Characterization of Ripe EFB Oil and Waste Frying Oil on the Properties of Starch Bio-based Plastic

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Abstract— **Much attention has been paid in recent years on research to replace petroleum-based commodity plastics with renewable materials in a cost-effective manner. The current study has therefore focused on the production of bio-plastic, primarily starch, from renewable materials. This paper presents the results of studies of the characteristics of the produced bio-plastic by using ripe empty fruit bunch (EFB) oil and waste frying oil (WFO). The potato starch-glycerol based with different amount of ripe EFB oil and waste oil (2.5, 5.0, 7.5 and 10.0 g) were prepared and drying at 40°C for 48 h. Mechanical properties, water solubility, water vapor permeability (WVP), biodegradation test and fourier transform infrared spectroscopy (FTIR) were performed in this study. As a result, the higher the amount of ripe (EFB) oil/WFO in starch bio-based plastic, increase the elasticity which the higher value is 16.5 MPa and 36.33% in elongation at break but decrease in tensile strength, solubility and water vapor permeability. FTIR exhibit that the intermolecular interaction in starch bio-based plastic occurred through C-O-H, O-H, C-H aliphatic and C=O groups. For biodegradation test, all starch bio-based plastic was no longer in soil after a week they were buried which means they have been degrade successfully.**

Keywords— **Bio-plastic, Starch ripe EFB oil bio-plastic (SREOB), *Starch waste frying oil bio-plastic (SWFOB), Ripe EFB oil, Waste frying oil (WFO).***

I. INTRODUCTION

Currently, people become conscious of the damaging effects on the environment materials of petrochemical plastic derived. Some of the researchers conducted some researches into plastic waste management on this world by searching an environmentally friendly ways to plastics. The environmentally friendly way is bioplastic, which it can be easily degraded by microorganisms' enzymatic actions and disposed in the environment. The table shows the comparison between bio-plastic and petro-plastic [1]:

Table 1: Comparison between Bio-Plastic and Petro-Plastic

	Bio-plastic	**Petro-plastic**
Renewable	Yes or partially	No
Sustainable	Yes	No
Break down in the environment	Biodegradable and/or compostable	Some degradable by polymer oxidation
Polymer range	Limited but growing	Extensive
GHG emissions	Usually low	Relatively high
Fossil fuel usage	Usually low	Relatively high

Approximately, percentage of the world's oil production is 4%. This 4% has been converted to plastics usually for used as external car panels and shopping bags while the other further few percent are used in industries of processing. The utilization of fossil fuels caused more costly and as oil runs out, the need for raw material replacement sources for the production of plastics are becoming more and more important [2].

A bio-plastic is made of renewable resources that come in part or in whole from biological sources such as potato starch, maize, soy, rice, sugar cane, wheat and vegetable oil. Bioplastics are made up of all sorts of applications and properties [1]. Today, a blend of potato starch with a compatible plasticizer such as glycerol is one of the most urgent and widespread bio-plastic [3].

Potato starch possesses excellent chemical stability and can be edible [4]. It is produced from low-cost, biocompatible, renewable basic resources. Bio-plastic from potato starch is described as isotropic, odorless, colorless, non-toxic and biodegradable [5].

Usually, plasticizer is used to modify biopolymers to improve processability and/or other applicable properties [6]. Glycerol and sorbitol are the most widely used polyol plasticizers for starch bio-based plastics due to the close similarity between their chemical structures and the structure of starch polymer. [7]. Glycerol is used as a plasticizing agent in this research. Some findings from other researchers have shown that glycerol improves the permeability of water vapor (WVP) and mechanical properties [8]. Glycerol has been reported to greatly increase the plasticity of the bio-plastic [9].

Empty fruit bunches (EFB), oil palm fibers and shells are the main solid waste in the processing of palm oil mills. Palm oil mills produce 14% fibers, 7% shells and 23% fresh fruit bunch (FFB) per ton. In 2008, the EFB alone contributed some 19.5 million tons [10]. Because of its large quantity, EFB has low commercial value and constitutes a disposal problem. EFB is traditionally burned, disposed of in landfills or composted with organic fertilizer. Burning of EFB is prohibited to prevent air pollution and other environmental problems. Therefore, it is important to use EFB optimally to solve these problems and to use the resource for valuable products at the same time.

