

Degradation of linear low-density polyethylene/poly(vinyl alcohol)/kenaf composites

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Abstract The interest of this study was to examine various kenaf (KNF) loadings and burial time, and their effects on degradability behavior of linear low-density polyethylene/poly(vinyl alcohol)/kenaf (LLDPE/PVOH/KNF) composites in soil. The LLDPE/PVOH/KNF composites with various KNF loadings were melt-mixed in a Thermo Haake internal mixer. The composites were buried in soil and their extent of degradability was assessed by tensile properties, morphological study, and weight loss and crystallinity measurements of the degraded composites. Fourier transform infrared spectroscopy was utilized to monitor the changes in chemical structure of the composites before and after degradation in soil. The results showed that with increase in KNF loading, the tensile modulus increased; meanwhile, the tensile strength and elongation-at-break of the degraded composites were dropped. Tensile properties of the degraded composites were decreased with prolonging soil burial time. The formation of cracks and pores was observed in field emission scanning electron microscope micrographs. By measuring the weight loss in LLDPE/PVOH/KNF composite samples before and after the soil burial testing and differential

scanning calorimetry measurements, it was found that the weight loss and crystallinity of the composites increased with soil burial time. The degradation of the composites was confirmed by the formation of a strong absorption band assigned to carbonyl groups.

Keywords Linear low-density polyethylene/poly(vinyl alcohol) · Kenaf · Composites · Soil burial · Degradability

Introduction

Polyethylene (PE) and polypropylene (PP) are among the popular thermoplastics that have attracted applications in various packaging industry, owing to low cost and their relatively good mechanical characteristics [1–3]. However, these polymer materials have aggravated significant environmental problems because of their stability, and that it may take hundreds of years to degrade when they are buried in typical solid waste sites [4–6]. Therefore, all over the world, the development of environmentally friendly polymer products is highly encouraged. Several attempts have been made to reduce the environment impact using degradable synthetic polymer and/or renewable natural polymer resources as the raw materials [7–9]. Renewable natural resources can be used in the form of natural fibers including cellulose, starch, and chitosan [10]. The introduction of renewable natural resources into the non-degradable polymers is the cheapest alternative to produce the partially degradable polymer composites [2]. Kenaf fiber, as a renewable natural resource, is widely utilized as filler/reinforcement in natural fiber polymer composites due to its low cost, biodegradability characteristics, high specific mechanical properties, and abundant availability [11–13].

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Generally, the biodegradation of a high molecular weight polymer is referred to a chemical degradation that is usually caused by naturally occurring microorganisms (fungi, bacteria and algae) via enzymatic action [14, 15]. Biodegradable materials may be degraded into carbon dioxide, biomass, and methane [14, 15]. Poly(vinyl alcohol) (PVOH), a widely used synthetic polymer, is utilized in plastic packaging, films or composites due to its biodegradability [1, 16, 17]. It was reported recently that various species of microorganisms (bacteria, fungi, yeast, and mould) can induce biodegradation of PVOH to occur [7, 14]. Nevertheless, PVOH itself is expensive to be used as a raw material in composite systems. Therefore, blending with a cheaper plastic material like PE can be cost saving. PE is well known for its hydrophobicity, high molecular weight, and resistance toward biological attack due to the lack of functional groups recognizable by microorganisms [18]. It is believed that the plastic materials, either from natural resources and/or biodegradable materials buried in landfilling environment, are subjected to microorganism attack. As a result, the plastic materials undergo degradation and will disintegrate with the removal of natural resources and/or biodegradable materials. Therefore, blending of PE/PVOH with a sufficient amount of natural fibers enhances the degradability of the resulting composites. The natural fibers become the target for the microbial attack and thus promote degradation activities of the resulting composites [19]. There are only few studies that show positive findings in the degradability tests of biocomposites, especially on natural soil burial [2, 20, 21].

The soil burial testing stimulates the real environment and is a realistic method for evaluating the biodegradation process in a waste landfill [15]. Sam et al. [22] evaluated the changes in properties of LLDPE/soya powder blend as a function of soya powder content and compatibilizer. They found that degradation rate of the blends in soil increased by increasing the soya powder composition, and the compatibilized blend degraded slower than the uncompatibilized blend. So far, there is no specific report regarding the degradation behavior of LLDPE/PVOH/KNF composites in case of soil burial exposure. The purpose of this study was to examine the degradability of LLDPE/PVOH/KNF composites subjected to soil burial exposure, and the degradation of the composite was investigated by tensile properties, FESEM, weight loss study, DSC, and FTIR. The outcome of this study is essential as a basis for future application of LLDPE-based composite. More importantly, it was discovered that LLDPE-based composite can be always modified according to any specific application.

