EFFECTS OF PREHYDROLYSIS ON THE PRODUCTION OF DISSOLVING PULP FROM EMPTY FRUIT BUNCHES

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WAN ROSLI, W. D., LEH, C. P., ZAINUDDIN, Z. & TANAKA, R. 2004. Effects of prehydrolysis on the production of dissolving pulp from empty fruit bunches. The necessity of expanding and diversifying value-added features of oil palm lignocellulosic waste in Malaysia has prompted an examination of its suitability as a raw material for dissolving pulp production. Introduction of prehydrolysis prior to any alkaline pulping process helps to decrease hemicellulose content and subsequently produce satisfactory high cellulose pulps. Empty fruit bunches (EFB) have been hydrolysed under a wide range of conditions and the resultant fibers determined for their yield and chemical properties. Results indicated that for all responses determined, namely, fiber yield, α-cellulose and viscosity, temperature gave the greatest effect particularly in the presence of acid, whilst time and acid level had lesser effects. It was also noted that, contrary to wood-based materials, prehydrolysing of EFB in the absence of acid is most effective. When pulped using a soda cooking cycle, an unbleached pulp with an α-cellulose of 96% and a Kappa number of 3.9 was obtained.

Key words: Cellulose pulp – α-cellulose – oil palm lignocellulosics – viscosity

WAN ROSLI, W. D., LEH, C. P., ZAINUDDIN, Z. & TANAKA, R. 2004. Kesan prahidrolisis terhadap penghasilan pulpa terlarut daripada tandan buah kosong. Keperluan untuk memperluas dan mempelbagaikan nilai tambah bahan buangan lignoselulosa sawit di Malaysia telah mendorong suatu penilaian dijalankan tentang kesesuaiannya sebagai bahan mentah dalam penghasilan pulpa terlarut. Prahidrolisis sebelum proses pempulpaan alkali dapat membantu mengurangkan kandungan hemiselulosa dan seterusnya menghasilkan pulpa selulosa yang berkualiti tinggi. Tandan buah kosong (EFB) telah dihidrolisiskan di bawah keadaan-keadaan tertentu dan penentuan hasilan serta sifat-sifat kimia telah dilakukan terhadap gentian yang diperoleh. Keputusan menunjukkan bahawa untuk semua parameter yang ditentukan, iaitu, hasilan gentian, α-selulosa dan kelikatan, suhu memberi kesan yang paling besar terutama jika dicampur asid, manakala masa dan aras asid memberi kesan yang lebih kecil. Tidak seperti bahan berasaskan kayu, prahidrolisis EFB tanpa kehadiran asid adalah lebih berkesan. Apabila dipulpakan dalam kitaran pemasakan soda, suatu pulpa takterluntur yang mempunyai α-selulosa setinggi 96% dan nombor Kappa 3.9 diperoleh.

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Introduction

The enormous quantities of lignocellulosic wastes available from oil palm industries such as trunks and fronds during replanting and pruning, and empty fruit bunches and mesocarp fibres during milling, provide a sustainable resource material for fibre-based industries in Malaysia. Paper grade pulps from oil palm fibres have since been established (Akamatsu *et al.* 1987, Khoo & Lee 1991, Wan Rosli *et al.* 1998). However, the suitability and feasibility of this material for production of high value-added products, especially with commercial comparable qualities, have yet to be ascertained.

