UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT AND CHARACTERISATION OF SUGAR PALM [Arenga pinnata (Wurmb) Merr.] FIBRE REINFORCED VINYL ESTER COMPOSITES

MUHAMMAD HUZAIFAH BIN MOHD ROSLIM

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

To my beloved father and mother for their invaluable sacrifices, encouragements and support throughout my life

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To my beloved wife for her love, patience and understanding

&

To my awesome 8 siblings

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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May 2019

Chairman : Mohd Sapuan Bin Salit, PhD
Faculty : Institute of Tropical Forestry and Forest Products

Recently, environmental issue have raised awareness many parties including researchers and now numerous research are done to replacing synthetic fibres with natural fibres as the main component in composites. Natural fibres are preferred compared to synthetic fibres due to several imminent advantages, such as biodegradable, light in weight, low in cost, and good mechanical properties. With such qualities, sugar palm tree is a plant that has been found to be suitable to be used to produce natural fibres. Hence, this research focused on the capability and the sustainability of sugar palm fibre (SPF) as lignocellulosic reinforcement for polymer composites. For that purposes, this research consist of four parts. In the first part, characterisations of SPFs obtained from three geographical locations i.e. Kuala Jempol (Peninsular Malaysia), Tawau (West Malaysia), and Tasik Malaya (Indonesia) in terms of its mechanical, thermal, physical, morphology, and chemical properties were done. The finding confirmed that, the highest chemical content of cellulose resulted in the highest strength and thermal stability of the fibre. Fibre originating from Kuala Jempol had the highest cellulose content of 44.53%, followed by Indonesia (44.47%) and Tawau (43.75%). Kuala Jempol fibre (233.28 MPa) also had the highest tensile strength, followed by Indonesia (211.03 MPa) and Tawau (201.30 MPa), which was affected by the cellulose content in the fibre. Then, the second part of this research investigated the effect of different fibre content, which are 10 wt.%, 20 wt.%, 30 wt.% and 40 wt.%. From the results, the additions of SPFs to vinyl ester decrease tensile and flexural strength of the composites, which are 25.1 MPa (10 wt.%), 12.5 MPa (20 wt.%), 9.7 MPa (30 wt.%) and 6.1 MPa (40 wt.%) for tensile strength, while for flexural strength, the results recorded 48.5 MPa, 24 MPa, 18.8 MPa and 2.5 MPa for 10 wt.%, 20 wt.%, 30 wt% and 40 wt.%, respectively. However, SPFs improve impact strength of the composites especially at 30 wt% with 5.4 kJ/m^2. The addition of SPFs also raise the water absorption and thickness swelling of the composites as the presence of fibres in the composites increase water retained in the composites. Besides that, thermal stability of the composites also reduces by the addition of the SPFs that shows the onset temperature of thermal degradation of 10 wt.% was at 270.83˚C, while 40 wt.% was at 196.67˚C. Next, the third part of this research is about the characterisations of SPFs obtained from three geographical locations reinforced vinyl ester composites on the physical, thermal, and mechanical properties. The SPFs were utilized as reinforcement material with a fixed loading of 10 wt.%. The reinforced VE composites were prepared using a wet lay-up compression moulding method. The physical properties examined were water absorption, thickness swelling, and moisture content. To determine the strength of the SPF composites, tests on the tensile, flexural, and impact strength related to mechanical properties were completed. A thermogravimetric analysis (TGA) was completed to observe the thermal properties. This study confirmed that the properties of the composites were affected by the strength of the fibre. The SPF/VE composites obtained from Kuala Jempol had the highest tensile, flexural, and impact strength compared to the SPF/VE composites from Indonesia and Tawau. In addition, SPF Jempol/VE also recorded the highest percentage of water absorption, thickness swelling, and moisture content. A comparison of thermal properties showed that SPF Tawau/VE had highest percentage of mass loss between fibres from the three geographic locations. Finally, the last part of this research is to investigate the effect of soil burial on physical, mechanical and thermal properties of SPF (SPF) reinforced vinyl ester (VE) composites. Neat VE and SPF/VE composites at weight ratio of 10 wt% SPF were prepared using wet hand lay-up method. The specimens were buried in the soil for 0, 200, 400, 600, 800 and 1000 hours to investigate the effect of soil burial to its physical properties (i.e. water absorption), mechanical properties (i.e. tensile, flexural and impact strength) and thermal properties. Obtained results indicated that, the longer soil burial period led to high water absorption of the composites. After 200 hours of soil burial, the SPF/VE composites water uptake increased 0.92% compared to neat VE which is 0.42%. Based on the results, soil burial reduces the mechanical strength of the composites. Tensile, flexural and impact strength decreased rapidly after 200 hours of soil burial compared to neat VE which were 28.24%, 8.04% and 14.83%, respectively. Thermal stability after 200 hours of soil burial increase temperature onset of the SPF composites compared to neat VE. Overall, soil burial increased water absorption, temperature onset of the SPF composites and at the same time decreased the tensile, flexural and impact strength of the composites.

