

PINEAPPLE LEAF FIBER EXTRACTION AND ITS APPLICATION AS
KRAFT PAPER

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ABSTRACT

Cultivation of pineapple in Malaysia produces wastes such as leaf and crown where these wastes are discarded or left in the field. A systematic pineapple leaf fiber (PALF) extraction method was designed to maximally utilize this waste and make pineapple plantation more sustainable. Therefore, these four-parts of studies were conducted in order to develop an effective fiber extraction method. In Part 1 (method selection), PALF from Josapine cultivar was successfully extracted via manual retting (combing), water retting, chemical extraction or mechanical-chemical extraction methods. The mechanical-chemical method was found to be the best method to produce the whitish pulp while larger yield (4.0 wt%) of fine and clean fibrils of PALF was obtained using 3 wt% of sodium hydroxide (NaOH). In Part 2, (extraction parameter), the PALF extraction was done by cooking the PALF in NaOH at 1 wt% to 11 wt% NaOH concentrations for 30 to 180 minutes. Scanning electron microscopy images showed that the structure of fibre was starting to deteriorate when the concentration of NaOH was more than 10 wt%. Thermogravimetric analysis revealed that at the concentration of less than 3 wt% NaOH was insufficient to remove the hemicellulose layer from the fiber. It was also found that the amount of lignin removal was constant at 7 wt% NaOH onwards, as revealed by the value of Kappa number. Fourier transform infrared spectroscopy shows that at 3 wt% NaOH was sufficient to remove the hemicellulose and lignin from the PALF. In conclusion, 3 wt% NaOH concentration, 90 minutes of cooking time at 90 °C were identified as the optimum parameter to be used in the delignification process. Further discovery revealed that the NaOH solution can be reused one time only for the extraction process without significantly affected the extracted PALF's properties. In Part 3 (kraft paper production), kraft papers produced using the PALF extracted at different NaOH concentrations (1, 3, 5, 7, 9, and 11 wt%) and NaOH solution recycle were characterized in terms of tensile strength, tear strength and water absorption properties. It was found that the average tearing force of the kraft paper reached its maximum (216604.8 mN) at the NaOH concentration of 9 wt% while the tensile strength was at its maximum (13 MPa) at 3 wt% NaOH. It was also observed that by recycling 3 wt% NaOH solution led to the reduction of PALF paper tensile strength from 12.6 MPa (fresh NaOH) to 11 MPa (1st recycle) and 6.5 MPa (2nd recycle). At 3 wt% NaOH concentration and only one time of NaOH recycling were good processing parameters for obtaining PALF kraft papers with balance properties. In Part 4, the kraft paper prepared at the selective parameter (3 wt% NaOH, 90 minutes cooking time at 90 °C) was used as a reinforcing layer in the epoxy resin. The kraft paper/epoxy composites were prepared at different number of layers i.e. 1, 2 and 3 layers. The properties of the composites were measured via tensile test and Izod impact test. Results showed that the insertion of the papers' layers made of NaOH extraction into the epoxy resin had reduced the density and the tensile properties of the composites. However, the impact properties of the composites improved corresponding to the fiber pull out as a result of weak fiber-matrix bonding. In contrast, the composite that used the papers made of manual retted PALF have weak impact strength due to the stronger fiber-matrix adhesion.

