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COMPARISON STUDY OF THE BIO-DEGRADATION PROPERTY OF POLYLACTIC ACID (PLA) GREEN COMPOSITES REINFORCED BY KENAF FIBERS

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ABSTRACT

Degradation of polylactic acid (PLA) was studied to investigate the best method of degradation to assist in reducing environmental pollution. Two condition of testing were conducted, which are natural weathering and landfill burial test. The rate of degradation was determined by weight loss, which was calculated once a month for six months. Natural weathering was achieved by exposing the samples to the natural environment, and this method was adopted according to ASTM D1435. Landfill burial testing was conducted by adopting ASTM G160-12 with a few modifications. The result shows that degradation was faster in a landfill burial condition. This was determined by the calculated weight loss, which was 2–4% of the total weight loss of PLA and its composites after six months' exposure to a natural weathering environment. In contrast, the landfill burial condition showed 4–17% weight loss after six months. The addition of natural fiber, whether bast or core fiber, assisted in the degradation of the composites. As well, the addition of natural fiber led to a 0.3–1.3% higher weight loss among the composites compared to neat PLA following natural weathering, while an 11–13% weight loss was recorded for composites exposed to the landfill burial condition. Natural weathering and landfill burial testing provide a time frame for the degradation of composite products. This is a good information for commercial composting facilities, providing data on the time frame required for material biodegradation.

Keywords: Biodegradation; Kenaf fiber; Landfill burial; Natural weathering; Polylactic acid

1. INTRODUCTION

In recent decades, the effects of globalization have created an extremely competitive atmosphere in all aspects of society. However, this flourishing competition must consider the harmony and balance between human needs and the environment to create a sustainable future (Suwartha et al., 2017). Researchers have played a role in identifying potential biomaterials to replace fossil fuel-based products. Polylactic acid (PLA) is a degradable polyester that has been widely used in various applications. PLA is completely mineralized to CO_2, water, and a small amount of biomass after four to six weeks' exposure to a composting condition with a temperature of approximately 60°C (Lunt, 1998; Drumright et al., 2000; Farrel et al., 2001; Itavaara et al., 2002). The degradation of PLA was studied to investigate the best method of degradation to assist in reducing environmental pollution. Although it is degradable, the process of PLA degradation could take longer than expected. This is due to the thousands of polymer chains that must be

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broken before continuing the process. Zhang et al. (2008), Li and McCarthy (1999), Tokiva and Jarerat (2004), and Auras et al. (2004) studied and proved that PLA degradation was completed in aerobic (composting) or anaerobic (biomethanation) environments over a period of six months to five years.

PLA degradation was affected by various factors, including moisture, heat, lights, microorganisms, etc. Otherwise, the sizes of the samples were also an important factor, where a smaller size experienced easier degradation than larger samples due to a higher surface area exposure to microorganism attacks (Kunioka et al., 2006; Kale et al., 2007). In addition, types of filler also reflect the degradation rate, where natural fiber reinforcement and nanoclay could increase and fasten the rate of composite degradability due to the hydrophilic nature of the material.

Various research studies have been conducted to investigate the degradability of PLA composites. Rudnik and Briassoulis (2011a) studied the degradability of PLA film at various thicknesses, as determined through real composting in a Mediterranean condition and laboratory simulation setup. They found that both conditions assisted in the degradation of PLA film; however, the laboratory simulation setup has a higher rate of degradability due to the control temperature and pH, which are favorable for microorganism reproduction. Cadar et al. (2012) studied the degradation of commercially available PLA, and they synthesized PLA under a controlled composting condition. They found that PLA degradation was dependent on the lactic acid content. Larger amounts of lactic acid content speed up the degradation rate of the composites. Sikorska et al. (2012) studied the effect of recycled PLA on degradation properties. PLA degradation was achieved in a composting pile and in water at 70°C. They reported that the multi-processing of PLA did not affect the rate of degradation. The results obtained indicate that hydrolytic degradation occurs preferentially via random ester bond scission. Degradation within a composting pile was completed after 42 days of incubation, while degradation in water decreased continuously starting from the beginning of the process.

