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Effects of LLDPE on Mechanical Properties, Degradation Performance, and Water Absorption of Thermoplastic Sago Starch Blends

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Abstract. The purpose of this study was to improve the mechanical and physical properties, degradation performance, and water absorption of a thermoplastic mixture of sago starch with the addition of Linear Low-Density Polyethylene (LLDPE). The method used is the grafting method where polyethylene is grafted onto Maleic Anhydride (LLDPE-g-MA). In this research, Thermoplastics Sago Starch (TPSS) was made with a mixture of Sago Starch (65%) and Glycerol (35%), added with water as a solution. Compatibilizer (PE-g-MA) was made by reacting LLDPE (88%) and Maleate Anhydride (9%) with an additional initiator of Benzoyl Peroxide (3%). The concentration of LLDPE varied from 10,15, 20, 25, and 30%. Substances were mixed into an internal mixer Thermo Haake Polydrive with a speed of 100 mm/minute, for 15 minutes. The concentration of 30% LLDPE produced the highest tensile strength and elongation at a break of 4.30 N/mm2 and 2.15%, respectively. Simultaneously, as LLDPE concentration increased, Young's Modulus decreased. This was powered by the morphology of the sampling surface, with the occurred reaction of adhesion interface or dispersion of LLDPE toward the whole surface with the assistance of compatibilizer as connecting substance between hydrophilic polymer and a hydrophobic polymer, which could improve the properties of mechanical. The formation of ester groups from the reaction of inter groups of hydroxyls derived from the starch with the groups of anhydride derived from the mixture of compatibilizer was visibly seen at the peak of 1693 cm^{-1}. The degradation performance of the TPSS: LLDPE mixture with a ratio of 70:30 showed good performance where the degradation continued to increase up to 42% on the 30th day of degradation in freshwater, 18% in seawater, and 17% in soil burial. While the water absorption test showed that the higher concentration of LLDPE, the lower speed of water absorption.

Keywords: Bioplastics; Compatibilizer; Coupling agent; Grafting; Starch

1. Introduction

In 2018, the production of global plastic was nearly 360 million tons (Barnaba *et al.*, 2020). Meanwhile, the production of bioplastic reached just 2.01 million tons or 0.56

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percent of global plastic production (European Bioplastics, 2017). The increasing market of bioplastics could reduce dependency on fossil-based source and transform to be a biobased society (Imre *et al.,* 2019; Geueke *et al.,* 2018). Nowadays, approximately 80% of the world's plastic production is not recycled (Blank *et al.,* 2020) and the usage of plastics polluting is on the rise (Sidek *et al.,* 2019). Bioplastic, which by definition is biodegradable and/or gained from a renewable source, is a sustainable alternative to conventional plastic and its production capacity is estimated to increase to2.43 million tonnes in 2024(European Bioplastics, 2018).

Bioplastics are made from natural polymeric materials such as starch, cellulose, and fats (Amni *et al.,* 2020; Hasan *et al.,* 2019; Silviana & Subagio, 2019; Judawisastra *et al.,* 2018; Suharty *et al.,* 2014). However, bioplastic cannot completely replace the function of conventional plastic yet. Starch-based bioplastic possesses some weaknesses, e.g., low mechanical properties, high water sensitivity (Kaewtatip & Tanrattanakul, 2012; Gáspáret *et al.,* 2005), and rigidness. It is due to the characteristic of Starch that tends to coagulate and create a stressed area that is concentrated, resulting in worse mechanical characteristics (Vinhas *et al.,* 2007; Pedroso & Rosa, 2005). This is because starch has a tendency to coagulate, creating a concentrated area of stress that degrades the material's mechanical characteristics, and it shows fast absorption at high concentrations of starch (Abdorreza *et al.,* 2011). Mixing thermoplastic starch with synthetic polymers such as polyethylene can increase its mechanical characteristics (Zaman & Beg, 2021; Majid *et al.,*2009; Wang & Sun, 2004).

However, due to the difference in polarity where natural polymers are hydrophilic/polar and synthetic polymers are hydrophobic/non-polar so that the two materials are incompatible, the Compatibilizer is needed to unite them through the grafting method. The addition of the compatibilizer is expected to increase the homogeneity of the mixed solution and improve the adhesion between the phase (Sari *et al.,* 2020; Imre & Pukánszky, 2013).

