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Effect of Starch Content on the Physico-Mechanical Properties of Recycled Polypropylene /Sweet potato Thermoplastic Starch Blend

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Abstract

Recycled polypropylene bottles (RPPB)/ thermoplastic sweet potatoes starch (TSPPS) with compatibiliser (polypropylene –grafted- maleic anhydride) was prepared by melt blending on the two roll mill, and effect of the thermoplastic starch loading of 0,10,20,30,40 and 50wt% (sample A-F) on the physical and mechanical properties of the blend has been reported. Starch from sweet potato peel was treated with 0.16% aqueous solution of sodium hydrogen sulphite as a preservative. pH, Ash content, moisture content and FTIR were used to characterise the produced sweet potatoes peel starch. Hardness, tensile strength and percentage elongation at break properties decrease as the thermoplastic starch content increases from 0-50wt. %. The impact strength was found to be improved at sample C (20wt%) as thermoplastic starch content increases when compared to the control sample (sample A) while the modulus of elasticity, density and water absorption results shows that there was an increase in these properties as thermoplastic starch content increases. Biodegradation result of the blend upon soil burial for 98 days shows that the higher the starch content in the blend, the faster the rate of degradation.

Keywords. *Compatibilizer, Physico- mechanical properties, Thermoplastic starch. Polypropylene, Polymer blend*

I. Introduction

Biodegradable plastics development is considered as the best approach to solving solid waste problems, which gave rise to an interest in the development of polymers that will deteriorate or degrade into benign by-products under composting environments or microorganism action [1]. Researchers in recent times have focused on generating new materials by the blending of a synthetic polymer with natural occurring biopolymers, which include starch, chitin and cellulose to produce polymers that are biodegradable [2]

The concept of combining two or more different polymers to obtain a new material system with the desirable features of its constituent is not new. Many systems based on the chemical combination of different monomers have been formulated over the years to produced varieties of polymer blend system in order to meet commercial application with minimum cost has increased rapidly [3]. Starch is found in many plant sources, such as corn, potato, rice, wheat, yam and tapioca are employed in the development of biodegradable polymer and blends in recent times. The polymer blend is a mixture of different polymers to produce a new material with new physical properties, [3, 4].

Gelatinisation of starch in the presence of plasticisers which include water and/or glycerol gives what is referred to as thermoplastic starch [5, 6]. Polypropylene is a petroleum-based polymer which is non-degradable in nature of which about 45millions tons of polypropylene are consumed globally in 2017 [7]. Plastic products waste disposal has become a challenging problem of public concern due to their non-degradability, and they cause environmental menace both to land and marine bodies [8, 9, and 10].

Sweet potatoes (*Ipomea batatas*) is a seasonal crop which grows in the tropical and subtropical region and mainly used as food. Nigeria, which is the third-largest producer of sweet potato in the world at 2015 [11] of about 3.8 million tons and only exported a limited quantity with primary production carried out in Plateau state Nigeria. This food crop has shown to contain 50-80% starch on a dry basis [12]. This crop, when used to prepare food, are mostly peel by slicing, and peels lie as waste and not adequately utilised. This research which presents the use of waste materials and inexpensive source of starch for polymer blend production opted for recycled polypropylene bottles and sweet potato peels to produced blend which is available and an attempt to address the problem of non-degradability of synthetic polymer and in turn address it environmental pollution problem and also reduce the dependence on petroleum-based polymer and will likely reduce the cost of production.