Cellulose pulp, also called dissolving pulp, is a chemically refined bleached pulp with high cellulose content (90–98%), low hemicellulose content, very little residual lignin (> 0.05%), low extractive and ash contents, and also with a uniform molecular weight distribution (Hinck *et al.* 1985). Dissolving pulp is an important starting material for the production of cellulose derivatives such as cellulose acetate, cellulose nitrate and cellulose ether, and of regenerated cellulose such as viscose rayon and cellophane film (Hinck *et al.* 1985, Hartler & Lindström 1987). The two major resources for the production of dissolving pulp are cotton linters and wood pulp, the latter being more important. With increased demand and cost of pulpwood, new alternatives for the production of dissolving pulp are being investigated. Non-woody raw materials from annual plants is one of these alternatives and is popular especially in developing countries where pulpwood stocks are scarce (Abdul Karim *et al.* 1994, Panth *et al.* 1997). For the preparation of dissolving pulps from lignocellulosics, it is necessary to remove the hemicelluloses and lignin by acid pulping or acidic prehydrolysis-alkali pulping (Hartler & Lindström 1987). In this work, oil palm empty fruit bunch (EFB), with an estimated eight million tones of biomass being generated yearly in Malaysia (MPOB 2001), is used as raw material in the production of dissolving pulp. EFB was selected as it is easily collected from palm oil mills and, thus, omits the cost of transportation from plantation sites. An earlier study of chemical pulping of EFB for pulp and paper making has shown that alkaline process using sodium hydroxide alone was most effective in terms of pulping efficacy and environmental friendliness, in comparison with either sulfite or kraft processes (Wan Rosli *et al.* 1998). Thus, in the present study soda pulping was adopted for the preparation of dissolving pulp, with some modifications. However, since hemicelluloses are detrimental to the quality of dissolving pulp it was necessary to remove them and also lignin by introducing a prehydrolysis stage before pulping. The effects of prehydrolysis variables, namely, temperature, time and acid level were investigated by determining the yield and chemical properties of the resultant EFB.

Materials and methods

Empty fruit bunch

Fruit bunches of oil palm (*Elaeis guineensis*) were sterilised and stripped of any oil palm fruit. The EFB underwent mechanical treatment to loosen the fibrous strands, which was later washed, cleaned, sorted and dried. The untreated fibres were then ground and passed through a 35–60 mesh, after which they were analysed for chemical characteristics. Results of the analysis are given in Table 1.

Table 1 Chemical analysis of untreated EFB

Constituent	Fraction
Alcohol-benzene extractives	2.3%
Holocellulose	82.5%
Lignin	17.2%
α-cellulose	60.6%
Ash	5.4%

Prehydrolysis

All prehydrolysis cooks were carried out in a 4-litre stationary stainless steel digester (NAC Autoclave Co. Ltd., Japan). The temperature of the cook was measured and controlled by a computerised thermocouple.

Moisture-free EFB fibres (200 g) were prehydrolysed using eight parts of liquor to one of fibre. Various combinations of temperature (T), time (t) and acid level (A_L) were used as shown in Table 2. Pressure was relieved during heating until the digester reached 100 °C. At the completion of the prehydrolysis the pressure was again relieved and the digester was immediately cooled to below 100 °C with cool air. After washing and air-drying, the yield of oven-dried fibre was determined.

Table 2 Treatments of prehydrolysis

Treatment	Temperature (°C)	Time (min)	Acid level (%)
I	140	120	1
II	155	120	1
III	170	120	1
IV	155	120	0.5
V	155	120	1.5
VI	155	60	1
VII	155	120	0
VIII	170	60	0

Soda pulping

Soda pulping was conducted using the same digester as in the prehydrolysis. A total of 150 g each of moisture-free untreated and prehydrolysed EFB fibres (basis of calculation) were cooked at a temperature of 170 °C. The ratio of liquor to fibre was 8:1. Time to reach cooking temperature was set for 90 min. Cooking was carried out for 90 min and alkali level was maintained at 25%. At the completion of each cook, the resultant pulp was washed and mechanically disintegrated in a three-bladed disintegrator for 1 min at a pulp consistency of 2.0%. Finally, the pulp slurry was screened on a flat-plate screen with 0.15 mm slits. Yield of pulp was determined on oven-dried fibre basis.

Analyses

Prior to the determination of chemical characteristics, fibres were first extracted using benzene-alcohol (2:1) to ensure that they are extractive-free. The holocellulose content was determined by using the method of Wise *et al.* (1946). Holocellulose, α-cellulose content and viscosity were determined according to Wise *et al.* (1946), Japanese Industrial Standard 8101 and TAPPI standard T230 om-89 respectively.

