

DEGRADATION ASSESSMENT OF FLEXIBLE POLYURETHANE FOAM COMPOSITES BASED ON PALM OIL IN SOIL

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<https://doi.org/10.23939/chcht18.04.558>

Abstract. This paper presents information on a comparison of the degradation of a flexible palm oil-based polyurethane foam with a commercial flexible polyurethane foam (control) in soil over a period of 30 days. The sample used in this study was a composite polyurethane foam containing about 50-60% palm oil polyol and has characteristics similar to conventional polyurethane foam. The novelty of this study is to investigate the degradation rate of a composite flexible polyurethane foam with a palm oil polyol content of 50-60%. As a result of the study, an environmentally friendly formulation of composite polyurethane foam was developed based on the following parameters (weight loss, FTIR, XRD, TGA, and SEM).

Keywords: degradation, hard segment, soft segment, urethane bond, weight loss.

1. Introduction

Polyurethane is formed from the reaction of polyether or polyester, which has an OH group with an isocyanate NCO group to create a urethane bond.¹ Various polyurethane products, such as coatings, foams, elastomers, and adhesives, are on the market.² The world needs flexible polyurethane foam (FPUF) around 31% of total polyurethane production.³ FPUF produces the most waste due to its wide use in automotive, furniture, and construction. This waste will continue to increase along with increasing demand for FPUF. This polymer is complex to decompose by microorganisms and impacts the environment. This situation can pollute the atmosphere in the long term.

Conventional FPUFs are synthesized from petroleum-derived polyols. Petroleum supplies are decreasing and cannot be renewed. Replacement of petroleum polyols with palm oil polyols is one way to increase the degradable composition. The problem arises

because using 100% palm polyol produces FPUF that is brittle and inflexible. In previous research, we synthesized FPUF with a palm oil polyol composition of up to 60%. Mixing palm oil polyol with PEG-400 and commercial polyol in a specific ratio creates good FPUF characteristics. The structural and physico-mechanical properties of composites can be improved by the use of PEG.⁴ Using PEG-400 40%: palm oil polyol 60% (FPUC1) and PEG-400 50% : palm oil polyol 50% (FPUC2) of the polyol system improved the elasticity of the FPUF composite.⁵ Using commercial polyol 40%: palm oil polyol 60% (FPUC3) and commercial polyol 50%: palm oil polyol 50% (FPUC4) of the polyol system improved development but was less flexible.⁶

Using petroleum-derived polyol is still necessary to make degradable FPUFC. The composition of organic and petroleum-derived polyols in the polyol system will influence the level of FPUFC degradation. Degradation in soil is carried out by microorganisms.⁷ Microorganisms produce enzymes that can hydrolyze ester bonds in polyester polyols into water-soluble monomers and oligomers, followed by mineralization of polymer fragments.^{8,9} The ether bonds in polyether are more resistant to microbial (fungal) attacks. The degradation of polyether polyurethane foam is much slower than that of polyester polyurethane foam.¹⁰

Polyurethane degradation is carried out in soil and water. Soil is a medium that provides many microorganisms that can decompose polyurethane foam. Different pH levels of soil affect the community of microorganisms found in the soil. Acidic soil has *Geomyces pannorum*, *Nectria* sp. strain BC11, *Cylindrocladiella parva*, *Penicillium inflatum*, and *Plectosphaerella cucumerina*. Neutral soil has *Alternaria* sp. strain 18-2, *Penicillium venetum*, *Neonectria ramulariae*, and *Penicillium viridicatum*.¹¹ The soil environment contains fungi that have the potential to decompose polyurethane. Polyurethane has many molecular bonds analogous to those found in biological macromolecules, and fungal genes encode various secreted hydrolases, enhancing possible unintentional degradation of polyurethane due to these enzymes. This research uses fertile soil with a neutral pH taken from the fertile soil of plantations.

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The degradation of FPUC is not yet known. Based on this, this study aimed to examine the degradation of flexible polyurethane foam composites from palm oil polyol combined with PEG-400 and commercial polyol in soil.