In this paper, the degradation of neat PLA and its composites was studied using natural weathering and the landfill burial method. Although PLA is one of the most degradable polymers, its degradation behavior could vary when exposed to different conditions. This study was conducted for six months, where the earliest testing period was the rainy season and the dry season occurred toward the end of this study. The weight loss and chemical changes of the composites were determined in terms of degradation properties. Morphological studies were conducted to observe the effect of weathering and landfill burial on the samples.

2. METHODS

2.1. Materials

Extrusion-grade PLA, ESUNTM, was supplied by Shenzen Bright China Industrial Co., Ltd., China, with a density of 1.31 g/cm^3. Kenaf bast and core fibers were collected from the Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia (UPM), Serdang. Chemical substances, such as sodium hydroxide (NaOH) and hydrochloric acid (HCl), were supplied by Merck Group Malaysia.

2.2. Production of the Composites

PLA and collected fibers (KBF and KCF) were dried before use. In addition, 2% w/w fiber loading was used for composite production. PLA and fibers were weighed and pre-mixed before being extruded. The extrudate was pelletized followed by compression molding at a temperature of 175°C for 8 min. Then, composites were left to cool before being cut for sample testing.

2.3. Degradable Testing

Degradable testing was conducted to measure the rate of degradation of neat PLA and PLA-kenaf green composite. Two different testing methods were used, including natural weathering and the landfill burial test. The rate of degradation was determined by the weight loss calculated once a month for six months. Samples were sized 63.5 mm × 12.7 mm × 3 mm.

2.3.1. Natural weathering

Natural weathering was conducted by exposing the samples to the natural environment, and the method was adopted according to ASTM D1435. However, a few modifications were made according to Danjaji et al. (2002). Samples were hung on a rack 100 cm above the ground, and the samples' distance from each other was approximately 4 cm. The rack was set up at a small area near the faculty. Samples were observed regularly to ensure the samples continued to hang on the rack and were spaced appropriately. Meteorological data during the testing period of air temperature, air moisture, and total precipitation were collected from the Malaysian Meteorological Department. Samples were left for six months to observe the natural weathering effect, and measurements were taken monthly. The setup of the natural weathering test is shown in Figure 1.

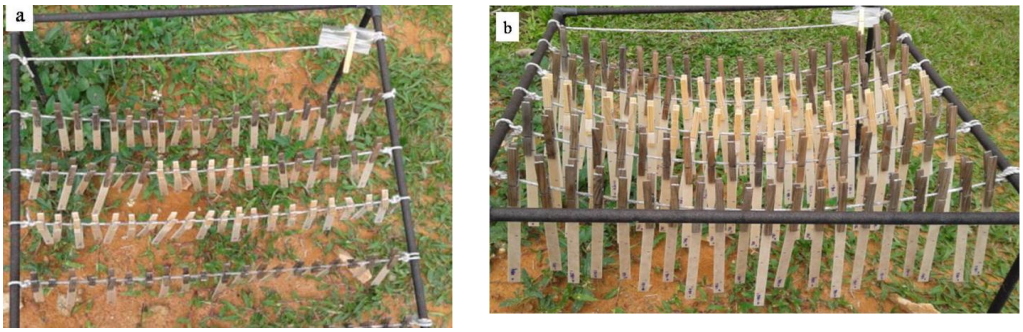

Figure 1 Natural weathering test setup: (a) Samples hanging vertically; (b) Arrangement of samples

2.3.2. Landfill burial testing

Landfill burial testing was conducted by adopting ASTM G160-12, and a few modifications were made following the method run by Rudnik and Briassoulis (2011a) and Fukushima et al. (2009). Samples were buried at a depth of 15 cm in a composting area of Universiti Teknologi MARA. An irrigation cycle was applied that consisted of one day of irrigation and one day of no irrigation. However, irrigation was interrupted during the raining period. The test setup is shown in Figure 2. Since this testing were conducted in nearby area of natural weathering testing similar meteorological data, such as air temperature, air moisture, and total precipitation during the testing period, were also collected from the Malaysian Meteorological Department. In addition, soil temperature, soil pH and soil moisture content was recorded by thermometer and soil pH and moisture meter at weekly period.

