PREPARATION AND CHARACTERIZATION OF SUSTAINABLE LIGNIN FROM OIL PALM EMPTY FRUIT BUNCH (OPEFB) FOR POLYLACTIC ACID (PLA) BIOCOMPOSITE MATERIAL IN 3D PRINTING

BY

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ABSTRACT

3D printing is one of the additive manufacturing technologies that has widely been used in the automotive and manufacturing industry. Polylactic acid (PLA) is one of the materials used in 3D printing, made up of linear polymeric structure, that resulted in lower mechanical properties compared to other polymer materials used such as polyamide (PA), acrylonitrile butadiene styrene (ABS), and polycarbonate (PC). The reinforcement of lignin into PLA is not only capable to improve its stiffness but also provides thermal stability and antioxidant properties in PLA/lignin biocomposite. However, the interfacial adhesion between PLA and lignin had reduced the tensile strength and elongation at break of this biocomposite. Hence, this study aimed to utilize the lignin from oil palm empty fruit bunch (OPEFB) by dioxane-based extraction in PLA/lignin biocomposite. A dioxane-based extraction method is one of the solvent extraction processes capable to extract the native structure of lignin from lignocellulosic biomass. OPEFB was used as the source of lignin due to its availability in South East Asia (SEA). The lignin extraction process was optimized by using one-factor-at-time (OFAT) and response surface method (RSM) optimization. The factors that been optimized were temperature (range: 70 to 90ºC), dioxane concentration (range: 90 to 97 %(v/v)), solvent/solid ratio (range: 6 to 10 ml/g), hydrochloric acid concentration (range: 0.1 to 0.5 M) and retention time (range: 40 to 140 min). The optimized factors were further used to extract lignin for PLA/lignin biocomposite. The PLA/lignin biocomposite samples were prepared with a lignin content of 0.5, 1.0, 1.5 and 2.0 *wt%* in filament and 3D printed form. The highest extraction yield of lignin was 10.64% by using 1,4-dioxane with 0.1M of hydrochloric acid (HCl) as an acid catalyst at 90ºC and 10 ml/g of solvent/solid ratio for 140 minutes. The extracted lignin consisted of 92% of acid-insoluble lignin and 0.1% of acid-soluble lignin. The Fourier-transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) confirmed the release of lignin with low contamination of cellulose and hemicellulose. Apart from that, lignin from OPEFB showed an additional carbonyl group in the chemical structure of lignin. Thermogravimetric analysis (TGA) showed that the extracted lignin started to degrade around 200ºC. The Young's modulus had increased 27% after the reinforcement of 0.5 *wt%* of lignin (PLAL0.5) compared to PLA. No reduction in tensile strength and elongation at break was observed during the tensile test. Lignin also acted as a nucleation crystallization agent, which could increase the crystallinity of PLA/lignin biocomposite and provide mechanical strength. The differential scanning calorimetry (DSC) confirmed that the crystallinity of PLA/lignin biocomposite was increased only after 1 *wt%* of lignin reinforcement (PLAL1.0). The 3D printing that involved the melting and cooling process further improved the degree of crystallinity (X_c) of PLAL1.0. Hence, the PLAL1.0 was selected as the best lignin content into PLA with the highest value of Young's modulus of 2.14 GPa. Also, no interlayer adhesion was observed in 3D printed PLAL1.0. The lignin from OPEFB by dioxane-based extraction successfully increase the stiffness without any reduction in the ductility of PLA for 3D printing application.