2. Experimental

2.1. Materials

The materials used in this research were commercial palm oil brand Sari Murni, H₂O₂ 50% from Bratachem, acetic acid 100% from Merc, sulfuric acid 98% from Smart Lab, ethylene glycol from Bratachem, sodium bicarbonate from Merc, HBr from Merc, KOH from Merc, DABCO BLV catalyst from Shandong Tonglan Chemical Co., Ltd., DABCO T-9 catalyst from Tianjin Hutong Global Trade Co., Ltd., silicone oil from Hubei Star Chem Co., Ltd., TDI 80 (80:20 mixtures of the 2,4 and 2,6-isomers) from Shandong Baovi Energy Technology Co., Ltd., distilled water, methylene chloride from Dow, diethylene glycol from Merc, polyethylene glycol-400 from Flourishing Chemical Prosperity, and commercial polyol from Joint Chemical Enterprise.

2.2. Methods

2.2.1. Synthesis of Palm Oil Polyols

200 mL of palm oil was heated at a temperature of 50-55°C with 0.3 moles of 47% acetic acid in a 250 mL 3-neck flask equipped with a stirrer, then concentrated sulfuric acid catalyst (98%) as much as 1% of the amount was added. The total oil was continuously stirred at a speed of 200 rpm, and then 50% hydrogen peroxide (dilute) was gradually stirred at 1.7 moles (still initiating and maintaining a temperature of 50-55°C). After adding H₂O₂, the temperature was held at 50°C for 1 hour. The epoxidized oil was separated with a separating funnel and then added with a saturated sodium bicarbonate solution until the pH was neutral. It was washed with hot distilled water two times. Next, the washing water was separated using a separating funnel. Sodium sulfate solution was added to absorb the remaining water and then filtered. The epoxidized palm oil obtained was slightly cloudy yellow.

10 mL of epoxidized palm oil reacted with 30 mL of ethylene glycol and then a sulfuric acid catalyst of 1% of the total reactant was added at a temperature of 50°C for 1 hour. After the reaction was stopped, the product was cooled to room temperature, and then hexane was added: reaction product = 1:1 and NaHCO₃ drop by drop until pH was neutral. It was then washed with distilled water to remove the remaining ring-opening compound, catalyst, and epoxy oil that did not participate in the reaction. The mixture was poured into a separatory funnel. The top layer was an organic layer containing a yellow product, and the bottom layer was the remaining reactant (ring-opener agent), which was clear. The top layer (organic layer) was purified with a rotary vacuum evaporator at 80°C to remove hexane (solvent).

2.2.2. Synthesis of FPUC

Foam synthesis was done by mixing bio polyol with commercial polyol and PEG-400 according to the treatment and additional materials (Table 1).

Then, the mixture was reacted with TDI 80 and stirred at 1200 rpm for 10 seconds. The reaction mixture was poured into a mold measuring 120 x 120 x 100 mm³ and placed in the oven at 70°C for 1 hour. Next, leave it at room temperature for 24 hours, and the foam is ready for observation.

2.2.3. Degradation

FPUC1, FPUC2, FPUC3, and FPUC4 measuring 2.5x1.5x1 cm³ was weighed. The samples were buried in fertile soil with pH of 7 and incubated at room temperature. Weight loss observations were carried out for one month at 0, 5, 10, 15, 20, 25, and 30 days. The degree of degradation was calculated based on weight loss (%) using a formula:

$$\text{Weight loss (\%)} = \frac{W_o - W_t}{W_o} \times 100$$

where W_o is the dry weight before the degradation, W_t is the weight after degradation at time t .

Table 1. FPUC formulations

Composition (pphp)	FPUC1	FPUC 2	FPUC3	FPUC4
Palm oil polyol	60	50	60	50
Commercial polyol	-	-	40	50
PEG-400	40	50	-	-
DABCO T-9 Catalyst	0.2	0.2	0.2	0.2
DABCO BLV Catalyst	0.53	0.53	0.53	0.53
NIAX L-618 silicone oil	1	1	1	1
Aquades	1	1	1	1
Methylene chloride	4.2	4.2	4.2	4.2
TDI	51.9	51.9	51.9	51.9

Note: pphp = part per hundred polyol

On the 30th day, all FPUC samples were observed using FTIR brand Perkin Elmer, X-Ray Diffraction (XRD) brand X'Pert PRO PANalytical MPD PW3040/60, SEM brand Hitachi S-3400N, and thermal analyzer DTG-60 brand Shimadzu.

3. Results and Discussion

This research develops previous research in synthesizing environmentally friendly flexible polyurethane foam composites from palm oil polyol combined with PEG-400 and commercial polyol. Flexible polyurethane foam composites were obtained with mechanical properties and expansion close to the characteristics of commercial flexible polyurethane foam from petroleum-derived polyols. Degradation was carried out using soil obtained from fruit plantations.

