

SCANNING ELECTRON MICROSCOPY FOR IDENTIFICATION OF PAPER STRENGTH DEVELOPMENT BY TWO VARIATIONS OF REFINER PLATE PATTERN

Y. M. Dermawan¹ and A. Ghazali¹

¹ School of Industrial Technology, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia.

The abundant oil palm Empty Fruit Bunch (EFB) was used as raw material in the low-cost, low environmental impact Alkaline Peroxide Pulping (APP) to produce pulp and paper. Pulping was performed by using two types of refiner plate pattern, 12716 and D2A505. For each plate pattern, samples were prepared by treatment with 2:2.5% (wt/wt) alkali-to-peroxide (AP) ratio applying different number of AP impregnation stages. For plate 12716, the resultant papers show tensile strength varying from 6.9 Nm/g to 16.22 Nm/g, tear strength varying from 3.6 mN-m²/g to 5.2 mN-m²/g and folding endurance varying from 2 to 25. As of plate D2A505, tensile strength of papers made from the produced pulp range from 6.93 Nm/g to 29.77 Nm/g, tear strength from 2.9 mN-m²/g to 5.34 mN-m²/g, and folding endurance from 2 to 1633. Examination of paper surface by Scanning Electron Micrograph (SEM) revealed an increase in the extent of what appeared under SEM as 'fibre coalescence', implying extensive overlapping and bonding between the produced pulp mass. For all sets of lower strength pulp network, the produced fibrous mass was predominantly fibre bundles without any sign of coalescence. SEM analysis also witnessed the presence of long individual fibres and fibre bundles having correlation with paper resistance to burst and folding. The established correlations demonstrate the important way scanning electron microscopic technique serve for comprehensive understanding of fibre and fibre bonding characters in paper quality assessment.

Keywords: alkaline peroxide pulping; APMP; EFB; impregnation; paper properties, SEM

INTRODUCTION

The oil palm empty fruit bunch (EFB) is an all-year abundant biomass generated from palm oil extraction from the species of *Elaeis guineensis*. Introduced as an ornamental plant of West Africa, *E. guineensis* was first brought to Indonesia in 1848 and to Malaya in 1911 [1]. As today's cash crop, the oil palm gradually contribute to the major revenue of the countries, placing them as world leading producer of palm oil and, of course, the major generator of EFB.

In the effort of utilizing this cellulosic biomass and prevent its accumulation, the concept of APMP [2-6] was mimicked and applied, initialising it as Alkaline Peroxide Pulping (APP). The properties of the produced pulp were studied and the surface morphology of the pulp network was examined. Scanning Electron Microscopic (SEM) analysis of the pulp network was seen as an effective tool for understanding fibre bonding and strength development. This paper reports the properties of APP pulp from EFB. The outcome of SEM analyses of the papers corresponding to the pulp produced by increasing the stages of alkaline peroxide (AP) impregnation and by refining with two variations of

refiner plate patterns are hereby discussed.

MATERIALS AND METHODS

Preparation of Raw Material

The vascular bundles of EFB were obtained in the form of bales. These were loosened, washed thoroughly and air-dried before grinding to 500-micron particles. Dewaxing was done by soaking the biomass particles in distilled water at 70°C for 30 minutes and this was to achieve 50% removal of the extractives components.

Alkaline Peroxide Impregnation

The alkaline peroxide (AP) was prepared by premixing hydrogen peroxide and sodium hydroxide. Reaction with 2:2.5% alkaline peroxide was allowed to stand for 40 minutes at 70°C before pressing the biomass to 20% residual moisture. This process is denoted as 1-stage impregnation while for treatment involving x times repetition of the said steps is denoted as x-stage impregnation. Repetition (x), however, was no more than four times and each time involves the application of fresh chemicals on the biomass.

*Corresponding author: Tel: +604-6532239, Fax: +604-6573678
E-mail: arniza@usm.my (Arniza Ghazali)

Refining Process and Making of Handsheet

Refining process was conducted by using the Sprout Bauer Refiner to allow fibrillation. The AP treated EFB was split into two portions and refined using refiner plate patterns 12716 and D2A505. The two portions of pulp were made into hand sheets in accordance to TAPPI Test Methods 1997 [7].