II. Materials and method

Recycled polypropylene bottles of code option 5 were washed, dried and shredded using a shredding machine, and sweet potato (*Ipomoea batatas*) peels were selected and washed both were obtained at a local eatery in Samaru, Zaria, Nigeria. Glycerol (1.26g/cm^3) and polypropylene grafted maleic anhydride (PP-g-MA) manufactured from Sigma-Aldrich with a melting point of 156˚C and density of 0.934 g/cm^3 and sourced from a local dealer in Lagos Nigeria; all chemicals were used as received (Fig.1)

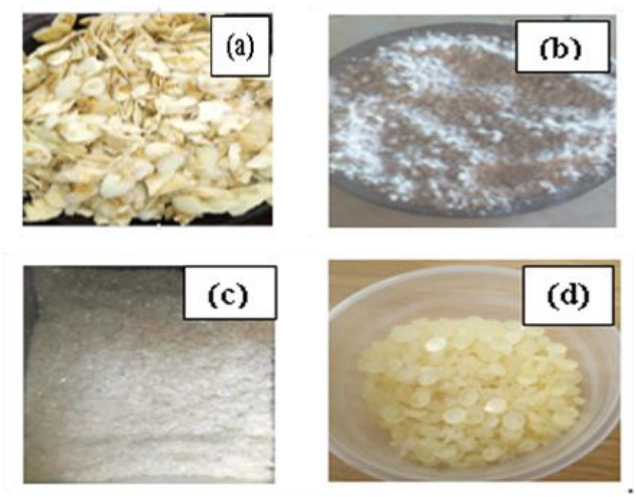

Fig.1 (a) sweet potato peels
(b) sweet potatoes peel starch
(c) shredded PP bottles
(d) PP-g-MA

A. Extraction of the starch from sweet potato peels

The potato peel was washed to remove dirt, then followed by treatment with 0.16% aqueous solution of sodium hydrogen sulphite for 12 hours as a preservative by a method prescribed by Kaur et al. [13]. The potato peel was washed, blended and sieved, the filtrate was allowed to settle down and then decanted. More water was added, and it was allowed to stand for 12 hours, and the decanting process was repeated to obtain the white fine starch powder which was dried in an oven at 45ºC for 5 hours.

B. Characterisation of sweet potato peel starch (SPPS)

a) Proximate analysis of SPPS

1) Moisture content determination

The moisture content of SPPS was determined by measuring weight loss after drying 5g of sample in an oven at 45°C for 35mins. Initial sample weight before drying W_o was taken an after drying W_f was recorded and was recorded until a constant weight is obtained:

$$\% \text{ moisture content} = \left(W_0 - W_f / W_0\right) \times 100\ \% \qquad (1)$$

2) pH determination

The pH meter is a scientific instrument that measures the hydrogen- ion activity in a water-based solution. Five grammes (5g) of SPPS was mixed with 10 ml of distilled water, the pH meter was immersed in the solution, and the reading was taken. These were determined according to ASTM D6657-2014.

c) Ash content

Three grammes (3 g) of the sample of SPPS was heated at the 500°C in a lab furnace as such that the organic content was burnt off by combustion, to yield the mineral and organic compound residues.

b) Fourier transform infrared spectrometer of SPPS

The extracted powder starch of known amount was placed in test cavity of the FTIR analyser which was analysed using Agilent Cary 630 FTIR spectrophotometer The FTIR spectrum of the sample was obtained at the wavelength in the range of 650-4000 cm^{-1}

C. Preparation of thermoplastic potatoes peel starch

Thermoplastic sweet potato peels starch (TSPPS) was prepared base on the method described by Favis et al. [14]. The Sweet potatoes peel starch, water and glycerol were mixed at ratio 50:25:25 respectively to form a slurry in a beaker. Then the slurry (Mix) was poured into a blender and mixed at 80°C at 50rpm. Thermoplastic gel form was cast in a 180mm x 150mm x3mm glass mould and place in an oven at 95°C for 12h. The cast thermoplastic gel was allowed to dry in the open air for 48h at 25°C.