3.1. Weight Loss

The structural arrangement and chemical composition of polymers are essential to determine degradation patterns.¹² Polyurethane consists of a hard segment

and a soft segment. The hard segment is formed from diisocyanate and chain extender, while the soft segment is constructed from long and aliphatic diol polymer chains. Hard segments form cross-links. Polyurethane soft segments are easy to decompose because they have easily hydrolyzed bonds such as ester bonds and urea bonds (amide bonds).¹³ The ratio of hard and soft segments influences the degradation rate of flexible polyurethane foam.

Observations on the 5th day of all samples FPUC and control were overgrown with fungus with white hyphae on the surface of the fertile soil (Fig. 1). The Fungi grew on materials because of carbon and nitrogen sources. Fungi that play a role in polyurethane biodegradation are *Aspergillus* (*Aspergillus fumigatus*), *Penicillium* (*Penicillium mandriti*, *Penicillium chrysogenum*, and *Penicillium roseopurpureum*), *Chaetomium*, *Cladosporium*, *Fusarium* (*Fusarium solani* and *Fusarium oxysporum*) and *Trichoderma*.^{14,15} These microbes produce enzymes. The enzymes that play a role in the polyurethane degradation process are esterase, lipase, and protease enzymes.^{16,17}

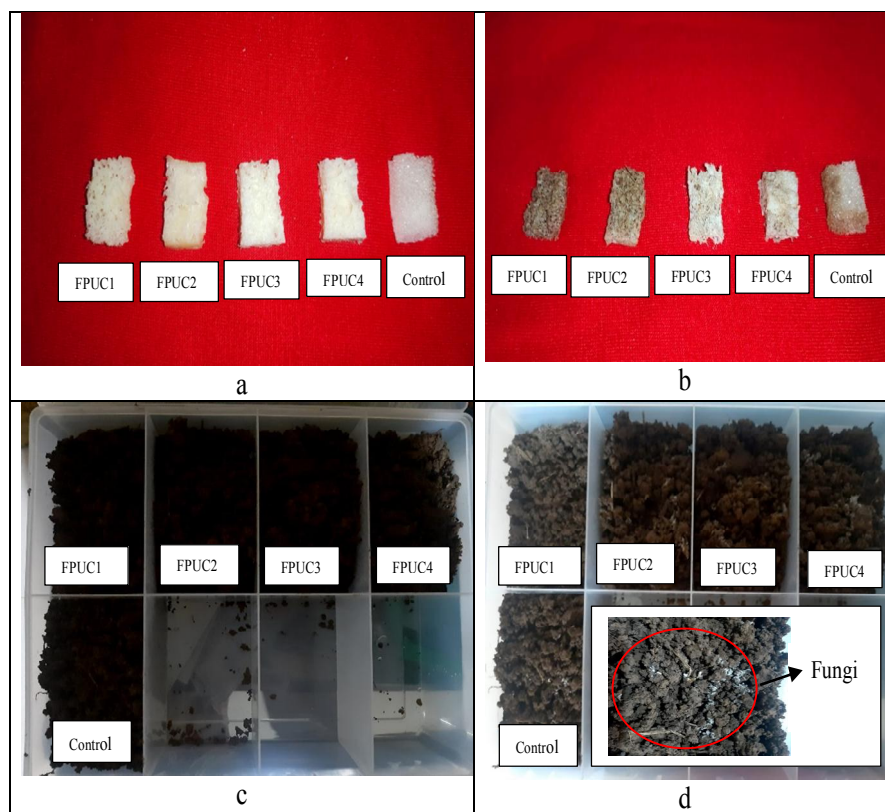


Fig. 1. Photo of flexible polyurethane foam sample before degradation (a), flexible polyurethane foam samples after degradation for 30 days (b), photo of sample photo of soil with flexible polyurethane foam samples with degradation time 0 day (c), and 5 days (d)

Fig. 2 showed that from 0 to 5 days, FPUC1 and FPUC2 had a high weight loss of 31% and 24%. This suggested that a high degradation rate occurred in FPUC1 and FPUC2. The fungal mycelium on the soil surface penetrated the material's pores. Fungi produced an esterase enzyme, which broke down the ester groups in palm oil polyols. After that, a wider foam pore diameter was formed, which made it easier for soil microorganisms to enter the foam cells and break the cross-links of the FPUC. This condition increased the degradation rate. This phenomenon can be stated that on the third day, the side chains in the molecular chain begin to break and are followed by the primary chain breaking.¹⁸

