

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

The Effects of Autohydrolysis Pretreatment on the Properties of OPT Pulps for the Production of Dissolving Pulp

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ABSTRACT

This preliminary study investigated an environmentally friendly method for fabricating cellulose-rich dissolving pulp from Oil Palm (*Elaeis guineensis*) Trunk (VBOPT) fibre. This method encompassed an autohydrolysis pretreatment followed by soda pulping procedures. The impact of autohydrolysis pretreatment on the separation of lignocellulosic components was scrutinised to facilitate the production of chemical cellulose. Autohydrolysis was performed on VBOPT fibre for 60 min at temperatures ranging from 140°C to 160°C, maintaining a solid-to-liquid ratio of 1:8. The yield of the prehydrolysed OPT varied between 73.5% and 91.5%. The chemical composition of the prehydrolysed VBOPT fibre comprised 70.6–80.0% holocellulose, 63.7–87.1% α -cellulose, 7.4–10.9% β-cellulose, 5.5–25.4% γ -cellulose, and 21.5–26.6% lignin. The prehydrolysed OPT was subsequently

ARTICLE INFO

Article history: Received: 22 February 2024 Accepted: 30 May 2024 Published: 30 July 2024

DOI: https://doi.org/10.47836/pjst.32.S3.03

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Keywords: Autohydrolysis, dissolving pulp, non-wood biomass, oil palm trunk, soda pulping

INTRODUCTION

Dissolving grade pulp is a cellulose-rich pulp normally known as dissolving pulp or chemical cellulose. Distinct from papermaking grade pulp, dissolving pulp possesses a higher level of cellulose purity with α -cellulose content of 90–98%, relatively low hemicellulose (<5%), less than 1% lignin and less than 0.1% ash content (Andrade & Colodette, 2014; Biermann, 1996). Dissolving pulp is commonly used by dissolving it in certain chemical solutions or undergoing chemical modification to produce regenerated cellulose (such as viscose rayon, cellophane, cellulose hydrogel) and cellulose derivatives including cellulose, carboxymethyl- and ethyl-cellulose) and other cellulose-based products such nano- and micro-crystalline cellulose) (Chen et al., 2016; Miao et al., 2014; Sixta et al., 2013).

In recent years, there has been a significant increase in the production of dissolving pulp, driven by strong market demand growth. The dissolving pulp production in 2020 was 8.86 million tons. It is expected to grow at a compound annual growth rate (CAGR) of 6.22% from 2020 to 2027, about 73% higher than papermaking pulp (CAGR of 3.6%). The rapid growth of dissolving pulp in the global market causes the demand for its primary sources, conventional pulp wood (both hardwoods and softwoods) and cotton linter, to rise, as well as their market price.

On the other hand, in the bioeconomy era, it is anticipated that the utilisation of dissolving pulp will experience a significant increase due to its prominence as a sustainable and renewable resource, primed to replace petroleum-based resources in the production of various commodities, chemicals and energy. Efforts are ongoing to explore new alternative sources for producing dissolving pulp to undertake the expected expanding demand (Batalha et al., 2012). Numerous potential non-wood biomass, including from agricultural residues (e.g. empty fruit bunch (EFB), plant stalks, tobacco and corn, sugarcane bagasse and grain straw, rice, and wheat), naturally growing plant (e.g. bamboo and dhaincha) and industrial crops (e.g. jute, and kenaf) have been subjected to investigation (Andrade & Colodette, 2014; Batalha et al., 2012; Behin & Zeyghami, 2009; Chen et al., 2016; Huang et al., 2019; Jahan et al., 2007; Sarkar et al., 2021; Wan Rosli et al., 2003). The production of dissolving pulp made from bamboo has already been successfully commercialised.

Among all the vegetable oil productions, palm oil contributes to the biggest world market, accounting for 35.7% (79.5 million tonnes) of the total global vegetable oil production (217.5 million tonnes). Malaysia is one of the largest palm oil producers in the world, standing as the second-largest oil palm producer (19 million tonnes), following Indonesia (47 million tonnes), with a harvested area covering approximately 5.65 million hectares. Based on Market Analysis Report (2024), the global palm oil market will be increased at a CAGR of 5.5% from 2024 to 2030. It indicated that the byproduct or processing waste from the oil palm industry would increase significantly (Ishak et al., 2021), which includes oil palm trunk (OPT), oil palm fronds (OPF), empty fruit bunches (EFB), and mesocarp fibres. Bukhari et al. (2018) reported that the oil palm industry generates about 85 million tons of lignocellulosic biomass residues annually from plantation and milling activities.