Results and discussion

Effects of prehydrolysis on EFB fibre

The introduction of acidic prehydrolysis prior to alkaline pulping produces pulp with high α-cellulose but considerably low hemicellulose content; the hemicelluloses are degraded to alkali-soluble substances. In addition, acidic prehydrolysis also increases the extractability of lignin during subsequent alkaline pulping process and hence, reduces the ash content substantially (Howard 1963).

Figure 1 shows the yield of fibre and α-cellulose content of each prehydrolysis condition (refer to Table 2). Prehydrolysis treatments I and VI had the highest yields (82.68 and 79.05% respectively), and also the lowest α-cellulose contents (70.18 and 66.56% respectively). Conversely, treatments III, V and VIII produced the lowest yields (65.36, 70.33 and 70.10% respectively) but the highest α-cellulose contents (86.48, 77.72 and 83.61% respectively). The low yield is attributed to the removal of hemicellulose during prehydrolysis while the reduction in α-cellulose denotes cellulose degradation.

α-cellulose content is the most important criterion in determining the purity of dissolving pulp. Prehydrolysed fibre with a higher α-cellulose content is preferred over higher yield. On this basis, treatments III, V, VII and VIII are favoured (Figure 1). Although treatment III gave the highest α-cellulose content, high reaction temperature with the presence of 1% acid for 120 min caused undesirable hydrolytic degradation of cellulose, which led to unacceptable reduction in viscosity (8.92 cP, Figure 2). This is important since these fibres will then undergo a pulping and bleaching stage which will further reduce the viscosity. It has been shown that a minimum viscosity of ca. 13 cP is required at this stage for a satisfactory preparation of dissolving pulps from EFB (Leh 2002). Treatment V, with the highest acid level of 1.5% and at a moderate reaction temperature of 155 °C for 120 min, also gave a satisfactory high yield and α-cellulose content. However, the relatively low viscosity of 10.13 cP made the pulp unsuitable for the present purpose.

It is also interesting to note that without the presence of acid (treatments VII and VIII), the fibres obtained were of acceptable α-cellulose content and also of higher viscosity compared with treatments using acid. Although mild acid is necessary for the removal of hemicelluloses in woody lignocellulosics (Brasch & Free 1964), this study showed that the presence of acid in the prehydrolysis of oil palm EFB was detrimental to cellulose degradation, especially when high reaction temperature was applied. In fact water hydrolysis was sufficient to reduce the

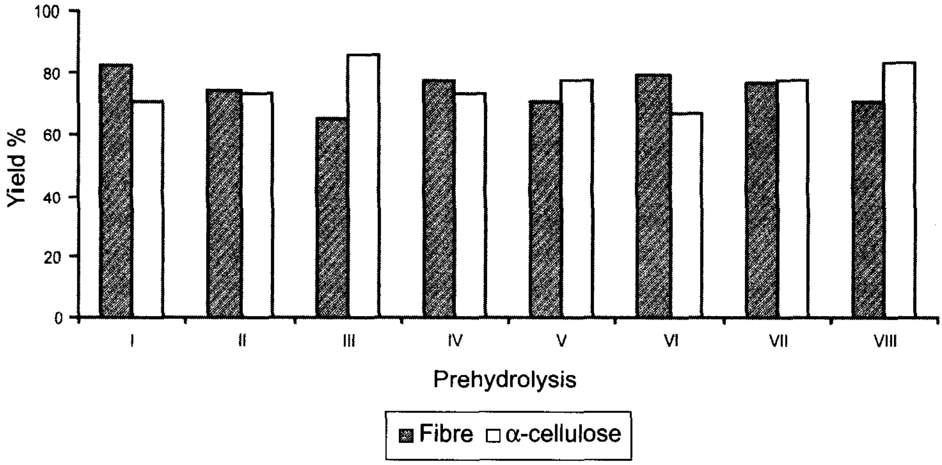

Figure 1 Yield of fibre and α-cellulose content for different prehydrolysis treatments (refer to Table 2)

