DEGRADATION OF OIL PALM EMPTY FRUIT BUNCHES (OPEFB) FIBRE DURING COMPOSTING PROCESS USING IN-VESSEL COMPOSTER

Wan Aizuddin Wan Razali,[a] Azhari Samsu Baharuddin,[a,*] Ahmad TarmezeeTalib,[a] Alawi Sulaiman,[c] M. Nazli Naim,[a] Mohd. Ali Hassan,[b] and Yoshihito Shirai [d]

Changes in the lignocellulosic structure of oil palm empty fruit bunches (OPEFB) during composting treatment using an in-vessel composter was investigated in this work. The composting process was completed within 40 days of treatment, and the final C/N ratio achieved was 13.85. Scanning electron microscopy (SEM) revealed that the structure of OPEFB material was severely degraded, especially during the thermophilic phase where the biodegradation process was most active. Close examination of the physicochemical and thermal analysis using X-ray diffraction (XRD), Fourier transform infrared (FTIR), and thermogravimetric and differential thermal analysis (TG/DTA) showed that the crystallinity size of the OPEFB structure decreased. This result was attributed to the removal of silica bodies from OPEFB materials. Also, the functional groups of cellulose, hemicelluloses, and lignin structures had changed throughout the composting period, and the most extensive degradation of cellulose was detected in the thermophilic phase. It was also found that the exothermic peak of the matured compost reduced most significantly compared to the raw OPEFB. In conclusion, the in-vessel composting system was able to enhance the degradation process of OPEFB materials for producing compost.

Contact information: a: Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia, MALAYSIA; b: Department of Bioprocess Technology, Faculty of Biotechnology and Biomolecular Sciences, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia, MALAYSIA; c: Faculty of Plantation and Agrotechnology, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia; d: Department of Biological Functions and Engineering, Graduate School of Life Science and System Engineering, Kyushu Institute of Technology, 2-4 Hibikino, Wakamatsu-ku, Kitakyushu, Fukuoka 808-0916, JAPAN;
* Corresponding author: azharis@eng.upm.edu.my

INTRODUCTION

At present there are 4.69 million hectares of land planted with oil palm in Malaysia, yielding millions of tonnes of oil palm fresh fruit bunches each year (MPOB 2009). The oil fraction in the oil palm fruits is usually in the range 18 to 22%, and the remaining being moisture and oil palm biomass such as oil palm mesocarpfibre (MF), palm kernel shell (PKS), and empty fruit bunches (OPEFB) are available for further exploitation. According to Tan *et al.* (2010), Malaysia is currently the second largest palm oil producer, with 406 currently active palm oil mills and a total annual processing

capacity of 92.78 million tonnes of oil palm fresh fruit bunches (OPFFB). It was reported that the breakdown of components in OPFFB is 21% palm oil, 7% palm kernel, 14% mesocarp fiber, 7% palm kernel shell, and 23% OPEFB (Omar *et al.* 2011). Therefore it is obvious why huge amounts of OPEFB are produced by the industry annually. With increasing demand for palm oil worldwide, the amount of oil palm biomass available is expected to grow, and composting is one of the best approaches to solve the abundance of this waste.

Composting is an aerobic biological process that converts organic materials into a more stable material with a lower content of degradable organic matter, thus giving rise to less phytotoxicity toward plants (Zhu 2006; Xiao *et al.* 2009). Much research has been successfully performed on oil palm biomass, such as co-composting OPEFB with partially treated palm oil mill effluent (POME) (Baharuddin *et al.* 2009), OPEFB with sewage sludge (Kala *et al.* 2009), OPEFB with palm oil mill decanter sludge and chicken manure (Kananam *et al.* 2011), palm oil mill sludge (POMS) with sawdust (Singh *et al.* 2010), and OPEFB with goat manure, cattle manure, and chicken manure (Singh *et al.* 2010). Despite that success, in another study, composting OPEFB has faced the difficulty of a low degradation rate due to the rigid structures of cellulose, hemicelluloses, and lignin in the OPEFB material (Kananam *et al.* 2011). OPEFB is a fibrous material, having a 40 to 50% moisture content (Singh *et al.* 2010). Historically, oil palm biomass such as OPEFB has been well known for its difficulty to degrade and therefore has been preferred for mulching in oil palm plantations or burning to obtain its fertile ash (Singh *et al.* 2010; Bahrain *et al.* 2012). Due to the nature of the palm oil processing, POME is also produced in huge quantities because the ratio of water requirement to the fresh fruit bunch (FFB) processed is usually 1:1. POME is a colloidal suspension of thick brownish liquids, which is comprised of 95 to 96% water, 0.6 to 0.7% oil and grease, and 4 to 5% total solids (Sulaiman *et al.* 2009; Hock *et al.* 2009). Standard treatment for POME is by anaerobic digestion in open ponds due to lower capital and operating costs. Moreover, the treated anaerobic sludge can be easily recovered and dried for fertilizer (Singh *et al.* 2010). The biogas produced from this technology can be further polished and used to generate electric power through the clean development mechanism (CDM) project, as previously reported (Sulaiman *et al.* 2009). In a previous study, treated anaerobic sludge was successfully added to OPEFB to enhance the composting process (Baharuddin *et al.* 2010).

There are four types of composting that have been widely applied in the industry, namely open static piles, turned windrows and piles, aerated static piles, and in-vessel systems (Hubbe *et al.* 2010). For special requirements, a furrow system and a windrow pile system have also been applied to OPEFB and POME sludge composting processes (Baharuddin *et al.* 2009, 2010; Yahya *et al.* 2010). However, for these systems, a longer composting period was required in addition to difficulties faced in controlling important parameters such as moisture content, temperature, and oxygen level (Xiao *et al.* 2009). On the contrary, an in-vessel composting system has many advantages over these systems because it requires less space and provides better control for agitation, aeration, and mixing of the compost materials (Kim *et al.* 2007). Usually for in-vessel composting systems, two phases are involved, which are a high-rate phase in the vessel composter and a curing phase in an exterior composting pile (Singh *et al.* 2009). However, up to this

point, there has been no reported study on composting OPEFB and POME anaerobic sludge using an in-vessel composting system for field scale operation. Since the composting processes involve changes of chemical composition, several techniques have been used to determine the stability of compost such as X-ray diffraction (XRD), Fourier transform infrared (FTIR) and thermogravimetric, and different thermal analysis (TG/DTA) (Baharuddin *et al.* 2011; Baffi *et al.* 2007). Therefore, this study was conducted to evaluate the changes in the lignocellulosic structure of OPEFB during composting by using an in-vessel composting system on a semi-commercial scale.