Table 1: Formulation table for polymer blend (PP/TSPPS/PP-g-MA) blend

Ingredients	Samples (wt. %)					
	a	b	c	d	e	f
Used Polypropylene bottles	100	90	80	70	60	50
Thermoplastic sweet potatoes peel Starch	0	10	20	30	40	50
PP-g- MA	0	3	3	3	3	3

D. Production of polymer blend (PP/SPPS/PP-g-MA)

Firstly, Recycled shredded polypropylene bottles were added in the mixing chamber of a two-roll mill at 160°C for 4mins until completely melted. Then thermoplastic potatoes peel starch and compatibiliser (PP-g-MA) was then added of the varying amount according to the formulation table presented in Table 1, mixing was continued for another 4mins until a homogenous dispersion is obtained. A strip of foil paper fitted into a mould and processing oil was rubbed on its surface before the polymer blend was placed on it, to avoid it sticking to the mould. a The sheeted samples were cured in a mould of 100 mm X 100 mm X 3 mm dimensions using a hydraulic hot press at a temperature of 160ºC and pressure of 3 bars for 7 mins., it was cooled at room temperature of 25°C (Fig.2).

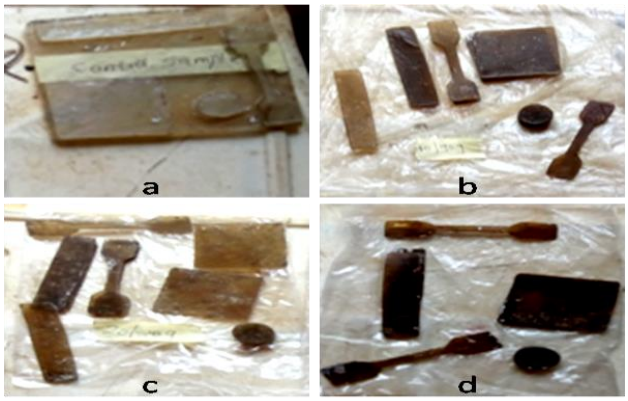

Fig.2 PP/TSPPS/PP-g-MA blend (a) 0wt% (b) 10wt% (c) 20wt % (d) 30wt%

Fig.2 (e) 40wt% (f) 50wt % of thermoplastic sweet potato peels starch

III. Physico- mechanical properties of the recycled PP/TSPPS/PP-g-MA polymer blend

A. Physical properties of the blend

a) Density

Mass and volume relationship Eqn.2 was used in determining the density of the blend. Sample of 3mm x 5mm x 5mm dimension was weighed using an electric weighing balance to obtain its mass (m) and vernier calliper was used to measure the volume (v):

Density (ρ) = M/V (2)

b) Hardness

Hardness test was carried out using a durometer tester model no—5019 on Shore A scale according to ASTM- D2240. The sample was placed at durometer sample carrier; the indenter was made to press and penetrate through the sample. The scale on the durometer indicates the hardness values at various points. This test was carried out in triplicate

c) Water absorption

Water absorption test was carried out according to ASTM D570-2010. Sample of 3mm x 10mm x 5mm dimension were cut and immersed in water at a 25°C for 48 h. The water absorbed was determine by reweighing the samples with the aid of a digital weighing balance.

$$\%\text{Water absorption} = \frac{W_1 - W_0}{W_0} \times \frac{100}{1}, \quad (3)$$

Where W_1 is the final weight (g), and W_o is the initial weight after and before immersion in water (g) respectively

B. Mechanical properties of the blend

The following evaluation was carried on the produced polymer blend using a standard procedure

a) Tensile properties

Tensile properties (tensile strength, elongation at break and modulus of elasticity) were measured according to ASTM D638-10 standard using Hounsfield Monsanto Tensometer model no 9875W at a cross speed of 10 mm/min and a gauge length of 33mm on specimen dimension 100mm x 20mm x 3mm. Tensile strength was determined using the formula below

$$Tensile\ strength = \frac{\text{force}}{\text{cross}-sectional area} = \text{F/A} \quad (4)$$

$$\%\ \text{Elongation at break} = (\Delta L/L) \times 100, \quad (5)$$

Where ΔL and L change in length and original length, respectively

$$\text{Modulus of elasticity} = {}^{\text{F/A}}/_{\Delta L/L} \quad (6)$$

b) Impact strength

Impact test was carried out on notched specimens to determine impact strength using a pendulum hammer of 4J, according to ASTMD256. Izod impact testing machine (model no. 6957) on a sample dimension of 55mm x 10mm x 3mm. Each sample was placed on the vice and clamped firmly.