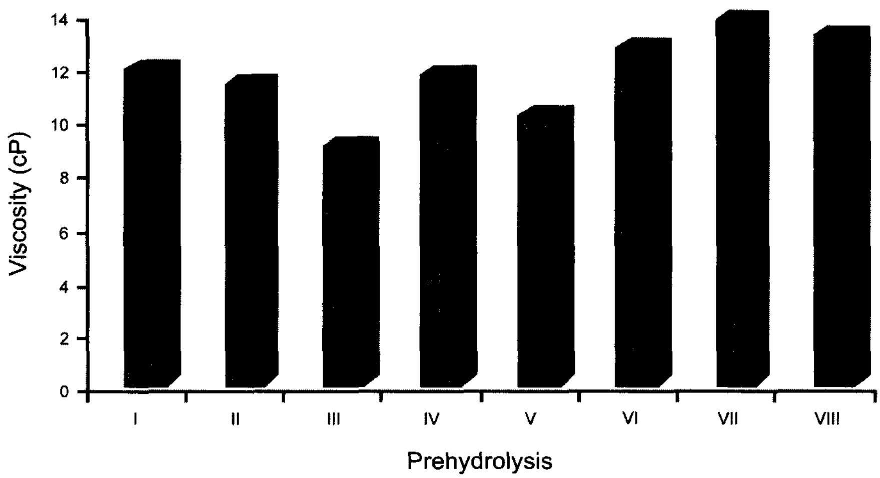

Figure 2 Viscosity of holocellulose for different prehydrolysis treatments (refer to Table 2)

hemicellulose to a satisfactory level, and at the same time, to minimise cellulose degradation.

The small difference of only 0.6 cP in viscosity in treatments VII and VIII suggested that the effect of reaction temperature on cellulose hydrolytic degradation during water prehydrolysis was not significant. However, the higher α-cellulose content in treatment VIII compared with treatment VII could be ascribed to the higher removal of hemicellulose as a result of the higher temperature during the prehydrolysis. These results also indicated that temperature played a more important role in removing the hemicellulose than time. Since α-cellulose content is the most important criterion for the production of dissolving pulp, we conclude that treatment VIII was the best condition for the preparation of prehydrolysed fibres to be used in the subsequent stages of this research.

Effects of prehydrolysis on soda pulp

In order to demonstrate further the effectiveness of prehydrolysis in producing dissolving pulps, a preliminary experiment was conducted where both the prehydrolysed and untreated fibres were subjected to the same soda pulping conditions. The results for the unbleached pulp are shown in Table 3. It was clear that prehydrolysis process had a positive effect on pulp quality. There was an increase in α-cellulose content and a huge reduction in kappa number (3.88 for prehydrolysed fibres compared with 13.16 for untreated). There was also a reduction in ash content, which affected the overall quality of the pulp. The positive effects of this stage were, however, accompanied with a loss in yield of pulp. This is probably due to the removal of the remaining hemicellulose and lignin. It is also possible that the cellulose component was being degraded as seen from the low viscosity. These analyses showed that prehydrolysis-soda process was beneficial to the pulping of EFB; pulp with a very high α-cellulose content, low kappa number and low ash content can be obtained. However, to achieve higher quality of dissolving pulp, an extensive investigation is needed to determine the optimum soda pulping conditions.

Table 3 Properties of soda pulp and prehydrolysed soda pulp

Pulp type	Screened yield (%)	Kappa No.	α-cellulose (%)	Viscosity (cP)	Ash × 10^{-2} (%)
Untreated	46.83	13.16	90.89	12.51	71
Prehydrolysed	30.62	3.88	96.43	10.19	14.5

Conclusions

Prehydrolysis of oil palm EFB fibres in the absence of acid is preferable within the range of process conditions employed in this study. Acidic prehydrolysis engendered hydrolytic degradation of cellulose to a lower molecular weight with low viscosity. High reaction temperature (170 °C) in the presence of acid produced prehydrolysed fibre with high α-cellulose content but with serious cellulose degradation. On the other hand, in the absence of acid, high reaction temperature only had a minor effect on cellulose degradation although a big reduction in hemicellulose was also noticed; this produced fibres with high α-cellulose content. Prehydrolysis produced positive effects on soda pulp quality, i.e. high α-cellulose content, low kappa number and low ash content. Of the three factors investigated, cooking temperature had the most influence on the parameters studied compared with time and acid level.

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