EXPERIMENTAL

Raw Materials

The pressed-shredded OPEFB was obtained from Seri Ulu Langat Palm Oil Mill, (Dengkil, Selangor, Malaysia) in the size range 15 to 20 cm. Two tonnes of the mentioned OPEFB were used in this study.

Seed Sludge/Microbial Seeding

The thickened POME anaerobic sludge was obtained from the closed anaerobic digester located at FELDA Serting Hilir Palm Oil Mill, Negeri Sembilan, Malaysia. The POME anaerobic sludge was collected from the bottom outlet of the settling tank attached to the digester. Six tonnes of POME anaerobic sludge were added to the OPEFB material throughout the composting process.
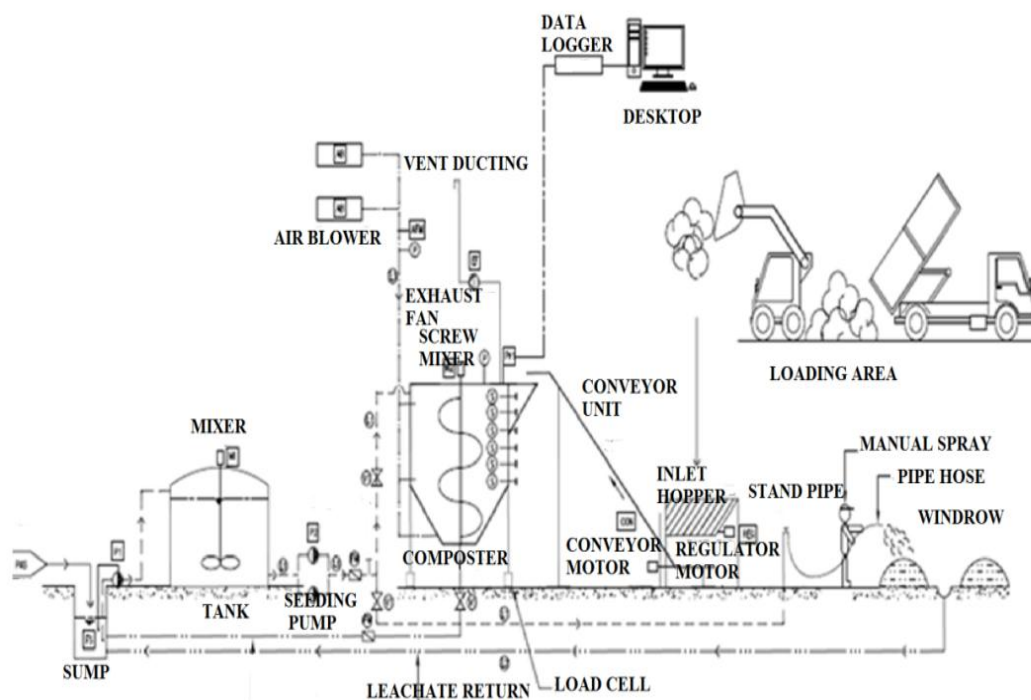

Fig. 1. Schematic diagram of the in-vessel composting system

In-Vessel Composting System Operation

This study was conducted for 5 months using a semi-commercial in-vessel composter located in Universiti Putra Malaysia (UPM), Serdang Selangor from the 1st of November, 2010 until the 10th of April, 2011. A schematic diagram of the system is shown in Fig. 1. There were six main components attached to the composter, namely OPEFB feeding, compost product discharge, materials mixing device, leachate collection, aeration, and carbon dioxide (CO_2) removal. The dimension of the composter was 8 meters in height by 3.4 meters in diameter. The effective volume of the composter was 10 cubic meters.

For the start-up operation, the OPEFB materials were fed into the composter through the hopper using a conveyor belt. At the same time, the mixing device was turned on to ensure thorough mixing. The POME anaerobic sludge was then added gradually using a pump by distributing it evenly at the top of the composter. The leachate run-off was collected in a sump and recycled to the storage tank. For aeration, air was introduced into the vessel composter using air diffusers attached at the interior wall located at different positions.

Sampling and Analysis

OPEFB samples for physicochemical and microbiological analysis were collected daily from the sampling ports attached to the composter and stored at -20°C prior to analysis. Changes of texture and colour were physically observed and recorded. Temperature and oxygen concentration in the composter were measured using a portable temperature and oxygen detector manufactured by Demista Instrument USA (Model CM2006, USA). Moisture content was determined by drying 1g of sample at 120°C until a constant weight was achieved using an AND MX90 Moisture Analyzer (MX90, Japan), and the pH value was measured using a pH meter (Model DELTA 320, Mettler Toledo, USA).

Biological oxygen demand (BOD), chemical oxygen demand (COD), total solids, total suspended solids, and volatile solids were determined according to the standard methods (APHA 2005). Carbon and nitrogen were analyzed using a CHNS Analyzer (Leeco, USA), and nutrients and heavy metal elements were analyzed using inductively coupled plasma (ICP)-OES (Perkin Elmer, USA). Viable microbial count was determined using the plate counting methodas proposed by Brock and Madigan (1991). Acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL) methods were used to determine the cellulose, hemicellulose, and lignin contents of OPEFBand POME anaerobic sludge as proposed by Goering and Van Soest (1970).

Scanning Electron Microscopy

The morphological structure of the compost sample was observed using scanning electron microscope (SEM) (S-3400N, Hitachi, Japan). SEM images of all the samples were taken at 500x magnifications for analysis.

X-Ray Diffraction Analysis

It is important to confirm the crystallinity and composition of OPEFB during the degradation process. Measurements of wide angle X-ray diffraction of OPEFB samples

were made using a Rigaku XRD-DSC-X II diffraction system. Cu-*Kα* radiation (0.15418 nm) was used as radiation source, and 2θ angles were recorded using a scintillation counter, in the 2θ interval between 10 and 40°C. The crystallinity of SiO_2 pattern was estimated using the Scherrer equation ($L = K\lambda/\beta\cos\theta$), where λ is wavelength of X-ray, β is the full width at half maximum, and θ is the Bragg angle). The value of β was estimated by fitting the XRD pattern obtained from analysed result.

Fourier Transform Infrared Analysis

Fourier transform infrared (FTIR) equipment (Perkin Elmer GX2000, USA) was used to quantify the chemical bonds between the functional group and OPEFB structure. The tested samples were subjected to the wave numbers within range 500 to 4000 cm^{-1} at a resolution of 4 cm^{-1}.

Thermogravimetric and Differential Thermal Analysis

Thermal behavior of the composting samples was analysed using a simultaneous thermal analyzer EXTAR 6200 TG/DTA system (Seiko instruments Inc., Japan) under a constant nitrogen flow (100 mL/min^{-1}). The thermogravimetry (TG)/DTA temperature was set in the range of 25 to 500 °C to investigate the mass loss of the samples at a heating rate of 9 °C/min^{-1}. Weight loss and heating rate were recorded continuously.