C. Biodegradability studies

a) Soil test

The soil was obtained from NILEST, Nigeria hostel refuge dump was analysed for the microbial present, 1 g of soil sample was added to 10ml to sterile water, and then mixed in a test tube. 0.1ml of the diluted sample was aspirated into a sterile Petri plate. The prepared media was gently poured into the plate and swirl to mix the sample; the inoculated plate was incubated at 37^0 C for 24 hours and observed for growth on the sample.

b) Soil burial degradation test

This test was carried out according to ASTM D5988, which has similarity to actual conditions of waste disposal. Biodegradability of PP/TSPPS/PP-g-MA blend was studied by weight remaining overtime in a soil environment. Samples were weighed and then buried in the soil at a depth of 10cm from the surface for 98 days(14weeks). The soil moisture was maintained by injecting water to keep the microorganisms active. The buried sample was weighed after every 7 days, and the weight loss was calculated.

IV. Results and discussion

A. Result for characterisation of SPPS

The result of sweet potato peels starch is presented in Table 1

Table 1: Proximate analysis result for SPPS

S/N	Parameter	Result	Specification
1	Moisture (%)	7.1	<20
2	Ash (%)	0.56	<1%
3	pH	6.4	4-7

From Table 1, the pH, moisture content and ash content met the required specification. Rowe et al. [15] reported that a starch suspension in water showed to have a pH of 4 - 7. The moisture content of potatoes peels starch powder was found to be 7.1%, which determine its quality, shelf life and storability [16]. Pelissari et al. [17] reported similar result for ash content for banana and plantain flour and also noted that starch powder with high moisture content is more prone to bacterial attack than low moisture content ones.