This research examined how different environments (freshwater, seawater, and soil) affected the tensile characteristics, morphology, water absorption, and biodegradation performance of LLDPE. The distribution morphology of LLDPE on the surface was also in evaluation.

2. Methods

2.1. Material

Sago starch Parang brand is a production of Warna Jaya Indonesia. Obtained from the traditional market of Serpong-Tangerang, Indonesia. Moisture content was 14%. Low Linear Density of Polyethylene (LLDPE) UF 1810S1 Pellet with density: 0.922 g/m^3, Melt Index (190^{0}C/2.16 kg): 1.0g/10 min, melting point 122^{0}C, obtained from PT. Chandra Asri Petrochemical Tbk (TPIA) Cilegon-Indonesia. Maleic anhydride for sinthesis Merck KGaA, 64271 Darmstadt Germany, Benzoyl peroxide Merck for sinthesis Merck KGaA, 64271 Darmstadt Germany and Glycerol Analytical Reagent merck Univar Produksi Ajax Finechem. These can beobtained from the Rudang shop Medan-Indonesia.

2.2. Preparation of TPSS

In order to prepare TPSS, sago starch and glycerol were reacted at a ratio of 65:35, and water was added at a concentration of up to 250% of the amount of sago starch. The mixture is heated to 100°C until it turns into gelatin. To reduce the water content in TPSS gelatin to 5%, it is oven-dried for 24 hours at 80°C (Majid et al,, 2009). Figure 1 depicts a flowchart of the process of TPSS preparation.

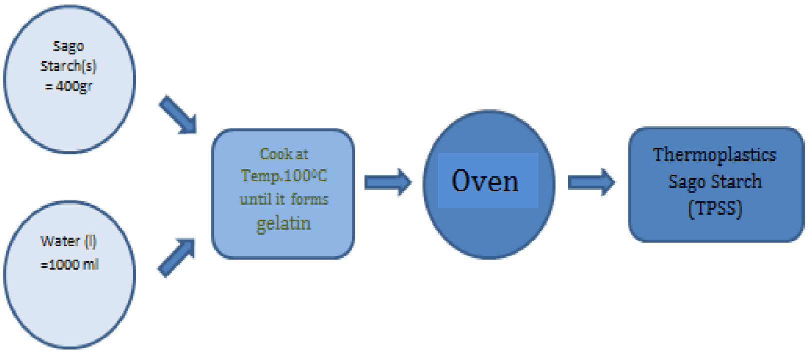

Figure 1 Flow diagram of the process of preparation of TPSS

2.3. Preparation of Compatibilizer

The blends were mixed with Haake Polydrive Thermo. LLDPE was first added into the mixing chamber, followed by Maleate Anhydride after 5 minutes of a mixture, then Benzoyl Peroxide (BPO) was added at last. The mixture was completed at 150^0C and a speed of 100 rpm, with the mixture's total time, being 15 minutes. The composition comparison of LLDPE: Maleate Anhydrous: Benzoyl Peroxide, the ratio was 88:9:3. The compound was removed, cooled, and cut into pellets. *Figure 2 illustrates a Flow Diagram of the Compatibilizer (LLDPE-g-MA) Preparation Process*.

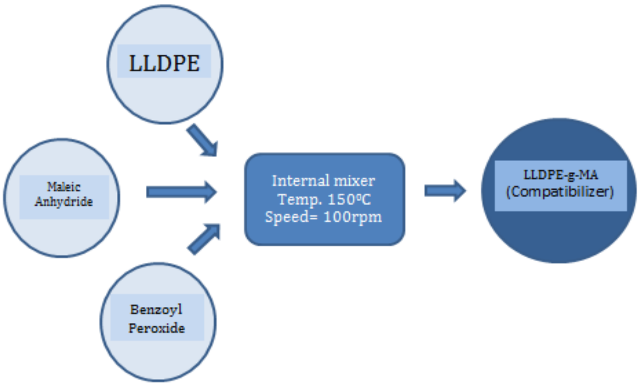

Figure 2 Flow diagram of the process of preparation of Compatibilizer (LLDPE-g-MA)