RESULTS AND DISCUSSION

Table 1 shows the characteristics of press-shredded OPEFB, POME anaerobic sludge, and the OPEFB material at the end of the composting period. According to Bernal *et al.* (2009), simple organic compounds such as carbohydrates, fat, and amino acids will degrade faster in the thermophilic phase of the composting process than the more resistant organic compounds such as cellulose, hemicelluloses, and lignin. Since OPEFB is a lignocellulosic-based material, a longer degradation time is needed to degrade the materials as observed in this study. It was also observed that the color of the OPEFB changed from dark yellow initially to slightly blackish, and the final texture turned soil-like with an earthy smell.

The final nutrient contents in terms of N, P, and K were measured and stabilized at 2.8%, 0.4%, and 2.8%, respectively. The nutrient results achieved were satisfactory, since according to Rihani *et al.* (2010), the N, P, and K values should be more than 1% in order to provide enough nutrients for plant growth. It was also discovered that the final matured compost had a low content of heavy metals; lead (0.5 mg/kg), chromium (21.9 mg/kg), and no cadmium and nickel were detected. This indicates that the final compost was safe to be used as a soil amendment and fertilizer. It was reported earlier that high concentrations of heavy metals can penetrate into the soil and may affect plant health and growth, soil properties and microorganisms, livestock, and also human health (Rihani *et al.* 2010).

Table 1. Characteristics of Pressed-Shredded OPEFB, POME Anaerobic Sludge Used in this Study and Properties of Compost at Day 40

Parameters	Value		
	OPEFB	POME Anaerobic Sludge	Final Compost
Moisture (%)	44.9	95.0	49.3
pH	5.9	7.4	7.5
C (%)	44.1	31.5	38.5
N (%)	0.6	4.7	2.8
C/N	77.7	6.7	13.8
Total solid (mg/kg)	-	51750.0	-
Suspended solid (mg/kg)	-	46830.0	-
Volatile solid (mg/kg)	-	30820.0	-
COD (mg/L)	-	60500.0	-
BOD (mg/L)		18100.0	-
Cellulose (%)	37.3	1.7	26.2
Hemicellulose (%)	27.8	5.6	15.4
Lignin (%)	16.5	53.1	33.3
Ash (%)	5.8	37.3	17.8
Compositions of Nutrients and Metal Elements			
Phosphorus (%)	0.1	0.8	0.4
Potassium (%)	1.4	4.0	2.8
Calcium (%)	0.2	1.9	0.8
Sulphur (%)	0.1	1.2	0.5
Magnesium (%)	0.1	1.2	0.5
Zinc (mg/kg)	22.4	122.8	68.0
Manganese (mg/kg)	26.4	333.6	160.3
Iron (%)	0.1	1.1	0.6
Copper (mg/kg)	19.6	142.9	49.0
Boron (mg/kg)	1.8	60.9	25.1
Cadmium (mg/kg)	N.D.	N.D.	N.D.
Chromium (mg/kg)	39.1	10.6	21.9
Plumbum (mg/kg)	0.2	1.2	0.5
Nickel (mg/kg)	N.D.	N.D.	N.D.

N.D.-Not Detectable

Composting Profiles of OPEFB

Temperature and bacterial count

Composting profiles for the temperature and total bacterial population measured in the in-vessel composter and windrow pile are shown in Fig. 2. In the figure, it is clearly shown that at the initial stage of the composting process, the temperature rose rapidly from 30 to 57 °C in less than 12 hours and thereafter maintained the thermophilic condition (60 to 70 °C) until day 7 in the in-vessel composter. After day 7, the temperature gradually reduced to a lower range and ended at 60 °C at the end of day 10. Once the thermophilic stage was completed, the OPEFB materials were unloaded and left to cure in the windrow. The temperature gradually decreased to 50 to 60 °C when introduced to the windrow pile and continued to drop to equal the surrounding temperature at the end of the composting period. The fluctuation of the temperature recorded throughout the composting process could not be avoided because of the POME

anaerobic sludge addition and mechanical turning effects. Tumuhairwe *et al.* (2009) reported that turning compost can cause a significant reduction of temperature build-up due to the heat transfer problem. A similar phenomenon was reported by Kalamdhad *et al.* (2009a, b) and Kumar *et al.* (2010) using an in-vessel composting system.

Initially, the rapid increase of temperature was attributed to the high population of bacteria (shown in Fig. 2), which is desirable for pathogen destruction. Bernard *et al.* (2009) and Jauraphy *et al.* (2005) reported that pathogens can be killed at 55 °C. Furthermore, only a few days are required to eliminate almost all pathogens, making the compost product safe for agricultural use (Hachicha *et al.* 2009). According to Hachicha *et al.* (2009), the rapid increase of temperature during the thermophilic phase is caused by the rapid breakdown of the available organic and nitrogenous compounds by microorganisms. However, as the organic matter stabilized, the microbial activity and the rate of organic matter decomposition became slower. Finally, a stable temperature was reached as less decomposition occurred.
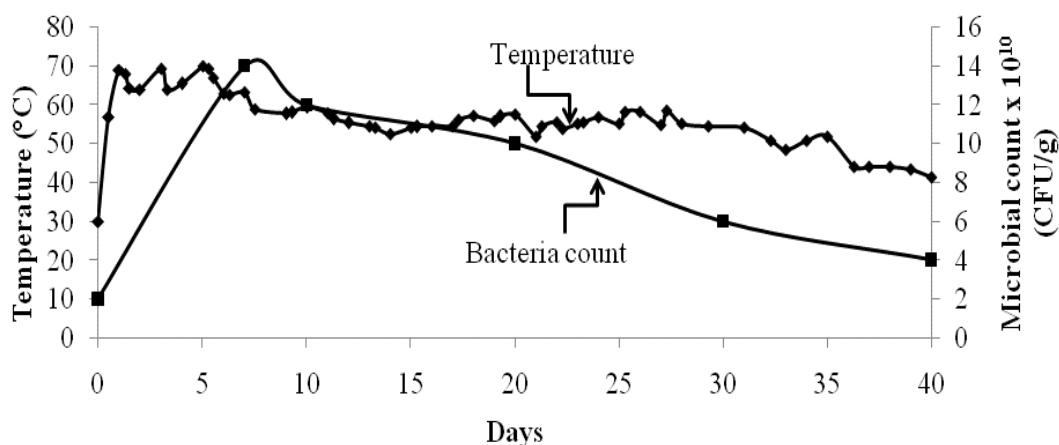

Fig. 2. Profiles for temperature and total bacterial population during the composting process

Moisture content, oxygen, and pH

Figure 3 shows the profiles for moisture, pH, and oxygen level throughout the composting period of the OPEFB materials. The arrows show the days at which POME anaerobic sludge was added for moisture, nutrient, and microbial supplementation. At the beginning of the composting process (days 1 to 7), the process was thermophilic, and thus high moisture evaporation occurred in the system. Therefore, a proactive measure was taken to supplement the moisture loss by adding POME anaerobic sludge into the composter on a more frequent basis (8 times), and the moisture content was stablised in the range 45 to 65%.