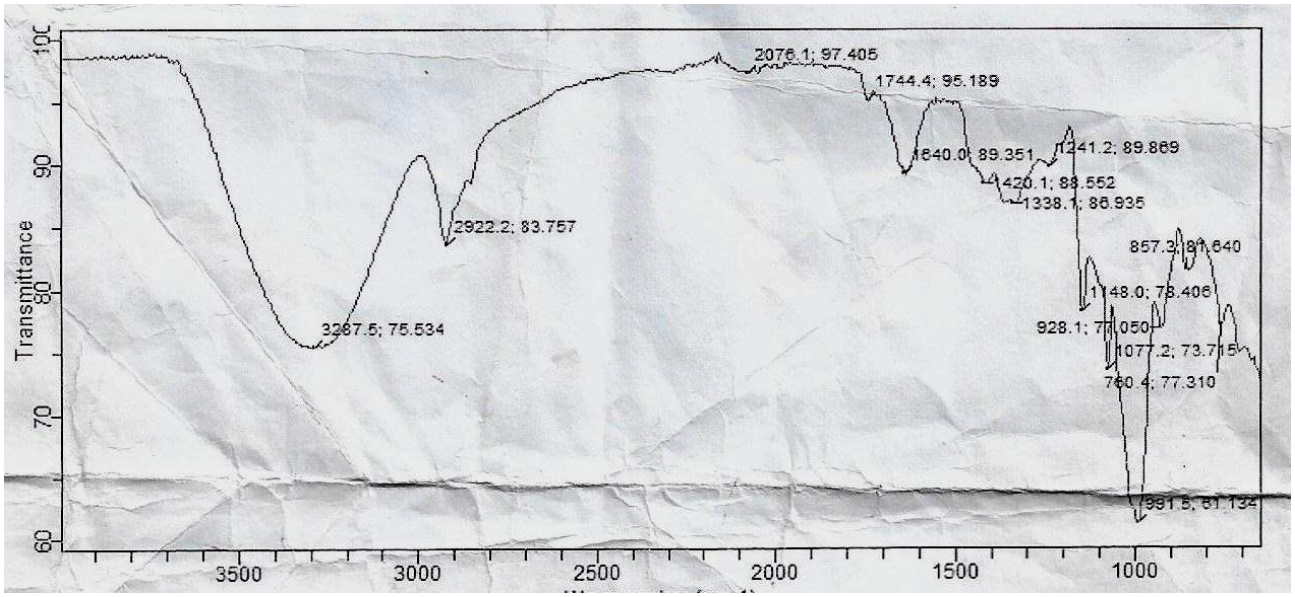

Fig.3: FTIR profile for extracted Sweet potatoes peel starch

FT-IR spectroscopy of SPPS

Fourier transform infrared spectra of SPPS in Fig.3 shows fundamental group responsible for each wavelength of absorption. The broad peak observed at 3287cm^{-1} is OH stretching typically of the hydroxyl group in starch the peak at 2922 cm^{-1} is C-H stretching, the peak at 1640 cm^{-1} is due to the adsorbed water - stretching typically of alkynes and the peak at 1338, is CH_2 twist and C-0-H bending,1077 cm^{-1} -928 cm^{-1} is C-0-C stretching and C-O-H bend, 857-760 cm^{-1} CH and CH_2 deformation which is similar to result reported by ([18,19 and 20]

B. Results for physical test

The density of the blend increases with increasing thermoplastic starch content from 0 to 50 wt. %. This is clearly as a result of the inclusion of denser materials; a similar trend was reported by Kormin et al. [21] and Srabayeeta et al. [22].Besides, poor compatibility or lower amount of compatibiliser used, poor bonding and dispersion between starch, pp and pp-g-MA could be a reason for this trend Fig.4.

Hardness properties presented in Fig.5 of the produced blends decreases as thermoplastic starch content increases sample with 10wt % of starch content has a relative value with the unfilled sample of 93 and 93.5 shores A respectively. The decrease in the hardness property of the blends with increase thermoplastic starch loading could be linked to poor interface adhesion, decrease the crosslink density of the thermoplastic starch on the recycled propylene chain. A similar trend was reported by Srabayeeta et al. [22]

Water absorption result presented in Fig.6 shows that there was an increase in water as thermoplastic starch content increases. This could be due to the presence of the O-H group in the amorphous region of the starch and the O-H group increases with increase in thermoplastic starch content in the produced blend. However, compatibilisation of this material significantly decrease the water uptake when compared to other studies of uncompatibilized starch Obasi et al. [23]. A similar trend was reported by Kormin et al. [21] in their work on LDPE incorporated with different starch sources

C. Results for mechanical properties

The results of the mechanical properties of the developed blend at starch loadings of 0-50wt% are shown in Figs. 7, 8, 9 and 10.

The effect of thermoplastic starch content (TPS) on the tensile strength is shown in Fig 7 and was found to decrease as thermoplastic starch content increases, with 10wt% loading having a comparable value when compared to the unfilled sample of 22.42MPa and 24.07MPa respectively, further increase in TPS content continuously decrease the tensile strength of the blend which may be caused by the polar group in starch hindering healthy bond formation between the thermoplastic starch and recycled PP. Also, maybe insufficient/ or non-optimum amount of compatibiliser was used (PP-g-MA). Agglomeration of starch within the polymer (PP) matrix may also lead to this behaviour and in turn, lead to low stress transfer. Yew et al. [24] and Obasi et al. [23] reported a similar trend.

The effect of TSPPS on percentage elongation at break of the blend is presented in Fig.8. It was found to decrease as the starch content increases from 10-50wt%this could be attributed to the polar nature of starch, agglomeration, cracks due to poor adhesion of starch on the polymer matrix, thereby soften or reduce the physical bonding of starch and matrix and reducing the elongation property of the blend as it has been stretched. Balakrishna et al. [25], Obasi et al. [23] and Kormin et al. [21] reported a similar trend.