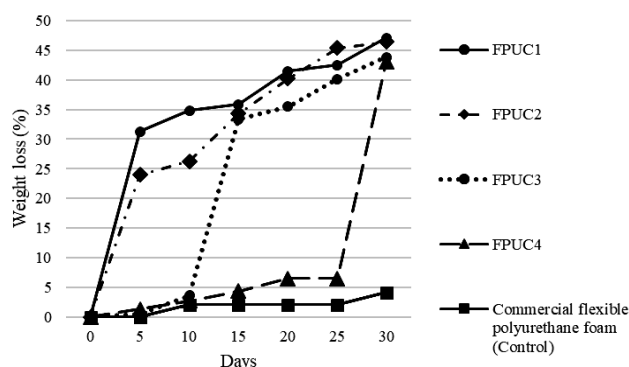


Fig. 2. Weight loss of FPUC and control during degradation in soil

FPUC3 had the highest weight loss from 10 to 15 days, namely 33%. At the same time, FPUC4 showed a slow degradation until day 25, reaching a rapid degradation of 43% by day 25-30. The significant difference between the degradation rates of FPUC3 and FPUC4 (Fig. 2) is due to increased commercial polyols in the foam composition. The nutrients available to the microbes decrease, resulting in fewer active microbial cells. The lower number of enzymes causes FPUC4 to take longer time to degrade than FPUC3. When compared, the degradation rate of FPUC3 is significantly different from FPUC4, while the degradation rate of FPUC1 is slightly different from FPUC2. This is due to the type of compounds used to synthesize the FPUCs. FPUC3 and FPUC4 contain crystalline commercial polyols (Fig. 4). FPUC1 and FPUC2 contain PEG-400, which is crystalline and plays a role in the formation of the soft segment of the foam. FPUC3 and FPUC4 synthesized from commercial polyols have crystalline properties and play a role in forming hard segments.¹⁷ Although commercial polyols and PEG-400 have crystalline properties, PEG-400 degrades more quickly than commercial polyols. Control had a prolonged degradation rate because very little microbial nutrition was available.

Polymer weight loss during the degradation process depended on the breakdown of each chain, which occurred in three ways: destruction, depolymerization, and degradation processes. High weight loss resulted in the release of compounds with low molecular weight, mainly occurring in the soft segment. The depolymerization process is the breakdown of polymers into smaller fragments or smaller macromolecules, while degradation represents the disintegration of polymers into smaller oligomer units. The polyurethane sample looks like crumbs on the last day of degradation.¹⁹ FPUC1, FPUC2, FPUC3, and FPUC4 had better degradability than the control sample.

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups in FPUC1, FPUC2, FPUC3, FPUC4, and control after degradation for 30 days were observed using Perkin Elmer brand FTIR (Fig. 3). FTIR analysis showed that all foam samples did not show the presence of amine groups ($-NH$) at wave numbers $3300-3250\text{ cm}^{-1}$, but carbonyl groups ($-C=O$) were detected at wave numbers 1693 cm^{-1} . This indicated that degradation of urethane bonds ($-OCONH-$) has occurred in all foam samples. All foam samples showed the presence of methylene groups ($-CH_2$) at wave numbers $2954-2853\text{ cm}^{-1}$.

All foam samples had no absorption peak with a wave number of 2355.07 cm^{-1} (indicating CO_2). This showed that the bonds had been degraded so that CO_2 was released from the foam cells. The disappearance of the isocyanate group absorption peak ($-NCO$) in the area around $2250-2300\text{ cm}^{-1}$ for all polyurethane foam samples due to the degradation process.

The intensity of the carbonyl peak at wave number 1735 cm^{-1} was not sharp, indicating the presence of lower ester groups in the degraded sample. An increase in the bending intensity of $-NH$ bending in the urethane group at a wave number of 1535 cm^{-1} was observed in the FTIR spectrum of the degraded sample. This observation suggested hydrolysis of ester bonds occurring during the degradation of these polymers. Some enzymes had genes that code for proteins with hydrolytic (esterase, urethanase) and oxidative activity (mono-, di-oxygenase, dehydrogenase), which were active during the foam degradation process.²⁰

3.3. X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) was used to detect crystalline compounds in the foam. XRD testing was carried out on all foam samples buried in fertile soil for 30 days.

