fig. 4.** The biodegradability of VPP and VPP/HBP in soil environment.

While in Figure 5, for rPP/HBP blends it is found that the weight loss for blend with HBP-TA is 9%, 12.2%, and 13.1% as the HBP increase from 5% to 20%, and the blend with HBP-MA increases the weight loss to 4.5%, 5.2%, and 5.8% as the HBP increase from 5% to 20%. While for the blends with HBP-AD and HBP-PA it's found that the weight loss is in the range of 3.9% to 4.25% for blend with HBP-AD and the weight loss are in the range of 4.8% to 5.4% for blend with HBP-PA.



**Fig. 5.** The biodegradability of rPP and rPP/HBP in soil environment.

From the above results, it's found that the blends with HBP-TA has the best results when compared with other blends this is due to the higher content of OH groups in HBP-TA. Also, it is

found that the result of rPP is better than VPP this is because rPP has shorter chains and lower entanglements which permit in better absorption of water and make the degradation easier. The presence of HBP molecule can reduce the polymer crystallinity, the reduction in the crystallinity means the polymer ability to withstand the degradation reduced, and the hydrolysis within amorphous regions are higher than crystalline regions. The presence of esters in HBP can result in faster degradation, these results are in agreement with Bakhshi and Agarwal [23], and Gu et al. [24].

### Conclusion

From the results above, it can be concluded that the FTIR results show the absence of chemical interaction between PP and the four HBPs and only physical interaction with a slight shifting in band values. In addition, it is found that the biodegradation in soil increased as the amount of HBPs increased and that the rate of weight loss in rPP blends was higher than that in VPP blends, and the same results were obtained from the water contact angle. Furthermore, the MFR increased as the HBPs increased while the viscosity decreased, indicating that the HBPs addition improved the processability of PP (VPP and rPP).

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