EFB is composed of 40.37% cellulose, 20.06% hemicellulose and 23.89% lignin [11]. EFB has high potential as a source of cellulosic derived products, such as cellulose fiber [12], nanocellulose, glucose, xylose and ethanol. As a source for bioplastic production, cellulose also has other potential uses. There is no plasticity feature of cellulose. The use of cellulose in the production of bio-plastic requires some modification. Cellulose nanocrystals (CNC) were derived from cellulose used in bio-plastic synthesis [13], nano-fiber cellulose (NFC), cellulose acetate butyrate, cellulose acetate and bio-PE. To increase physical properties or as a filler, such as starch, cellulose derivative was mixed with other biopolymers matrix [12]. Figure 1 shows 3 categories of oil palm [14].

Fig. 1: From Left to Right: Unripe, Ripe, Overripe.

Waste frying oil (WFO) is a complex and heterogeneous waste from restaurants and, to a lesser extent, food processors and households. Interest for this waste has grown profoundly in recent decades and a strong competition has begun to grab this raw material [15].

This research aims to produce bio-based plastics using WFO and ripe EFB oil. Other than that, to study and characterize the tensile strength, solubility, water vapor permeability (WVP), biodegradation test and fourier transform infrared spectroscopy (FTIR) of the produced bio-plastics.

II. METHODOLOGY

A. *Materials and chemicals*

Potato starch and ripe EFB oil were purchased from one of the local supermarkets which stated in Shah Alam, Selangor. WFO was taken from household. Glycerol and distilled water were obtained from Chemical Engineering Laboratory in Uitm Shah Alam, Selangor.

B. *Casting of starch bio-based plastic*

The starch bio-based plastic solution was prepared by dissolving 8 g of potato starch with 2 ml of glycerol in 100 ml of distilled water. Potato starch was dissolved in 100 ml of distilled water at 150°C for 20 min. When starch and distilled water formed a homogeneous solution, 2 ml of glycerol was added followed by different amount of ripe EFB oil and WFO (2.5, 5.0, 7.5 and 10.0 g) at 150°C for about 10 min until well mixed. Next, all prepared solutions namely as starch ripe EFB oil bio-plastic (SREOB) and starch waste frying oil bio-plastic (SWFOB) were poured out into petri dishes and kept at 40°C for 2 days in a drying oven with air convection.

Table 2: Starch Bio-Based Plastics Composition

Sample code	**Potato starch (g)**	**Glycerol (ml)**	**Ripe EFB oil (g)**	**Waste frying oil (g)**
TPS$_0$	8.0	2.0	-	-
TPS-R$_{2.5}$	8.0	2.0	2.5	-
TPS-R$_{5.0}$	8.0	2.0	5.0	-
TPS-R$_{7.5}$	8.0	2.0	7.5	-
TPS-R$_{10.0}$	8.0	2.0	10.0	-
TPS-W$_{2.5}$	8.0	2.0	-	2.5
TPS-W$_{5.0}$	8.0	2.0	-	5.0
TPS-W$_{7.5}$	8.0	2.0	-	7.5
TPS-W$_{10.0}$	8.0	2.0	-	10.0

C. *Mechanical properties*

An ASTM D882-10 (v10.013.0) was used to perform mechanical tests. The SREOB and SWFOB used for tensile testing were cut with scissors to 50 mm x 25 mm. For each SREO and SWFO, the tensile strength, the break elongation and the Young's modulus were determined with a test speed of 50 mm / min and an initial distance between the 30 mm claws. At break, the percentage of strain was calculated by:

$$\text{Percent elongation} = \frac{\text{elongation at rupture}}{\text{initial gage length}} x\ 100 \qquad (1)$$

D. *Solubility*

Water solubility of each SREOB and SWFOB were measured as described by [16] with some modifications. To determine the initial dried SREOB and SWFOB, each of them were measured and dried at 53°C for 24 hours. They were then placed in a 30 ml distilled water petri dish and stored for 48 hours at room temperature. Unresolved dry SREOB and SWFOB were determined by removing the pieces of them from the petri dish and rinsing them gently with distilled water before drying for 24 hours at 53°C. The solution to the SREOB and SWFOB have been calculated as follows:

$$(\%) = \frac{(Wb - Wa)}{Wb} \times 100 \qquad (2)$$

where Wb is the dry weight of the SREOB and SWFOB before submersion and Wa is the dry weight of the SREOB and SWFOB after submersion.

E. *Water vapor permeability (WVP)*

The water vapor permeability of SREOB and SWFOB have been tested with 15 g of silica gel using the glass measuring cells. All SREOB and SWFOB were tied over the test cells' mouth and kept in a desiccator with distilled water (RH= 100 %). During 7 days, the measuring cells were weighted every 6 hours [17]. Water vapor permeability (WVP) rate was calculated as follows [18]:

$$WVP = \frac{g}{tA} x \frac{X}{\Delta P} \qquad (3)$$

Where the mass flux (g/tA) is; A is the permeation area, g is the weight gain, and t is the total hourly time. The term g/t was calculated by linear regression in steady state between the points of weight gain and time; X is the average sample thickness, and the difference between pure water and ambient vapor pressure containing silica gel.