Abstrak tesis dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN DAN PENCIRIAN KOMPOSIT VINIL ESTER DIPERKUAT GENTIAN IJUK [Arenga pinnata (Wurmb) Merr.]

Oleh

MUHAMMAD HUZAIFAH BIN MOHD ROSLIM

Mei 2019

Pengerusi : Mohd Sapuan Bin Salit, PhD
Fakulti : Institut Perhutanan Tropika dan Produk Hutan

Baru-baru ini, isu alam sekitar telah meningkatkan kesedaran ramai pihak termasuk penyelidik dan kini banyak penyelidikan dilakukan untuk menggantikan gentian sintetik dengan gentian semulajadi sebagai komponen utama dalam komposit. Gentian semulajadi memiliki beberapa kelebihan seperti mudah terurai, ringan, murah, dan sifat mekanikal yang baik. Dengan kualiti seperti itu, pokok enau adalah tumbuhan yang sesuai untuk digunakan sebagai gentian semulajadi. Oleh itu, kajian ini memberi tumpuan kepada keupayaan dan kelestarian gentian enau atau dikenali sebagai ijuk sebagai tetulang lignoselulosa bagi komposit polimer. Untuk tujuan itu, kajian ini terdiri daripada empat bahagian. Pada bahagian pertama, ciri-ciri gentian ijuk diperoleh dari tiga lokasi geografi iaitu Kuala Jempol (Semenanjung Malaysia), Tawau (Malaysia Barat), dan Tasik Malaya (Indonesia) dari segi sifat mekanik, haba, fizikal, morfologi dan kimia telah dikaji. Penemuan itu mengesahkan bahawa, kandungan kimia tertinggi selulosa menghasilkan kekuatan tertinggi dan kestabilan haba gentian. Gentian yang berasal dari Kuala Jempol mempunyai kandungan selulosa tertinggi sebanyak 44.53%, diikuti oleh Indonesia (44.47%) dan Tawau (43.75%). Gentian Kuala Jempol (233.28 MPa) juga mempunyai kekuatan tegangan tertinggi, diikuti oleh Indonesia (211.03 MPa) dan Tawau (201.30 MPa), yang terjejas oleh kandungan selulosa dalam gentian. Seterusnya, bahagian kedua kajian ini mengkaji kesan kandungan gentian yang berbeza, iaitu 10 wt. %, 20 wt. %, 30 wt. %dan 40 wt. %. Dari hasil kajian, penambahan enau ke ester vinil mengurangkan kekuatan tegangan dan lenturan komposit, iaitu 25.1 MPa (10 wt. %), 12.5 MPa (20 wt. %), 9.7 MPa (30 wt. %) dan 6.1 MPa (40 wt. %) untuk kekuatan tegangan, manakala untuk kekuatan lenturan, hasilnya mencatatkan 48.5 MPa, 24 MPa, 18.8 MPa dan 2.5 MPa untuk 10 wt. %, 20 wt. %, 30 wt. % dan 40 wt. %. Walau bagaimanapun, gentian enau meningkatkan kekuatan impak komposit terutama pada 30 wt. % dengan 5.4 kJ / m^2. Penambahan gentian enau juga meningkatkan penyerapan air dan kadar pembengkakan komposit kerana kehadiran gentian dalam komposit meningkatkan air yang disimpan dalam komposit. Selain itu, kestabilan haba komposit juga dikurangkan dengan penambahan enau yang menunjukkan suhu permulaan degradasi haba sebanyak 10 % pada suhu 270.83 ˚C, manakala 40 % pada tahap 