Experimental

Materials

LLDPE with a density of 0.92 g/cm^3 and a melt flow rate of 1 g/10 min at $190 \text{ }^\circ\text{C}$ was obtained from PT. Lotte Chemical Titan Nusantara, Indonesia. PVOH with a density of 0.269 g/cm^3 and a molecular weight of $89,000\text{--}98,000 \text{ g/mol}$ was obtained from Sigma-Aldrich (M) Sdn. Bhd., Malaysia. KNF was supplied by National Kenaf and Tobacco Board (LKTN), Kelantan, Malaysia. KNF with an average particle size of $75 \mu\text{m}$ was used and dried in a vacuum oven for 24 h at temperature of $80 \text{ }^\circ\text{C}$ before being utilized. The weight ratio of 60:40 (by wt%) was kept constant for LLDPE/PVOH. Melt mixing process was performed in a Thermo Haake internal mixer (Model R600/610 from LMS Scientific Solution Sdn. Bhd., Malaysia) at a temperature of $150 \text{ }^\circ\text{C}$ and at a rotor speed of 50 rpm. During the mixing process, the torque values were recorded for each sample. The composites were then molded into thin sheets (1 mm) using an electrically heated hydraulic press (GoTech Testing Machine, Model KT-7014 A from GoTech Testing Machine Inc., Taiwan) at $150 \text{ }^\circ\text{C}$.

Natural soil burial test

The extent of degradability of LLDPE/PVOH/KNF composites was evaluated using the simple soil burial test. The soil burial test method in this study was conducted by referring to Roy et al. [3], Yussuf et al. [20], Ismail et al. [23], and Zuhair and Anwar [24]. The dumbbell-shaped composite samples were put inside a polybag covered by normal garden soil. The samples were buried to a depth of 10 cm and located outside natural environment with an average temperature around $30 \text{ }^\circ\text{C}$ and 80% relative humidity. The garden soil was sprayed with water at regular time intervals to maintain the humidity. The average data of temperature and rainfall were obtained from the nearby meteorology station in Butterworth (latitude $5^\circ 21' \text{N}$, longitude $100^\circ 24' \text{E}$) as shown in Table 1. Soil burial for 3 and 6 months was done because PVOH is a synthetic biodegradable polymer and KNF is a natural resource which is also biodegradable. The samples were recovered from the soils after 3 and 6 months, respectively, and were cleaned with distilled water to remove the soil debris from the surface. Next, the samples were subjected for drying at temperature of $60 \text{ }^\circ\text{C}$ in vacuum oven until a constant weight was attained.

Tensile tests

The tensile testing was performed using a Universal testing machine (LMS Scientific Solution Sdn. Bhd., Malaysia)

Table 1 Average temperature and rainfall data obtained from meteorology station in Butterworth

Time (month)	Average	
	Temperature (°C)	Rainfall (mm)
January 15	27.7	1.5
February 15	28.6	0.8
March 15	29.0	3.4
April 15	28.6	11.6
May 15	29.2	6.5
June 15	28.9	6.9
July 15	28.6	5.5
August 15	28.0	7.0
September 15	28.0	14.2
October 15	28.0	10.2
November 15	27.3	10.6
December 15	28.1	5.1

following the ASTM D638-Type IV. The crosshead speed and gauge length were kept constant at 5 mm/min and 50 mm, respectively. The tests were carried out on 1-mm-thick dumbbell-shaped specimens at 25 ± 3 °C. A total of five readings of the tensile results (strength, modulus, and elongation-at-break) were taken.

Morphology

A Zeiss Supra-35VP FESEM (Carl Zeiss NTS GmbH, Germany) was used to observe the surface of the degraded samples after subjected to soil burial. A thin layer of gold was coated on the surface of degraded samples to avoid electrostatic charging and poor resolution. The samples were mounted onto the aluminum stage for inspection.