In general, oil palm fruits can be harvested after three years post-planting and reach maximum yield in the 12th–13th year of their 25-year lifespan (Abdullah & Sulaiman, 2013). When the oil palm trees reach the unproductive stage, replantation becomes necessary. It is estimated that 74.5 tonnes of OPT per hectare (on a dry-weight basis) will be generated in replantation areas. Based on an annual replantation rate of 5% of the current plantation areas, about 21 million tonnes of felled OPT would be available (Bukhari et al., 2018). Under the common practice, felled OPT is left within the plantation area for natural decomposition. However, this method of decomposing takes about five to six years to fully decompose the felled OPT, posing challenges to replanting activities and attracting insect pests, which may inhibit the growth of new trees. Besides, open burning is a ban imposed by the Malaysian Government in 1998 (A1102 Act/Environment Quality Act 2001) due to environmental pollution issues (ASEAN, 2003); thus, burning of felled OPT within the plantation should not be a viable option for managing this biomass waste. Thus, the prudent approach to handle this massive biomass is to convert it into value-added products.

A previous study reported that OPT comprises 62% vascular bundles; the remaining percentage is parenchyma. Chemical composition analysis of the vascular bundles revealed that they consist of 56.91% cellulose, 25.47% hemicellulose, 11.91% lignin and 15.33% extractive (Lamaming et al., 2015). Given its high cellulose content, comparable to bamboo, vascular bundles of OPT present great potential as an alternative source for producing dissolving cellulose.

The conventional methods for producing dissolving pulp from pulpwood include the acid sulphite pulping process and the prehydrolysis kraft pulping process. However, both processes raise environmental concerns due to the emission of sulphur-containing compounds, such as sulphur dioxide (SO₂), which can lead to acid rain formation. Considering this concern, adopting sulphur-free pulping should be a priority, particularly when utilising non-wood biomass for dissolving pulp production. Different from papermaking pulp production, the attainment of high-purity α -cellulose is critical in producing dissolving pulp, where non-cellulose impurities, besides lignin, hemicellulose (γ -cellulose), and even degraded cellulose (β -cellulose), can significantly impose negative impacts on its application. Owing to the amorphous nature and low degree of polymerisation (200–300) of branched hemicellulose, acidic pretreatment before alkaline pulping can effectively eliminate it.

This study evaluates the feasibility of utilising vascular bundles of OPT to produce dissolving pulp. Moreover, environmentally sustainable processes are implemented to ensure commercial viability, incorporating non-toxic chemicals and diminishing chemical consumption. Therefore, water hydrolysis, also known as autohydrolysis, is adopted in conjunction with soda-pulping. Since autohydrolysis is a process solely utilising water at elevated temperatures to treat the biomass to remove hemicellulose through the catalytic effect of the self-generated organic acids, it enhances sustainability. As a preliminary investigation, this study aims to examine the effects of autohydrolysis pretreatment parameters on subsequent soda pulp properties. Hence, the result of this study is expected to provide fundamental insight into autohydrolysis pretreatment towards OPT, of the potential to maximise yields and α -cellulose content and minimise kappa number and ash content in the resultant pulp.

MATERIALS AND METHODS

Materials

Kluang Pilot Plant, Johor, Malaysia, provided the oil palm trunk (OPT) vascular bundles (VBOPT). The raw OPT, collected from a palm oil plantation, underwent an OPT fibre extraction process, which included chipping, screw pressing, drying, and vascular bundlesparenchyma separation processes. To enhance the purity of the vascular bundle used in this study, the VBOPT received was further sieved to remove the residual parenchyma portion.

