

MECHANICAL PROPERTIES OF RENEWABLE POLYMER OF THERMOPLASTIC POLYETHYLENE BLENDED WITH WASTE OIL POLYMER AT PROLONGED ULTRA-VIOLET IRRADIATION

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Abstract: Recycling of waste oil into polymeric substances has become increasingly important for many applications in agriculture and packaging as it is a renewable resource derived from natural plants, such as sunflower, cotton, linseed or oil palm. Apart from natural palm oil, waste oils derived from industrial activities are converted into monomers and crosslinked with hardener to form solid polymers, namely waste oil polymer (WP). WP is then mixed with low-density polyethylene (LDPE) and high-density polyethylene (HDPE) at ratio of 5%, 10%, 15%, 20%, 25% and 30%. The mixing of both polymer substances using injection molding was carried out to produce solid shape dumbbell specimens. The specimens were exposed under Ultra Violet (UV) irradiation at different durations starting from 500, 1000, 1500, 2000, 2500 and 3000 hours. The mechanical properties show that the WP composition ratios and UV irradiation exposure times play significant roles in controlling mechanical properties of two-matrix polymers blended with LDPE and HDPE of synthetic polymers. The tensile strength and elongation at break of WL5 blend were higher than the neat LDPE, with the percentage increment of 11% and 3.7% respectively before and after UV irradiation exposure at 500 hours. Meanwhile, the tensile strength and young's modulus generally decreased for WH while the elongation at break increases for all composition ratios, thus providing the opportunity to modulate polymer properties.

Keywords: High density polyethylene, low density polyethylene, renewable polymer, mechanical properties, ultra-violet irradiation.

Introduction

The manufacture of polymeric materials from renewable resources has been growing significantly due to dwindling petroleum resources. In this context, vegetable oils have become one of the alternatives since they are abundant and relatively low cost (Gerard *et al.*, 2013). Demands for sustainable development using renewable resources rather than petroleum-based ones have brought forward a new method in composite synthesis and development. In addition, renewable polymers in which a natural ingredient has been partially substituted for a petroleum-derived compound, have gathered much attention from researchers (Roumeli *et al.*, 2015).

Waste oil polymers (WP) include all vegetable oil such as palm oil, rapeseed oil, soybean oil, sunflower oil and many more. The main constituents of waste oil polymers are triacylglycerols, which are the product of esterification of glycerol with three fatty acids. Fatty acids account for 95% of the total weight of triglycerides and their content is characteristic for each waste oil (Roumeli *et al.*, 2015; Min Min *et al.*, 2008; Martinez *et al.*, 2015). In the polymer industry, waste oil, which represents a major potential source of chemicals, has been utilized as an alternative feedstock for sustainable monomers.

Nevertheless, Polyethylene (PE) waste takes a thousand years to degrade after disposal, causing a negative environmental impact

(Enriquez, 2016; Burlein & Rocha, 2014). Therefore, the introduction of biodegradable polymers in the form of thermoplastic Low Density Polyethylene (LDPE) and High Density Polyethylene (HDPE) materials may be considered as an alternative in the development of more environmentally friendly materials (Burlein & Rocha, 2014). In plastic packaging, Low Density Polyethylene (LDPE) and High-Density Polyethylene (HDPE) are commonly used as blending materials for packaging due to good mechanical properties and good processability, including recyclability, moldability, high flexibility, low cost and low friction during compounding. LDPE is resistant to stress cracking and had excellent impact properties (Burlein & Rocha, 2014), while HDPE has lower stiffness, good chemical resistance and barrier properties (Trigui *et al.*, 2012).

The degradation, either photo, thermal or biological, may change bond scission, chemical transformation and the formation of new functional groups of the polymers that break and cross-link with the polymer chains, the formation of carbonyl (C=O) and vinyl (CH=CH₂) groups, as well as changes in the conformation of the polymer (Romo *et al.*, 2015). It also changes the polymer based on its strength, color and shape by specific process. Degradation of mechanical, optical or electrical characteristics are changes in material properties that change the polymer's crazing, cracking, erosion, discoloration or phase separation.