F. *Fourier transform infrared spectroscopy (FTIR)*

Using a Perkin Elmer Spectrum One FTIR Spectrometer, the SREOB and SWFOB absorbance spectrum were recorded. Measurements of FTIR spectroscopy have been made. The peaks, as shown in the chart, indicated that the SREOB and SWFOB included the functional groups.

G. *Biodegradation tests*

On the open space yard in UiTM Shah Alam, the biodegradation test was conducted in normal soil. SREOB and SWFOB were buried at a depth of 5 cm x 5 cm from the surface and kept water every 2 days at maximum holding capacity [19] for up to 2 weeks. The reading for pH, temperature and moisture of soil were taken.

The SREOB and SWFOB were cleaned with a soft brush after 2 weeks and dried at 60°C for 4 hours [19] under the same environmental conditions used to record the initial weights.

The weight loss was expressed as a percentage (Wloss%) according to equation as follows:

$$Wloss\% = \frac{W0 - W1}{W0} x100 \qquad (4)$$

where W0 represents the initial sample weight (before the biodegradation starts) and W1 represents the biodegraded sample weight at each sampling time.

III. RESULTS AND DISCUSSION

A. *Starch bio-based plastic*

Starch bio-plastic obtained in this study is shown in Figure 2, which a colored plastic turn from white to very light yellowish. The resulting of starch bio-based plastic color depended on the amount of the ripe EFB oil and WFO used. This is influenced by the color of both oils which are yellow.

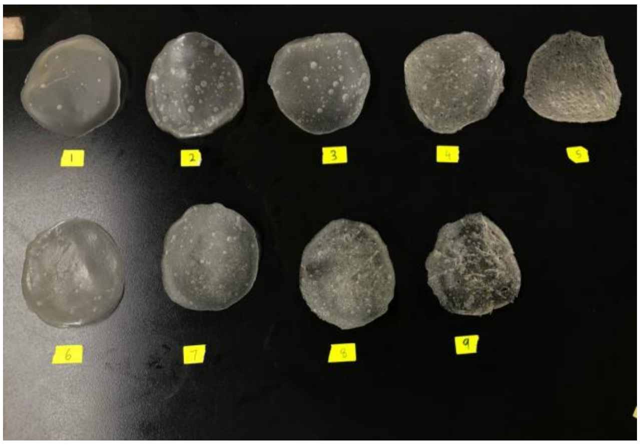

Fig. 2: Results of starch bio-plastic

B. *Mechanical properties*

The Young's modulus (E), tensile strength (TS) and percentage of elongation at break (%EAB) values of the starch bio-based plastics are shown in table.

Table 3: Mechanical properties of bio-plastics

Types of Sample	Thickness (mm)	Young's Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)
TPS0	0.444	99.7	6.59	20.03
TPS-R2.5	0.554	76.7	4.21	26.93
TPS-R5.0	0.554	61.5	2.86	27.23
TPS-R7.5	0.740	32.2	2.56	28.80
TPS-R10.0	1.032	29.0	1.38	28.83
TPS-W2.5	0.449	87.3	6.31	25.93
TPS-W5.0	0.434	51.6	2.78	29.97
TPS-W7.5	0.393	50.8	2.08	30.97
TPS-W10.0	0.646	16.5	2.04	36.33

Young's module (E) is the ratio of the tensile force at the extension break. The lower the modulus of young value, the more elastic the bio-based plastic samples are tested and vice versa if the higher the modulus of young value, the lower the elasticity value but the higher the tensile strength value [20]. Young's module (E) is a measure of an elastic deformation or resistance under load. It relates strain (force per unit area) along an axis or line to strain (proportional deformation). The fundamental principle is that when compressed or extended, a material undergoes elastic deformation and returns to its original shape when the load is removed [21]. Figure 3 shows Young's modulus (E) significantly decreased with increasing amount of WFO and ripe EFB oil. TPS-W10.0 showed the lowest value in Young's modulus (16.5 MPa) followed by TPS-R10.0 (29.0 MPa). According to [6] The presence of the oil will increase the elasticity of the bio-plastic starch. It can be stated that the higher the amount of oil that be used, the more elastic of the SREOB and SWFOB produced.