Once the temperature had dropped after day 7, less moisture evaporation occurred, and therefore the frequency of POME anaerobic sludge addition was reduced to only 12 times for the next 23 days. This strategy was successful, whereby the moisture content was recorded satisfactory in the range 45 to 68% until the end of the composting process.
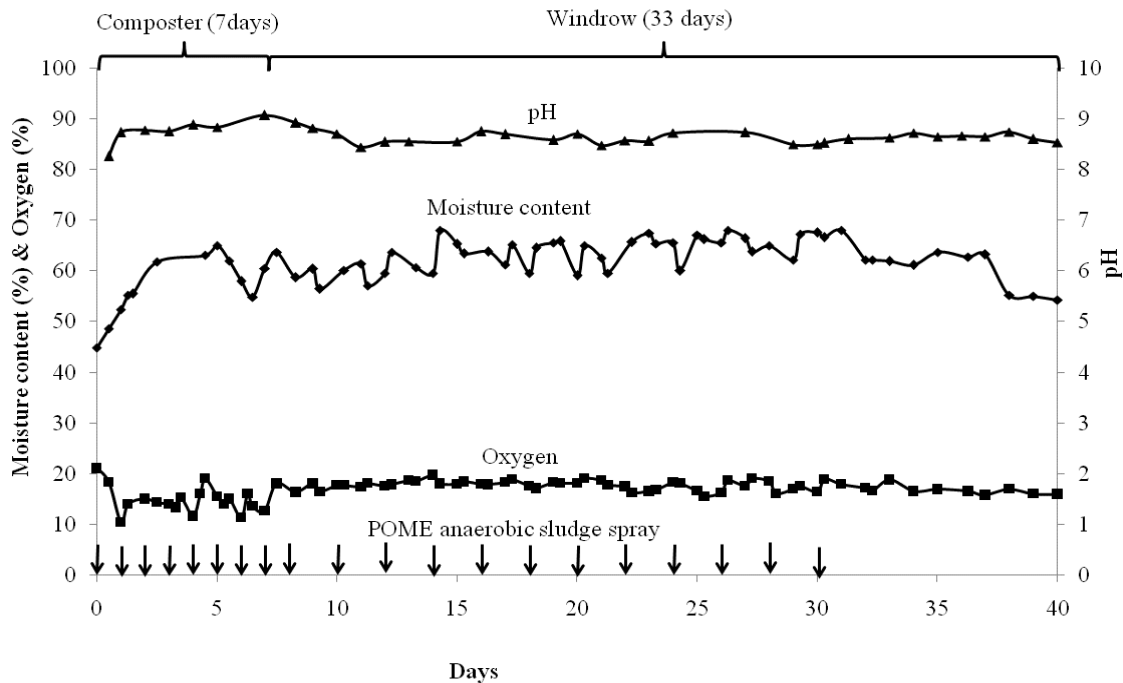
Fig. 3. Profiles for moisture, pH, and oxygen level during the composting process

Maintaining an optimum oxygen level is very important to ensure active microbial activity and the degradation processes are efficient (Rihani *et al.* 2010). In this study, the oxygen level was controlled using a blower to ensure a level higher than 10%. At the beginning of the composting process (until day 7), higher microbial activity was observed as reflected by the thermophilic temperature achieved. At this stage, oxygen was heavily consumed by the bacteria and was recorded at the lowest levels. The oxygen level in the range 10 to 15% was recorded, as shown in Fig. 3. However, due to frequent aeration, the oxygen level was maintained above 10% most of the time. The relationship between microbial activity and oxygen consumption for organic matter decomposition was in agreement with the previous study by Rihani *et al.* (2010).

Meanwhile, there was no difference in the pH value recorded during the composting from days 1 to 40. The pH values recorded were in the range of 8.2 to 9.0, except for a slight increase initially during the thermophilic stage in the in-vessel composter. This phenomenon was reportedly due to the increase in ammonia content generated by the biochemical reactions of nitrogen-containing materials (Kalamdhad *et al.* 2009a). An increase in pH also attributed to the increase of thermophilic bacteria (Ahmad *et al.* 2011). According to Tumuhairwe *et al.* (2009), changes in pH from acidic to alkaline could be attributed to microbial activity and the ammonification process that lead to the formation of NH_4OH, which caused the increasing pH in compost. The continuous addition of POME anaerobic sludge for microbial and alkalinity supplementation to the composting materials proved to be a good strategy whereby pH values were recorded almost stable in the range 6.0 to 8.5 and reported to be suitable for most types of plants (Hachicha *et al.* 2009).

C/N ratio

　　　　Figure 4 shows the profiles for nitrogen (N), carbon(C), and the C/N ratio throughout the composting process. It was found that the content of N had gradually increased while C decreased throughout the composting period. According to Bernal *et al.* (2009), this phenomenon was due to the decomposition of organic matter as a result of microbial action. It causes the C/N ratio to decrease over time. OPFFB material is composed of lignocellulosic structures, and it accounts for the major portion of the whole biomass. Cellulose and hemicellulose, which have a higher C content, are usually degraded earlier than lignin by bacterial action in the thermophilic stage in the composter. Lignin, which is more resistant toward bacterial degradation, will be degraded later (Jouraiphy *et al.* 2005). Basically the main function of lignin is to provide strength to the plant and resistance against the microbial degradation process (Tuomela *et al.* 2000; Said-Pullicino *et al.* 2007). Thus, the mechanical turning process (by helical screw impeller) inside the composter helped to significantly accelerate the lignin degradation process. Moreover, proper mixing due to helical screw impeller action inside the composter also helped to prevent N loss by volatilization. This is consistent with the findings summarized by Hubbe *et al.* (2010), that N loss will reduce if mechanical turning is applied according to a schedule and moisture content was maintained within favorable ranges. In addition, according to Tumuhairwe *et al.* (2009), the temperature decrease occurring as a result of turning the compost was beneficial in preventing N losses because the increase in pH during the thermophilic phase caused N losses due to volatilization.
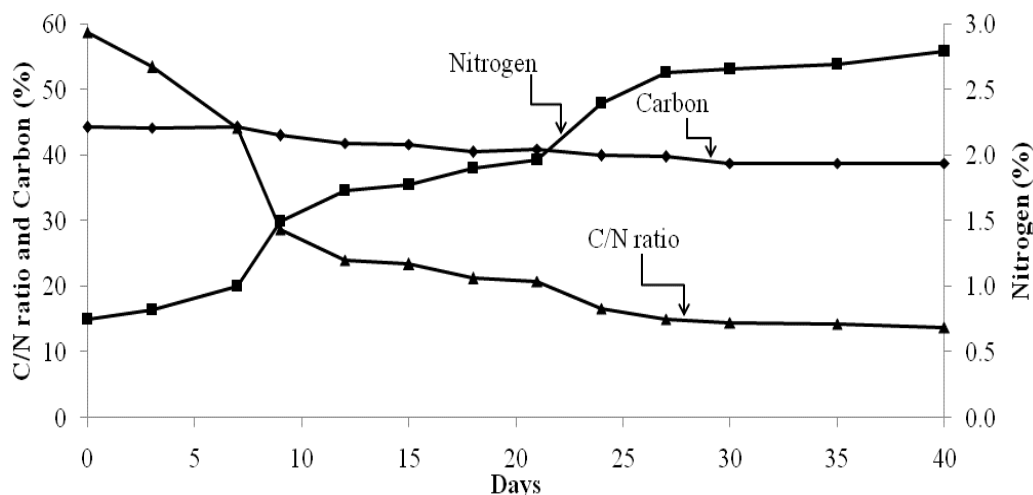