ABSTRAK

Penanaman nanas di Malaysia menghasilkan sisa seperti daun dan jambul di mana sisa ini dibuang atau pun dibiarkan di ladang. Kaedah pengekstrakan gentian daun nanas (PALF) yang sistematik telah direkabentuk bagi memaksimumkan penggunaan sisa ini dan menjadikan penanaman nanas lebih lestari. Maka, kajian yang terdiri daripada empat bahagian ini telah dijalankan untuk membangunkan satu kaedah pengekstrakan yang efektif. Di bahagian 1 (pemilihan kaedah), PALF daripada kultivar *Josapine* telah berjaya diekstrak dengan kaedah rautan manual (sikatan), rendaman air, pengekstrakan kimia atau pengekstrakan mekanikal-kimia. Kaedah pengekstrakan mekanikal-kimia didapati merupakan kaedah terbaik bagi menghasilkan pulpa yang keputihan serta hasil yang banyak (4.0 wt%) dan gentian PALF yang halus dan bersih diperolehi menggunakan 3 wt% NaOH. Di bahagian 2, (parameter pengekstrakan), pengekstrakan PALF dilakukan dengan memasak PALF pada kepekatan NaOH antara 1 hingga 11 wt% selama 30 hingga 180 minit. Imej mikroskop elektron imbasan menunjukkan bahawa gentian mula merosot pada kepekatan NaOH melebihi 10 wt%. Analisis termogravimetrik menunjukkan bahawa kepekatan NaOH kurang dari 3 wt% tidak cukup untuk mengalihkan lapisan hemiselulosa daripada gentian nanas. Didapati bahawa jumlah penyingkiran lignin adalah malar pada 7 wt% dan ke atas, seperti yang telah ditunjukkan oleh nilai nombor Kappa. Spektroskopi inframerah jelmaan Fourier menunjukkan pada 3 wt% NaOH cukup untuk menyingkirkan hemiselulosa dan lignin daripada PALF. Kesimpulannya, 3 wt% kepekatan NaOH, 90 minit masa memasak pada 90 °C telah dikenalpasti sebagai parameter optima untuk digunakan dalam proses delignifikasi. Penemuan selanjutnya menunjukkan bahawa larutan NaOH boleh dikitar semula hanya sekali tanpa menjejaskan sifat-sifat PALF yang diekstrak. Di bahagian 3 (penghasilan kertas kraf), kertas kraf yang terhasil daripada PALF yang diekstrak pada kepekatan NaOH (1, 3, 5, 7, 9 dan 11 wt%) dan kitar semula larutan NaOH telah dikaji berdasarkan sifat kekuatan tegangan, kekuatan koyakan dan penyerapan air. Didapati bahawa purata daya koyakan kertas kraf mencapai tahap maksima (216604.8 mN) pada kepekatan 9 wt% NaOH manakala kekuatan tegangan maksima (13 MPa) tercapai pada 3 wt% NaOH. Juga diperhatikan bahawa kitar semula pada 3 wt% larutan NaOH menyebabkan penurunan pada kekuatan tegangan kertas PALF daripada 12.6 MPa (NaOH segar) kepada 11 MPa (kitar semula sekali) dan 6.5 MPa (kitar semula kali kedua). Maka, 3 wt% kepekatan NaOH dan penggunaan semula larutan NaOH kali pertama adalah parameter pemprosesan yang baik untuk mendapatkan kertas PALF dengan sifat yang seimbang. Di bahagian 4, kertas kraf telah disediakan pada parameter yang telah dipilih (3 wt% NaOH, 90 minit masa memasak pada 90 °C) telah digunakan sebagai lapisan penetulang dalam resin epoksi. Komposit kertas kraf/epoksi telah disediakan pada jumlah lapisan yang berbeza iaitu 1, 2 dan 3 lapisan. Sifat-sifat komposit telah diukur melalui ujian tegangan dan hentaman Izod. Keputusan menunjukkan bahawa penyelitan helaian kertas yang diperbuat secara pengekstrakan NaOH telah mengurangkan ketumpatan komposit dan sifat tegangan. Bagaimanapun, sifat hentaman komposit bertambah baik kerana kejadian tarik keluar gentian disebabkan kelemahan ikatan gentian-matriks. Sebaliknya, komposit yang menggunakan kertas yang dihasilkan melalui kaedah rautan manual mempunyai sifat hentaman yang lemah disebabkan oleh kekuatan rekatan gentian-matriks.

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LIST OF ABBREVIATIONS

AQ	-	anthraquinone
DEG	-	diethylene glycol
DMF	-	dimethylformamide
DSC	-	differential scanning calorimetry
EDTA	-	ethylene diamine tetra acetic acid
FAO	-	Food and Agriculture Organization
FTIR	-	Fourier Transform Infrared
MPIB	-	Malaysian Pineapple Industry Board
M	-	manual retted
MC	-	mechanical-chemical retted
NFC	-	nanofibrillated cellulose
OCC	-	old corrugated container
OPEFB	-	oil palm empty fruit bunch
PAL	-	pineapple leaf
PALF	-	pineapple leaf fiber
PANI	-	polyaniline
TCF	-	totally chlorine free
CaCO ₃	-	calcium carbonate
H ₂ O ₂	-	hydrogen peroxide
Na ₂ S	-	sodium sulphide

LIST OF SYMBOLS

K	-	Kappa number
f	-	factor correction to a 50% permanganate consumption, dependent on the value of p
w	-	weight of moisture free pulp in the specimen, g
p	-	amount of 0.1 permanganate actually consumed by the test specimen, ml
b	-	amount of thiosulfate consumed in the blank determination, ml
a	-	amount of thiosulfate consumed by the test specimen, ml
N	-	normality of thiosulfate
X	-	mass of the PALF obtained from the extraction process, g
Y	-	initial mass of PAL used in the extraction process, g
m_1	-	wet weight, g
m_2	-	conditioned weight, g
ρ_c	-	calculated density of solid, g/cm ³
A	-	weight of the solid in the air, g
B	-	weight of the solid in the auxiliary liquid (distilled water), g
ρ_0	-	density of the auxiliary liquid at the given temperature, g/cm ³

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Increasing global industrialization led to significant consumption of paper especially for the purpose of packaging, which accounts for over 41 % of paper usage globally (Beckline, Yujun, Eric et al., 2016). According to the pulp and paper capacities survey 2013 – 2018 by the Food and Agriculture Organization of the United Nations (FAO, 2014), the global paper consumption will increase to 500 million tons in year 2025 which corresponds to an annual paper consumption growth of 1.6 %. The intensive paper consumption leads to significant depletion in natural resources especially wood due to rapid deforestation. Conventionally, paper was produced from the hardwood pulp which caused the deforestation to occur as these sources of hardwood took up several years before mature. Hence, research has been conducted continuously to search different source of suitable pulp for paper especially for packaging purposes.