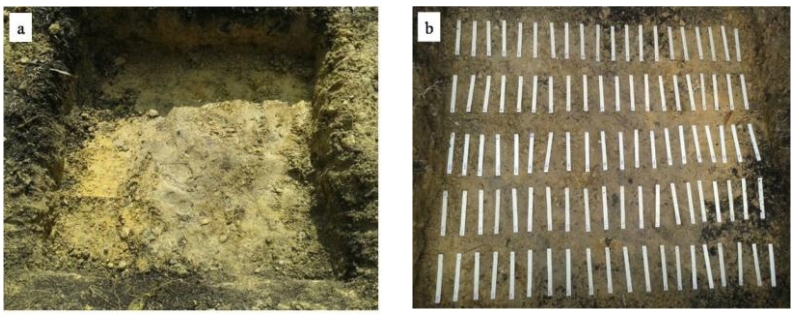

Figure 2 Landfill burial test setup: (a) Area of testing; (b) Arrangement of samples

3. RESULTS AND DISCUSSION

3.1. Natural Weathering Test

Figure 3 illustrates the air temperature and humidity for the testing period. Data were collected from the Malaysian Meteorological Department based on the nearest station in Subang, latitude 3°08' N and longitude 101°33' E. The testing period was from October to March. The first three months was the rainy season, while the last three months was the dry season, with no rain for a few months.

The below Figure 3 also shows that the humidity during the earlier testing period was approximately 90%, with temperatures ranging between 26–28°C. In contrast, the humidity for the last three months was nearly 60%. Meanwhile, the temperature during the latter period was also highest, reaching 29°C during the fifth month of the testing period.

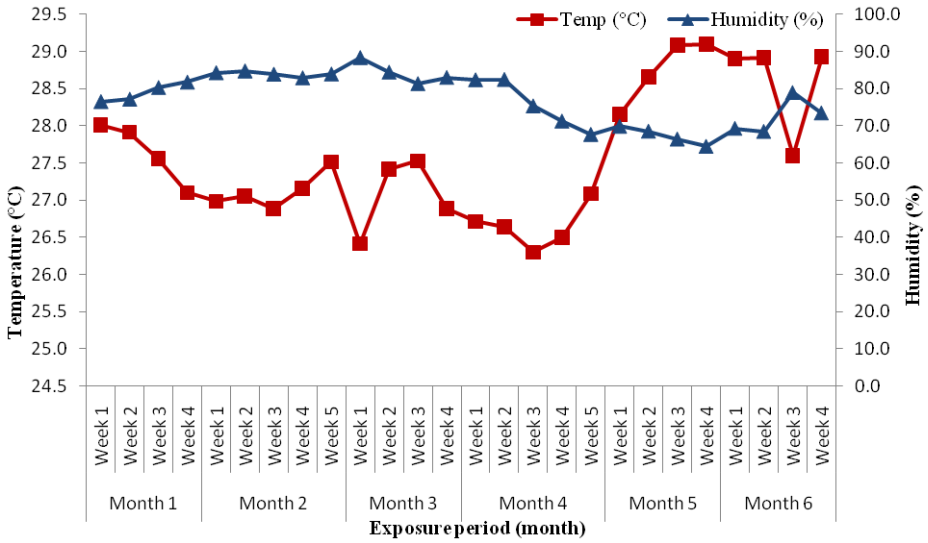

Figure 3 Air temperature and humidity during the testing period

3.1.1. Weight loss

Figure 4 shows the percentage of weight loss of kenaf bast composite (KBC) and kenaf core composite (KCC) with neat PLA as a control sample. The percentage of weight loss of the composites was higher than that of neat PLA, as was expected. The addition of natural fiber to the composites enhances the degradation of the composite. This is due to the cellulose content of kenaf fiber, leading to higher water absorption, which has a synergetic effect on the biodegradation rate, thus increasing the degradation rate (Yussuf et al., 2010). In addition, Wang et al. (2008) and Parra et al. (2006) explain that the disruption of polymer chains by the addition of fibers could accelerate the biodegradation of the composites.