2.4. Preparation of TPSS/LLDPE/PE-g-MA blends

The mixture preparation of TPSS / LLDPE/PE-g-MA was the final phase in this research, in which the whole phases that have been prepared above would be mixed here, such as TPSS, LLDPE, and compatibilizer. The compound was produced by mixing TPSS and LLDPE with the comparison of 90:10, 85:15, 80:20, 75:25, and 70:30. PE-g-MA was used with an amount of 10 wt. % based on the weight of TPSS (Majid *et al.,* 2009). Figure 3 shows the process flow diagram of the TPSS/LLDPE/LLDPE-g-MA (compatibilizer) preparation.

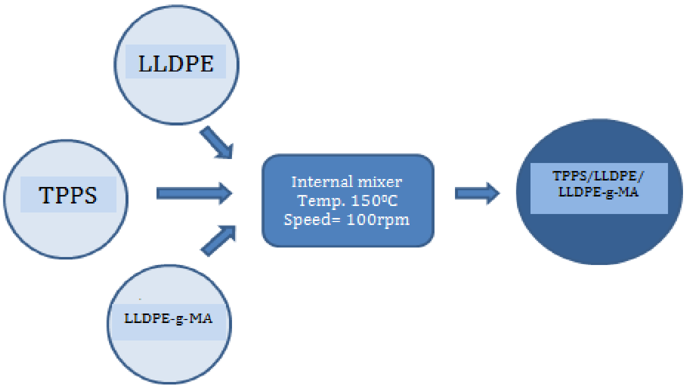

Figure 3 Flow diagram of the process of preparation of TPSS/LLDPE/LLDPE-g-MA (Compatibilizer)

2.5. Compression Molding

The blend of TPSS / LLDPE was printed with hydraulic pressure compression at a temperature of 150^{0}C for 15 minutes. The pressure was increased to its maximum every 5 minutes. All compression molded sheets by ASTM D638 Type 1.

2.6. Tensile Properties

A tensile test was carried out by examining the machine of Universal Band Tensilon with the specimen of ASTM D638 Type 1. The temperature in the chamber was 25^{0}C, humidity on 60% RHdn, 10mm speed/minute. 3 Specimens were used to obtain the average value of Tensile Strength, Elongation at Break, and Modulus Young.

2.7. Spectroscopy Fourier Transform Infra-Red (FTIR)

Fourier transform infrared spectroscopy (FTIR; Perkin Elmer System 2000) was utilized to obtain qualitative information on the groups and chemical characteristics of TPSS / LLDPE blends. For each spectrum, 45 scans with a resolution of 4 cm-1 were recorded consecutively with Wd: 9:5 mm. The sample was measured in the form of a 1-millimeter-thick layer produced through hot-press molding.

2.8. Test of Morphology

The scanning test of Electron Microscopy (SEM) was carried out to evaluate the morphology surface of the blended sampling of TPSS / LLDPE. The sampling was dried up in the oven to reduce water content and avoid electrostatic filling as well as worse resolution. By the time of sampling surface inspection, it was installed on the piece of aluminum stubs and sputter-coated with a thin layer of gold.

2.9. Test of Water Absorption

The sampling sheet was cut into a dimension of 20mm x 20mm (Obasi *et al.*, 2015) with a thickness of 3mm. It was then washed with aquadest and dried up to 80% for 12 hours, cooled down in a desiccator, and weighed to gain initial weight. It was then immersed into aquadest for ten days with a chamber temperature of 28-30^{0}C and reweighed to gain weight afterward.

2.10. Test of Biodegradability

The compatible blend of TPSS / LLDPE with a dimension of 20mm x 20mm with a thickness of 3 mm was examined with different condition control (Spaccini *et al.*, 2016; Maran *et al.*, 2014). The first sampling was stored in a container containing just fresh water. The second one was immersed in seawater. The test for weight loss was done every 10, 20, and 30 days.

2.11. Test of Soil burial

The sampling was buried at a 10mm depth from the surface of alluvial soil (Henry & Isaac, 2014) which was placed in the holed box. Regularly wet it to keep its humidity. The weight loss of the samples was monitored at regular intervals of 10, 20, and 30th days. The test was carried out by washing the sample with aquadest, drying it up at chamber temperature until its weight remained constant, and then weighing it.