196.67 ˚C. Kemudian, bahagian ketiga kajian ini menkaji sifat ijuk yang diperoleh dari tiga lokasi geografi bertetulang komposit vinil ester pada sifat fizikal, haba, dan mekanik. SPFs digunakan sebagai bahan pengukuhan dengan peratusan tetap sebanyak 10%. Komposit VE telah disediakan menggunakan kaedah pengadun basah dan pembentukan mampatan. Ciri fizikal yang diperiksa adalah penyerapan air, kadar pembengkakan, dan kandungan lembapan. Untuk menentukan kekuatan komposit SPF, ujian pada tegangan, lenturan, dan kekuatan hentakan yang berkaitan dengan sifat mekanik telah dilakukan. Analisis termogravimetrik (TGA) telah dibuat untuk mengetahui sifat ketahanan haba. Kajian ini mengesahkan bahawa sifat-sifat komposit itu dipengaruhi oleh kekuatan gentiannya. Komposit SPF /VE yang diperoleh dari Kuala Jempol mempunyai kekuatan tegangan, lenturan, dan kesan tertinggi berbanding komposit SPF /VE dari Indonesia dan Tawau. Di samping itu, SPF Jempol /VE juga mencatatkan peratusan tertinggi penyerapan air, kadar pembengkakan, dan kandungan lembapan. Perbandingan sifat terma menunjukkan bahawa SPF Tawau / VE mempunyai peratusan tertinggi kehilangan berat antara gentian dari tiga lokasi. Akhirnya, bahagian terakhir penyelidikan ini adalah untuk mengkaji kesan pengebumian tanah ke atas komposit vinil ester (VE) yang diperkuat gentian enau (SPF) oleh sifat fizikal, mekanikal dan terma . Komposit rapi VE dan SPF / VE pada nisbah berat sebanyak 10 % SPF telah disediakan menggunakan kaedah pengadun basah dan pembentukan mampatan. Spesimen dikebumikan di dalam tanah selama 0, 200, 400, 600, 800 dan 1000 jam untuk menyiasat kesan pengebumian tanah kepada sifat fizikalnya (iaitu penyerapan air), sifat mekanik (iaitu kekuatan tegangan, lenturan dan kekuatan impak) dan haba hartanah. Hasil yang diperoleh menunjukkan bahawa, tempoh pengebumian tanah yang lebih lama menyebabkan penyerapan air yang tinggi komposit. Selepas pengebumian tanah selama 200 jam, SPF / VE menggabungkan pengambilan air meningkat 0.92 % berbanding dengan VE yang kemas iaitu 0.42 %. Berdasarkan keputusan, pengebumian tanah mengurangkan kekuatan mekanikal komposit. Kekuatan tegangan, lenturan dan kesan menurun dengan cepat selepas pengebumian tanah selama 200 jam berbanding dengan VE kemas yang masing-masing adalah 28.24 %, 8.04 % dan 14.83 %. Kestabilan haba selepas 200 jam pengebumian tanah menaikkan suhu komposit SPF berbanding dengan VE yang kemas. Secara keseluruhan, pengebumian tanah meningkat penyerapan air, permulaan suhu komposit SPF dan pada masa yang sama mengurangkan kekuatan tegangan, lenturan dan kesan komposit.

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Thank you.