The small percentage of weight loss in the second month was related to the breaking of thousands of polymer chains and an area of higher crystallinity caused by hydrolytic scission (Rudnik & Briassoulis, 2011a). The rate of weight loss increased gradually after the second month until the end of the testing period. However, the increment in the percentage of weight loss was small, with a mere 0.1% difference between each month. The small amount of weight loss was due to the samples' size, which was 3 mm in thickness. Kale et al. (2007), Kunioka et al. (2006), and Rudnik and Briassoulis (2011b) reported that the samples' size was an important factors, affecting the rate of degradation of the composites. Larger sample sizes require a longer degradation time than small sample sizes due to the presence of more chains to be degraded.

As shown in Figure 4, the total weight loss of neat PLA after the six-month testing period was the lowest, at only 2.49%, while KBC and KCC showed total weight losses of 3.79% and 2.78%,

respectively. In general, there is small gap between the total weight loss of neat PLA and its composites under the natural weathering condition. This result shows that neat PLA, KBC, and KCC were not significantly affected when exposed to the environment, even after six months' exposure, and this result reveals the possibility of using this material as an outdoor product.

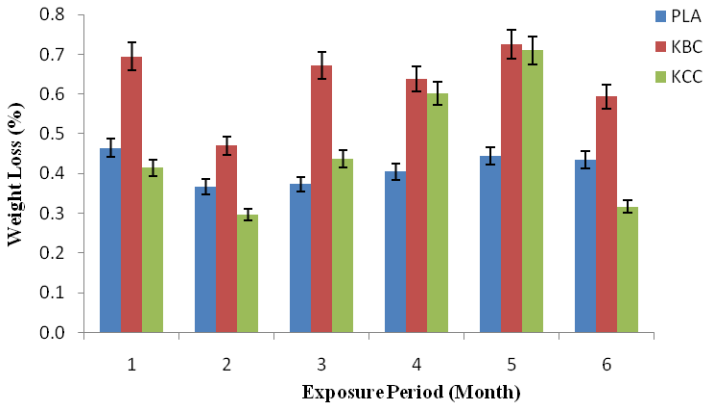

Figure 4 Weight loss of neat PLA, KBC, and KCC following the natural weathering test

3.1.2. Microscopic observation

Microscopic observations of the surface of naturally weathered samples are shown in Figure 5. The surfaces of control KBC, KCC, and neat PLA exhibit a smooth surface, as shown in Figures 5a, 5c, and 5e. Six months of exposure created voids and blisters across the full surfaces of KBC and KCC. The blister effect was clearly seen on KBC and KCC, as compared to neat PLA. Rather than blister on the surface, a small crack that pointed towards the centre of KBC and KCC was observed, exposing the fiber inside the composites, as shown in Figures 5b and 5d. This result was similar to that achieved by Danjaji et al. (2002), who showed that cracks were visible across the entire surface of the samples. However, no colour changes were visible on samples exposed to natural weathering. The surface of neat PLA after six months' exposure only shows a void beneath the surface (Figure 5b). The natural weathering condition reveals that Malaysian weather does not affect PLA composites. It shows beneficial results that PLA and its composites can be used as outdoor products, as degradation of this material will take longer, even when exposed to the weather.