Therefore, the aim of this work is to take advantage of LDPE, HDPE and waste oil polymer itself, by blending the LDPE / HDPE polymer with WP to enhance the mechanical properties. Furthermore, the WP is expected to degrade faster compared to petroleum-based polymers and to propose a new method for melt-mixing of thermoset renewable polymers with thermoplastic polymers as composites materials. Thus, the two types of thermoplastic of LDPE and HDPE are blended with WP in granulate forms and named WL and WH respectively. The granulate was injected through injection molding to produce dumbbell specimens. Furthermore,

the specimens were exposed to UV irradiation at a constant temperature of 50°C at 500, 1000, 1500, 2000, 2500 and 3000 hours. UV radiation may cause photooxidative degradation which results in breaking of the polymer chains, produces free radical and reduces the molecular weight, causing deterioration of mechanical properties and leading to useless materials. The advantage of this study is to examine the reduction rate of time degradation for WP blended with petroleum-based thermoplastic after an unpredictable time.

Materials and Methods

Low Density Polyethylene (LDPE)

Low density polyethylene (LDPE) with a density of 0.922 g/cm³, melt index of 5 g/10 min and vicat softening point of 93 °C was supplied by Lotte Chemical Titan (M) Sdn. Bhd.

High Density Polyethylene (HDPE)

High density polyethylene (HDPE) with a density of 0.957 g/cm³, melt index of 4 g/10 min and vicat softening point of 124 °C was supplied by Lotte Chemical Titan Sdn. Bhd.

Waste Oil Polymer (WP)

Waste cooking palm oil was used to produce the waste oil polymer (WP). The conversion process is started with the preparation of a catalyst to generate the epoxies from the unsaturated fatty compound. The second reaction is the opening of the ring acid catalyst of the epoxies to form polyols or bio monomer (Rus *et al.*, 2013; Mohid *et al.*, 2013; Ghani, & Rus 2013; Rahim *et al.*, 2013; Rus *et al.*, 2015a; Rus *et al.*, 2015b; Sulong *et al.*, 2013). Then, the bio-monomer was mixed with cross-linking agent flexible Polymethane Polyphenyl Isocyanate (Modified polymeric-MDI) with a viscosity of 120-160 cps, specific gravity at 1.18-1.20 g/ml and renewable monomers at room temperature (renewable polyol) (Rus *et al.*, 2013; Mohid *et al.*, 2013; Ghani, & Rus 2013; Rahim *et al.*, 2013; Rus *et al.*, 2015a; Rus *et al.*, 2015b; Sulong *et al.*, 2013).

Blended Waste Oil Polymer (WL and WH)

The monomer was heated in an oven at 80°C until liquidized. Bio-monomer was mixed with cross-linking agent Modified polymeric-MDI. The liquid bio-monomer and isocyanate was stirred with a ratio of 1:0.5 to produce waste oil biopolymer (WP). After that, LDPE and WP were mixed together until homogenous sy. The LDPE-WP blend was left for 30 minutes to solidify for the injection-moulding process named as WL. The processes are repeated for HDPE composition and named as WH. The composition of WL and WH are 5, 10, 15, 20, 25 and 30%.

Injection Moulding

Dumbbell specimens of WL and WH are prepared in accordance with ISO 527-2 (5A) using injection molding (Nissei Horizontal Screw Type Injection Molding NP7). The specific condition: temperature in nozzle = 185°C (LDPE), 205°C (HDPE), temperature of first cylinder = 175°C (LDPE), 180°C (HDPE), temperature of second cylinder = 165°C (LDPE and HDPE) and injection pressure = 43.3 MPa (LDPE), 96.6 MPa (HDPE).

Ultra Violet Irradiation Exposure

Artificial aging is to expose the specimens to UV irradiation using UV Weatherometer to simulate the solar irradiation. Other parameters, for example temperature at 50°C and relative humidity remain under control at 50%. The UV Weatherometer is Haida International Equipment Co., LTD and is equipped with a UV Lamp Tested Model HD-703. Tests were conducted using ISO 4892-3 at interval times of 500, 1000, 1500, 2000, 2500 and 3000 hours.

Universal Tensile Machine

Mechanical properties of the specimens were determined by tensile strength, percentage elongation at break and young modulus. Tensile tests were conducted using Universal Tensile Machine AG-I, Shimadzu in accordance with ISO 527-2 (5A). Five specimens were analyzed with testing speed at 5 mm/min and the load

was 10 kN at a cross-head speed of 50 mm/min⁻¹.

Scanning Electron Microscope

A scanning electron microscope (SEM JEOL JSM-6390LV) with an accelerating voltage of 10 kV was used to analyze the morphologies of selected molded specimens. Samples of the scanning electron microscopy (SEM) were taken from the cross-section at the center of the fractured molded tensile bar. Fractured surface of the samples was mounted on a brass holder using JFC-1600 and sputtered with platinum.