Experimental

Autohydrolysis Pretreatment

All autohydrolysis experiments utilised a stationary, 4-litre stainless-steel digester produced by NAC Autoclave Co. Ltd., Japan. The digester was equipped with a computercontrolled thermocouple and did not have external circulation mixing. The autohydrolysis was conducted by inserting 350 g (on a dry weight basis) of VBOPT into the digester vessel, and water was added at a liquor-to-material ratio of 8:1. The reaction temperature was maintained within a required range of 140–160°C for 60 min. Upon completing the autohydrolysis process, the autohydrolysed VBOPT was rinsed in a stainless-steel mesh filter, spin-dried, kept in a sealed plastic bag, and subsequently stored in a refrigerator at 4°C for future use. The spent liquor was collected for pH assessment, and the yield was calculated based on the oven-dried fibre.

Soda Pulping

The soda pulping of prehydrolysed VBOPT was carried out in the same digester described earlier (autohydrolysis). An amount of 200 g (on a dry weight basis) of prehydrolysed VBOPT was cooked at a constant liquor-to-fibre ratio of 6:1 and a time-to-maximum temperature of 90 min, time-at-temperature of 60 min for a reaction temperature of 160°C using 25% alkali charge based on the dry weight of prehydrolysed VBOPT. After the pulping process, the resulting pulps and spent liquor were collected. The spent liquor was tested for pH, and the obtained pulp was disintegrated with a tri-blade mixer for 1 min at a pulp consistency of 2.0%. Subsequently, the pulps were screened using Somerville Screener with a flat-plate screen with a slit width of 0.15 mm. The yield was quantified based on dry weight basis.

Chemical Composition Analysis

The air-dried untreated and prehydrolysed VBOPTs were prepared by grinding into small particles using a RETSCH cutting mill SM100 and sieving into a range of size of (0.595–0.25 mm) by passing through a 35–60 mesh pore size. The chemical compositions of the untreated VBOPT and parenchyma cell (in powder form), prehydrolysed VBOPT, and prehydrolysed-soda pulp were examined according to standard test methods as follows: The extractive-free untreated VBOPT was prepared following the TAPPI 24 cm - 97 method with slight modifications, wherein ethanol-toluene served as the solvent for the extraction. However, this procedure was not applied to the prehydrolysed VBOPT because, during the autohydrolysis pretreatment, most of the organic compounds soluble in organic solvent to biomass would likely result in higher extractive content than the original biomass, as the lignocellulosic composition, which experienced structural changes, may also dissolve in the solvent.

Thus, this procedure could yield faulty results for chemical composition analyses. The lignin content of untreated and prehydrolysed VBOPT was determined following TAPPI 222 om-02—Acid Insoluble Lignin, while the kappa number of prehydrolysed-soda pulp was evaluated according to TAPPI-236—Kappa Number of Pulp. The cellulosic contents viz. holocellulose, α -cellulose, β -cellulose, and γ -cellulose were examined using methods described by Wise et al. (1946) and the Japanese Standard Method JIS 8101 methods, respectively. All analyses were performed in triplicate, and results were presented in mean with standard deviation. Equations 1–9 correspond to the calculation in Tables 2 and 3. The flowchart with the process condition of this research work is shown in Figure 1.

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For untreated biomass:

(α, β, γ) Cellulose _{UTB basis} of UTB (%) = $(\alpha, \beta, \gamma)_{UTB}$ (%) × Holocellulose _{UTB} , (%)/100	Eq. 1
For pretreated biomass:	
$Holocellulose_{UTB \ basis}$ (%) = $Holocellulose_{PTB}$ (%) × (Yield_{PTB}, %/100)	Eq. 2
(α, β, γ) Cellulose _{PTB basis} (%) = $(\alpha, \beta, \gamma)_{PTB}$ (%) × Holocellulose _{PTB} , (%)/100	Eq. 3
(α , β , γ) Cellulose _{UTB basis} (%) = (α , β , γ) _{PTB} (%) × Yield _{PTB} , (%)/100	Eq. 4
Klason lignin $_{UTB \text{ basis}}(\%) = Klason \text{ lignin}_{PTB}(\%) \times (Yield_{PTB}, (\%)/100)$	Eq. 5
Reduction of y cellulose content, (%)	
$= \frac{[\gamma Cellulose_{UTB \ basis} \ of \ UTB(\%) - \gamma Cellulose_{UTB \ basis} \ of \ PTB(\%)]}{[\gamma Cellulose_{UTB \ basis} \ of \ UTB(\%) \times 100]}$	Eq. 6
Reduction of Klason Lignin, (%)	
= $\frac{[Klason \ lignin_{UTB \ basis} \ of \ UTB, (\%) - Klason \ lignin_{UTB \ basis} \ of \ PTB, (\%)]}{[Klason \ lignin_{UTB \ basis} \ of \ UTB \ (\%) \times 100]}$	Eq. 7