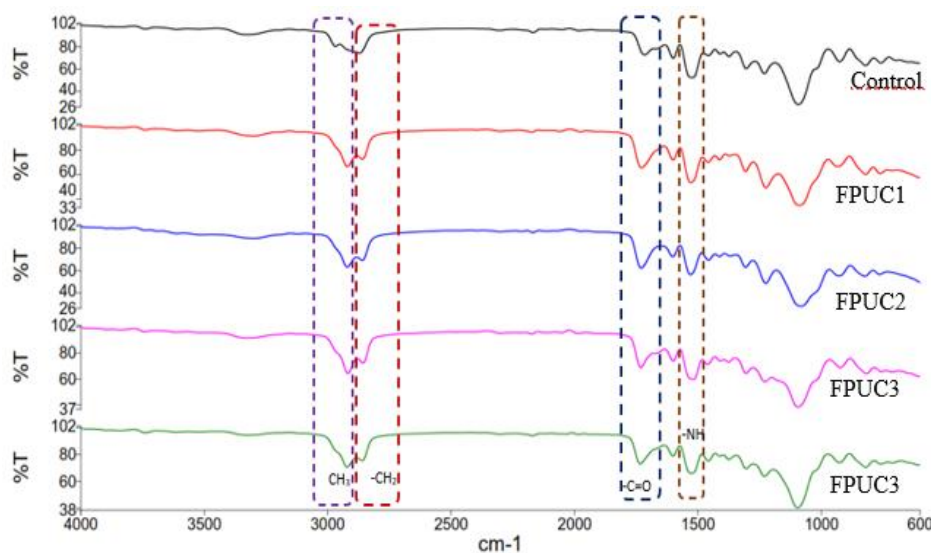


Fig. 3. FTIR spectrum of FPUC1, FPUC2, FPUC3, FPUC4, and control after degradation in soil for 30 days

Fig. 4 showed the presence of crystal-shaped diffraction peaks with a value of $2\theta = 27.22^\circ$ and $2\theta = 26.79^\circ$ which indicates the presence of PEG-400 at FPUC1 and FPUC2. PEG-400 plays a role in providing flexible properties (elongation at break for PEG 40% = 111% and PEG 50% = 121%) and increasing cross-link strength (tensile strength for PEG 40% and 50% = 60 kPa) in flexible polyurethane foam.⁵ Adding PEG-400 improves the structure of flexible polyurethane foam based on palm oil. This shows that using PEG-400 increases the crystalline properties of the foam. FPUC3 and FPUC4 had crystalline diffraction peaks with values of $2\theta = 19.68^\circ$ and $2\theta = 21.33^\circ$ which indicate the presence of a polymer suspected to be commercial polyols. The tensile strength test results show that the use of 40% and 50% commercial polyol has values of 200 kPa and 210 kPa. Elongation at break in flexible polyurethane foam with 40% and 50% commercial polyol composition are 57% and 49%.⁶ Polyurethane foam with the addition of commercial polyol has a stronger structure than PEG-400. The crystalline properties of commercial polyols are stronger than PEG 400. Palm oil polyols had degraded, whereas PEG-400 and commercial polyols crystalline required more extended degradation. Polyesters were much more susceptible to biological degradation than polyethers. Palm oil polyols were faster biodegradable polyesters than polyethers (PEG-400 and commercial polyols). PEG-400 is a crystalline soft segment that degrades more slowly than amorphous soft segments. The crystalline structure of commercial polyol degrades more slowly compared to PEG-400. Consequently, FPUC1 and FPUC2 degrade faster than FPUC3 and FPUC4.

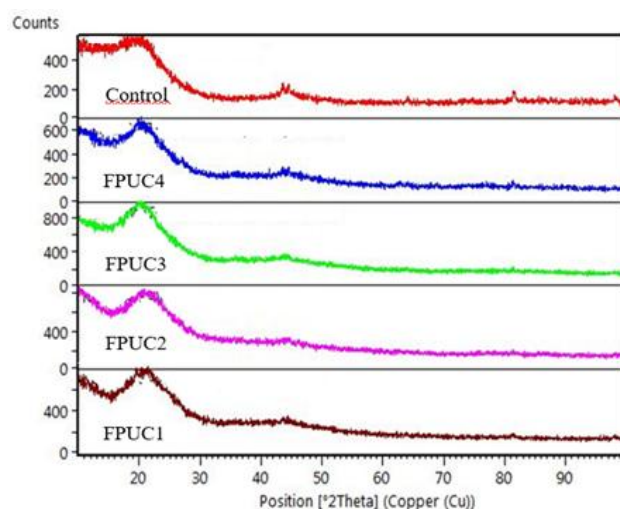


Fig. 4. XRD graphs of FPUC1, FPUC2, FPUC3, FPUC4, and control after degradation in soil for 30 days