Fig. 4. Profiles for nitrogen, carbon, and C/N ratio throughout composting process

　　　　Figure 4 clearly shows that the C/N ratio of OPEFB reduced significantly from 58 (at day 0) to 44 (at day 7). As mentioned earlier, this happened because of the high temperature during the thermophilic phase in the in-vessel composter. Once the OPEFB material was transferred to the windrow, a smaller reduction of C/N was observed as a result of the lower operating temperature. The final C/N ratio at day 40 was calculated to be 13.85. This is satisfactory because matured compost material usually has a C/N ratio of 15 or less (Baharuddin *et al.* 2009; Hock *et al.* 2009).

LignocellulosicStructure Changes of OPEFB

Scanning electron microscopy

Scanning electron microscopy (SEM) analysis was conducted to understand the degradation of the OPEFB lignocellulosic structure throughout the composting period. Micrographs for OPEFB at different stages of the composting process are shown in Fig. 5. The micrographs revealed that the surface condition of the raw OPEFB material was initially hard and rough, and the pores were filled with silica bodies (indicated by arrows) that acted to protect the plant structure and enhance its mechanical strength (Law *et al.* 2007). Bacteria are unicellular with sizes ranging from 0.5 to 3.0 µm, and therefore they have a high surface/volume ratio, which allows rapid transfer of soluble substrates into the cell (Tuomela *et al.* 2000). However, due to the availability of silica bodies inside the pores, the penetration of microbes into the cellulose layer in the internal tissue is restrained, as reported by Wong *et al.* (2008). Therefore, in normal conditions this will hinder the degradation process of OPEFB.
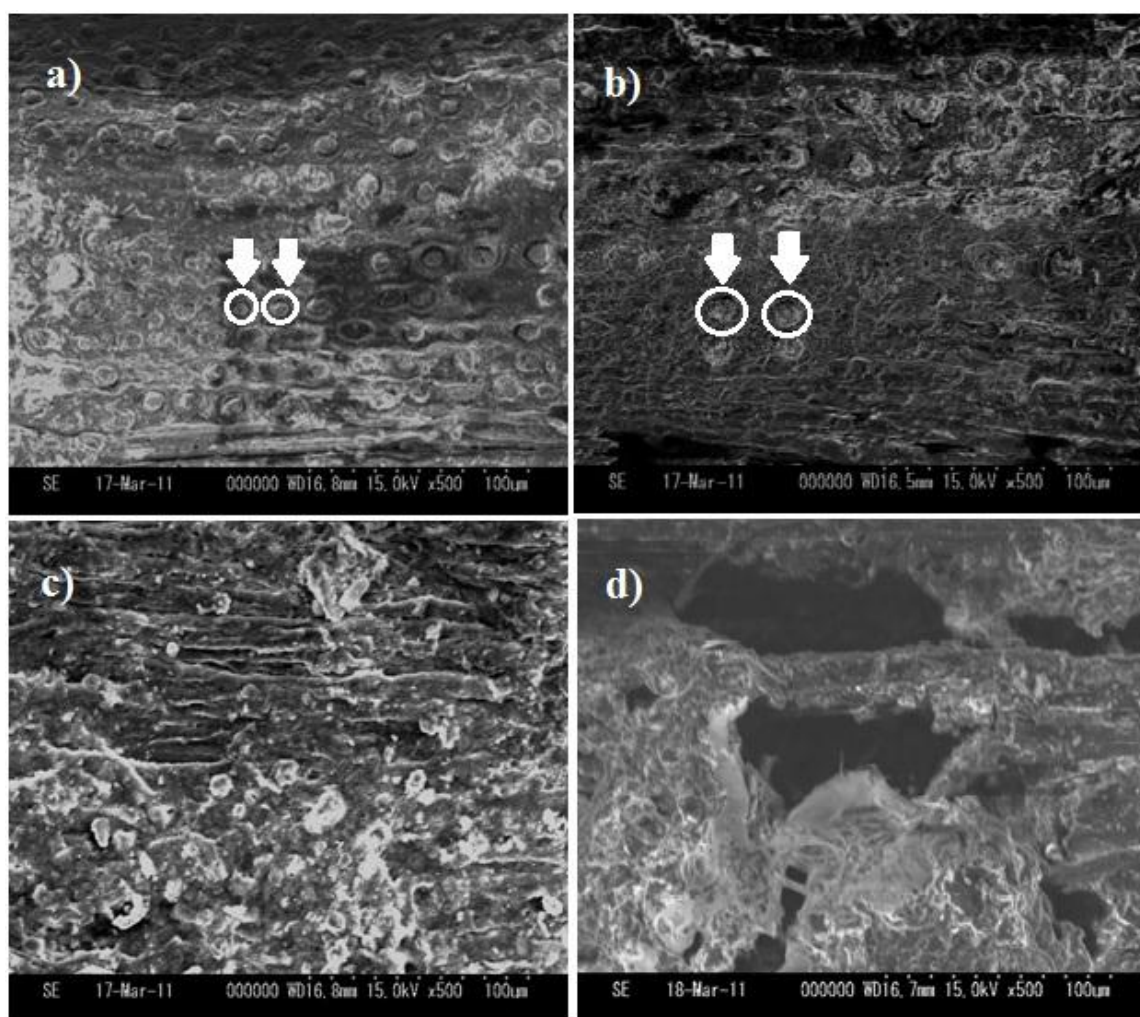

Fig. 5. SEM micrograph (500x magnification) for the raw press shredded OPEFB (a), OPEFB material at day 7 (b), OPEFB material at day 20 (c), and OPEFB material at day 40 (d)

After 7 days exposed to thermophilic temperatures inside the composter and frequent mechanical turning, the silica bodies inside the pores were partially removed, as clearly observed in Fig. 5(b). Based on an earlier study by Baharuddin *et al.* (2011) and Bahrin *et al.* (2012), the removal of silica bodies was due to the active microbial action and frequent movement of OPEFB materials. As clearly shown in Figs. 5(b) and 5(c), the silica bodies were partially removed after 7 days and almost completely removed after 20 days of composting. After 40 days of composting, the OPEFB were almost completely degraded, and many holes were created and no more silica bodies were apparent, as shown in Fig. 5(d).