Weight loss

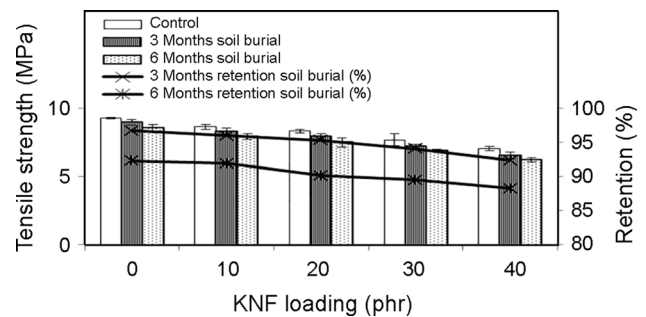
The biodegradability was evaluated by measuring the weight loss in samples before and after the soil burial testing. The equation below was used to calculate the weight loss of the degraded samples:

$$\text{Weight loss (\%)} = \frac{W_0 - W_1}{W_0} \times 100, \quad (1)$$

where W_0 and W_1 are the weights of the samples before and after the soil burial test, respectively.

Differential scanning calorimetry (DSC)

A PerkinElmer DSC 7 thermal analyzer (PerkinElmer Sdn. Bhd., Malaysia) was used to study the crystallinity of the degraded composites. It was carried out under a nitrogen

**Fig. 1** Effect of 3 and 6 months of natural soil burial on tensile strength of LLDPE/PVOH/KNF composites at different KNF loadings

atmosphere according to ASTM D3418-03. A sample of 5–10 mg was capsulated in an aluminum pan and exposed to the heating and cooling cycles. The heating and cooling rates were maintained at a constant rate of 10 °C/min, in a temperature range of 30–170 °C. The area under the endothermic curve was used to calculate the heat of fusion. As the heat of fusion is directly proportional to the amount of crystalline LLDPE in the sample, it decreases as KNF loading increases. The crystallinity percentage can be calculated from the following equation:

$$\text{Crystallinity (\%)} = \frac{\Delta H_f^*}{W_f \times \Delta H_f^0} \times 100, \quad (2)$$

where ΔH_f^0 is the heat of fusion for fully crystalline LLDPE (290 J/g) [1], ΔH_f^* is the experimental heat of fusion for the composites sample, and W_f is the weight fraction of LLDPE in the composites.

Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra of the degraded samples were obtained using an FTIR-PerkinElmer System 2000 spectrometer (PerkinElmer Sdn. Bhd., Malaysia). The spectrum resolution was 4/cm in a spectral range of 4000–550/cm.

Results and discussion

Tensile properties and morphology

Figure 1 illustrates the effect of 3 and 6 months of natural soil burial on the tensile strength of LLDPE/PVOH/KNF composites at various KNF loadings. The decrement in tensile strength with increasing KNF loading was attributed to the poor interfacial adhesion between KNF and LLDPE/PVOH, as reported in our previous study [25]. Meanwhile, the decrement in tensile strength after 3 and 6 months of

soil exposure was a result of degradation of composites due to the leaching of KNF and PVOH from composites, when they interacted with the moisture in soil. This led to formation of pores as shown in the FESEM study. The pores acted as stress focal points and also were channels for the water and/or microbes to penetrate further into the polymer chain [23]. Therefore, the composites started to lose their structural strength and their ability to transfer stress was reduced effectively. Another reason that contributed to the tensile strength decrement could be due to the microorganism attack on composite surface that induced polymer chain scission. Sam et al. [22] reported that the reduction of tensile strength after soil burial exposure was due to the presence of polymer chain scission resulting from degradation. According to Mohan and Srivastava [15], scission of polymer chains is one of the major reasons for reduction of mechanical properties. The retention of tensile properties for LLDPE/PVOH/KNF composites at different KNF loadings

after 3 and 6 months of soil burial exposure is also presented in Fig. 1. It is obvious that the tensile strength retention for the composites after 6 months of soil burial is lower than that for the composites after 3 months of soil burial. This result shows that longer soil burial period tends to induce greater deterioration and results in higher degradability in the composites sample.

Figure 2 shows the effect of 3 and 6 months of natural soil burial on elongation-at-break of LLDPE/PVOH/KNF composites at various KNF loadings. The trend of elongation-at-break of the composites was decreased with increasing KNF loading. Similar findings were reported by Obasi and Onuegbu [18], and Pang et al. [25]. Meanwhile, the decrement trend observed with soil burial exposure time was due to the action of microorganism attack that reduced the polymer chain length. As a result, the flexibility and the elongation-at-break of the composites were decreased. The elongation-at-break retention for the composites after 6 months of soil burial exposure was lower than that for the composites after 3 months of soil burial exposure, indicating higher degradability for composites with prolonged soil burial period.