3.4. Thermogravimetric Analysis (TGA)

TGA analysis used a Shimadzu brand DTG-60 thermal analyzer. This analysis aimed to determine the stability of the foam to heat after degradation for 30 days. Fig. 5 shows that FPUC1, FPUC2, FPUC3, FPUC4, and control have two stages of weight loss. The first stage of weight loss occurred due to the degradation of hard segments and weak bonds of $O=C-NH$, leading to the formation of primary or secondary isocyanate amines.²¹ The bond energy of $C-N$ was weaker than that of $C-O$ and $C-C$ because the hard segment had a lower molecular weight than the soft segment. The molecular chain segments of polyurethane begin to degrade, releasing isocyanates,

polyols, and other pyrolysis intermediate products.²² Stage 2 caused maximum weight loss due to the degradation of soft segments containing polyether

chains (PEG-400 and commercial polyols). High oxygen content in the soft segments (ester bonds) leads to significant heat absorption.²³

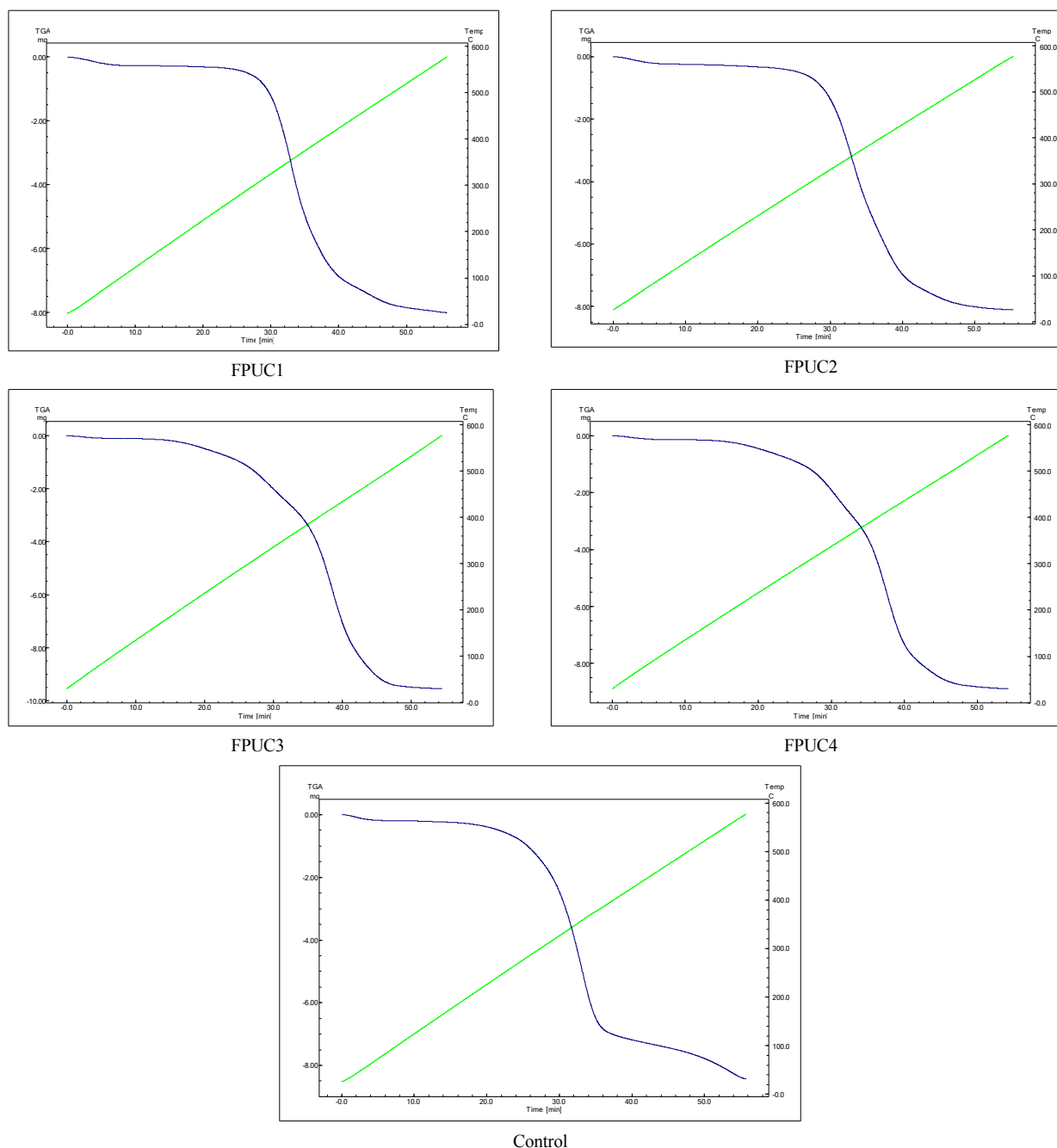


Fig. 5. TGA graphs of FPUC1, FPUC2, FPUC3, FPUC4, and control after degradation in soil for 30 days

FPUC1 had a weight loss of 2.78% at 180°C and a weight loss of 77.93% at 530°C. FPUC2 had a weight loss of 3.30% at 180°C and a weight loss of 78.07% at 530°C. A weight loss of 1.50% at 180°C and a weight loss of 94.25% at 530°C are observed for FPUC3. FPUC4 had a weight loss of 1.77% at 180°C and a

weight loss of 87.02% at 530°C. The control sample showed weight loss of 2.80% at 180°C and 89.90% at 530°C. These results indicated that all flexible polyurethane foams lost dimensional stability due to breaking chemical bonds (urethane bonds and ester bonds) and forming oligomers.

3.5. Scanning Electron Microscope (SEM)

Observation of foam morphology used SEM brand Hitachi S-3400N. Fig. 6 shows the morphology of all flexib-

le polyurethane foam samples synthesized from palm oil polyols combined with PEG-400 and commercial polyols degraded during 30 days in fertile. The content of palm oil polyol was more sensitive to attack by microorganisms.