X-ray diffraction analysis

Silica bodies are actually embedded in the OPEFB strands and they are formed by the accumulation of silica ions (Law *et al.* 2007). From the SEM results, it was clear that most of the silica bodies were removed after 40 days of composting. The main components of EPEFB materials are cellulose, hemicelluloses, and lignin. According to Bledzki and Gassan (1999), hemicellulose is the weakest component because the structures are made of weak matrix polysaccharides and amorphous structures. Because they are so weak, hemicellulose greatly contributes to the degradation of OPEFB materials during the composting process. Jouraiphy *et al.* (2005) stated that lignin resists biodegradation and only undergoes partial biotransformation, while hemicellulose is the component that was subject to the most extensive degradation. In our hypothesis, the existence of silica bodies is due to the increase of Si ion concentration, as carbonaceous substances are consumed by microbes. This will contribute to the nucleation of ionic mineral on the OPEFB matrix, as reported by Kalia *et al.* (2011).

Figure 6(a) shows a comparison of SiO_2 peak intensity in OPEFB at different composting periods. It can be observed that most of the carbon structure that was contained in the cellulose and hemicelluloses components was in the amorphous form at the beginning and tended to become depleted toward the end of the composting period. In raw OPEFB, a SiO_2 peak was not found, indicating that strong Si bonding with plant cell matrix (in the form of silica bodies) protects and gives mechanical strength to the OPEFB structure (Law et al. 2007).

Increase of SiO_2 to the point of saturation during the degradation process shows that the SiO_2 was in crystal lattice form. Along with the process, a broad peak of amorphous carbon ranging from 20 to 25° started to deplete on day 30. The depletion was expected to be coming from the degradation of hemicellulose and cellulose. Further investigation by using the Scherrer equation revealed that the crystal size increased from an average 34 nm to 45 nm after 7 days of composting. However from day 7 onward, the crystal size decreased from an average of 45 nm to 20 nm (Fig. 6(b)). Removal of large SiO_2 crystals on the OPEFB matrix occurred due to the frequent turning process during the windrow stage. It was earlier discovered that the high temperature during the thermophilic phase and frequent mechanical turning throughout the composting loosened the SiO_2 crystals embedded on OPEFB matrix (Baharuddin *et al.* 2010). Smaller SiO_2 crystal size at the end of the composting process might be due to nucleation of Si ion inside the siliceous pathway in the OPEFB strands. This could not be removed by the turning process (Law *et al.* 2007).
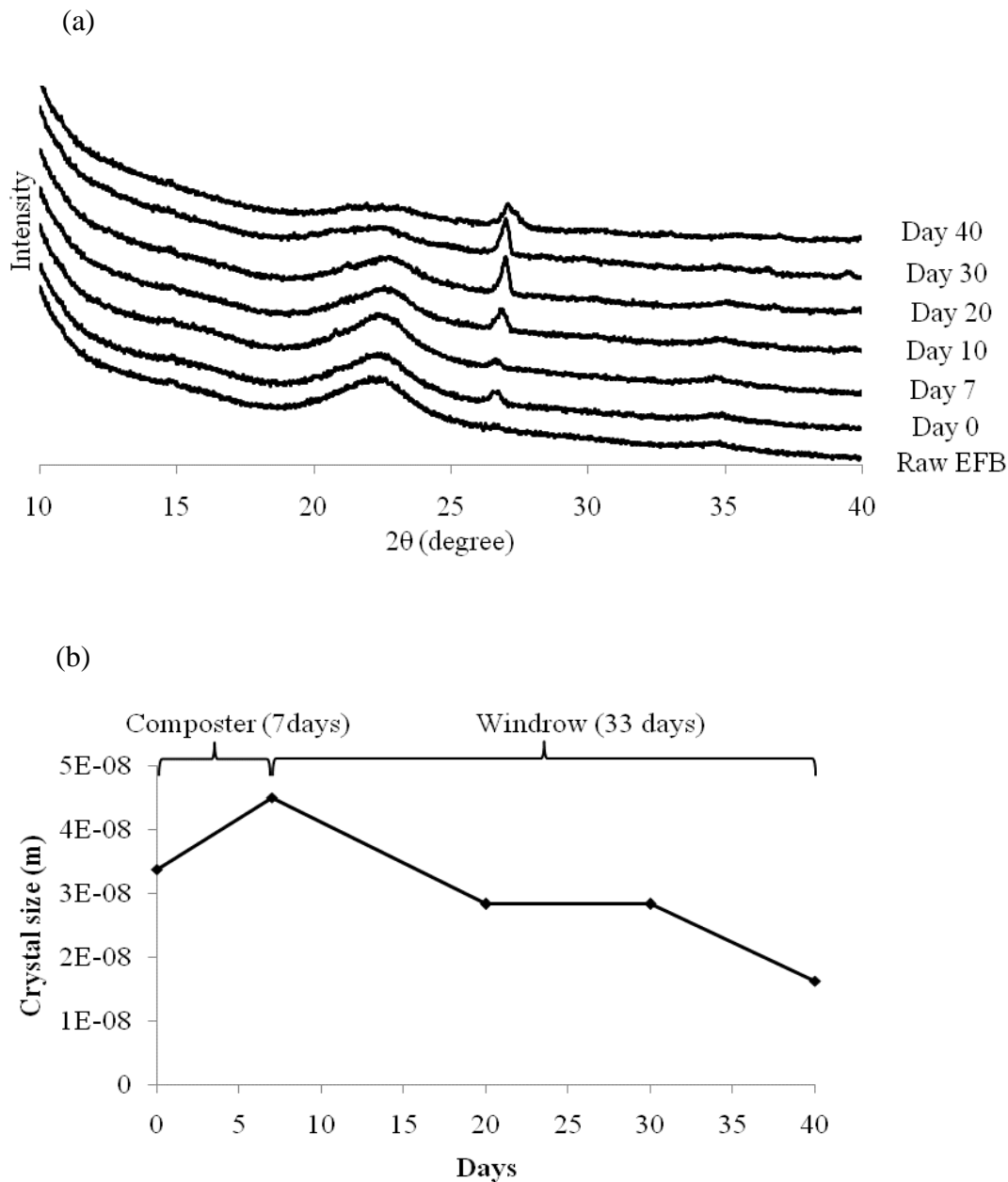
(a)