2.12. Analysis of Thermogravimetric (TGA)

The implemented analysis of Thermogravimetric was STA module with channel TGDTA 7300. The measurement was done at 7.73 mg with a heating rate of 10^0C/minute. The heating was begun at 30^0C to 650^0C. The rate of nitrogen gas was 50 mL/min.

3. Result and Discussion

3.1. Mechanical Properties

The Tensile Strength (TS), Elongation at Break (EB), and Young's Modulus (YM) from the blend of TPSS/LLDPE Compatibilized, as shown in Figure 1, showing the affecting result of additional LLDPE, at the combination of PE –g-MA as a compatibilizer on the tensile strength and elongation at break, experienced continuous increase with the increased concentration of LLDPE from 3.20 N/mm^2 become 4.30 N/mm^2 and 0.72% to 2.15% at the maximum concentration at LLDPE 30% concentration (Matzinos *et al.*, 2001)as can be seen in Table 1. When the young modulus decreased from 447N/mm2 to 201N/mm2 due to the higher concentration of LLDPE, the young modulus decreased as well. Figure 4-6 illustrates the Tensile Strength (TS), Young's Modulus (YM), and elongation break charts of TPSS/LLDPE compatibilizer blends.

Table 1 Mechanical Properties Test of TPSS/LLDPE-Compatibilizer

LLDPE Content (%)	Tensile Strength (N/mm^2)	Elongation at Break (%)	Young Modulus (N/mm^2)	Reference
0	1.80	0.38	479	(Majid *et al.*, 2009)
10	3.20	0.72	448	
15	3.39	1.25	271	
20	3.60	1.43	252	
25	3.82	1.60	239	
30	4.31	2.50	205	

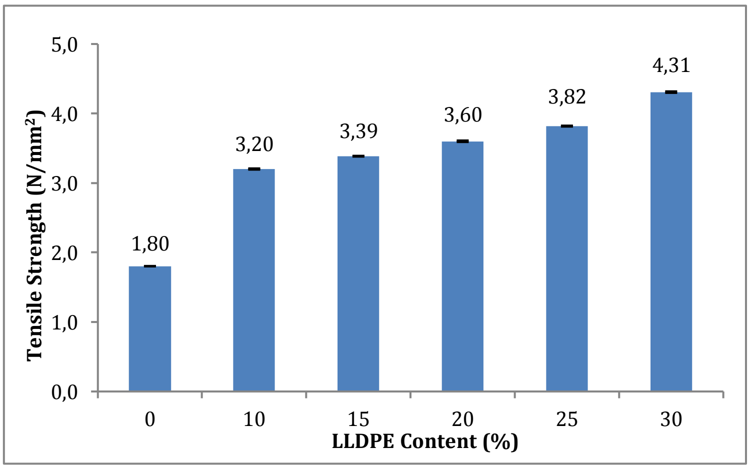

Figure 4 Tensile strength (TS) and Young's Modulus (YM) test of TPSS/LLDPE compatibilizer blends