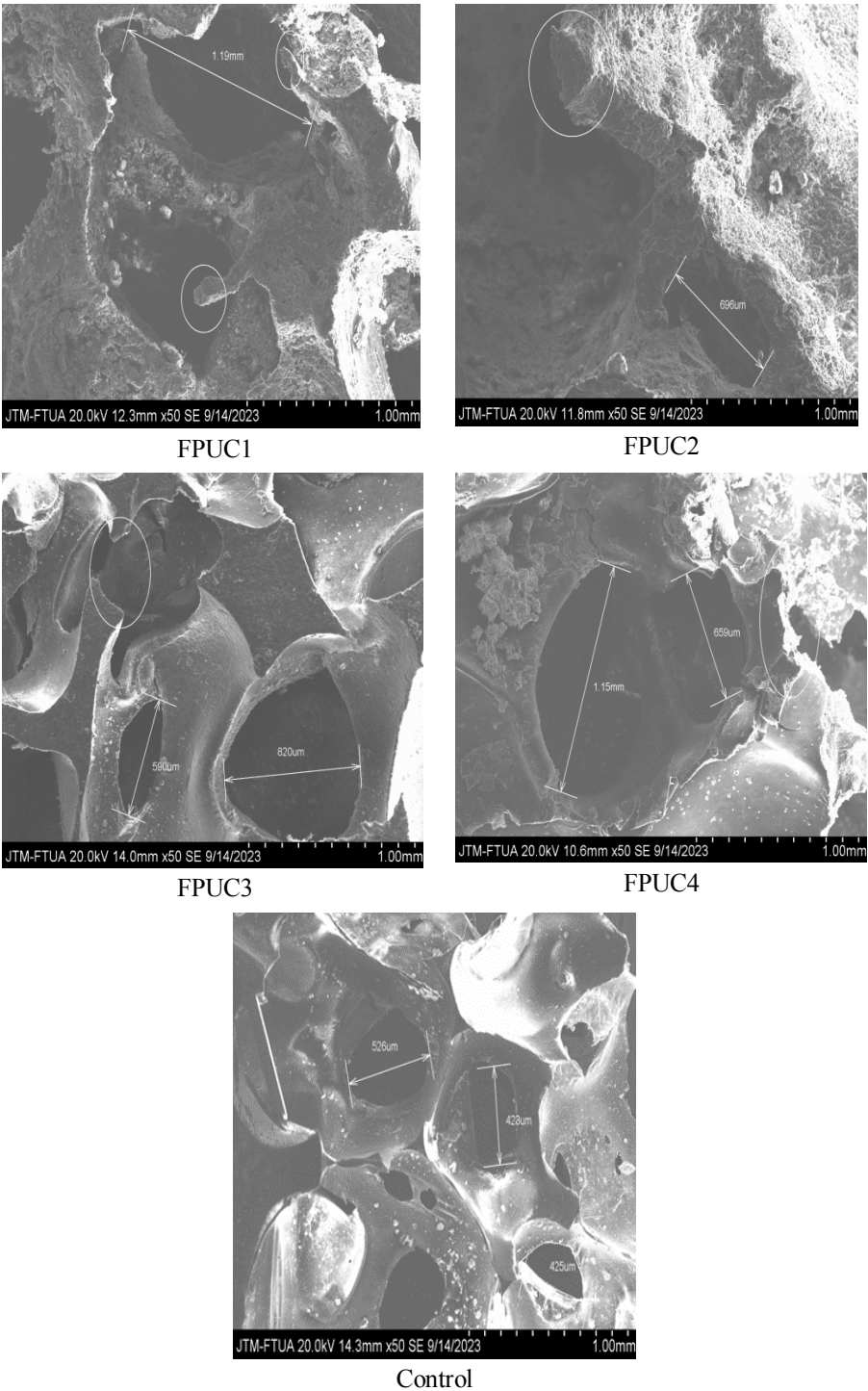


Fig. 6. SEM of FPUC1, FPUC2, FPUC3, FPUC4, and control after degradation in soil for 30 days

It was indicated by the rough surface of the foam and the broken cell skeleton, resulting in increased pore size, which led to the cracking of the material. Evidence of degradation on the surface of flexible polyurethane foam is the appearance of cracks, voids, holes of various sizes, and surface irregularities. The cellular structure became irregular and contained large cells with thick cell walls, increasing the number of large interconnected pores. The most apparent degradation was seen in the thinnest part of the material - the cell window membrane, which started to disappear completely, forming layers of porous structures and interconnecting small cells into more giant cells.

Control slightly degraded due to weight loss, although Fig. 6 did not show a broken cell framework. Massive polymeric supports and edges of large cells were preserved or only lightly damaged.

4. Conclusions

Palm oil-based flexible polyurethane foam composite can replace conventional flexible polyurethane foam (control) because it has similar characteristics. This research has successfully proven the degradation ability of FPUC. FPUC1 (60% palm oil polyol: 40% PEG-400) had a higher degradation rate (weight loss of 47%) compared to other FPUCs and controls. FPUC degradation occurs due to enzyme activity produced by soil microbes. FTIR, XRD, TGA, and SEM tests show that polyester (palm oil