خلاصة البحث

تعد الطباعة ثلاثية الأبعاد إحدى تقنيات التصنيع بالإضافة التي تم استخدامها على نطاق واسع في مجال التصنيع وصناعة السيارات. حمض البوليلاكتيك (PLA) هو أحد المواد المستخدمة في الطباعة ثلاثية الأبعاد، ويتكون من هيكل بوليمري خطي، مما يؤدي إلى انخفاض خصائصه الميكانيكية مقارنة بالمواد البوليمرية الأخرى المستخدمة مثل البولي أميد (PA)، وأكريلونيتريل بوتادين ستايرين (ABS)، والبولي كربونات (PC). إنّ تقوية (PLA) باللجنين لا تقتصر على تحسين صلابته فحسب، بل توفر أيضاً ثباتاً حرارياً وخصائص مضادة للأكسدة في المركب الحيوي (PLA/ اللجنين). ومع ذلك، فإن الالتصاق السطحي بين (PLA) واللجنين قد قلل من قوة الشد والاستطالة عند الكسر لهذا المركب الحيوي. ومن هنا، هدفت هذه الدراسة إلى استخدام اللجنين المستخلص من العناقيد الفارغة لثمار زيت النخيل (OPEFB) باستخدام الديوكسان في مركب (PLA/ اللجنين). وطريقة الاستخلاص المعتمدة على الديوكسان هي إحدى عمليات الاستخلاص بالمذيبات القادرة على استخلاص البنية الأصلية للجنين من الكتلة الحيوية اللّجنوسليلوزية. وقد استخدمت عناقيد (OPEFB) كمصدر للجنين نظراً لتوافرها في جنوب شرق آسيا. وتم تحسين عملية استخلاص اللجنين باستخدام عامل واحد في كل مرة (OFAT) وطريقة التحسين: سطح الاستجابة (RSM). وكانت العوامل التي تم تحسينها هي درجة الحرارة (النطاق: 70 إلى 90 درجة مئوية)، وتركيز الديوكسان (النطاق: 90 إلى 97٪ (حجم/ حجم))، ونسبة المذيب/ الصلب (النطاق: 6 إلى 10 مل/ غم)، وتركيز حمض الهيدروكلوريك (النطاق: 0.1 إلى 0.5 مولار)، ووقت الاستبقاء (النطاق: 40 إلى 140 دقيقة). وقد تم استخدام العوامل المحسّنة كذلك لاستخلاص اللجنين للمركب الحيوي (PLA/ اللجنين). حيث تم تحضير عينات المركب الحيوي (PLA/ اللجنين) بمحتوى من اللجنين بنسبة 0.5 و1.0 و1.5 و2.0 وزن٪ على شكل خيوط مطبوعة ثلاثية الأبعاد. وقد كانت أعلى إنتاجية لاستخلاص اللجنين 10.64٪، وذلك باستخدام 1، 4-ديوكسان مع 0.1 مولار من حمض الهيدروكلوريك (HCl) كمحفز حمضي عند 90 درجة مئوية و10 مل/ غم من نسبة المذيب/ الصلب لمدة 140 دقيقة. ويتكون اللجنين المستخلص من 92٪ من اللجنين غير القابل للذوبان في الحمض و0.1٪ من اللجنين القابل للذوبان في الحمض. وقد أكد تحليل فورييه الطيفي للأشعة تحت الحمراء (FTIR) والمسح المجهري الإلكتروني (SEM) إطلاق اللجنين مع انخفاض تلوث السليلوز والهيميسليلوز. وبعيداً عن ذلك، أظهر اللجنين من (OPEFB) مجموعة كربونيل إضافية في التركيب الكيميائي للجنين. كما أظهر التحليل الحراري الوزني (TGA) أن اللجنين المستخلص بدأ في الانحلال عند حوالي 200 درجة مئوية. وزاد معامل يونج بنسبة 27٪ بعد التقوية بمقدار 0.5 وزن ٪ من اللجنين (PLAL0.5) مقارنة مع (PLA). ولم يلاحظ أي انخفاض في قوة الشد والاستطالة عند الكسر أثناء اختبار الشد. كما عمل اللجنين أيضاً كعامل تبلور للتنوّي، حيث يمكنه أن يزيد من تبلور المركب الحيوي (PLA/ اللجنين) وينتج قوة ميكانيكية. كما أكد مسعر المسح التفاضلي (DSC) أن تبلور المركب الحيوي (PLA/اللجنين) قد زاد فقط بعد 1 وزن٪ من تقوية اللجنين (PLAL1.0). إنّ عملية الطباعة ثلاثية الأبعاد التي تضمنت كذلك عملية الصهر والتبريد أدت إلى تحسين درجة التبلور (Xc) للجنين (PLAL1.0)، لذلك، تم اختيار (PLAL1.0) كأفضل محتوى لجنين في (PLA) مع أعلى قيمة لمعامل يونج البالغ 2.14 جيجا باسكال. كذلك لم يلاحظ أي التصاق للطبقة البينية في المطبوع ثلاثي الأبعاد (PLAL1.0). لقد نجح اللجنين من (OPEFB) عن طريق الاستخلاص القائم على الديوكسان في زيادة الصلابة دون أي انخفاض في ليونة (PLA) في تطبيق الطباعة ثلاثية الأبعاد.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science in Engineering

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DECLARATION

I hereby declare that this thesis is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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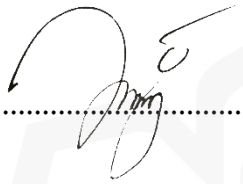
Signature  Date .....09/12/2021.....