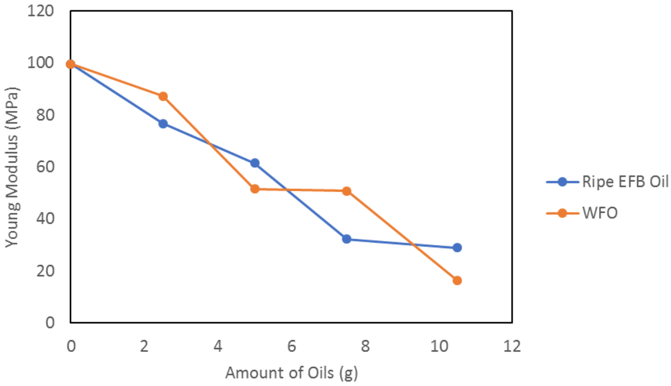

Fig. 3: Graph of Young's Modulus with Different Types and Amount of Oils

The sample's tensile strength is a specific strength obtained before breaking/tearing from the maximum pull. This measurement is designed to determine the magnitude of force needed to reach the maximum pull point on each sample surface area. The change in mechanical properties is characterized by the effect of plasticizers that weaken the intermolecular forces between the chains of adjacent macromolecules, increase the free volume and cause mechanical resistance to decrease [22]. Thus, the increase in the plasticizer concentration causes a reduction of the tensile strength due to the decrease in the intermolecular interactions. According to table, the best values for tensile strength (TS) was found in TPS-W2.5 which the value is (6.31 MPA). Based on the results of this tensile test that shown in Figure 4, it showed that high amount of ripe EFB oil and WFO are very influential on the decreasing of the value of tensile strength starch bio-based plastic produced. It can be stated that, the lower the amount of oil used, the higher the tensile strength.

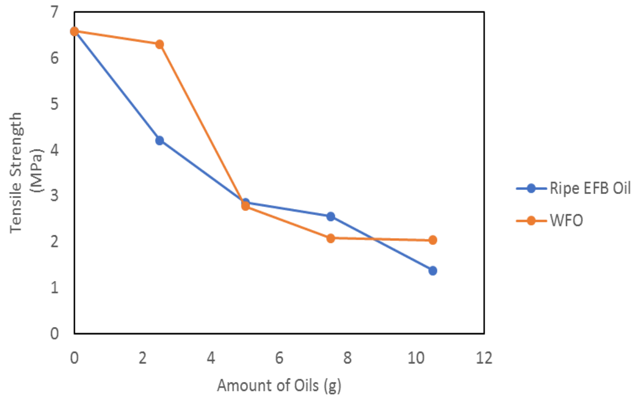

Fig. 4: Graph of Tensile Strength with Different Types and Amount of Oils

Break elongation is a measure of the flexibility and is defined as the ability to deform before breaking [18]. Regarding the results obtained in Table 3, the TPS-W$_{10.0}$ displayed highest value for percentage of elongation at break (%EAB) which 36.33% while the lowest (%EAB) is at sample TPS-W$_{2.5}$ which 25.93%. Figure 5 shows gradually increase of elongation at break with increase of oil. This is possible because there are more polymer chain entanglements per chain, resulting in higher break elongation. Increasing oil was also due to the reduced rigidity associated with plasticizer concentrations, resulting in high elongation at breakage [23].