From the result presented in Fig.9, it could be seen that modulus of elasticity of the blend produced increases as the starch content increases from 91MPa to 210MPa as the thermoplastic starch loading increases from 0 - 50 wt. %, this is a result of the starch hindering the mobility of the chain of the recycle polypropylene matrix thus contributing to the rigidity and stiffening

effect of the blends of which the PP-g-MA also help to improve the bond characteristics [23].

The effect of TSPPS on the Impact strength was presented in Fig.10 The result shows that a decrease in impact strength value from 0.152J/mm to 0.105J/mm as the thermoplastic starch content increase from 0 to 50wt%. The composite sample containing 20% thermoplastic starch shows the highest impact strength of 0.135J/mm when compared to other starch loadings. The decreased in these properties may be a due agglomeration of the starch on the matrix. Poor interfacial bonding and this may tend to produce cracks upon impact. A similar trend was reported by Kormin et al. [21].

D. Result for degradation test

Soil analysis test shows the bacterial acting on the soil *Bacillus subtilis* is a kind of bacterial also known as the hay bacillus or grass bacillus in a gram-positive, vogues test detect the activeness of the bacterial, methyl red test detect the ability of the organism to produce and maintain acids and product from glucose fermentation, catalyse test shows the oxygen that protects the microorganism, oxidase test shows the bacterial produced at specific cytochrome, lactose test shows weather can utilise particular carbohydrate in the soil.

The result presented in Fig. 11 shows the level of degradation for all sample buried over time in terms of the percentage weight remaining after degradation. The results obtained show that the degradation increase with increase thermoplastic starch contents from 10 to 50wt% throughout 98days, that is the lower the rate of degradation of the blend the higher per cent weight remaining. The blend containing 40 and 50wt% of starch degraded faster in the first 7 weeks (34days), and then there was a gradual decrease in the next 7 weeks. The weight remaining gradually decreases as the burial time increases, and after 98 days of study, the per cent weight remaining for 40 and 50wt% thermoplastic starch content are 72.84% and 71.07% respectively.

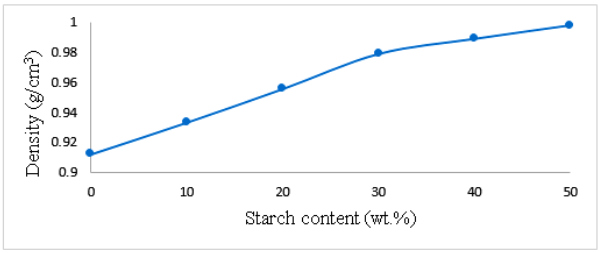

Fig. 4 Effect of starch content on the density of PP/TSPPS/PP-g-MA blend

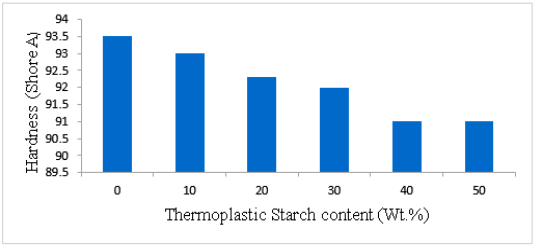

Fig. 5 Effect of starch content on the hardness property of PP/TSPPS/PP-g-MA blend

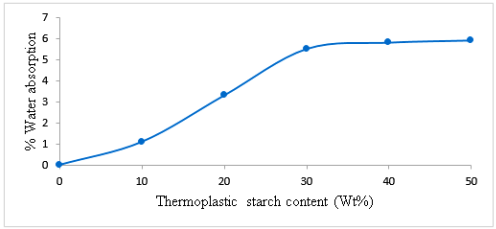

Fig. 6 Effect of starch content on the percent water absorption of PP/TSPPS/PP-g-MA blend