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
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This is to confirm that:

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Signature: ______________________________
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Prof. Ir. Dr. Mohd Sapuan Salit

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Name of Member of Supervisory Committee:
Assoc. Prof. Dr. Zulkiffle Leman

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Name of Member of Supervisory Committee:
Dr Mohamad Ridzwan Ishak

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LIST OF ABBREVIATIONS

ADF	Acid detergent fibre
ANOVA	Analysis of variance
ASTM	American Society for Testing and Materials
DOA	Department of Agriculture
DTG	Derivative thermogravimetric
DSC	Differential scanning calorimetry
HVAC	Heat ventilation air conditioning system
MAH	Maleic anhydride
MEKP	Methyl ethyl ketone peroxide
MPOB	Malaysian palm oil board
NaOH	Sodium hydroxide
NFRP	Natural fibre reinforced plastic
NDF	Neutral detergent fibre
-OH	Hydroxy
OPF	Oil palm frond
OPT	Oil palm trunk
PE	Polyester
PP	Polypropylene
PLA	Poly lactic acid starch
PHAs	Polyhydroxyalkanoates
PBS	Polybutylene succinate
SEM	Scanning electron microscope
SPF	Sugar palm fibre
SPF/VE	Sugar palm fibre reinforced vinyl ester
SPB	Sugar palm bunch
SPT	Sugar palm trunk
SPF	Sugar palm frond
SPS	Sugar palm starch
TGA	Thermogravimetric analysis
TAPPI	Technical Association of the Pulp and Paper Industry
VE	Vinyl ester

LIST OF SYMBOLS

A	Average fibre area
dB	Decibel
F	Force to failure
GPa	Gigapascal
Hz	Hertz
J	Joule
kV	Kilovolt
kN	Kilonewton
M_0	Initial weight
M_1	Final weight
MPa	Megapascal
rpm	Revolutions per minute
µm	Micrometer
T	Tensile
T_0	Initial thickness
T_1	Final thickness
T_{peak}	Highest peak temperature for decomposition
T_{onset}	Onset temperature
T_{max}	Maximum peak temperature
T_{end}	Complete decomposition
$T_{10\%}$	Temperature at 10% mass loss
Wt%	Weight percentage
W_0	Initial weight
W_1	Final weight

CHAPTER 1

INTRODUCTION

1.1 Background

The fibre reinforced composites offer required mechanical features such as light weight but high stiffness and strength and corrosion resistant. These materials have wide application such as in the aircraft, automotive, aerospace, military, construction, marine, oil and gas and many industries (Madeo, 2015). Synthetic fibre such as Kevlar, aramid, glass fibre and others, are the initial material developed for fibre reinforced composites. In spite of its unique advantages such as environmental stability, thermal insulation and conductivity, synthetic fibres are made up of fossil fuels and required high energy consumption for production, processing and extraction (Begum & Islam, 2013). Besides that, synthetic fibre is a drawback to the environmental issue since it is non-degradable. This has led to many studies on natural fibres to replace synthetic fibres as reinforcement materials. Increasing interest on natural fibres as reinforcement materials are due to availability from renewable resources, low cost, light weight and biodegradable.

Sugar palm fibres (SPF), which are the black fibrous that wrapped around sugar palm tree, are the main material used in this research. This tree can easily found in the wild in Malaysia and the fibres from this tree are usually discarded by the farmers as it can disturb the process of sap and fruit harvesting. It can lead to the increasing of green waste and environmental problem since the only options to the farmers are to let it decay on the ground or burn it. Thus, developing bio composites from SPF can help to reduce green waste and help to reduce environmental problems (Sanyang et al., 2015). Previously, numerous research were done on different types of plant such as jute, kenaf, sisal, bamboo, coconut, hemp, ramie and flax (Rohit & Dixit, 2016)

Therefore, this study was conducted to investigate the properties of sugar palm (*Arenga pinnata*) fibre reinforced vinyl ester reinforcement from different geographical location, which are from Kampung Kuala Jempul, Negeri Sembilan, Kebun Rimau Sdn Bhd, Tawau, Sabah and Kampung Naga, Tasik Malaya, Indonesia. The effect from different location towards properties of sugar palm fibres were evaluate in terms of physical, thermal, and mechanical properties. After that, SPFs reinforced composites were prepared and the physical, mechanical, thermal and environment degradation were analysed. At the end of this research based on the result obtained, the best fibre can be identified to be used as a raw material for any suitable product or composites.