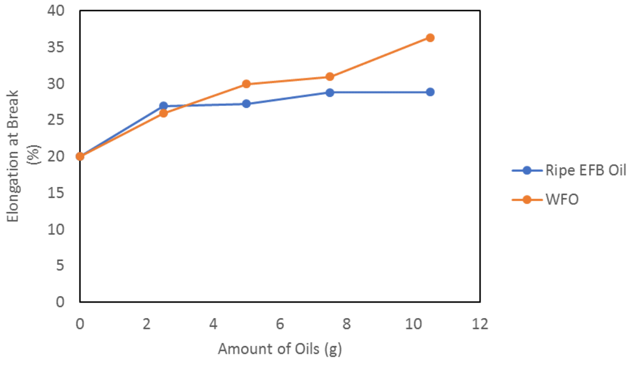

Fig. 5: Graph Of (%EAB) with Different Types and Amount of Oils

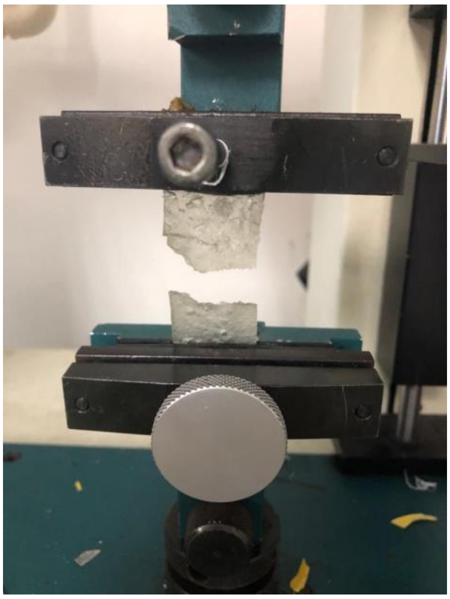

Fig. 6: Mechanical properties test

C. *Solubility*

Water is a small size polar compound that tends to favor sorption as well as diffusivity in dense materials. This capacity makes these materials more moisture-permeable than non-condensable gasses [24]. Water solubility testing is useful in determining sample stability under different conditions. Table 4 shows the sample weight following the drying process and the percentage of bio-based plastic solubility starch.

Table 4: Weight of Samples after Drying Process and Percentage of Solubility

Sample	**After Drying Process**		**Percentage of Solubility (%)**
	Weight of sample before submersion, W_b (g)	**Weight of sample after submersion. W_a (g)**	
TPS$_0$	0.47	0.37	21.28
TPS-R$_{2.5}$	0.61	0.50	18.03
TPS-R$_{5.0}$	1.10	0.91	17.27
TPS-R$_{7.5}$	0.37	0.31	16.22
TPS-R$_{10.0}$	0.50	0.43	14.00
TPS-W$_{2.5}$	0.44	0.36	18.18
TPS-W$_{5.0}$	0.62	0.51	17.74
TPS-W$_{7.5}$	0.46	0.40	13.04
TPS-W$_{10.0}$	0.47	0.42	10.64

Figure 7 shows the graph of the percentage of solubility which gradually decrease with increase of ripe EFB oil and WFO. The interaction between starch, glycerol and also the two oils has affected water solubility. Pure base oils are highly soluble in relation to the ratios of paraffin, naphthenic and aromatic compounds [25]. At the molecular level, the forces of absorption are binding forces in the oil matrix between the molecules of water and the molecules.

On the other hand, polymers' molecular weight plays a major role in their solubility. The solubility of a polymer decreases in a given solvent at a particular temperature as molecular weight increase. The same behavior is also noted as the degree of interconnection increases, as strongly interconnected polymers inhibit the interaction between polymer chains and solvent molecules, preventing these polymer chains from being transported into solutions. [26].

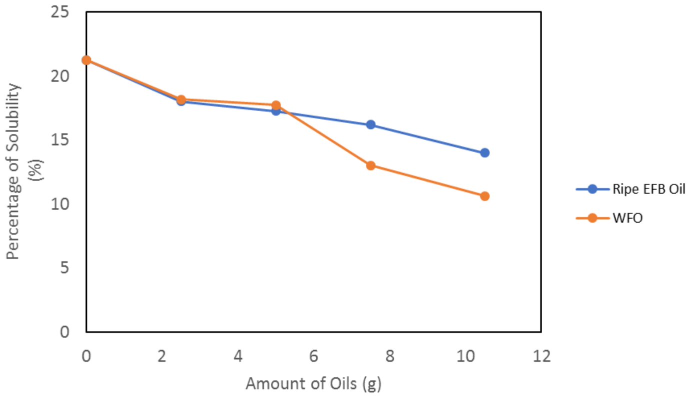

Fig. 7: Graph of Percentage of Solubility with Different Types and Amount of Oils