FESEM micrographs (Fig. 3) illustrate the surfaces of LLDPE/PVOH/KNF composites after 3 and 6 months of soil burial exposure. It can be seen that the degradation on the surface of composites has become more intense with increasing KNF loading and soil burial time. Formation of cracks and greater number of pores were observed, due to more effective degradability (as shown in Fig. 3). LLDPE/PVOH matrix (Fig. 3a) shows that crack appears after 3 months of soil burial exposure and the degradation continues with the presence of small pores on the composites surface after 6 months of soil burial exposure. It can be seen

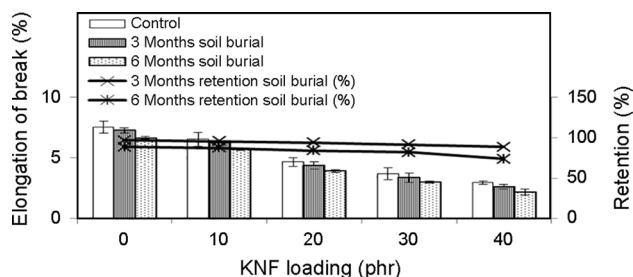


Fig. 2 Effect of 3 and 6 months of natural soil burial on the elongation-at-break of LLDPE/PVOH/KNF composites at different KNF loadings

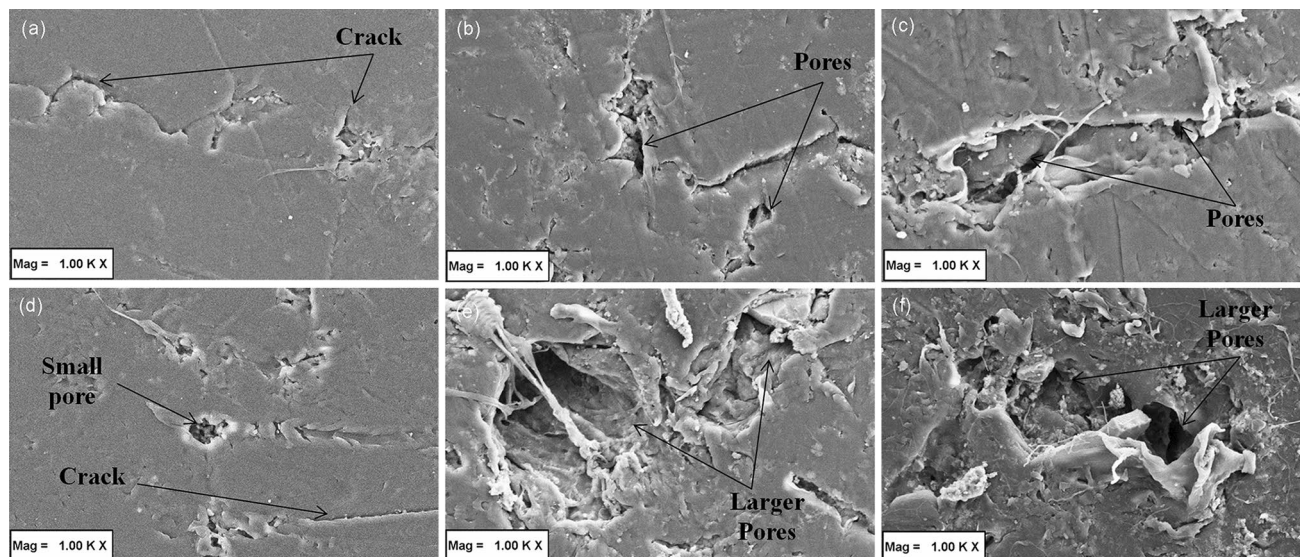


Fig. 3 FESEM micrographs of LLDPE/PVOH/KNF composites after 3 and 6 months of soil burial **a, d** 0 phr; **b, e** 10 phr; and **c, f** 40 phr KNF loading, respectively

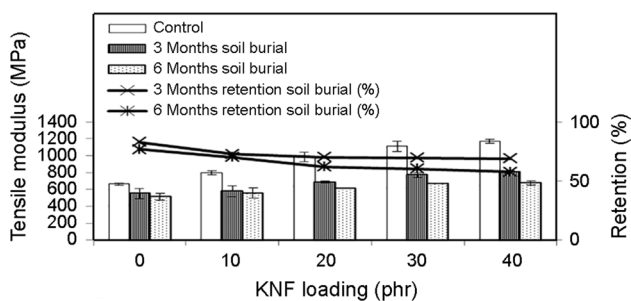


Fig. 4 Effect of 3 and 6 months of natural soil burial on the tensile modulus of LLDPE/PVOH/KNF composites at different KNF loadings

in Fig. 3e, f that the surface degradation has become more severe after 6 months compared to that after 3 months of soil burial exposure. This finding supported the lower retention of tensile strength as shown in Fig. 1. There was more and larger pores observed, especially for the composites with the highest KNF loading, i.e., 40 phr. It is obvious that KNF in the composites was seen from the very large pores formed after 6 months of soil burial exposure. Again, this confirmed the higher degradability of the composites after prolonged soil burial time.