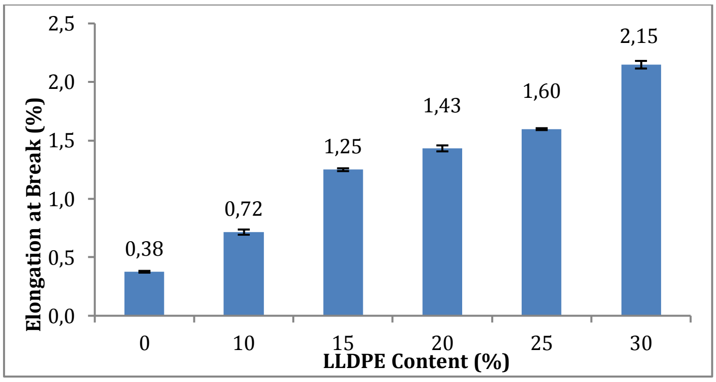

Figure 5 Elongation at Break test of TPSS/LLDPE compatibilizer blends

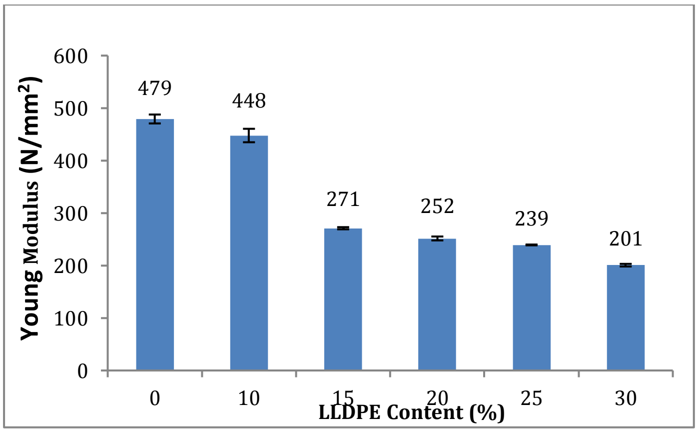

Figure 6 Young Modulus test of TPSS/LLDPE compatibilizer blends

3.2. FTIR

In the TPSS spectrum, there was a wide peak of characteristics at 939-1058 cm^{-1} as the strain peak of COO, which together with hydroxyl at 3000-3675 cm^{-1}. While the spectrum of copolymer that was used as a compatibilizer shows the peak of anhydride groups at 1707 cm^{-1}. The blended peak of TPSS/LLDPE compatibilizer emerged at 1084 cm^{-1} and 1148 cm^{-1} identified as Ester groups which were formed from the reactions of hydroxyl groups at 3265 cm-1 in the TPSS spectrum with the anhydride groups in the spectrum of PE-g-MA at 1693 cm^{-1}. Likewise, previous studies reported the formation of an ester group. (Majid *et al.*, 2009; Bikiaris & Panayiotou, 1998). The FTIR spectra of TPSS, PE-g-MA, and TPSS/LLDPE Compatibilized blends are shown in Figure 7.

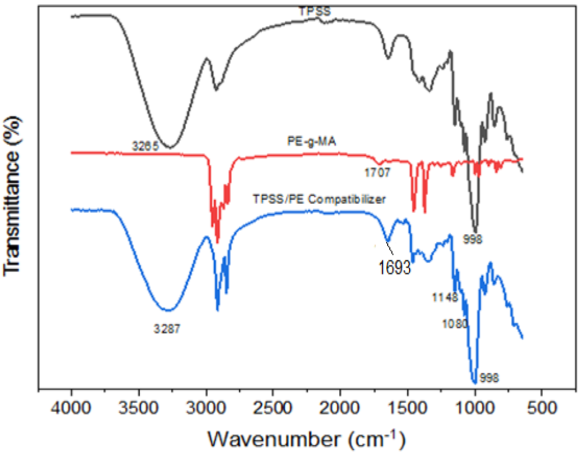

Figure 7 The Spectra of TPSS, PE-g-MA, and TPSS/LLDPE Compatibilized blends

3.3. Test of Morphology

Morphology affects the mechanical properties of a product and polymer dispersion evenly, indicating that interface and adhesion have increased. On the other hand, the occurrence of agglomeration in the polymer, shows that there was a worse interface reaction, in which agent dispersion, formed the copolymer with its kind. Differences in polarity were one of the causative factors. As in this TPSS/LLDPE blend, TPSS was the main component of the major blend on the polarized matrix. While LDDPE was the minor component of the dispersed phase, which was expected to be the non-polarizing reinforcing agent. Figure 8 shows how the difference in polarity led to an uneven distribution, which shows that the LLDPE is grouped or aggregated. (Rodriguez-Gonzalez *et al.,* 2003; Yoo *et al.,* 2001). To unite the natural polymer of hydrophilic with the synthesis polymer, which is hydrophobic, the coupling agent needs an ordinary connector, known as a compatibilizer. In the case of LLDPE, this minor polymer was evenly distributed throughout the blend, providing evidence of interface activity in a heterogeneous polymer blend. This is supported by the fact that bioplastic's mechanical properties increase when compatibilizers are added. Figure 9 shows an SEM image of a compatible blend of TPSS/LLDPE at a 20% LLDPE concentration, scaled to 1:150.