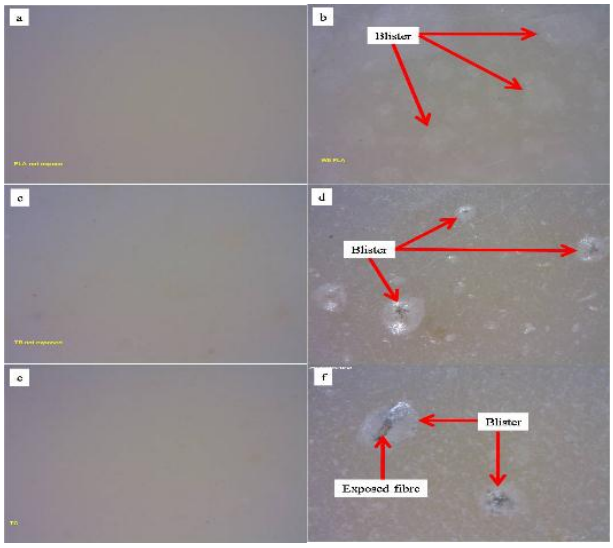

Figure 5 Microscopic observation of natural weathering samples: (a) PLA control; (b) PLA exposed; (c) KBC control; (d) KBC exposed; (e) KCC control; (f) KCC exposed

3.2. Landfill Burial Test

Figure 6 shows the soil temperature and pH for the testing period. Soil pH showed an inconsistent trend, ranging from 4–7 during the testing period. According to Andersson et al. (2010), the decrease in pH was due to the formation of acidic degradation products that migrated into the water. However, an increase in the soil pH up to 7 was due to a higher soil water content, which was determined by moisture meter to have neutralized the soil's acidity. In addition, the PLA degradation products were carbon dioxide, water, and humus (Fukushima et al., 2009). The soil water contents for October to March were 99%, 98%, 95%, 90%, 43%, and 77%, respectively, showing that the soil in the testing area was moist for the entire testing period.

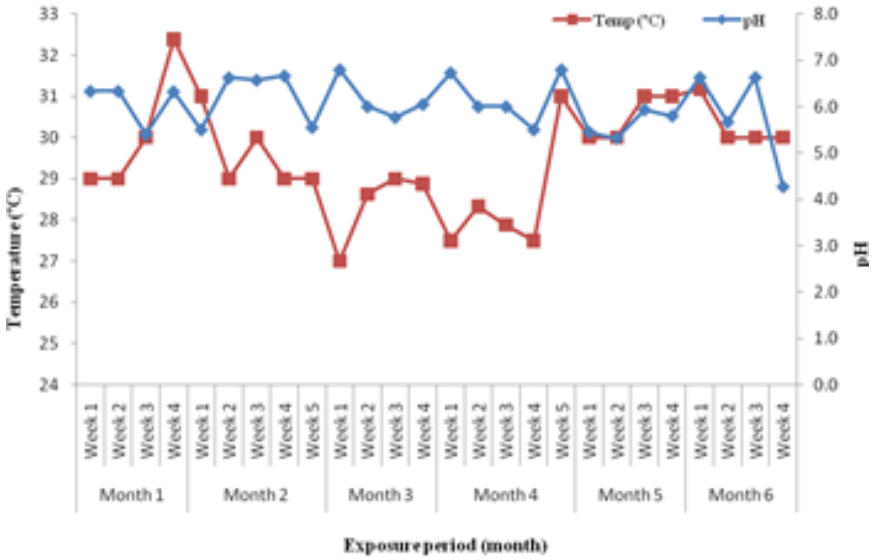

Figure 6 Soil temperature and pH during landfill burial testing

3.2.1. Weight loss

The percentages of weight loss of KBC and KCC following the landfill burial test are shown in Figure 7. The weight loss of neat PLA and its composites gradually increased from the first month of exposure until the sixth. The degradations of KBC and KCC were similar, gradually increasing with the time of exposure and leading to a higher weight loss than neat PLA. Although the weight loss of neat PLA was shown to be the lowest, it generally increased with the time of exposure. The greater weight loss of KBC and KCC indicates that the incorporation of natural fiber assisted in composite degradation, as cellulose fiber attracts microorganism attacks. Lu et al. (2004) explain that plant fibers are degraded into carbohydrates by numerous microorganisms existing in nature; for example, brown rot fungi can decompose cellulose and hemicellulose, while white rot fungi can decompose lignin (Gao & Tang, 1996).