Results and Discussion

Injection Molding Parameters

The injection-molding temperatures of blended polymers were based on TGA results. Each zone was ensured not to exceed 238°C so that the WP does not decompose during the injection moulding process. Then, by referring to the melting temperatures and typical injection molding temperatures of both pure LDPE and HDPE, the initial parameters were set based on TGA result. The next several temperature values with increasing WP composition were adjusted to produce non-defective samples such as flash, short shot, sink marks, flow lines, burn marks, vacuum voids and jetting.

According to Table 1, the WL was able to be processed at the same parameters as neat LDPE, regardless of the addition of WP. This is advantageous to the processing of environmentally friendly plastics and will not require any additional cost. However, further development of this area should consider processing at lower temperatures, require lower injection pressure and enhance productivity by reduction of cycle time (Wiwin *et al.*, 2014). Due to lower temperature and injection pressure required for processing, less energy is consumed and thermal degradation of WP is reduced.

Meanwhile for the WH, the temperature had to be adjusted to ensure the melted polymer fully fills the mold. All specimens injected in IM were individually examined for any defects such

Table 1: The injection-molding parameters used for WL and WH blended

Parameter Setting		WL	WH
Injection Pressure [MPa]		43.40	43.40
Injection Rate [cm ³ /s]		15.00	15.00
Holding Pressure [MPa]		43.40	43.40
Holding Time [s]		2.00	2.00
Cooling Time [s]		10.00	10.00
Injection Time [s]		2.00	2.00
Cycle Time [s]		22.00	22.00
Temperature [°C]	Feed	50.00	50.00
	Rear 1	165.00	170.00
	Rear 2	170.00	175.00
	Middle	175.00	180.00
	Front	180.00	195.00
	Nozzle	185.00	205.00

as flash, short shot, sink marks, flow lines, burn marks, vacuum voids and jetting. According to the results, the parameters used for neat HDPE were the same with the addition of different percentages of WP content. The temperature condition of HDPE is different from LDPE due to the material properties of LDPE and HDPE although both are ethylene polymers (Matmatch, 2020).

Mechanical Characterization of WL and WH before UV Irradiation Exposure

The tensile strength decreased with respect to the WP percentage ratio as illustrated in Figure 1. The tensile strength of WL was slightly lower

as compared to the WH. The tensile strength slightly decreased as the ratio of WP increases from 5% to 30%. Variation in values is due to different types of polyethylene used for WP. WL graph pattern was almost a straight line as the tensile strength values change with respect to WP composition ratio. The highest tensile strength value of WL5 is 9.93 MPa compared to the neat LDPE which is 8.91 MPa. The tensile strength of WL keeps decreasing for WL10, WL15, WL20, WL25 to the lowest value of 7.31 MPa at WL30. However, the tensile strength of the WH decreased more significantly as the value changed from WH5 28.66 MPa to 16.40 MPa at WH30. The change in value is 27%

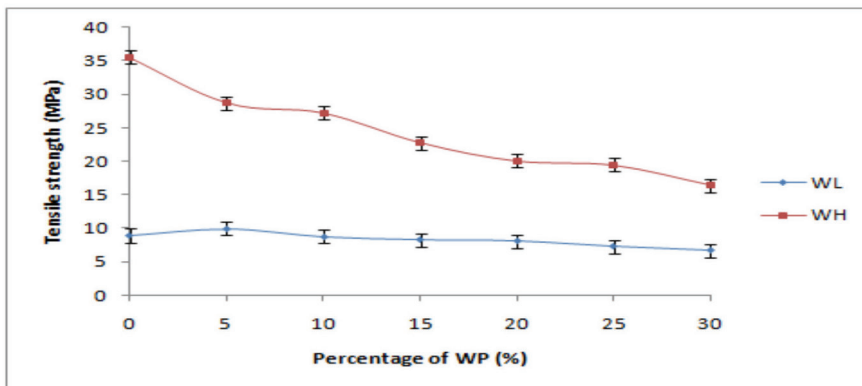


Figure 1: Tensile strengths for WL and WH with different percentage of WP composition ratio

lower compared to neat HDPE, which is 35.54 MPa. Although LDPE and HDPE are both thermoplastic ethylene polymers, they differ in several properties and uses. A clear difference is that LDPE has more branching than HDPE. Branching happens during polymerisation, where the polymer chains are bound to secondary polymer chains by replacing an atom in the primary chain with a monomer group (Matmatch, 2020). That weakens polymer intermolecular forces. That is why HDPE has a higher strength-to-density ratio than LDPE with an increased tensile strength (Matmatch, 2020).