Figure 4 shows the effect of 3 and 6 months of natural soil burial on the tensile modulus of LLDPE/PVOH/KNF composites at various KNF loadings. There was found an increasing trend in tensile modulus with increasing KNF loading. The increment was due to KNF which had stiffening effect on the composites [25]. Sam and Ismail [2] also found a similar trend in tensile modulus of linear low-density polyethylene/soya powder blends. Meanwhile, the tensile modulus was observed to decrease with increasing soil burial time. The decrement was due to the reduction in polymer chain mobility after the degradation by microorganism attack. The tensile modulus retention is lower for the composites after 6 months of soil burial exposure than the composites after 3 months of soil burial exposure (Fig. 4), once again indicating better degradability for the composites with longer soil burial exposure.

Weight loss

Figure 5 shows the effect of 3 and 6 months of natural soil burial on the weight loss of LLDPE/PVOH/KNF composites at various KNF loadings. The degradation of composites can be determined by measuring their weight loss after certain condition and period. According to Sam et al. [22], the direct way to examine the polymer biodegradability was to measure the weight loss of the degraded composites. From Fig. 5, the weight loss percentage of the composites increased with increasing KNF loading. The result is in agreement with the tensile test discussed earlier. The

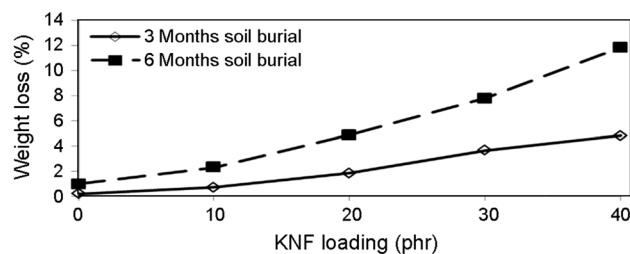


Fig. 5 Effect of 3 and 6 months of natural soil burial on the weight loss of LLDPE/PVOH/KNF composites at different KNF loadings

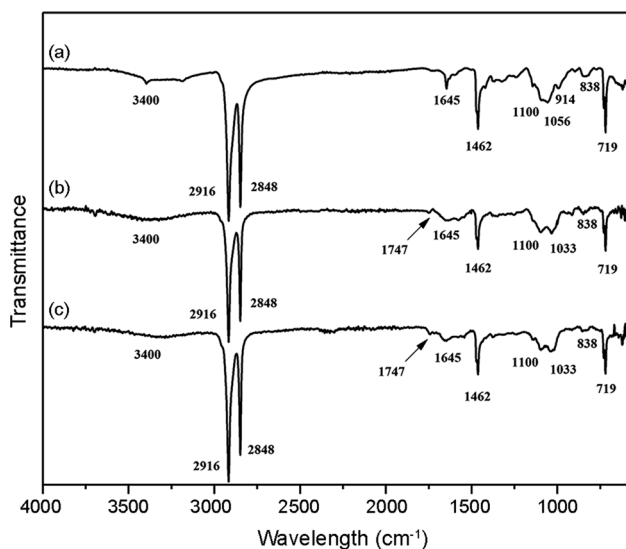
degradation of the composites is faster than that of the LLDPE/PVOH matrices, indicating the possibility of KNF leaching from the composites upon interaction with the moisture in soil. However, the extent of degradation of the composites is less, where the microorganism attack only takes place on the surface of the composites and penetration of oxygen through soil environment is limited [2, 19]. Figure 5 also shows that the weight loss percentage is higher for the composites after 6 months of soil burial exposure compared to the composites after 3 months of exposure, at all KNF loadings. This is due to the attack of microorganism which is extended to bigger surface area of the composites with prolonged soil burial time.