1.2 Problem statements

Since 1990s, many industries growing interest in natural fibre as a material in producing a new type of product as a result of increase awareness on environmental friendly products and to reduce the cost of synthetic fibre such as carbon, Kevlar, aramid etc. Major drawbacks to use synthetic fibre are the dependant on fossil fuel, which is non-renewable resources, can't be degraded when disposed and consume high cost and energy for processing. Other than problems coming synthetic fibre, another motivation to explore natural fibre as new reinforcement material are to coop with agricultural waste. One of the largest sectors that produce waste in Malaysia is from agriculture. The common practises to clear the agriculture waste among farmers are allowed to decay on the ground, on-farm burning, burial, stockpiling and landfilling. This research is expected to reduce environmental problems as mentioned by using agricultural waste as a new material for composites. Besides that, natural fibres have good fibre properties and can be as a substitute to the synthetic fibres especially in lightweight structure in numerous applications.

Natural fibre that is focused in this study is SPF. Traditionally, the villagers simply harvest the fruit and the sap from the tree and remove the fibres that cover the tree because it makes it difficult to climb. Based on observation, the unwanted fibres will be burned or abandoned by the villager. The used of the fibre for composites production can help to minimize the waste and can be a source of income for the villagers and create employments opportunities for rural area. It is important to understand the physical, thermal, mechanical, and chemical properties of SPFs before it is used in industrial applications such as in automotive, aircraft, and household item.

According to Lamidi et al. (2017) and Stanton et al. (2012), geographical location has certain impact to the properties of plant. This might due to climate, and soil characteristics. However, there is no study that investigates the relation between geographical locations with the properties of the plant. While, numerous studies claimed that certain properties of plant can affect the properties of the reinforced composites (Atiqah et al., 2017; Radzi et al., 2017; Fairuz et al., 2015; Nadlene et al., 2015). This research served as initial study to find out the effect of geographical location to the plant fibres and composites.

Previously, several researches were done investigation on SPF. For example, Atiqah et al. (2017) have done studied on sugar palm reinforced polyurethane composites, Norizan et al. (2017) sugar palm yarn fibre reinforced unsaturated polyester composites and Radzi et al. (2018) on roselle/SPF hybrid reinforced polyurethane composites. In addition, Haameem et al. (2016) have carried studied to investigate the effect of mechanical properties of napier grass (short and long fibre) reinforced polyester composites. From the results, the long fibre composites possess higher tensile strength than short fibre composites. This is similar with the finding from Leman et al. (2005) that shown the impact strength of long fibre composites possess higher value than short fibre composites. However, there is no study is done on properties SPF reinforced vinyl ester composites. Consequently, more research is needed in this area of study in order to determine the influence of location on the fibre properties. In addition, the effect of soil burial on physical, thermal and mechanical properties of SPF reinforced vinyl ester composites also been proposed to determine the degradation behaviour of the composites.

1.3 Research objective

The aim of this research is to investigate the properties of *Arenga pinnata* reinforced vinyl ester composites obtained from different geographical locations. The specific objectives of this research are:

- To determine physical, chemical, mechanical and thermal properties of sugar palm fibres from different geographical locations.
- To investigated the best ratio of fibre loading sugar palm fibre reinforced vinyl ester composites.
- To evaluate physical, mechanical and thermal properties of sugar palm fibre reinforced vinyl ester composites.
- To determine physical, mechanical and thermal properties of soil buried sugar palm fibre reinforced vinyl ester composites.

1.4 Significance of study

1. The findings from the current study are to explore the potential of new resources of natural fibres especially SPF for used as fibre in reinforced composite.
2. The successful development of natural fibres such as SPF will enhance living standards for farmers dealing with sugar palms in rural areas especially in Malaysia.
3. In addition, this study is expected to expose the potential of SPF in green or eco-friendly product development.
4. The development of SPF composites is expected to reduce environmental pollution during disposal.