Much efforts have been invested to study different non-wood alternatives as potential candidates for paper production. One of the potential replacements of wood in paper pulp production is pineapple leaf fiber (PALF), which is abundantly available in Malaysia (Daud, Hatta, Kassim et al., 2013; Yusof, Ahmad and Wahab., 2012). There are many attempts and researches that have been done on the utilization of PALF as cellulosic materials either for pulp production or as reinforcement in the natural fiber based polymer composites.

Researchers utilized pineapple biomass especially pineapple leaves to produce PALF/polypropylene (PP) composites (Arib, Sapuan, Ahmad et al., 2006), PALF/glass vinyl ester (VE) hybrid biocomposites (Zin, Abdan, Mazlan et al., 2019), PALF/polyester composites (Senthilkumar, Saba, Chandrasekar et al., 2019), silane treated kenaf/PALF phenolic hybrid biocomposites (Asim, Jawaid, Abdan et al., 2016), PALF, napier and hemp fibres filled scratch resistant epoxy composites (Ridzuan, Abdul Majid, Khasrib et al., 2019), sound adsorption material (Putra, Or, Selamat et al., 2018) and soil cover (Sarah, Rahman, Majid et al., 2018).

Pineapple leaf (PAL) serves as an attractive option for pulp making as pineapple plant can produce up to 1.5 kg of leaves per plant which is left in the field after cultivation (Sibaly and Jeetah, 2017). Furthermore, PALF possesses high cellulose content (66.2%) and low lignin content (4.2%) which resulted in good mechanical properties against the potential abrasion and tear when it is used as a packaging material (Mohamed, Sapuan, Shahjahan et al., 2009; Daud et al., 2013). Thus far, PALF has been used as the raw materials for pulping by Wutisatwongkul and co-workers (2016) where varies soda-pulping conditions were studied. Daud et al. (2013) studied on the suitability of napier grass and PALF for papermaking production where the study stated that both fibres exhibited great potential as substitute for pulp production.

1.2 Problem Statement

In Malaysia especially in the state of Johor, pineapple cultivation has contributed much to the income for the country. According to Malaysian Pineapple Industry Board (MPIB), there are more than 10 types of pineapple cultivators that are widely cultivated in the country, Josapine, Moris, Sarawak, MD2 and N36, to name a few. The main parts of a pineapple plant include the fruit, crown, stem and leaves. However, there is almost 50 % of the pineapple parts are covered by the leaves which are left unwanted during the harvest. During harvest, the unwanted by-products of pineapple are usually thrown away or burnt in the field as shown in Figure 1.1 hence generated wastes and air pollution (Sarah et al., 2018; Kasim, Selamat, Aznan et al.,

2015 and Hazarika, Gogoi, Jose et al., 2017) as this burning can take up to 2 to 3 days for completion. Open burning at the field was found to be the cheapest way to get rid of the wastes before next plantation as it does not acquire much labour. Thousand tons of PAL were produced and discarded every year yet only a small amount of it was used as feed stock for energy production (Asim, Khalina and Jawaaid et al., 2015). In order to reduce the negative impact of the generated wastes, research works have been done to convert them into value-added products, as mentioned in the introduction.



Figure 1.1 Open burning of pineapple field in Pontian (a) Before cultivation (b) After cultivation

Effective fiber extraction method must be established in order to enhance the feasibility of PAL usage as a non-wood alternative for pulp production (Daud et al., 2013; Yusof et al., 2012). The conventional extraction method often falls into three categories namely, mechanical method, chemical method and mechanical-chemical method. So far, reports on the results obtained by utilizing separate, different methods

are available (Uddin, Miah, Jalil et al., 2018; Sarah et al., 2018; Laftah and Rahman., 2015 and Munawar, Umemura, Tanaka et al., 2008) but a systematic study which covers the comparison of all the aforementioned methods and the selection of the best processing parameters is still a gap in the field that needs to be filled.