Differential scanning calorimetry (DSC)

Table 2 shows the DSC results of LLDPE/PVOH/KNF composites with various KNF loadings, after 3 and 6 months of soil burial exposure. The heat of fusion (ΔH_f^0) of the fully crystalline LLDPE was 290 J/g [1]. Based on Table 2, the melting temperature (T_m) did not significantly change with increasing KNF loading and soil burial time. This explains that there is no new crystalline phase formation during degradation [21, 22]. Table 2 also depicts the decrement in crystalline temperature (T_c) and crystallinity (X_c) of the composites with increasing KNF loading. This is due to the addition of KNF which has split polymer chains of LLDPE/PVOH matrices. Therefore, higher KNF loadings tend to provide more disordered arrangement of polymer chains and, consequently, the decrement in composite crystallinity [26, 27]. Sam et al. [26] also reported that the addition of soya powder hindered the crystallization of LLDPE during cooling. On the other hand, the T_c and X_c were found to be increased for all KNF loadings with prolonging soil burial time. This is because of the interaction of KNF and PVOH with the moisture in soil, and subsequently leaching out from the composites and formation of pores. The increase in crystallinity of natural filler–polymer composites after soil burial degradation test was in agreement with the results obtained by Yaacob et al. [21] and Sam et al. [22].

Table 2 DSC results of LLDPE/PVOH/KNF composites with different KNF loadings, after 3 and 6 months soil burial exposure

KNF loading (phr)	T_m (°C)		T_c (°C)		H_f^* (J/g)		Crystallinity, X_c (%)	
	3 months	6 months	3 months	6 months	3 months	6 months	3 months	6 months
0 KNF	125.5	125.4	107.1	107.5	41.1	42.2	23.6	24.3
10 KNF	125.7	125.1	107.0	107.3	28.4	33.2	18.0	21.0
20 KNF	125.3	125.2	107.0	107.2	24.0	29.9	16.6	20.6
30 KNF	125.5	125.3	107.0	107.0	21.8	27.0	16.3	20.2
40 KNF	125.5	125.1	106.9	107.0	17.9	24.8	14.4	19.9

**Fig. 6** FTIR spectra of **a** 0 month, **b** after 3 months; and **c** after 6 months of soil burial exposure of LLDPE/PVOH/KNF composites at 40 phr KNF loading

Fourier transform infrared (FTIR) analysis

Figure 6 illustrates the FTIR spectra of LLDPE/PVOH/KNF composites at 40 phr KNF loading before and after 3 and 6 months of soil burial, respectively. The FTIR spectrum of the control sample (Fig. 6a) shows the characteristic peaks of hydroxyl (O–H) stretching (3400/cm), CH bending (2916/cm), CH₂ stretching (2848/cm), O–H bending of adsorbed water (1645/cm), CH₂ bending (1462/cm), C–O stretching (1056/cm), C–OH stretching (914/cm), CH vibration of cellulose (838/cm), and C–C stretching vibration of CH₂ groups (719/cm) [22, 27, 28]. Referring to Fig. 6b, c, the characteristic peaks within the range of 1700–1750/cm (carbonyl group) are spotted after soil burial exposure. Moreover, the peak intensity of this carbonyl group, which was found to be increased for the composites after 6 months of soil burial (Fig. 6c), indicates that the degradability of the composites was higher than that of the composites after 3 months of soil burial. The formation of carbonyl groups after soil burial exposure confirmed that the degradation occurred

and the polymer chemical structure changed [22]. Similar findings on the formation of carbonyl peaks after degradation were reported by Muniandy et al. [19], Yaacob et al. [21], Sam et al. [22], and Sarifuddin et al. [28]. Furthermore, the intensity of spectral peaks that attributed to KNF (1645, 1100, 1056, 1033, 914, 838, and 719/cm) was found to be decreased as a function of soil burial time. These decrements in peak intensities could be an indicator of the reduction of KNF fraction upon degradation. Onggo and Pujiastuti [27] also reported a similar finding, where the spectral feature intensity (1015–1059/cm) of KNF was decreased as a function of weathering time. According to Moriana et al. [29], the decrease in intensity and broader fingerprint peaks (1500–600/cm) of degraded materials with respect to non-degraded materials was assigned to chemical structure decay.

Conclusion

From this investigation it was concluded that the LLDPE/PVOH/KNF composites were able to degrade in natural soil burial medium. The results showed that the addition of KNF and prolonged soil burial time accelerated the degradation of the composites. The tensile properties of the composites decreased, while the weight loss and crystallinity percentage increased with prolonging the soil burial exposure time. In addition, the FESEM results revealed composite degradation by the appearance of crack and pores in composite surface. The FTIR results confirmed the degradation of the composites by the emerging carbonyl group absorption.

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