Mechanical and Testing

The mechanical testing conducted in this study were folding endurance (TAPPI 511), burst index (TAPPI 403), tensile index (TAPPI 494), tear resistance index (TAPPI 414) and zero span strength (TAPPI 231).

RESULTS AND DISCUSSION

Examination of Hand Sheet Surface by SEM

a. Refiner plate 1

Applying the different plate pattern has apparently created different pulp properties as evident from SEM of hand sheets. Refiner plate pattern 12716 has only one refining zone attached with blades at the entering (Fig. 1a). SEM of the corresponding hand sheet surface shows predominance of vascular bundles generated by 1-

stage impregnation (Fig. 1b). The evidence of silica lying on these vascular bundles reflects the effect of insufficient softening of the biomass from the 1-stage impregnation. Failure to liberate silica bodies (A) is plausibly due to the presence of lignin, which added difficulty to silica liberation because of its natural gluing effect to biomass components [8]. In the said hand sheet, only a small portion of the desirable well fibrillated fibres were encountered.

The predominance of fibre bundles implies the high stiffness of the biomass and this had prevented the cell wall from detaching (C). It turned out, therefore, that only a small amount of fibrils was formed (Fig. 1b). In the absence of the favoured fibrillar structures, only a weak fibre to fibre bonding (D) and poor overall network strength could be established.

With 4-stage impregnation, most of the fibres are loosened due to sufficient EFB softening. Count of silica bodies on EFB was also reduced (E). This means that 4-stage impregnation offered better chances of Si liberation than the 1-stage impregnation. The mechanical effects of multiple-stage impregnation are also portrayed by the flattened silica craters, suggesting a more powerful effect of 4-stage AP impregnation in comparison to the 1-stage impregnation. The pulp refined using plate pattern 12716 resulted in intensive shearing of fibres, leading

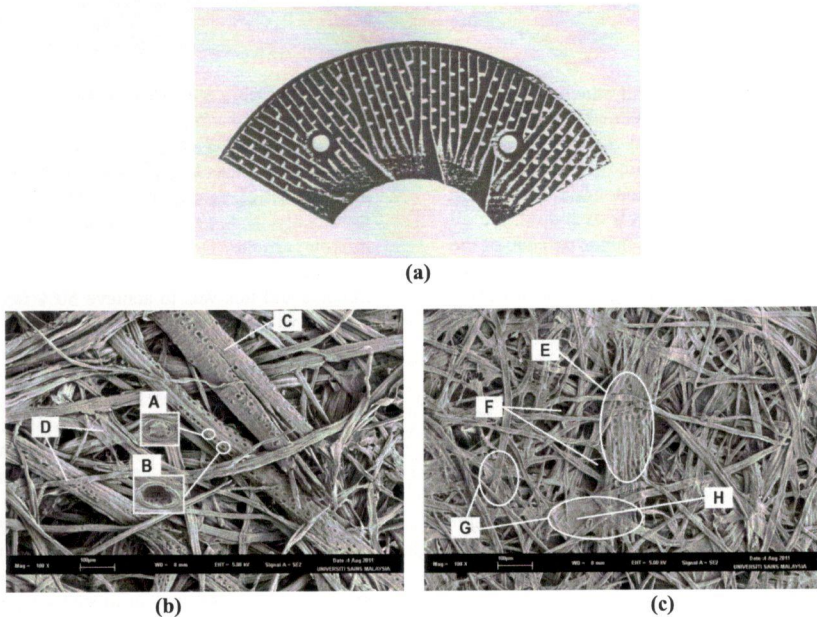


Fig.1. SEM of hand sheet surface showing the network of pulp produced by refining EFB with refiner plate pattern 12716 in (a), while the micrographs show surface of hand sheets formed by the network of pulp produced from (b) 1-stage AP impregnation and (c) 4-stage AP impregnation.

to pulp mass of loosened ribbon like fibrils. These high volume fibre were likely to cause sheet porosity (Fig. 1c) without the gap filling effects of fines. Plate pattern 12716 produced pulp with long fibre and good degree of fibrillation and this creates good fibre network. As a result, network of fibers appears more uniform on the paper surface [9]. The reduced gap sizes (F) and improved fibre bonding as a result of internal fibrillation was the features noticed at such areas as (G). As internal fibrillations are facilitated by extractive-rich cell wall peeling, 4-stage AP impregnation has thus removed significantly higher extractives than did the 1-stage AP impregnation.