Meanwhile, the percentage elongation at break of WL and WH shows opposite graph trend compared to the tensile strength. The percentage elongation at the break of the polymer blended was increased with the addition of WP content. As shown in Figure 2, for WL, the value of percentage of elongation at break started to increase slightly at WL5 and decreased at WL10, WL15, WL20, WL25 and WL30. The optimum value of percentage elongation at break is at WL5 (70.22%) and WL10 (69.33%) which are higher than neat LDPE (67.64%). On the other hand, the maximum percentage elongation at break is at WH5 (70.22%), which is higher than neat HDPE (37.83%). The increasing value of percentage of elongation at break may be due to yielding before breaking because of interfacial adhesion between WP and HDPE matrix.

In Figure 3, the young's modulus showed a similar trend with tensile strength. For WL, it declined insignificantly alongside the increase of WP. The highest young's modulus is 79.86 MPa and the lowest value is 60.35 MPa for WL and WL30 respectively. Meanwhile for WH, the highest young's modulus is 366.95 MPa for HDPE and the lowest is WH30, which is 224.74 MPa. During mechanical testing, loads were not directly transferred to the matrix but to WP for enhanced mechanical properties which required good interactions between the WP and the HDPE. In addition, the restriction of polymer chain movements contributes to the decrease in tensile strength as well as elongation and young's modulus due to increase in percentage ratio of WP, causing restriction on polymer chain movements.

According to previous studies, when filler and matrix bonding are not properly blended, specimens appeared unable to withstand shear strain and elongations at rupture (Tamboli et al., 2014; Souza et al., 2011; Xuefeng et al., 2014). WP imixed with thermoplastic results in distribution of tensile and compressive micro stresses present in WH. The tensile stresses are more likely to occur at higher fractions and it is possible that these stresses may generate interface cracking between matrices. The possibility of a tensile stress near the periphery of the flakes is low (Tamboli et al., 2014; Satomayor et al., 2014).

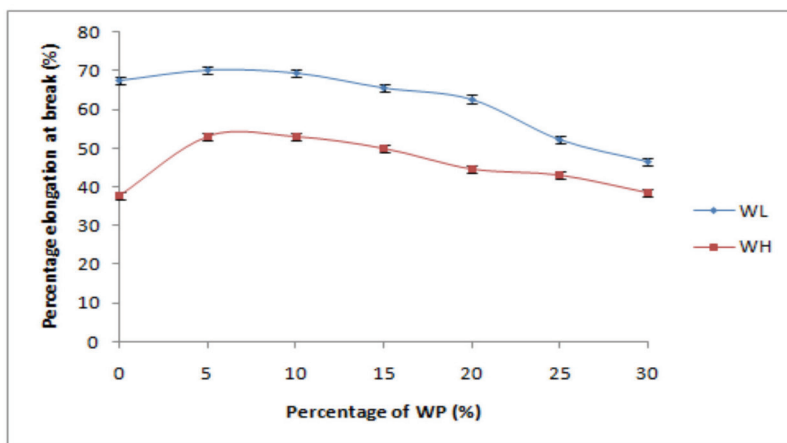


Figure 2: Percentage elongation at break for WL and WH with different percentage of WP compositions ratio

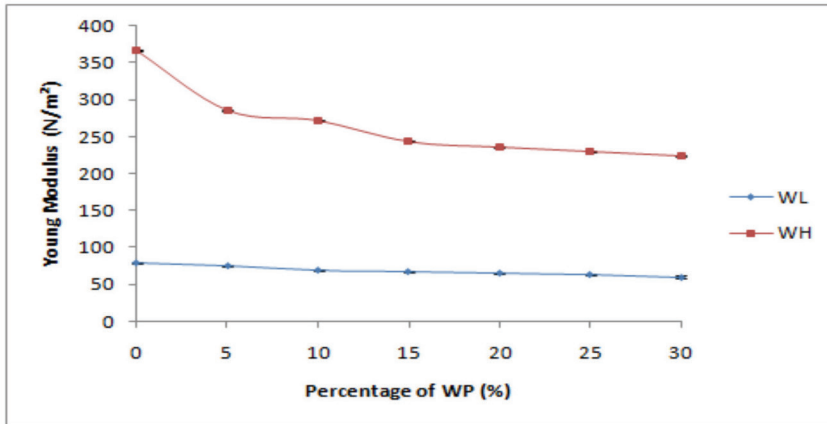


Figure 3: Young's Modulus for WL and WH with different percentage of WP composition ratio

Mechanical Properties WL after UV Irradiation Exposure

The WP content, cross-linker and UV irradiation exposure have influenced the bond strength of the polymer, and mechanical strength or elasticity of the WL is strongly dependent on the percentage ratio of WP. Thus, the variation in tensile strength and percentage elongation at break may be due to the changes in chain-scission ratio occurring during UV irradiation (Khoramnejadian & Radhnezhad, 2016; Hoque *et al.*, 2013).