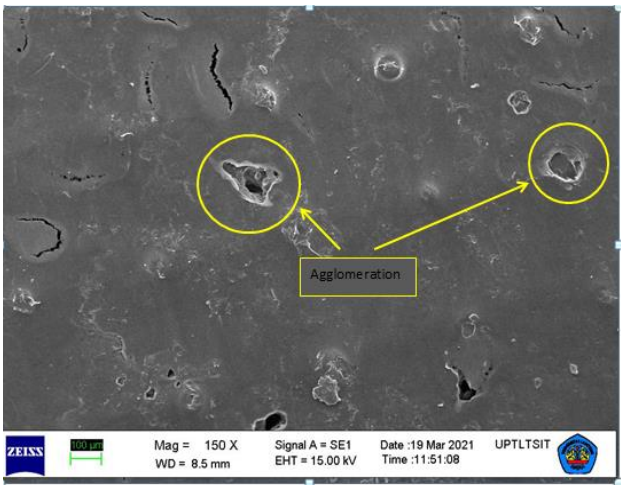

Figure 8 SEM Image of TPSS/LLDPE Uncompatibilized blends, (20% concentration of LLDPE) with scale 1: 150

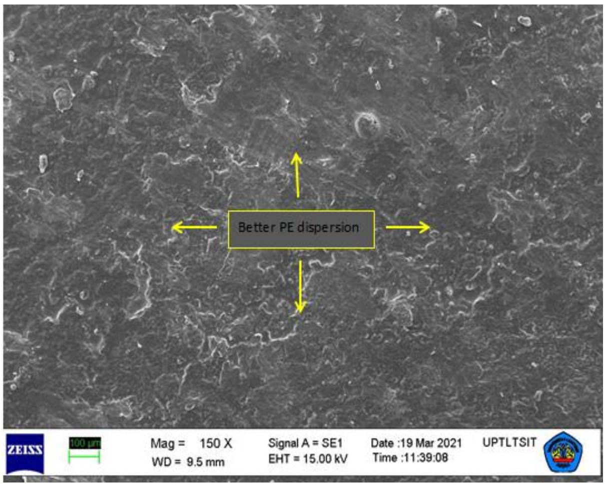

Figure 9 SEM Image of TPSS/LLDPE Compatibilized blends, (20% concentration of LLDPE) with scale 1: 150

3.4. Water Absorption Test

The balance of TPSS water absorption was increased to 120% as can be seen in Figure 10. This resulted from the hydrophilic nature of the starch due to the many hydroxyl groups available to react with the water (Vinhas *et al.,* 2007). The compatibilizer blend of TPSS and LLDPE demonstrates that the balance of water absorption decreased as LLDPE concentration increased. This was probably due to the decrease in the number of hydroxyl groups from starch as the concentration of TPSS decreased, which could have reacted with a water molecule (Obasi *et al.,* 2015; Majid *et al.,* 2009; Gáspár *et al.,* 2005).