1.5 Scope and limitation of study

The scope of this study is focusing on the properties of *Arenga pinnata* reinforced vinyl ester composites. The studies of this research were divided into four stages, which first stage is comparison between SPF obtained from three different geographical locations. The determination on material composition properties includes physical (density, water absorption, diameter and moisture content), thermal (TGA and DSC), chemical (cellulose, hemicellulose, lignin and ash) and mechanical (single fibre tensile test). The second stage of this research is determination the effect of different fibre loadings of SPF reinforced vinyl ester composites on the properties such physical (water absorption, thickness swelling, moisture content, and SEM), thermal (TGA), and mechanical (tensile stress, flexural and impact). Different fibre loadings that were prepared are 10%, 20%, and 30% (fibre by weight) by using a wet hand lay-up process. The third stage of this research is manufacturing the material which is SPF obtained from three different geographical locations reinforced vinyl ester composites. The sugar palm filler loading consisted only 10% loading. Finally, the effect of soil burial on physical, thermal and mechanical of SPF/VE were investigates.

1.6 Structure of thesis

The structure of this thesis is in accordance with the alternative thesis format of Universiti Putra Malaysia, which is based on journal publication. Each research chapter (journal paper) represents a separate research on its own: 'Introduction', 'Materials and methods', 'Results and discussion', and 'Conclusions'. The details of the thesis structure are presented as follows:

Chapter 1
Problem statements and objectives are presented in this chapter. The significance of the research work and the scope of research are also presented in this chapter.

Chapter 2
This chapter presents a comprehensive literature review on the areas related to the topic of this research. In addition, the research gaps obtained from the review were also clarified within the chapter.

Chapter 3
The methodology used in this research for the preparation of materials, testing procedures, and data collection is presented in this chapter.

Chapter 4
This chapter presents the first article "**Comparative study on chemical composition, physical, tensile, and thermal properties of sugar palm fibre (Arenga pinnata) obtained from different geographical locations**". In this article, the properties of SPF (*Arenga pinnata*) obtained from different geographical locations in terms of chemical composition, physical, tensile, and thermal properties were investigated.

Chapter 5
This chapter presents the third article "**Effect of fibre loading on the physical, mechanical and thermal properties of sugar palm fibre reinforced vinyl ester composites**". In this article, the effect of different fibre loading on physical, mechanical, and thermal properties of sugar palm composites was investigated.

Chapter 6

This chapter presents the second article "**Comparative study of physical, mechanical, and thermal properties on sugar palm fibre (Arenga pinnata (Wurmb) merr.) reinforced vinyl ester composites obtained from different geographical locations**". In this article, properties of SPF (*Arenga pinnata*) obtained from different geographical locations reinforced vinyl ester composites in terms of physical, mechanical, and thermal properties were investigated.

Chapter 7

This chapter presents the fourth article "**Effect of soil burial on physical, mechanical and thermal properties of sugar palm fibre reinforced vinyl ester composites.**" In this article, the effect of soil burial on physical, mechanical and thermal properties of SPF reinforced vinyl ester composites was investigated.

Chapter 8

This chapter presents the overall conclusions from the whole study as well as future recommendations for further improvement of this study.

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M.I Ammar, **M.R.M Huzaifah**, S.M. Sapuan, M.R Ishak and Z. Leman and M.L Sanyang. Development of sugar palm fibre reinforced vinyl estercomposites IN *Natural Fibre Reinforced Vinyl Ester and Vinyl Polymer Composites: Characterisation, Properties and Applications*. Elsevier Science & Technology, 2018. (Published)

Other presentations (poster/product/exhibition/showcase)

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***Award & Consultancy**

Silver Medal, Pameran Rekacipta Penyelidikan & Inovasi (PRPI2016)
National Book Award 2018, The Best General Book (Category of Flora and Fauna) Title of book: Pokok Enau: Potensi dan Pembangunan Produk ISBN 978-967-344-747-3 Authors: Mohd Sapuan Salit, Mohamad Ridzwan Ishak and Zulkiflle Leman Co-Authors: Ahmad Ilyas Rushdan, **Muhammad Huzaifah**, Muhammad Ammar Ishak, Mohamad Hafiz Adha Ishak, Indra Yana, Publisher: UPM Press, Serdang, Selangor. Malaysia

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