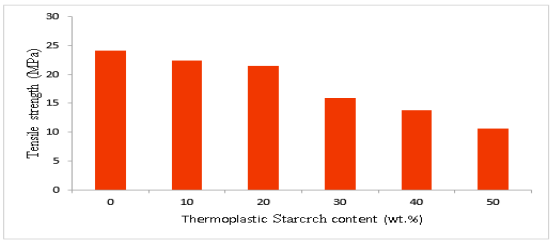

Fig. 7 Effect of starch content on the tensile strength of PP/TSPPS/PP-g-MA blend

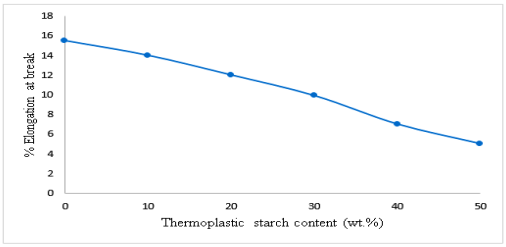

Fig. 8 Effect of starch content on the % elongation at break of PP/TSPPS/PP-g-MA blend

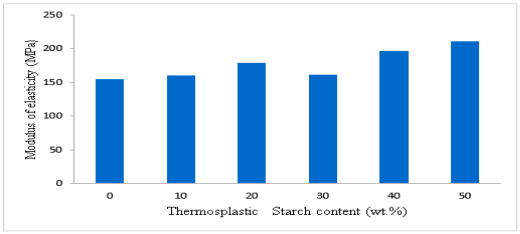

Fig. 9 Effect of starch content on the modulus of elasticity of PP/TSPPS/PP-g-MA blend

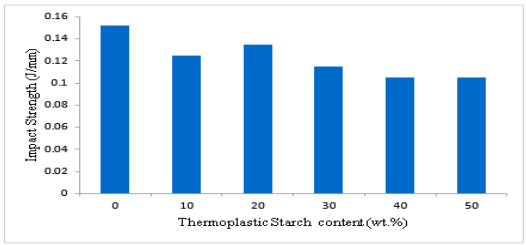

Fig.10 Effect of starch content on the impact strength of PP/TSPPS/PP-g-MA blend

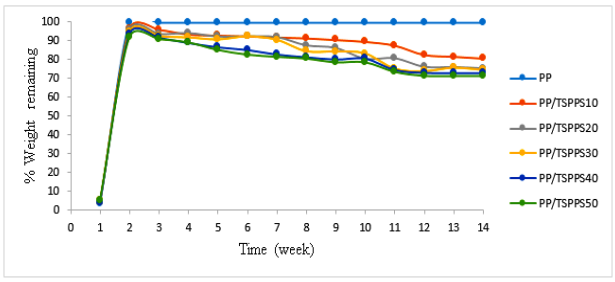

Fig.11 Percent weight remaining for the PP/TSPPS/PP-g-MA blend

V. Conclusion

In this study, the effect of thermoplastic starch content on the polypropylene/ sweet potatoes thermoplastic starch compatibilised with PP-g-MA has been successfully prepared using waste potato peel and recycled pp bottles from the environment in order to address environmental pollution caused by this polymeric material. The results obtained has confirmed that physicomechanical properties of the PP/TSPP/PP-g-MA were significantly affected by the amount of starch content in the produced blend. Within the limit of the variation presented in this study, optimum properties of the blend were obtained at sample b(10wt%) of starch content mostly, for tensile strength, per cent elongation at break, impact strength, hardness, water absorption and density (22.4MPa, 14%,0.125J/mm, 93 shore A,1.102%, 0.9334g/cm^3)compared to sample f (5owt%) of 10.54MPa,5.0%, 0.105J/mm, 91shore A, 5.9%, 0.998 g/cm^3, with sample with 50wt% starch having the highest modulus of elasticity of 210Mpa. The biodegradation result shows that the rate biodegradation of the blends was faster as starch content increases from 10-50wt% due to increased microbial activities.

VI. Acknowledgements

The authors are grateful to the Nigerian Institute of Leather and Science Technology, and mechanical department of Ahmadu Bello University, Zaria, Nigeria whose facilities were used in this study.

VII. Conflict of interest

The authors declare no conflict of interest.

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