(b)

Fig. 6. XRD analysis of composting process (a) and a comparison of crystal size by using Scherrer equation (b)

Fourier transform infrared analysis

Figure 7 shows infrared spectra of the OPEFB materials at different composting periods. This approach was used to verify the presence and changes in the organic groups, mainly due to lignin, cellulose, and hemicellulose. Smidt and Meissl (2007) stated that bands that represent organic molecules decrease continuously and disappear in the original infrared spectrum. In our hypothesis, the band disappearance was contributed by the degradation of specific components in OPEFB structure or the transformation of

some functional group to become more stable substances such as humic acid (Amir *et al.* 2010). The bands located at 2916 cm^{-1} and 2845 cm^{-1} were referred to as the hydrogen vibrations of C-H group of aliphatic methylene group (Bernabé *et al.* 2011) and were expected to be coming from the degradation of hemicellulose. Hemicellulose is usually more easily degraded by microbes because the structures are made of weak matrix polysaccharides, they are amorphous, and the structures mainly contain monomer sugar (Bledzki and Gassan 1999). Jouraiphy *et al.* (2005) reported that during the composting process, microorganisms utilized aliphatics and carbohydrates such as polysaccharides, cellulose, and hemicelluloses to meet their energy requirement, and thus hemicellulose tends to disappear more easily than cellulose or lignin. The absorption band at 1730 cm^{-1} indicates that metabolic products such as aldehydes, ketone, and ester were initially available in the OPEFB structure (Tandy *et al.* 2010). Depletion of a broad peak belonging to the hydroxyl group or O-H band at 1635 cm^{-1} was noticed after several days of composting, and the trend remained unchanged after 20 days of composting. The decrease of hydroxyl groups in the OPEFB caused a reduction in the hydrophilicity of the material and several degree changes in contact angle aspect. Kalia *et al.* (2011) stated that the structure of cellulose can absorb large quantities of water. Hence, the disappearance of the hydroxyl group was due to the evaporation of water from the OPEFB structure throughout composting process. Disappearing bands at 1590 cm^{-1} and 892 cm^{-1} might come from the double bond C=C and carbonate groups C-O, respectively. Double bond changes in OPEFB structure can be explained by the microbial degradation process that turned complex OPEFB carbohydrate structures into more simple carbon chains (Castaldi *et al.* 2005). Bands at 1260 to 1240 cm^{-1} were referred to carboxylic acids groups and tertiary amides, which were visible in the raw OPEFB and disappeared by the end of the composting process. The band at 1507 cm^{-1} was attributed to the aromatic skeletal structure of lignin. Throughout the process, some unidentified bands disappeared at day 40, which is expected from the degradation process for lignin.
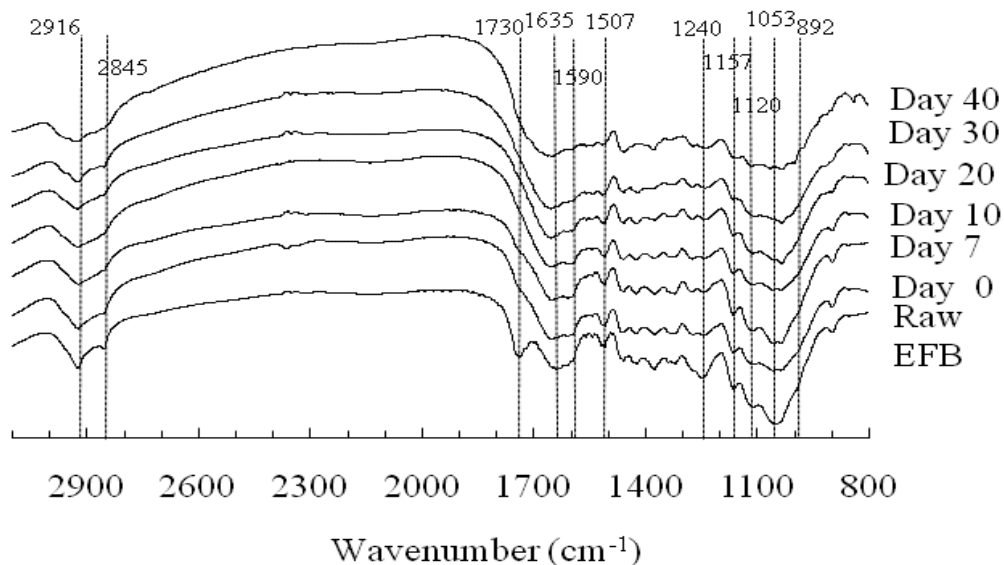

Fig. 7. Comparison and assessment of final composts by referring to infrared spectroscopic characteristics

According to Wong *et al.* (2008), lignin is the most difficult component to decompose. The bands at 1157, 1120, and 1053 cm^{-1} are attributed to S-O and polysaccharide compounds. Inorganic sulfates were contributed by POME anaerobic sludge and polysaccharides, which were contributed along the process. Disappearance of absorption bands at 1100 to 1050 and 950 to 900 cm^{-1} were contributed by silica ingredients, such as Si-O-Si and SiO-H (Smidt and Meissl 2007). Previously, it was also reported that in matured compost samples, silica compounds and carbohydrate absorbance bands had disappeared (Jouraiphy *et al.* 2005).

Thermogravimetric and differential thermal analysis

Figure 8 shows the thermal responses of OPEFB samples at different composting stages. In the temperature range between 130°C and 180°C, all samples underwent a dehydration process and the most tightly bound water molecule was released. Some volatile components were also released from lateral chains, together with other low molecular weight compounds (Baffi *et al.* 2007). The high volatile matter content inside OPEFB made it easy to burn. Hence, the final matured products exhibited small reduction in weight lost due to the existence of stable structure formation (Omar *et al.* 2011). Samples' weight losses in the range between 180 to 410°C and 400 to 600°C were reported to be associated with both the first exothermic peak, which may refer to the cellulosic component, and a second exothermic peak, which may correspond to lignin having more complex organic molecules (Baffi *et al.* 2007). Figure 8(a) shows the residual weight ratio at different stages in the composting process. A comparison of residual weight ratios shows the decomposition process occurs in one-step increments. According Bernabé *et al.* (2011), the increase in the residue could be attributed to the mineralization process that occurs throughout the treatment.