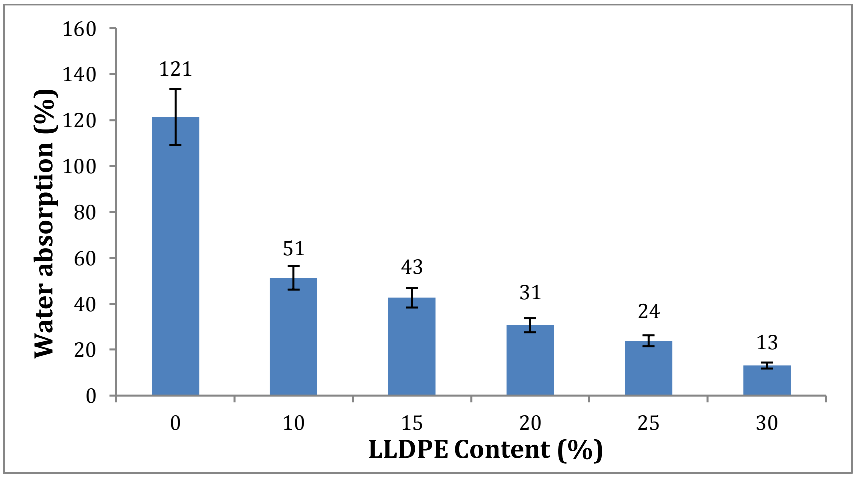

Figure 10 Equilibrium of water uptake test for TPSS/LLDPE Compatibilized blends

3.5. Biodegradability Test

In this degradation test, the three conditions were shown in figure 11-13, namely, freshwater immersion, seawater immersion, and being buried in the ground. In general, it could be said that the sample size weight of compatible blend-based bioplastics made from TPSS and LLDPE was going down as the quantity of concentrated LLDPE and time went up. For example, the sampling weight of freshwater decreased from 46 to 42% at concentrated LLDPE 10 to 30% of concentrated LLDPE until the 30th Day. Meanwhile, the decrease of seawater sampling weight from 56 to 18% at a concentrated LLDPE of 10- 30%, as well as the decreased percentage of soil buried sampling achieved 50% at a concentrated LLDPE of 30%, with the decomposition of 17% at the 30th Days. The more concentration of LLDPE contained in bioplastic, the smaller percentage of degradation rate (Abdullah *et al.,* 2013). Table 2 contains specific data.

Table 2 Performance of degradation in 3 conditions

Condition	Time (day)	LLDPE Concentrations						Reference
		0%	10%	15%	20%	25%	30%	
Fresh Water	10	17	46	43	40	38	36	(Ashok *et al.,* 2018)
	20	33	58	53	49	44	40	
	30	56	64	57	53	48	42	
Sea Water	10	15	47	43	38	11	9	
	20	31	52	48	45	17	14	
	30	52	56	52	49	21	18	
Soil Burial	10	14	34	32	23	15	13	
	20	29	47	43	37	19	15	
	30	49	50	47	46	23	17	