For autohydrolysed-soda pulp:

(α, β, γ) Cellulose _{PTB basis} (%) = $(\alpha, \beta, \gamma)_{PTSP}$ (%) × Screen Yield, (%)/100	Eq. 8
(α, β, γ) Cellulose _{UTB basis} (%) = $(\alpha, \beta, \gamma)_{PTSP}$ (%) × Screen Yield _{UTB} (%)/100	Eq. 9

UTB = Untreated biomass PTB = Pretreated biomass UTB basis = Based on untreated biomass PTB basis = Based on pretreated biomass PTSP = Pretreated biomass-soda pulp



Figure 1. The flowchart with process condition of the research work

RESULTS AND DISCUSSIONS

Chemical Composition Analyses of Oil Palm Trunk

In contrast to wood, OPT, as a non-wood biomass, appears in two distinct fractions after milling: the hard woody vascular bundles particles and the powder-like parenchyma cells,

including a minor portion of other softer tissue components. Figure 2 shows the vascular bundles and residual parenchyma cells separated from the raw sample of vascular bundles received from the factory. The chemical compositions of these two factions are presented in Table 1. The results reveal that the vascular bundle faction contains lower percentages of extractive (4.1%) and lignin (19.3%) contents but higher holocellulose content (79.4%) compared to parenchyma cells, which exhibits percentages of 6.4%, 23.2% and 76.5%, respectively.



OPT fibre supply by Kluang plant





OPT vascular bundles

OPT parenchyma

Figure 2. OPT Vascular bundles and parenchyma cells separated from OPT fibre supplied by Kluang Pilot Plant

Moreover, one can see that the vascular bundles show a higher cellulose content (a total of α -cellulose and β -cellulose) (60.0%) and lower hemicellulose content (γ -cellulose) (40.0%) in comparison to the parenchyma cells (48.8% and 51.2%, respectively). The findings suggested that the vascular bundles of OPT (VBPOT) demonstrated high potential as raw material for dissolving pulp production. The segregation of residual parenchyma cells from the VBOPT could improve the pulping efficiency because parenchyma cells will require higher pulping chemicals and result in lower pulp yield. Parenchyma

cells exhibit a high affinity to absorb chemicals and are easily degraded due to their size and thin cell wall structure (Paul & Kasiviswanathan, 1998).

Table 1

Chemical composition of OPT fibre (vascular bundles and parenchyma)

Chemical composition	Vascular bundles	Parenchyma	
Extractives*, %	4.1 ± 0.4	6.4±1.0	
Holocellulose [§]	82.7±0.9	$81.7{\pm}1.0$	
Holocellulose*, %	79.4	76.5	
α- Cellulose [#] , %	$54.8{\pm}0.5$	45.0±0.2	
β- Cellulose [#] , %	5.2 ± 0.3	$3.8{\pm}0.8$	
γ- Cellulose [#] , %	40.0±0.3	51.2±0.9	
Lignin content [§] , %	20.1±0.2	24.8±0.4	
Lignin content*, %	19.3	23.2	

§ Percentage calculated based on extractive-free biomass

* Percentage calculated based on original biomass

Percentage calculated based on holocellulose

Effect of Autohydrolysis Pretreatment on VBOPT

The effects of autohydrolysis at varying temperatures on the properties of VBOPT are detailed in Table 2. Autohydrolysis is an effective pretreatment of biomass utilising only water at elevated temperatures to hydrolyse and remove hemicellulose. Notably, the pH of the spent liquor decreased from 3.84 to 3.42 (equivalent to about 0.0013 M to 0.0085 M of acetic acid) as temperature increased from 140°C to 160°C. During autohydrolysis, biomass is heated to an elevated temperature (140°C and higher) within a closed system to achieve high pressure. Under this condition, water produces hydronium ion (H₃O⁺), which penetrates the biomass, causing the degradation of the susceptible component—primarily amorphous hemicellulose. As a result, its principal functional group—acetyl group, undergoes hydrolysis and releases acetic acid (Carvalheiro et al., 2016).