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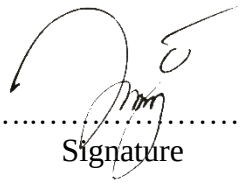

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LIST OF ABBREVIATIONS

2-MTHF	2-methyl tetrahydrofuran
3D	3 dimensional
ABS	Acrylonitrile butadiene styrene
AEL	Alkaline extracted lignin
ANOVA	Analysis of variance
ASTM	American Society for Testing Material
BDE	Bond dissociation energy
CPO	Crude palm oil
CPOPC	Council of Palm Oil Producing Countries
DES	Deep eutectic solvent
DPPH	2,2-diphenyl-1-picrylhydrazyl
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetric
EDS	Electron diffraction spectroscopy
FESEM	Field emission scanning electron microscopy
FTIR	Fourier-transform infrared
HBA	Hydrogen bond acceptor
HBD	Hydrogen bond donor
HTSt	High temperature short time
KL	Klaxon lignin
L	Linear term
MWL	Milled wood lignin
OFAT	One-factor-at-a-time
OPEFB	Oil palm empty fruit bunch

OPFFB	Oil palm fresh fruit bunch
PA	Polyamide
PC	Polycarbonate
PLA	Polylactic acid
PVA	Polyvinyl alcohol
Q	Quadratic term
RSM	Response surface method
RSPO	Roundtable on Sustainable Palm Oil
SEM	Scanning electron microscopy
TG	Thermogravimetric
TGA	Thermogravimetric analysis
USA	United States of America
UTM	Universal testing machine
UV	Ultraviolet

LIST OF SYMBOLS

ºC	degree Celsius
% (*v/v*)	percentage volume per volume
C	Carbon
Cl	Chlorine
H	Hydrogen
H_2S	Hydrogen Sulfide
HCl	Hydrochloric acid
K_2O	Potassium oxide
M	Molarity
N	Nitrogen
NaOH	Sodium hydroxide
NaHS	Sodium hydrosulfide
NH_3	Ammonia
O	Oxygen
OH	Hydroxyl
P_2O_5	Phosphorus pentoxide
Ru/TiO2	Ruthenium/Titanium dioxide
SiO_2	Silica bodies (silicon dioxide)
T_g	Glass transition temperature
T_m	Melting temperature
T_c	Crystallization temperature
wt%	Weight percentage
X_c	Degree of crystallization

CHAPTER ONE
INTRODUCTION

1.1 BACKGROUND OF STUDY

3D printing gains huge interest as an additive manufacturing technology in recent years. 3D printing is a creation of a physical object, designed by a graphical construction of computer-aid design and built with successive addition of material (Shahrubudin et al., 2019). First developed by Charles W. Hull in 1986 with the name of stereolithography, now 3D printing is also used in medical applications (Maroulakos et al., 2019). Among the medical applications that use 3D printing are bone reconstruction, rehabilitation, and regeneration (Maroulakos et al., 2019). In the manufacturing industry, 3D printing outputs are used as prototypes before proceeding with real production (Attaran, 2017). Low-melting-point polymers such as polylactic acid (PLA), polyamide (PA), acrylonitrile butadiene styrene (ABS), and polycarbonate (PC) are used as materials in 3D printing. Among them, PLA has been used widely as it is a toxic-free chemical and it does not cause any irritation on human sensitive skin (Z. Liu et al., 2019). The biodegradability of PLA also has been taken into consideration in material selection due to environmental preservation (Z. Liu et al., 2019).

However, the mechanical strength of the PLA could become a limitation compared to other polymeric materials in 3D printing (Z. Liu et al., 2019). This was due to the simple linear molecular structure of PLA, meanwhile, ABS, PA, and PC contain aromatic and branched graph structures (Z. Liu et al., 2019). Hence, composite studies with the reinforcement of potential filler have been performed to overcome this limitation. PLA biocomposite has been studied for years to increase the selectivity of

using biodegradable PLA in 3D printing (Ishii et al., 2018; Matsuzaki et al., 2016; Palmero et al., 2018). The potential use of lignin from the lignocellulose as a filler in the composites can increase the mechanical strength of PLA (Zhang et al., 2019). This is because lignin has an aromatic structure to support the linear structure of the PLA and improve the stiffness in the PLA/lignin biocomposite material (Bajpai, 2018; Z. Liu et al., 2019). Not only that, the reinforcement of lignin into PLA also could provide thermal stability and antioxidant properties in the PLA/lignin biocomposite (Domínguez-Robles et al., 2019; Mimini et al., 2019). Also, lignin is the second most abundant renewable polymer that could be obtained from biomass and agricultural waste (Yearla & Padmasree, 2015).