Figure 4 shows the pattern changes for tensile strength of WL under UV irradiation exposure. The overall trend of tensile strength for LDPE increased. For WL5 and WL10, with addition of 5 and 10% of WP but decreased at WL15. For WL20 to WL30, the increment in

strength values were also observed at lower UV irradiation exposure hours but decreased at 1500 hours until 3000 hours.

Figure 5 shows the trend changes of percentage of elongation at break after UV irradiation of WL. The percentage of elongation at break for neat LDPE is lower compared to WP. However, when blended with WL, the value starts to increase at WL5. Percentage of elongation at break pattern decreased after prolonged UV irradiation exposure together with the increment addition of WP.

The young's modulus is strongly dependent on temperature. Viscoelasticity describes a complex time-dependent elastic response, which exhibits hysteresis in the stress-strain curve when the load is removed. Young's modulus for WL blends after UV irradiation exposure

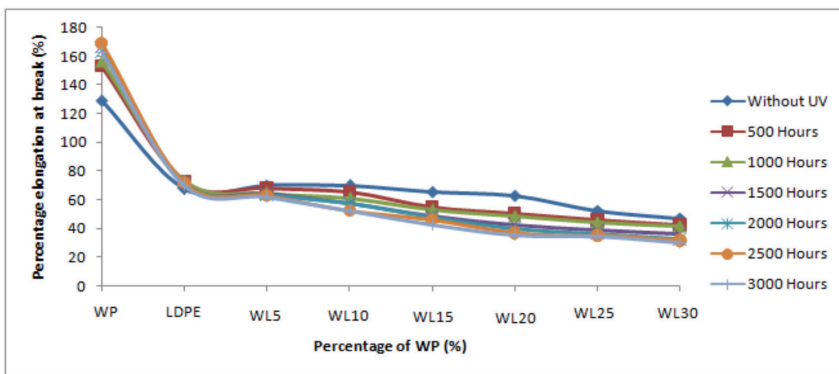


Figure 4: Tensile strength of WL after UV irradiation exposure hours

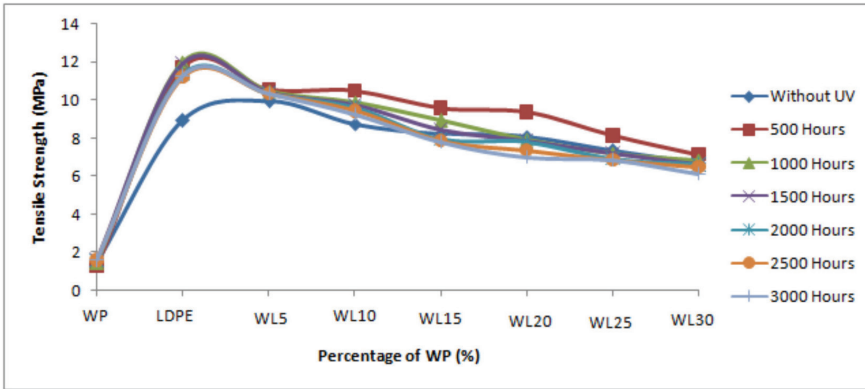


Figure 5: Percentage elongation at break of WL after UV irradiation exposure hours

as shown in Figure 6, with the overall value of young’s modulus decreased. However, for WL5 and WL10 the value of young’s modulus increased at 500 hours to 1000 hours and decreased afterwards. For WL15 to WL30, the value of young modulus increased at 500 hours but decreased after 1000 hours and afterwards.

The decreasing tensile strength and young’s modulus value may be attributed by the occurrence of chain-scission processes as a result of photo oxidation during UV irradiation. However, the mechanisms which govern the photo-oxidation processes in polymer compound specimens are not simple, since there are many factors that influence the processes. According to previous studies, these include the intensity and energy of UV rays, impurities, particle

size of the used biopolymer and free radicals formed during the synthesis or processing of the specimens (Khoramnejadian & Radhnezhad, 2016).

Mechanical Properties WH after UV Irradiation Exposure

The trend of tensile strength for WH increased at the first 500 hours of UV irradiation before it slightly decreased to 1000 hours of UV irradiation exposure as shown in Figure 7. Afterwards, the values of tensile strength for WH fluctuates. This result is similar to a previous study which found that tensile strength at yield point remains stable or increased slightly till 1000 hours of UV radiation and gradually reduces in all samples (Grigoriadou *et al.*, 2013).