A minor quantity of formic acid is also formed from carbohydrate-degradation products in biomass (Chen, 2015). The release of these organic acids catalyses further hemicellulose hydrolysis, resulting in the liberation of more organic acids, including simple sugars. Hence, the drop in pH of spent liquor can be attributed to the generation of more acid (Alén, 2015), while an increase in temperatures promoted greater acetic acid release.

Table 2

	Pt	Prehydrolysed VBOPT		
Chemical composition	140°C	150°C	160°C	
Spent liquor pH	3.8±0.1	3.7±0.0	3.4±0.0	
Solid yield, %	91.5±1.5	82.7±1.0	73.5±1.3	
Extractives, %	-	-	-	
Holocellulose, %				
PTB basis	80.0±0.5	77.6±1.0	70.6±1.3	
UTB basis	73.2	64.2	51.9	
α- Cellulose, %				
Based on holocellulose	63.7±1.0	71.5±0.5	87.1±0.7	
PTB basis	51.0	55.4	61.5	
UTB basis	46.6	45.9	45.2	
β- Cellulose, %				
Based on holocellulose	10.9±0.5	8.4±0.6	7.4±0.5	
PTB basis	8.7	6.5	5.2	
UTB basis	8.0	5.4	3.8	
γ- Cellulose, %				
Based on holocellulose	25.4±0.5	20.1±1.5	5.5±0.7	
PTB basis	20.3	15.6	3.9	
UTB basis	18.6	12.9	2.9	
Reduction	43.8	61.0	91.2	
Klason lignin, %				
PTB basis	21.5±1.5	23.6±0.3	26.6±0.5	
UTB basis	19.7	19.5	19.6	

Chemical composition of prehydrolysed vascular bundles of OPT

PTB basis: calculated based on pretreated vascular bundles, UTB basis: calculated based on untreated vascular bundles

The declining trend in the solid yield with the increased autohydrolysis temperature implies that more components in VBOPT were degraded and dissolved. Although based on the holocellulose content, it is obvious that the loss of biomass is mainly attributed to the carbohydrate component. The substantial decrease of γ -cellulose content (hemicellulose) in the holocellulose from 40.0% (or 33.1% based on original biomass) to 25.4% (18.6%), 20.1% (12.9%) and 5.5% (2.9%) when the autohydrolysis temperature were 140°C, 150°C and 160°C, respectively (Nguyen & Trinh, 2022; Peng et al., 2019).

It is very interesting to see that by subjecting the VBOPT to a temperature of 160°C for 60 min, the reduction of hemicellulose was achieved up to 91.2% (Table 2), followed by 61.0% under 150°C and 43.8% under 140°C. By correlating the decline in pH of spent liquor to the efficiency of hemicellulose removal, it is agreeable that by determining the pH of the autohydrolysis spent liquor, one can anticipate the efficiency of hemicellulose elimination.

Since the percentages of other types of cellulose (α - & β -celluloses) are interdependent with γ -cellulose, the reduction in the latter will increase the percentage of α -cellulose. The implication of simple autohydrolysis as pretreatment of VBOPT prior to the soda pulping process, even at 140°C, possesses a significant effect on increasing the cellulose content (63.7%), with the highest achieved under 160°C (87.1%).

One more attractive point to highlight is the increase of lignin content from 21.5% to 26.6% (based on prehydrolysed VBOPT) when the autohydrolysis temperature increased from 140°C to 160°C. The increase of lignin content in the prehydroylsed VBOPT is mainly due to the loss of holocellulose content, which is more prominent in the delignification reaction. As demonstrated in Table 2, when the lignin content is quantified based on untreated original biomass, the lignin contents were in the range of 19.5–19.7%, which is slightly lower than the VBOPT's lignin content counted based on extractive-free biomass (20.1%).