In the previous studies, several methods have been developed to extract the lignocellulose from the biomass. Among the solvent extraction methods used were alkaline treatment, deep eutectic solvent (DES) treatment, and organosolv treatment (Tian et al., 2017). It is important to take note that, the different types of lignin extraction processes could provide a different structure of lignin for its potential usage (Tang et al., 2020). Oil palm empty fruit bunch (OPEFB) is one of the available lignin sources in South East Asia (SEA) that could be used in the extraction of lignin (Hamzah et al., 2019). OPEFB waste is made of dry matter such as cellulose, hemicellulose, and lignin. Maryana et al. (2019) stated that OPEFB contains 15.36% lignin, 20.27% hemicellulose, 42.56% cellulose, and 21.10% water-soluble compound. The complex matrix of lignocellulose in plant function as a support for plant structure and as a defense mechanism from pathogens (Cragg et al., 2015). With high lignocellulose content, the handling of OPEFB waste must be managed properly. Improper handling of OPEFB could lead to anaerobic digestion and the release of toxic gases toward the surrounding

area (Muna et al., 2019). In conjunction with that, the complex lignocellulose structure from OPEFB is favorable for re-use as filler in composite material and various other applications. The use of OPEFB in lignin extraction process also one of the sustainable methods to reduce the agricultural waste produced.

1.2 PROBLEM STATEMENT

Although the reinforcement of lignin into the PLA could provide a wide advantage, poor interfacial adhesion between the lignin and PLA had limited the mechanical strength of this biocomposite (Hong et al., 2021). Even with the increment of Young's modulus, the elongation at break and tensile strength were reduced after the reinforcement of lignin into PLA (Gkartzou et al., 2017). The hydroxyl content in the chemical structure of lignin is the factor in the poor interaction between the PLA and lignin (Hong et al., 2021; Obielodan et al., 2019). Different types of extraction processes could provide different yields and structures of lignin that would cater toward its various applications (Tang et al., 2020). Another approach was used in this study, where 1,4-dioxane had been used as an extraction solvent in the lignin extraction process from OPEFB. The extraction using 1,4-dioxane could extract the native structure of lignin from lignocellulosic biomass with low carbohydrate contamination (Lu et al., 2017; Saha et al., 2019). With low contamination of cellulose and hemicellulose, dioxane-based extraction could reduce hydroxyl content in lignin. However, the extraction method using dioxane has not been reported on OPEFB as a raw material for the extraction of lignin. Hence, an optimization process was needed to extract the highest yield of lignin from OPEFB by using 1,4-dioxane, followed by the characterization of lignin.

Apart from that, high lignin content was not in favor of a better mechanical strength of PLA/lignin biocomposite. The higher the lignin content, the Young's modulus of PLA/lignin biocomposite begins to drop (Gkartzou et al., 2017). The lignin content below 5 *wt%* was recommended to provide a better Young's modulus of this biocomposite material (Obielodan et al., 2019). In conjunction with that, the reinforcement of lignin lower than 5 *wt%* was used in this study for the PLA/lignin biocomposite. Also, the tensile test for PLA/lignin biocomposite was prepared in filament and 3D printed form. Surface morphology and thermal/crystallization analysis also could provide a better understanding of the interaction of lignin and PLA in PLA/lignin biocomposite.

1.3 IMPORTANCE OF STUDY

In the current study, PLA/lignin biocomposite for 3D printing was done on different types of lignin (Mimini et al., 2019). Among the problem encountered was the reduction in ultimate strength and elongation at break due to the poor surface adhesion between PLA and lignin (Hong et al., 2021). Hong et al. (2021) stated that lignin-reinforced biocomposite is still a challenge in the production of polymer filaments. Theoretically, lignin is hydrophobic and could blend well with other hydrophobic materials such as PLA (Patel & Parsania, 2018). In conjunction with that, the selection of the lignin extraction method is crucial since it affects the structure of the extracted lignin. 1,4-dioxane could extract the lignin without any major modification on the microstructure of lignin during the extraction process (Lu et al., 2017). The reinforcement of lignin from OPEFB extracted by 1,4-dioxane into PLA for 3D printing at low lignin content was a novelty of this study, which could potentially promote the use of non-toxic biomaterial for 3D printing.