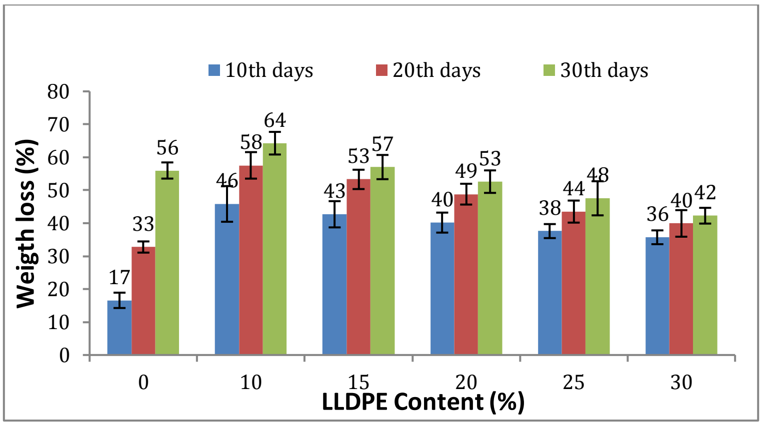

Figure 11 Degradation test of TPSS/LLDPE Compatibilized blends in freshwater

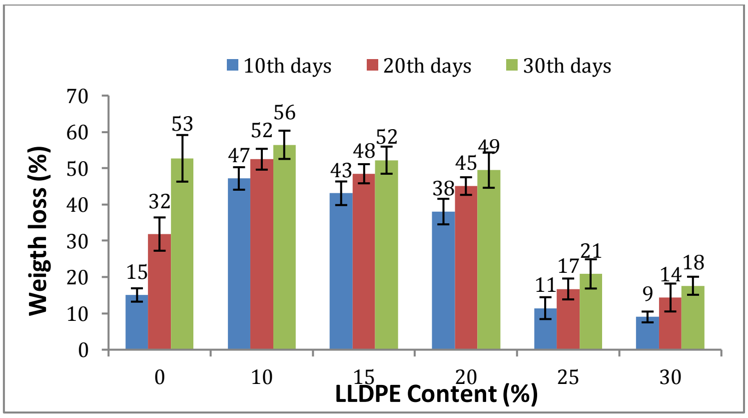

Figure 12 Degradation test of TPSS/LLDPE Compatibilized blends in Seawater

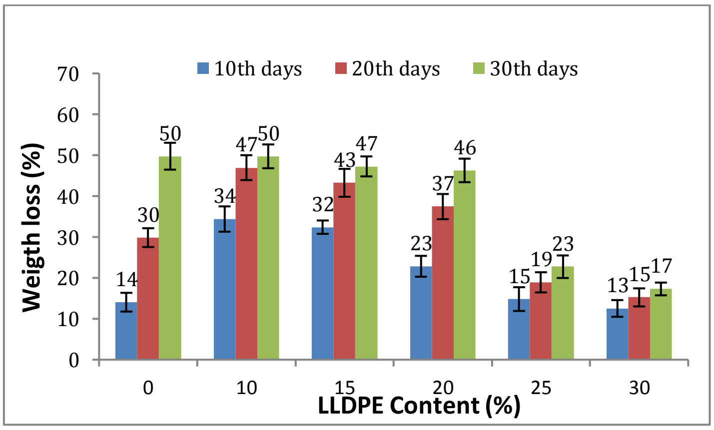

Figure 13 Degradation test of TPSS/LLDPE Compatibilized blends in Soil Burial

3.6. Analysis of Thermogravimetric (TGA)

The Thermogravimetric Analysis technique was used to determine the thermal decomposition and stability of the mixed TPSS/LLDPE compatibilizer film. Thermal decomposition occurs in four stages. The initial stage of degradation occurs at a temperature of 201.1^{0}C with a mass loss of 17.1%. At this stage, there is evaporation or dehydration of water into H_2 and O_2 gases at a temperature of 100^{0}C with a mass loss of 6.36%. According to the findings (González Seligra *et al.*, 2016), plasticizer compounds (glycerol) evaporate between the first and second stages at temperatures between 125 and 290°C. This thermal decomposition will continue along with the increase in temperature into compounds to other compounds. The initial thermal decomposition of plasticized starch occurs at around 300⁰C, during the second stage. At this stage, hydrogen groups are removed, and the starch carbon chain is decomposed and depolymerized (Nascimento *et al.,* 2012). Furthermore, in the third stage, polyethylene begins to degrade at a temperature of 400°C, producing gas and hydrocarbon oil (Abdullah *et al.,* 2013). The curve of TGA shows that degradation of TPSS /LLDPE compatibilized blends does not show a significant reduction in each phase. This indicates that the amount of mass loss in each temperature increase at each stage is almost the same (stable). The results of the TGA curve are presented in Figure 14.

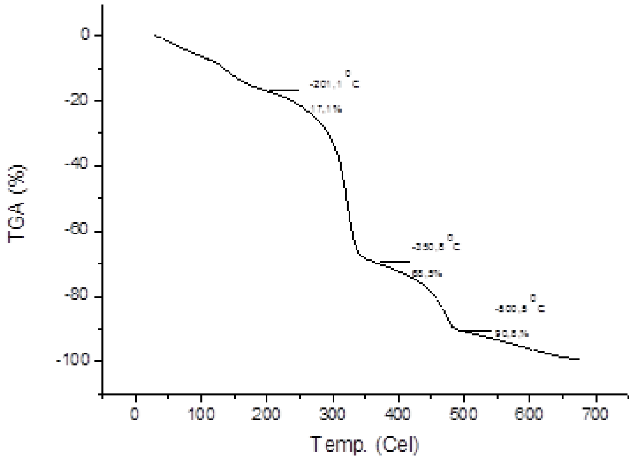

Figure 14 The TGA Spectrum of TPSS/LLDPE Compatibilized blends

4. Conclusions

The addition of LLDPE as a strength agent followed by a compatibilizer between the blend of TPSS and LLDPE could inadvertently rectify the weaknesses of bioplastics, such as their low mechanical properties and high water absorption. The increase in concentrated LLDPE or the decrease in concentrated TPSS could reduce the water absorption percentage. Meanwhile, the degradation rate of blended TPSS/LLDPE decreased because the hydroxyl groups of TPSS getting less reacted with water molecules or were degraded by water. In this case, dispersed particles of LLDPE in the compatible blend are significantly better than those in the incompatible one. Future studies will focus on TPPS/LLDPE/Compatibilizer blends.

Acknowledgments

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