The finding agrees with a recent review conducted by Lu et al. (2021); it was discovered that only a minimal amount of lignin was removed from the biomass after undergoing autohydrolysis pretreatment. According to previous researchers, lignin undergoes fluidisation within the temperature range of approximately 120–200°C. During high-temperature pre-treatments, the fluidised lignin coalesces into smaller particles, separating from the cellulose and moving from the native cell wall to the bulk liquid phase. As the coalesced lignin solidifies upon cooling, it re-deposits on the surface of the biomass. This results in a biomass with an enriched surface lignin. It is important to note that this process does not remove lignin from the biomass but instead re-allocates it (Donohoe et al., 2008; Kristensen et al., 2008; Takada et al., 2019).

Effect of Autohydrolysis Pretreatment on Pulp Properties

The three prehydrolysed VBOPTs (PVB) treated under varying autohydrolysis temperatures and untreated VBOPT (UVB) were cooked using a soda pulping process to produce pulp. Based on the data obtained (Table 3), prehydrolysed-soda pulping, even the one treated at 140°C produced pulp (PVB140) approaching dissolving properties with 89.4% α -cellulose and 5.3% of γ -cellulose compared to the pulp produced without undergoing prehydrolysis (87.2% and 12.1%, respectively). The significant effects of autohydrolysis were clearly shown by PVB160 (prehydrolysis under 160°C), where its α -cellulose content was 98.1% while γ -cellulose was less than 0.5%.

On the other hand, all the kappa numbers of pulp produced from prehydrolysed VBOPT were below 10.0. It indicated that prehydrolysed lignin was more easily degraded by the subsequent soda pulping process. It might be due to the hydrolysis of biomass increasing the hydroxyl group in lignin and thus enhancing the delignification during the alkaline pulping process. Nevertheless, since the prehydrolysis process will enhance the dissolution of the biomass during the pulping process due to the removal of the remaining hemicelluloses and lignin, yield losses are attributed to it. The calculated pulp yield is based on the untreated original biomass shown in Table 3. The dissolving pulp produced from VBOPT is 30.5–35.4, within the range of the dissolving pulp yield produced from pulpwood. These analyses show that the autohydrolysis-soda process was beneficial to the pulping of VBOPT. However, extensive investigation is needed to determine the optimum autohydrolysis-soda pulping condition.

Soda Pulping Condition		160°C, 60 min, 25% of NaOH		
			PVB	
Pulp Properties	UVB	140°C PVB140	150°C PVB150	160°C PVB160
Screen yield, %	32.0±0.7	46.4±0.9	42.8±0.4	41.5±1.5
Yield based on UTB, %	32.0	42.5	35.4	30.5
α-Cellulose, %	87.2 ± 0.2	89.4±1.1	90.8±0.2	98.1±0.5
β-Cellulose, %	$0.8{\pm}0.2$	5.3±1.0	$2.4{\pm}0.8$	1.5 ± 0.1
γ-Cellulose, %	12.1±0.8	5.3±0.7	$6.9{\pm}0.5$	$0.4{\pm}1.1$
Kappa number	12.0±0.6	9.6±1.3	$6.4{\pm}0.7$	4.5±0.1

Table 3

Characteristics of untreated OPT-soda pulp and prehydrolysed VBOPT-soda pulp

UVB: untreated vascular bundles, PVB: pretreated vascular bundles

In this study, the autohydrolysis-soda process shows excellent potential for utilising the vascular bundles of the oil palm trunk to produce dissolving pulp. Autohydrolysis pretreatment at an elevated temperature of 160°C eliminated hemicellulose from the

biomass. Therefore, the subsequent soda pulping can produce dissolving grade pulp with very high α -cellulose content and low hemicellulose and lignin content. Statistical analysis and optimisation of the process conditions should be conducted to further understand the interactive effects of the autohydrolysis and soda pulping process parameters.

ACKNOWLEDGEMENT

Sincere gratitude for the support the Science and Technology Research Partnership for Sustainable Development (SATREPS) provided in collaboration with Universiti Sains Malaysia (203/PTEKIND/67811002) for this project. SATREPS is a Japanese government program that promotes international joint research. It is structured as a collaboration between the Japan Science and Technology Agency (JST) and the Japan International Cooperation Agency (JICA).

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