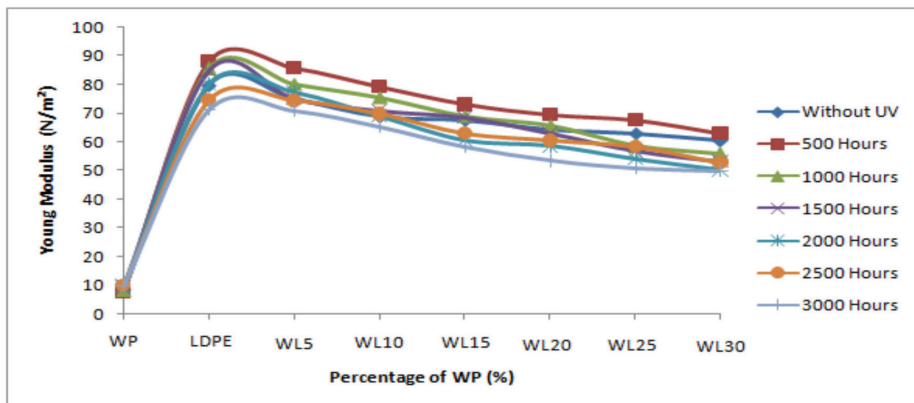


Figure 6: Young’s Modulus of WL after UV irradiation exposure hours

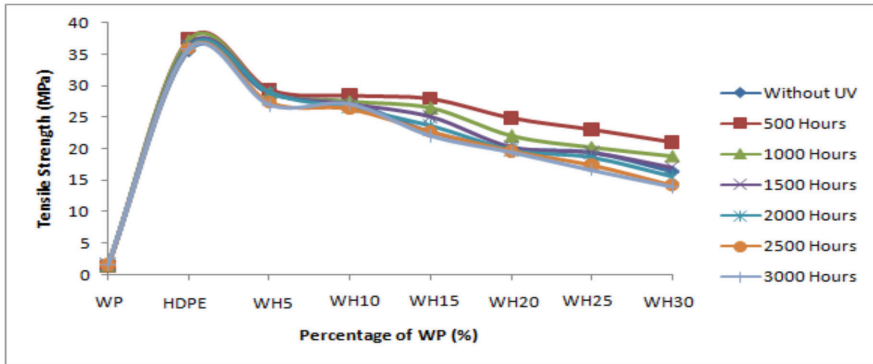


Figure 7: Tensile strength of WH after UV irradiation exposure hours

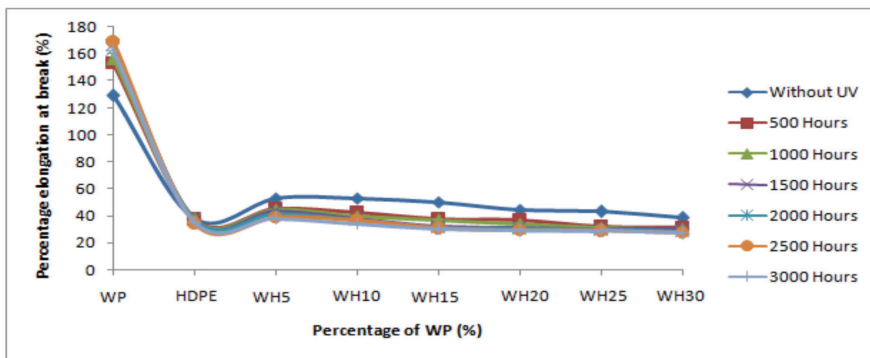


Figure 8: Percentage elongation at break of WH after UV irradiation exposure hours

The percentage of elongation at break as shown in Figure 8 for WH were higher compared to neat HDPE, which is similar to the result of unexposed samples. The percentage of elongation at break for WH blend decreased upon UV irradiation exposure.

The result of young's modulus is illustrated in Figure 9. Increase in UV irradiation exposure hours causes dramatic decrease for WH5. This is due to increasing WH blends that causes decreasing stiffness. HDPE has low strength and stiffness due to the orderly branch distribution that promotes HDPE crystallization and restricts chain rotation, producing a stronger but more brittle material (Grigoriadou *et al.*, 2018). The introduction of WP into the HDPE chain disturbs the order and leads to a more rigid material. The few branches in the HDPE structure allows the polymer chains to pack themselves closely together, so the intermolecular forces

are stronger, resulting in a dense and highly crystalline material of moderate stiffness (Gulmine, 2003; Suljovrujic, 2013; Benoit *et al.*, 2017).