polyols) degrades more easily than polyether. The crystalline structure of PEG-400 and commercial polyols causes a longer degradation time than palm oil polyols (amorphous). The surface morphology of the degraded FPUC1 looks rough. So, FPUC1 can be said to be more environmentally friendly and has the potential to be utilized by various industries.

Acknowledgements

This research was funded by the DIPA Faculty of Agricultural Technology 2023 under Contract Number 01C/PL/PN-UNAND/FATETA-2023.

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Received: February 12, 2024 / Revised: June 18, 2024 /

Accepted: July 04, 2024

ОЦІНКА ДЕГРАДАЦІЇ В ҐРУНТІ ГНУЧКИХ ПІНОПОЛІУРЕТАНОВИХ КОМПЗИТИВ НА ОСНОВІ ПАЛЬМОВОЇ ОЛІЇ

Анотація. У даній статті подано інформацію про порівняння деградації гнучкого пінополіуретану на основі пальмової олії з комерційним гнучким пінополіуретаном (контрольним зразком) у ґрунті протягом 30 днів. Зразок, використаний у цьому дослідженні, – це композитний пінополіуретан, що містить близько 50-60% поліолу пальмової олії і має характеристики, подібні до звичайного пінополіуретану. Новизна цього дослідження полягає у вивченні швидкості деградації композитного гнучкого пінополіуретану з вмістом поліолу пальмової олії 50-60%. У результаті дослідження було розроблено екологічно безпечну рецептуру композиційного пінополіуретану на основі наступних параметрів (втрата маси, FTIR, XRD, TGA та SEM).

Ключові слова: деградація, жорсткий сегмент, м'який сегмент, уретановий зв'язок, втрата маси.