As the extraction process could affect the properties of the extracted lignin, most studies tried to find the best method of the lignin extraction process. From the selection of the most suitable extraction solvents, the extraction of lignin has evolved to a two-stage treatment to achieve the optimum extraction process (Lu et al., 2017). Apart from the extraction process, the types of lignocellulosic biomass could also affect the quality of the lignin. The extraction of lignin in various types of lignocellulosic biomass has been conducted in many studies (Karmanov et al., 2020; Li et al., 2017; Zhang et al., 2020). In Malaysia, OPEFB is one of the lignocellulosic biomasses frequently been used as a source for lignin extraction. This is due to the availability of OPEFB in Malaysia as the second-largest contributor in CPO production. Lignin from OPEFB also had been extracted with various types of solvents, such as alkaline soda and ethanol organosolv (Tian et al., 2017). This study had utilized another organic solvent, 1,4-dioxane in the extraction of lignin from OPEFB with HCl as an acid catalyst.

1.4 RESEARCH OBJECTIVES

The objectives of this study are as follow:

1. To optimize the lignin extraction process from OPEFB using 1,4-dioxane as extraction solvent to gain a high extraction yield.
2. To analyze the extracted lignin by morphology, spectroscopy, and thermogravimetric analysis.
3. To study the effect of lignin from dioxane-based extraction in PLA/lignin biocomposite as a filament and 3D printed sample with the lignin content of 0.5, 1.0, 1.5, and 2.0 *wt%*.

1.5 SCOPE OF STUDY

This study focused on optimization of the lignin extraction process by using 1,4-dioxane from OPEFB to gain a high yield of lignin for the preparation of PLA/lignin biocomposite material in 3D printing.

i. Lignin extraction

The process utilized 1,4-dioxane as an extraction solvent in extracting lignin from OPEFB. The optimization technique had been done which included several factors such as temperature (range: 70 to 90ºC), the concentration of dioxane (range: 90 to 97 %(*v/v*)), the concentration of HCl (range: 0.1 to 0.5 M), retention time (range: 40 to 140 min) and solvent/solid ratio (range: 6 to 10 ml/g). The factor level for optimization was taken from the previous treatment with a different kind of lignocellulosic biomass. The optimization techniques used were one-factor-at-a-time (OFAT) and response surface method (RSM) toward the response, extraction yield (%).

ii. Characterization

The properties of the extracted lignin were characterized for its morphology, spectroscopy, and thermogravimetric analysis. Morphology analysis on scanning electron microscopy (SEM) of extracted lignin was performed. Spectroscopy analysis was undertaken to determine the functional group present in the extracted lignin by using Fourier-transform infrared (FTIR) spectroscopy. The thermogravimetric analysis (TGA) observed the degradation behavior of the extracted lignin with increasing temperature.

iii. PLA/lignin biocomposite

The extracted lignin was reinforced into the PLA in the preparation of PLA/lignin biocomposite material. The samples were prepared in filament and 3D printed form

with various lignin content at 0.5, 1.0, 1.5, and 2.0 *wt%*. The universal testing machine (UTM) was utilized to measure the tensile properties. The fracture surface was observed by scanning electron microscopy (SEM) for morphology analysis. The PLA/lignin biocomposite proceeded for thermal analysis. Thermogravimetric analysis (TGA) observed the degradation behavior of PLA/lignin biocomposite filament with increasing temperature. Differential scanning calorimetry (DSC) showed the glass transition (T_g), crystallization (T_c), and melting temperature (T_m) of PLA/lignin biocomposite.

1.6 THESIS ORGANIZATION

This thesis was completed with five chapters. Chapter One was a brief explanation of the background of the study which was related to 3D printing in additive manufacturing. The benefits of use and limitation of PLA as a polymeric material in 3D printing also have been discussed briefly. This chapter also explained the potential use of lignin within OPEFB as a filler for biocomposite material in 3D printing. Problems statements, objectives, scope, and importance of study were stated clearly in this chapter.

In Chapter Two, the beneficial use of lignin compared to other lignocellulose components for the PLA biocomposite study had been explained. Also, the current problem regarding PLA/lignin biocomposite had been discussed. The structure of lignin linkage in lignocellulose and its monomer was well illustrated in this chapter. Apart from that, the availability of OPEFB in Malaysia and its neighboring countries was explained thoroughly. The literature review on the different lignin extraction methods and the outcomes from the previous study was discussed with its extraction mechanism.

The materials and apparatus used during the research were specified in Chapter Three. The experimental procedure was described in detail on the preliminary treatment