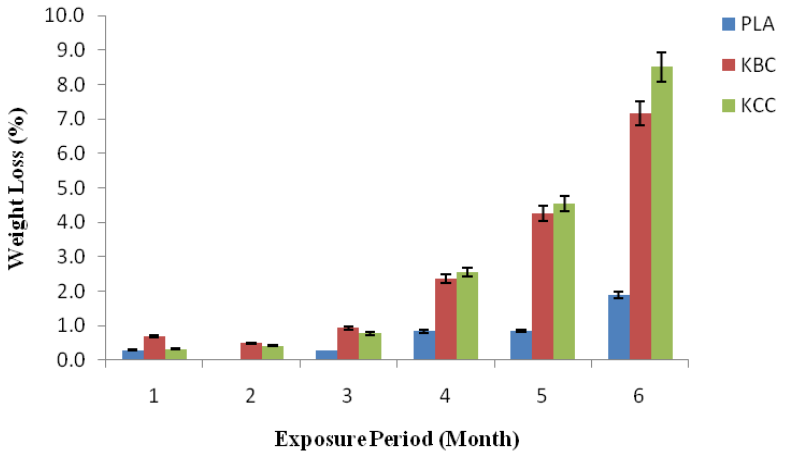

Figure 7 Weight loss of neat PLA, KBC, and KCC following the landfill burial test

Earlier stages of exposure reveal lower weight loss for all samples. Rudnik and Braissoulis (2011a) explained that the primary mechanism of PLA degradation was hydrolysis, catalyzed by temperature, followed by a bacterial attack on the fragmented residues. Fukushima et al. (2009) explain further that the degradation of composites under soil occurs by two process: (i) the hydrolysis of a high molecular weight into a lower molecular weight chain that was accelerated by an acid or base condition associated with temperature and moisture; and (ii) microorganism catalysis of degradation by hydrolytic scission of the ester group into acid and alcohol, which finally converts a low molecular chain into carbon dioxide, water, and humus. Cadar et al. (2012) reported that a lower degradation during an earlier stage of exposure indicates that the hydrolytic degradation process is causing a decrease in molecular weight. PLA is slowly degraded in ambient temperature soils, which is probably due to the slow rate of hydrolysis at low temperatures, water content, and the relative scarcity of a PLA-degrading organism (Pranamuda et al., 1997; Ho et al., 1999; Shogren et al., 2003; Rudnik & Braissoulis, 2011a).

As shown n Figure 6, soil temperature varied from 27–33°C, while soil water content ranged from 43–99%. Rudnik and Briassoulis (2011a) reported that PLA degradation is favorable at a temperature around 60°C. In addition, Fukushima et al. (2009) suggested that a lower weight loss might be due to the adherence of degradation products to the surface of the composites. They also mention that degradation activity was proceeding from the interior part, but the diffusion rate of product degradation was relatively slow. Kale et al. (2007) explained that the slower degradation rate for a real soil burial was due to the slight reduction in oxygen available in the soil.

As shown in Figure 7, a higher weight loss was recorded for composites than neat PLA. After six months of exposure, neat PLA had a weight loss of only 4.14%, while KBC and KCC recorded 15.89% and 17.09% weight losses, respectively. Although small amounts of fiber (2%) were incorporated into the PLA, the weight loss was improved at 11.75% and 12.95% for KBC and KCC, respectively. It shows that natural fiber attracted a microorganism attack that caused faster degradation of the composites compared to neat PLA (Yussuf et al., 2010). It is expected that higher fiber loading will boost composite degradation.

3.2.2. Microscopic observation

Figure 8 shows a comparison of the control samples and the samples after six months buried in a landfill. Control KBC, KCC, and neat PLA exhibited smooth surfaces before testing, but six months of being buried in a landfill showed colour changes and surface deformation on all samples of KBC, KCC, and neat PLA. The whitening effect indicates the beginning of hydrolytic degradation of the polymer chain that changed the refraction index of the samples due to water absorption and the presence of byproducts of the hydrolytic process (Li & McCarthy, 1999; Liu

et al., 2000; Li et al., 2001). The whitening effect on the exposed composites was observed even after one month of exposure. Shogren et al. (2003) indicated that colour changes were due to the beginning of microbial growth on the samples, which led to degradation of the samples.