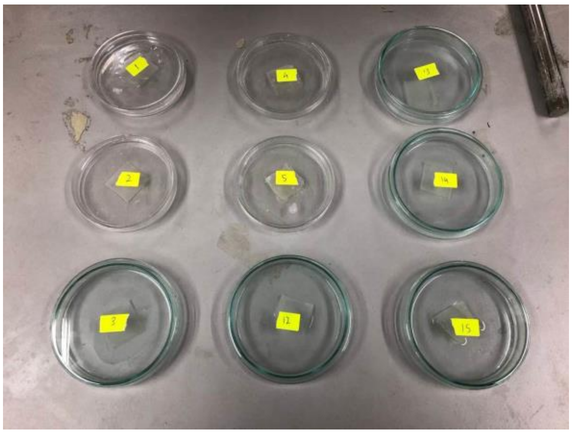

Fig. 8: Water Solubility Test

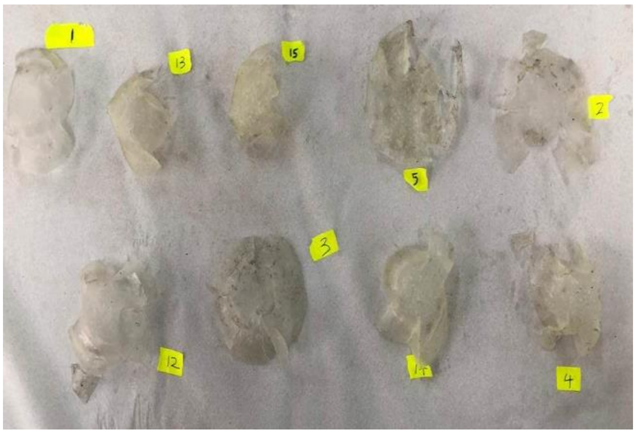

Fig. 9: Starch Bio-Based Plastic after WVP Test

D. *Water vapor permeability*

The results of water vapor permeability in starch bio-based plastic can be useful in understanding possible mechanisms of mass transfer and solvent and polymer interactions [27]. According to irreversible process thermodynamics, the driving force of water transfer through a sample is the water chemical potential difference. The water chemical potential difference is proportional to the difference in water vapor concentration between the two faces when the process occurs at constant temperature and pressure. [28]. The values of weight initial and final sample and also WVP rate are shown in Table 5.

Table 5: Weight of samples and WVP rate

Sample	**Weight of sample (g)**		**WVP**
	Initial (0 hour)	Final (167 hours)	**(g.mm.h^{-1}·m^{-2}kpa^{-1})**
TPS0	3.04	5.74	2.093 x 10^{-6}
TPS-R2.5	5.07	7.37	1.783 x 10^{-6}
TPS-R5.0	3.70	5.90	1.706 x 10^{-6}
TPS-R7.5	2.44	4.64	1.706 x 10^{-6}
TPS-R10.0	4.40	6.50	1.628 x 10^{-6}
TPS-W2.5	3.27	5.67	1.861 x 10^{-6}
TPS-W5.0	4.27	6.67	1.861 x 10^{-6}
TPS-W7.5	4.72	6.92	1.706 x 10^{-6}
TPS-W10.0	4.22	6.32	1.628 x 10^{-6}

The water vapor interacts strongly with polymer structure for most starch bio-based plastic, resulting in diffusion and solubility coefficients depending on the driving force [29]. The increase in weight of starch bio-based plastic WVP samples is due to high relative humidity attributable to high sample moisture content. The increased moisture content of the samples could have resulted in swelling as shown in Figure 9, resulting in expansion of the biopolymer matrix, which increased water vapor diffusion through SREOB and SWFOB [18].

Figure 10 shows the WVP rate diagram. The results show a slight decline in the rate of WVP with an increase in oils. The main mechanisms involved in the transfer of water molecules through the starch bio-based plastic are adsorption, diffusion and desorption. The microstructure affects these mechanisms as determined by chemical composition, crystallinity and plasticizer [30]. Addition of hydrophobic substances such as palm oil could increase film hydrophobicity, reducing water vapor adsorption and also sample migration [31]. It can be stated that the addition of palm oil into starch bio-based plastic in combination with same amount of glycerol could decrease WVP of starch bio-based plastic effectively.

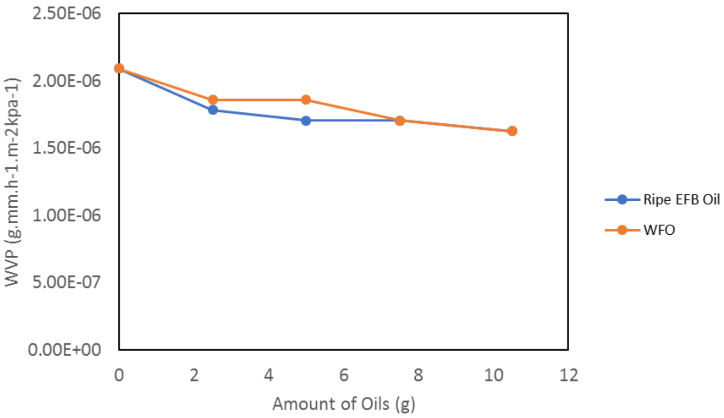

Fig. 10: Graph of WVP Rate with Different Types and Amount of Oils

E. *Fourier transform infrared spectroscopy (FTIR)*

The importance of FTIR spectroscopy in molecular structure identification stems from the large amount of information content obtained and the possibility of assigning certain absorption bands related to their functional groups. The FTIR spectrum of ripe EFB oil and WFO shows similar spectrum characteristics, can be related to those of triglycerides as triglycerides are their main component. All the values of used and unused palm cooking oil spectra is in in the range of the reported Table 6 [32].