All compost samples in thermograms indicated weight loss within the temperature range of 250 to 360 °C. All weight loss might be attributed to carbohydrate or specifically cellulose derivative combustion (Otero *et al.* 2002). The carbohydrate combustion was expected to be coming from hemicellulose and cellulose carbon chain disassociation (Alvarez and Vazquez 2002). The weight loss difference observed in thermal decomposition thermograms agreed with the XRD and FTIR results. The curve change also shows that the decomposition of the OPEFB structure occurs continuously during the composting process. Details of weight loss periods of each sample during the composting time were recorded and are shown in Table 2.

Table 2. Weight Losses of OPEFB Degradation Corresponding to the Respective Peaks Shown in the Thermograms (Fig. 8(b))

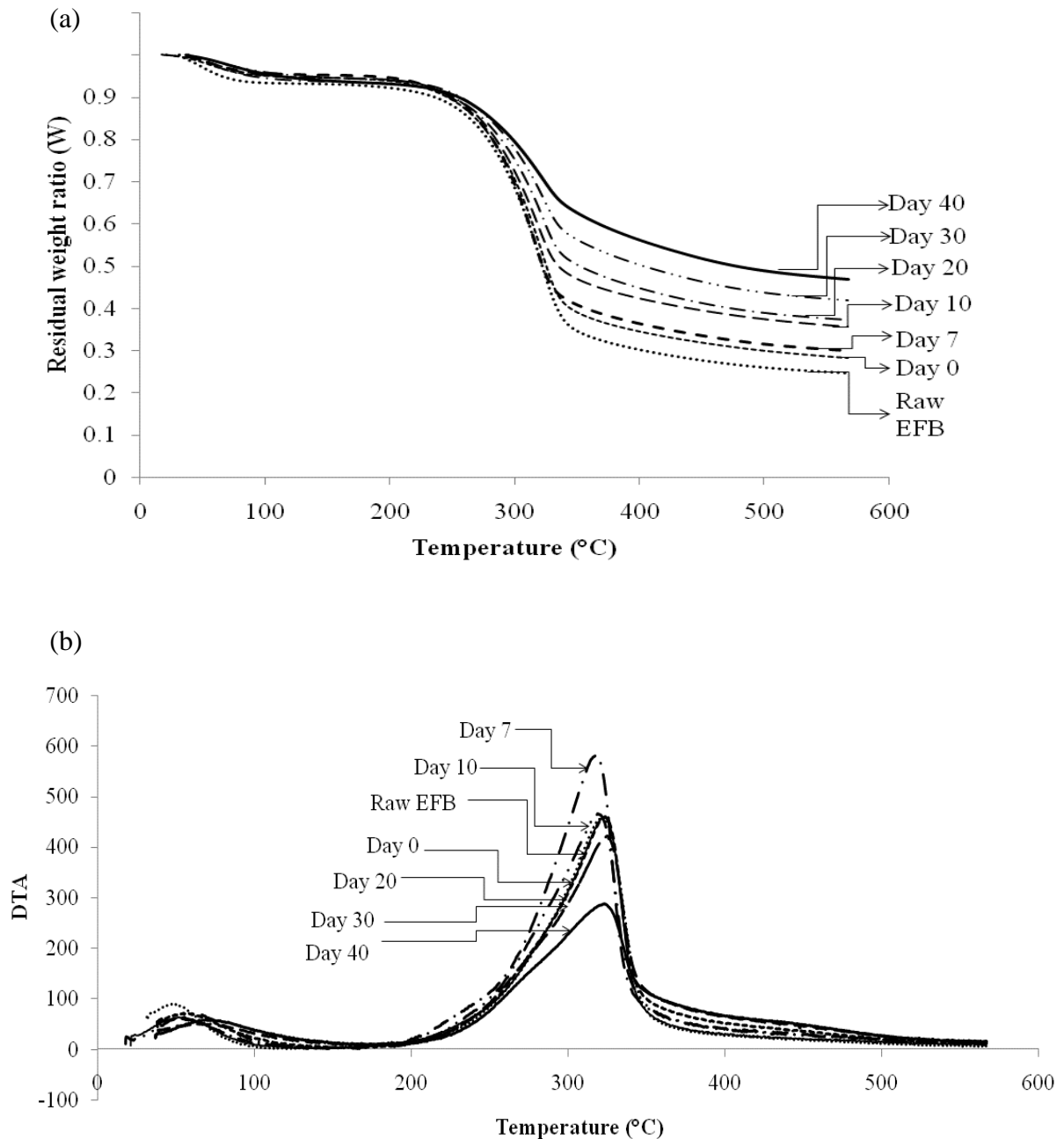| Samples	200 to 300°C	300 to 400°C	200 to 500°C
Raw OPEFB	0.24	0.38	0.66
0	0.24	0.36	0.64
7	0.24	0.58	0.62
10	0.22	0.30	0.57
20	0.19	0.31	0.55
30	0.16	0.28	0.50
40	0.13	0.24	0.44

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(a)

(b)

Fig. 8. TG data comparison of composed OPEFB with raw OPEFB (a) and DTA data comparison of composed OPEFB with raw OPEFB (b)

Figure 8(b) shows OPEFB decomposition reaction conducted with differential thermal analysis (DTA) for different OPEFB composting stages. The heat change of each sample indicates a specific activation energy with varied peak height or enthalpy values. Peak variation could be used to determine the activation energy of each OPEFB decomposition reaction later. The figure shows that all indicated peaks occurred in the

range 320 to 350 °C, which corresponded to decomposition of the OPEFB main structure, as explained previously. The highest peak was generated by the day 7 sample, which means that the highest degradation of the cellulosic component occurred inside the vertical in-vessel composter. As the OPEFB matured, the intensity of the exothermic peak decreased significantly compared to the raw OPEFB. The immature compost might consist of a low carbohydrate component, such as hemicelluloses, which tend to disappear in a stabilized composted sample (Li *et al.* 2001). After 40 days of composting, the sample showed minimal weight loss, indicating that the degradation process of OPEFB became slow due to low microbial activity at the end of composting process (Marhuenda-Egea *et al.* 2007). Furthermore, the minimal weight loss was also contributed by lower molecular components degradation of OPEFB in comparison to the initial materials.

CONCLUSIONS

1. Composting of OPEFB using an in-vessel composter followed by windrow composting was shown to be a feasible method to produce consistent compost with satisfactory N, P, and K (2.8, 0.4, and 2.8) contents. A composting period of 40 days with a final C/N ratio of 13.85 was achieved.

2. XRD results showed evidence of silica body removal from OPEFB, which can be related to the reduction of crystal size. FTIR results also verified cellulose, hemi-celluloses, and lignin changes throughout the composting process by determining the chemical compound changes in the OPEFB compost materials. Thermal analysis results showed that the highest degradation for cellulosic components occurred in the composter during the thermophilic phase, and the intensity of the exothermic peak of the matured compost decreased significantly compared to the raw OPEFB.

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