In addition, cracks were present on the surface of the neat PLA, which indicated a microorganism attack on the samples. This was obvious when kenaf fiber reinforced PLA, where the fibers acted to attract microorganisms. Similar results were revealed by Cho et al. (2011), where a rougher surface and many holes were formed on the surface of neat PLA and its composites, and the diameter of the holes was increased as the degradation time increased, which indicated pronounced degradation.

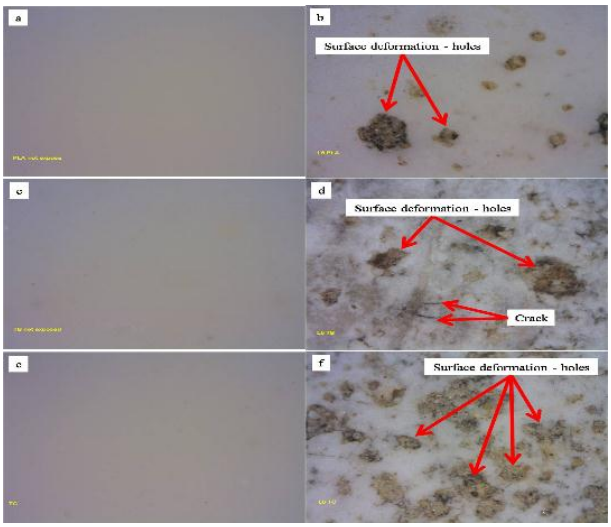

Figure 8 Microscopic observation of landfill burial samples: (a) PLA control; (b) PLA exposed; (c) KBC control; (d) KBC exposed; (e) KCC control; (f) KCC exposed

There were also black spot present around the cracks, which shows evidence of fungi attacking the samples. A heavy fungal attack was due to exposure to optimum temperature and moisture conditions that favour the growth of fungi with the presence of lignocellulosic materials (Abdul Khalil & Ismail, 2001). Yussuf et al. (2010) added that cellulose content leads to higher water absorption, which would increase the rate of degradation. Additionally, under real soil conditions, plant roots and worms could contribute to the mechanical fragmentation and disintegration of the samples (Rudnik & Briassoulis, 2011a; Rudnik & Briassoulis, 2011b).

The presence of surface cracks and black spots on the surface of KBC, KCC, and neat PLA led to a reduction in composite strength. Although no mechanical testing was conducted, while handling the samples, their brittleness increased with an increase in how long degradation was applied. Samples became soft and broke with even small forces applied. This result is similar to Rudnik and Briassoulis (2011a), where they reported the PLA film was brittle even after one month of soil burial.

Referring to Figure 7, neat PLA, KBC, and KCC were able to degrade under this soil condition. However, the rate was slower than reported by other researchers due to the lower temperature and lack of degradation microorganisms in the real soil condition as compared to the composting condition (Yussuf et al., 2010; Rudnik & Braissoulis, 2011a). Moreover, sample thickness influenced the biodegradation rate of the samples (Starnecker & Menner, 1996; Yang et al., 2005). This result was quite similar to research done by Yussuf et al. (2010), where a slower rate of degradation was observed at early exposure, increasing as the degradation time increased.

4. CONCLUSION

PLA-kenaf green composite degradation was determined using two different tests, which are natural weathering and the landfill burial test. Both conditions show degradation of the samples occurred, but PLA-kenaf green composite degradation was faster in a landfill burial condition. This was determined by the calculated weight loss, which is approximately 2–4% of the total weight loss of PLA and its composites after six months' exposure to a natural weathering environment. In contrast, the landfill burial condition shows 4–17% weight loss after six months buried in a landfill. The addition of natural fiber, whether bast or core fiber, assisted in the degradation of the composites. As well, the addition of natural fiber led to a 0.3–1.3% higher weight loss among composites, compared to neat PLA in a natural weathering condition. Meanwhile, an 11–13% higher weight loss was recorded for composites exposed to the landfill burial condition than for neat PLA. The addition of natural fibers acts to attract microorganisms and increase water absorption, which assist in the hydrolysis of polymer and enhance the degradation rate of the composites.

5. ACKNOWLEDGEMENT

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