The tensile strength, percentage of elongation at break and young's modulus of polymer blend fluctuated with the increasing ratio of WP. It is believed that the WP content, cross-linker and UV irradiation have influenced the bond strength of the polymer. According to Benoit *et al.*, 2017, the bond strength of polymers tends to increase ductility to HDPE properties. This behaviour suggests that mechanical strength or elasticity of the WH blend is highly dependent on the concentration of WP. Thus, the variations in strength and elongation at break may be due to the changes in chain-scission/cross-linking ratio occurring during UV irradiation (Gulmine, 2003; Suljovrujic, 2013; Benoit *et al.*, 2017).

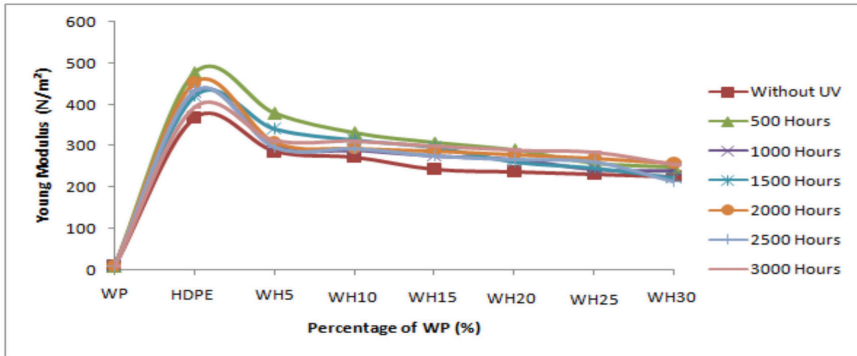


Figure 9: Young's Modulus of WH after UV irradiation exposure hours

The decreasing mechanical property values are caused by the properties of HDPE that have crystalline phases which normally absorb UV irradiation. Nonetheless, this crystalline phase breaks or cleaves the weakened chemical bonds or molecular chains, leading to shorter chains, which in turn causes the plastic material to become more brittle (Grigoriadou *et al.*, 2018; Benoit *et al.*, 2017). This phenomenon is due to the defects of motion in the crystalline regions and the motions of disordered chain segments at the surface of polymer crystals.

The radical decay was accelerated by the increasing degree of branching caused by decreasing crystallinity, and the UV irradiation considerably increases/decreases (fluctuate) with HDPE crystallinity/branching (Hoque, 2013). Thus, the initial differences in the structure of LDPE and HDPE, such as branching and crystallinity, have a significant influence on the induced changes of UV radiation, as well as their structure and properties.

In LDPE, the amorphous content is higher than HDPE, rendering the material more susceptible to oxidative degradation and cross-linking (Gulmine, 2003; Benoit *et al.*, 2017). Low loading of WP for both LDPE and HDPE show remarkable characteristics of increased tensile strength.

Morphological of UV Irradiation Exposure

The fracture was analysed to understand it at a particular magnification. Additionally, this is to show the homogeneity of the injected sample blends. Various magnifications were observed, but for the purpose of this study, the images were taken using scanning electron microscope (SEM) at a magnification of 100x.

The images show the overall structure of the sample that was injected. As shown in Figure 10 and Figure 11, the fracture is ductile. This is due to the significant elongation or necking before break. The deduction was confirmed by the plastic deformation that can be seen by the tear lines on the fracture surface (Xuefeng *et al.*, 2014).

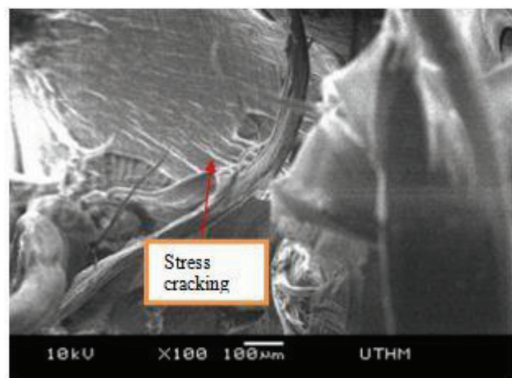


Figure 10: SEM image for WH20



Figure 11: SEM image for WL5

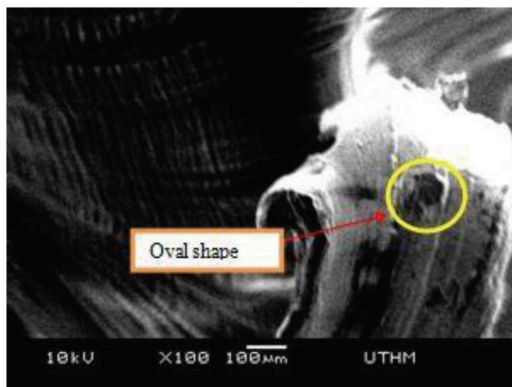


Figure 12: SEM images for LDPE

Nevertheless, due to the partially melted LDPE structure, there may have been some stress points which could be observed. The oval shaped structure that appears in LDPE SEM shows these structures are not attributable to the WP. Rather, they could be impurities or unmelted LDPE, as shown in Figure 12.