Table 6: Functional Groups in the Spectrum of Extra Palm Oil

Frequency (cm^{-1})	**Functional group assignment**
3005	Cis double-bond stretching
2924 and 2852	Asymmetrical and symmetrical stretching vibration of methylene (CH_2) group
1743	Ester carbonyl functional group of the triglycerides
1465	Bending vibrations of the CH_2 and CH_3 aliphatic groups
1417	Rocking vibrations of CH bonds of cis-disubstituted olefins
1402	=C-H bending vibration
1377	Bending vibrations of CH_2 groups
1236 and 1160	C-O stretching
1117 and 1098	Stretching vibration of the C-O ester group
1030	C-O stretching
962	Bending vibration of CH functional

	groups of isolated transolefin
850	$=CH_2$ wagging
721	Overlapping of the methylene ($-CH_2$) rocking vibration and to the out of plane vibration of cis-disubstituted olefins

As in Figure 11 and 12 show the spectrum of all SREOB and SWFOB have band at range 3276.57 to 3285.89 cm^{-1} are attributed to the stretching vibrations of -OH group and the C-H bond in $-CH_2$ respectively which is related to hydroxyl groups present on starch–glycerol bio-plastic [33]. At 2922.93 cm^{-1}, 2922.69 cm^{-1}, 2852.97 cm^{-1} and 2853.66 cm^{-1} resulting asymmetrical and symmetrical stretching vibration of methylene (CH_2) group. The C=O group of triglycerides resulting a stretching vibration at range 1743.81 cm^{-1} to 1744.46 cm^{-1}. At band 854.97 cm^{-1} and 856.15 cm^{-1} resulting $=CH_2$ wagging while the bands at range 1464.28 cm^{-1} to 1464.68 cm^{-1} resulting from the bending vibrations of CH_2 and CH_3 aliphatic groups. All SREOB and SWFOB with amount of 5.0, 7.5 and 10.0 g of ripe EFB oil and WFO have band at range 720.90 cm^{-1} to 721.38 cm^{-1} which resulting overlapping of the methylene ($-CH_2$) rocking vibration and to the out of plane vibration of cis-disubstituted olefins.