On the other hand, the black liquor produced from the delignification process which consists mainly of sodium hydroxide (NaOH) was regarded as pulping discharged. There were attempts to recover portion of this black liquor for energy generation purpose (Vakkilainen and Välimäki, 2014; Tran and Vakkilainen, 2016). The black liquor was recovered to obtain the pulping chemicals such as NaOH, sodium sulphide (Na₂S) and calcium carbonate (CaCO₃) where the chemicals were returned to the digester for reuse in pulping and serve as a fuel for steam production to generate energy. However, the recovery process was only applicable to big scale pulp production and it could be quite expensive due to the machineries involved. Therefore, recycling of NaOH solution in the extraction process can be an interesting topic to be focused on.

Natural fiber has been used in producing reinforced epoxy composites; for instance, Sand, Oliveira and Cândido et al. (2019) used bamboo/woven kenaf mat to produce epoxy hybrid composites. Others include sugar palm/epoxy composites (Bachtiar, Sapuan and Hamdan, 2008), abaca/epoxy laminated composites (Sinha, Narang and Bhattacharya, 2018), laminates of napier grass fiber/polyester (Haameem, Majid and Afendi et al., 2015), and jute/PALF/glass fiber reinforced epoxy composites (Reddy, Kumar and Rama et al., 2018). However, application of PALF paper as reinforcing material for epoxy has not been found. This justifies the need for the related studies.

This study was conducted so that the selection of PALF extraction method and determination of the best PALF extraction parameters can be done systematically. Four commonly used PALF extraction methods were screened through based on the PALF yields and morphology. After that, effects of processing parameters such as NaOH concentration and cooking time on the PALF yield and morphology were studied. Besides, effects of utilizing recycled NaOH was also investigated.

The extracted PALF was then used to produce kraft paper via conventional papermaking method. The kraft paper was then incorporated into epoxy resin to produce PAL/epoxy composites in order to study its effectiveness as reinforcing material.

1.3 Objectives of the Study

The aim of this study was to identify the most suitable extraction method and processing conditions for PALF extraction which was used for the production of kraft paper and epoxy composites. This aim is subdivided into the following objectives:

- (a) To determine the best PALF extraction method based on dry pulp yield and fiber morphology.
- (b) To study the effects of NaOH concentrations, cooking time as well as NaOH solution recycling on the dry pulp yield, morphological and thermal properties of the extracted PALF.
- (c) To characterize the PALF kraft paper produced at different NaOH concentrations and NaOH recycling level in terms of their water absorption, tear and tensile properties.
- (d) To study the effects of PALF kraft paper incorporation on density, tensile, impact and morphological properties of epoxy composites.

1.4 Scope of the Study

The whole study was divided into four parts which are PALF extraction method selection, extraction parameters studies, PALF paper characterizations and PALF/epoxy composites characterizations. Josapine cultivator was used as the source of PALF.

Four extraction methods namely, manual retting, water retting, chemical extraction and mechanical-chemical extraction were investigated. The extraction parameters studied were NaOH solution concentrations (1 to 11 wt%) and cooking time (from 30 to 180 minutes). Selections of the most suitable method and extraction parameters were done based on the percentage of dry pulp yield, morphological and thermal properties. In the study of the effects of NaOH solution recycling, the NaOH solutions were recycled up to 2 times only.

PALF paper was produced by using conventional papermaking process where 16 g of pulp was used to produce each sheet. Characterizations and properties of paper were determined based on tearing properties, percentage of water absorption and tensile properties.

Move on to the next part, PALF/epoxy composites were produced by hand lay-up process. Epoxy and hardener were kept at 2:1 for each formulation. PALF paper produced from manual retting (without NaOH) and chemical-mechanical (with NaOH) were used and each composite consists of 1, 2 or 3 plies of PALF paper. Properties of the PALF/epoxy composites were determined based on density measurement, physical and morphological properties.

Testing involved in each part of the study are as follows:

- (i) Extraction method selection
 - Percentage of dry pulp yield
 - Morphological properties (SEM)

- (ii) Fiber extraction parameter selection
 - Percentage of dry pulp yield
 - Morphological properties (SEM)
 - Functional groups identification (FTIR)
 - Kappa number determinations
 - Thermal properties (TGA)

- (iii) Paper production and characterizations
 - Tearing test
 - Water absorption test
 - Tensile properties

- (iv) PALF Paper/epoxy composites performance testing
 - Density measurement
 - Tensile properties
 - Impact properties
 - Morphological properties (SEM)

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LIST OF PUBLICATIONS

Indexed Journal

1. **Munthoub, D. I.**, Abdul Rahman, W. A. W., Lew, J. H., A. Majid, R. and Lai, J. C. (2020). Effects and extraction method on dry pulp yield and morphological properties of pineapple leaf fibre. *Malaysian Journal of Fundamental and Applied Sciences*. 16(3), 378-383. **(Indexed by SCOPUS)**