Through these pictures, different structures have been identified but it can be inferred that the WP content in the compound helps it to elongate before breaking, in addition to the ductile properties of pure LDPE. Less energy is consumed and thermal degradation of WP is minimized due to the lower temperature and injection pressure required for processing. This can be proven through the long strands that were formed at each fracture. With this property, these compounds can be used in various applications requiring tough or highly ductile materials.

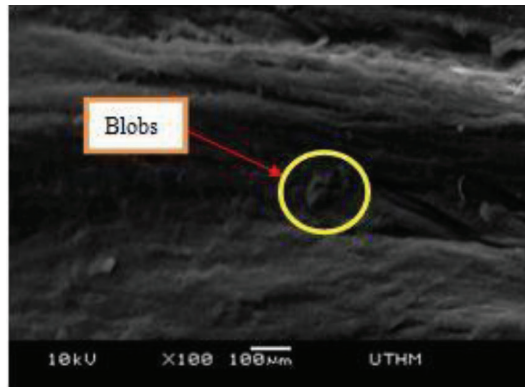


Figure 13: SEM image for WL15 at 3000 hours UV irradiation exposure

In addition, in the WP/LDPE compounds linked to flexible cross-linkers as described in Figure 13, blobs of material that are inhomogeneous to the entire sample are seen. Such blobs may be marked by the foamy structure as crosslinkers, which may not have fully blended with the compound. It might also be due to the processing condition during injection molding. Since the blobs are not clearly seen blended with the crosslinker in the WP/LDPE, it may be due to the fact that the mechanical properties of the cross-linker are slightly better than that of the flexible cross-linker.

Meanwhile, the SEM for WP / HDPE samples is shown in Figure 14. Based on the pictures, the structures may not be fully homogeneous since some rough surfaces are visible. It is also possible to observe some

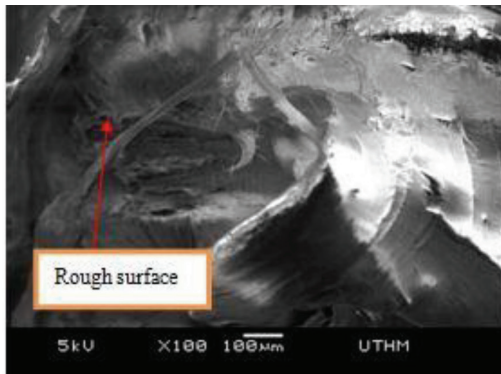


Figure 14: SEM image for WH10 at 2000hours UV irradiation exposure

strands, which display plastic deformation and ductile failure. No foamy structures were seen compared to the WP / LDPE images, which show that the temperature used in the injection molding is approximately accurate.

These findings may help to support the fact that not only the composition of WP, but also the parameters used in injection molding, mainly temperature, affects the mechanical properties of the formed WP/HDPE blend.

Conclusion

New green polymer blends based on different ratios of WP to LDPE and HDPE were successfully produced using melt mixing of Injection Molding. The processing conditions of WL and WH, were started with the initial conditions of standard LDPE and HDPE thermoplastic. The processing temperature during injection molding is based on standard LDPE and HDPE. The corresponding mechanical properties of the blend were examined using tensile strength, percentage elongation at break and young's modulus before and after UV irradiation exposure. The tensile strength and percentage elongation at break of WL5 blend were higher than the neat LDPE, with the percentage increment of 11.0% and 3.7% respectively of unexposed and exposed to UV irradiation at 500 hours. Meanwhile, higher than 5% WP content in the thermoplastic ratio

saw decreasing the tensile strength, percentage elongation at break and young's modulus for WL. The tensile strength and young's modulus generally decreased for WH while the elongation at break increases for all composition ratios. Apart from the changing ratios of WL and WH, the processing temperature also influenced the mechanical properties of the blend. Based on previous studies, it was believed that there is cross-linking and chain-scission between WP and LDPE or HDPE during the UV irradiation exposure.

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