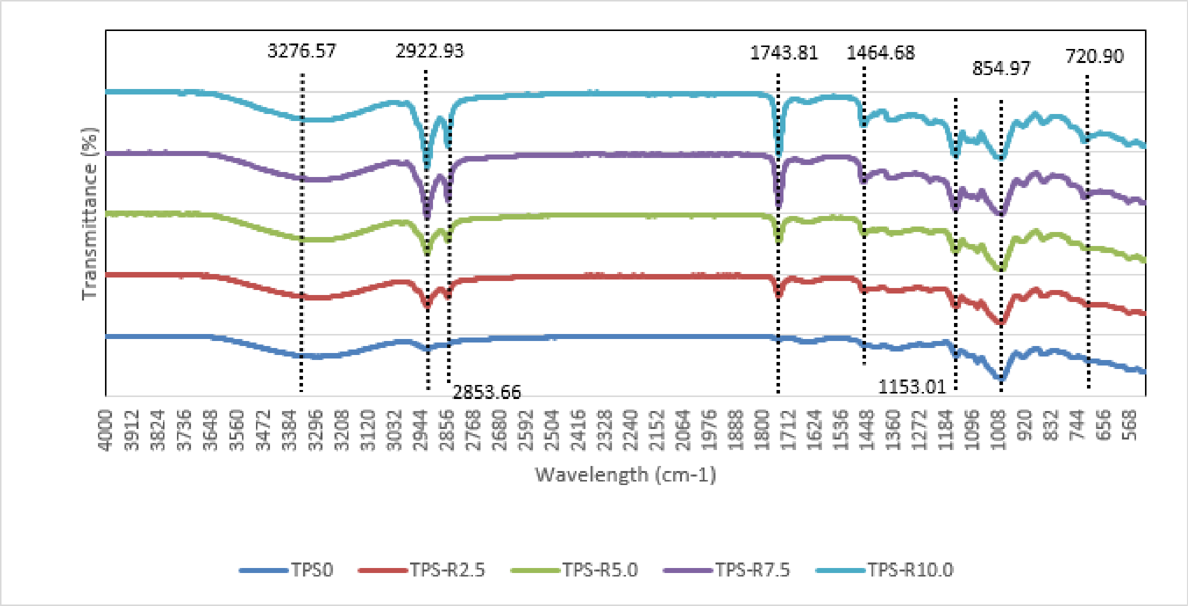

Fig. 11: FTIR spectra of ripe EFB oil

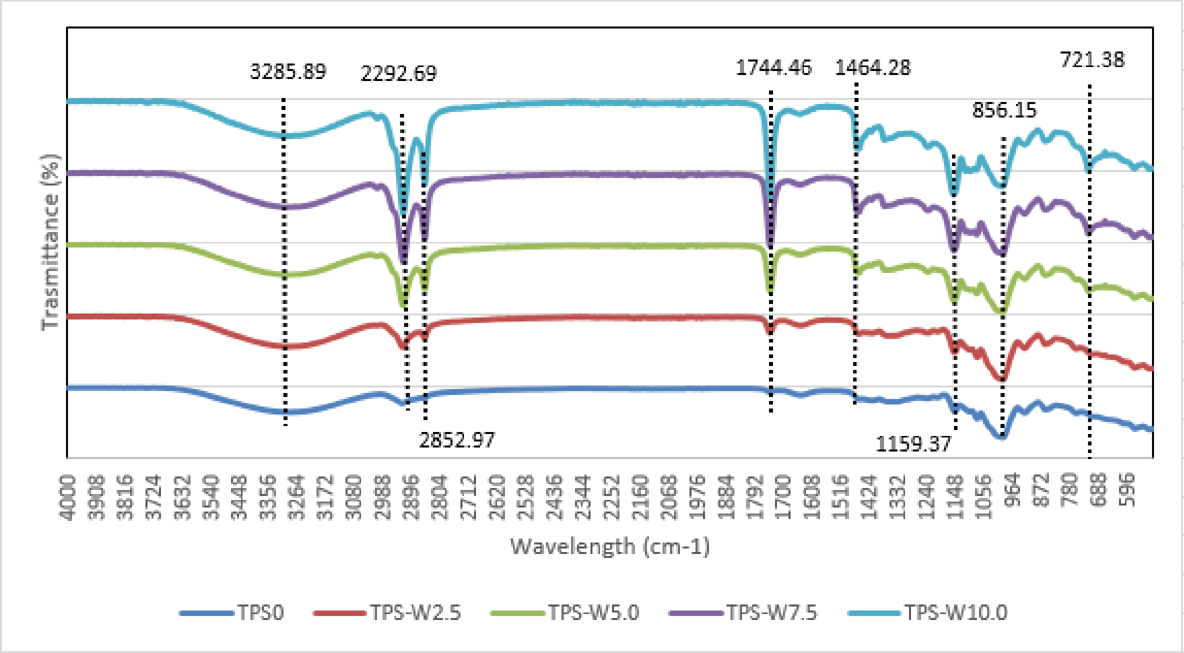

Fig. 12: FTIR spectra of waste frying oil

F. Results of biodegradation tests

The term "biodegradable" is simply defined as the ability of living organisms to break down the molecular components of a material into smaller molecules so that the carbon contained in that material can eventually be returned to the biosphere [34]. The biodegradation test was performed and buried all SREOB and SWFOB in normal soil with depth of 5 cm from the surface. The SREOB and SWFOB were buried within a period of 2 weeks as shown in Figure 13. Based on Figure 14, it is seen that all SREOB and SWFOB have been totally biodegradable, where they were no longer in the soil. The mechanism of degradation is associated with the biological activity present in the soil and the result shows that the enzymatic degradation occurred in all SREOB and SWFOB effectively.

Fig. 13: Starch Bio-Based Plastic before Buried with 5cm Depth

Fig 14: Results of Biodegradation Test after a Week

IV. CONCLUSION

Mechanical properties, water solubility, water vapor permeability, fourier transform infrared spectroscopy (FTIR) and potato starch/ripe EFB oil and glycerol-plasticized potato starch/WFO biodegradation test was assessed. Starch bio-based plastic with increasing amounts of WFO and ripe EFB oil improves the elasticity and percentage of break elongation (%EAB) of starch bio-based plastic due to more polymer chain entanglements per chain. The decrease in tensile strength with an increase in the amount of ripe EFB oil/WFO showed that increased use of oils would reduce a material's resistance to breaking under starch bio-based plastic tension. The value decreases with increasing amounts of ripe EFB oil/WFO for solubility and water vapor permeability testing. This is due to the very limited solubility of base oils, which is related to the ratios of paraffin, naphthenic and aromatic compounds and affected by the microstructure as determined by chemical composition, crystallinity and plasticizer. The functional groups of each sample were observed using FTIR, where the intermolecular interaction occurred through C-O-H, O-H, C-H aliphatic and C=O groups in starch bio-based plastic. Biodegradation test was successfully conducted as all bio-based starch plastic was degraded after a week associated with the present biological activity.

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