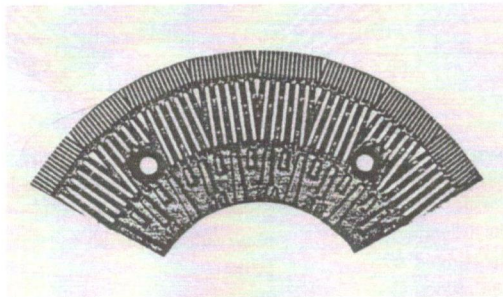
b. Refiner Plate 2

SEM of hand sheet made from pulp derived from refining EFB with plate D2A505 also shows

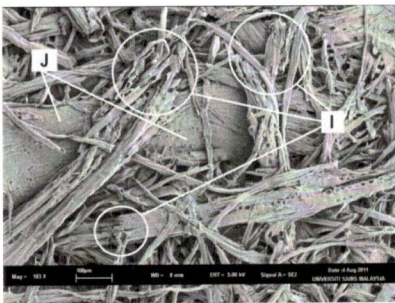
predominance of vascular bundle, generated by 1-stage AP impregnation (Fig.2). Some of them are ruptured, which might give rise to fines generation. Big fibre bundles with high counts of silica craters were still visible, implying the stiffness of the biomass. This is attributable to more powerful effect of the three refining zones of D2A505 plate pattern. The three zones are:

- Rough zone located near pulp entry (centre),
- Medium zone located after rough zone,
- Fine zone located after medium zone to the pulp exit.

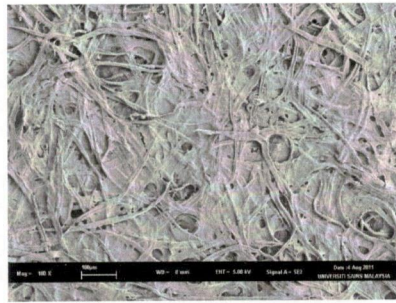
The predominance of the large medium zone was the possible cause of the unfavourable poor defiberation features.



(a)



(b)



(c)

Fig.2. SEM of hand sheet surface showing the network of pulp refined with plate pattern D2A505as shown in (a), giving pulp network from (b) 1-stage AP impregnation and (c) 4-stage AP impregnation.

With D2A505 plate pattern, 4-stage AP impregnation gave an improved pulp mass. Fibre bundles, silica body and silica craters were no longer found on the sheet surface. The number of fibrils was

greater and fibre-to-fibre bonding was mostly promoted by the internal fibrillation of the biomass.

As earlier reported [10] fines co-generated from the refining process also aided the networking amongst fibres. The friction from shearing effect of

plate D2A505 during refining caused peeling and damage of the outer cell wall, removing the primary wall and thus, creating internal fibrillation. This denotes lignin softening, which caused fibres to have more flexibility and better collapsibility, giving a smoother hand sheet surface with an almost complete coverage of the sheet surface with the unravelled microfibrils. Apparently, the increased number of impregnation stages caused an increase in inter-fibre bonding and an increase in fines generation. This

results in fibre coalescence, which is a typical reflection of good paper strength arising from good fibre networking.

Strength Development

Table 1 shows hand sheets properties improvement created by refining the AP-treated EFB with two types of refiner from 1-stage AP impregnation and 4-stage AP impregnation.

Table 1. Hand sheets properties

Plate Pattern	12716		D2A505	
	1	4	1	4
Canadian Standard Freeness (ml)	742 ± 0.0	697 ± 0.0	488 ± 3.5	139 ± 7.1
Tensile Index (Nm/g)	6.91 ± 0.8	16.15 ± 1.4	6.90 ± 0.7	29.80 ± 2.3
Folding Endurance	2.13 ± 0.3	14.30 ± 2.8	1.90 ± 0.6	1633 ± 657
Burst Index (kPa·m ² /g)	1 ± 0.0	1 ± 0.1	0.49 ± 0.0	2.02 ± 0.1
Tear Index (mN·m ² /g)	3.60 ± 0.1	4.99 ± 0.3	2.90 ± 0.1	5.27 ± 0.2

a. Refiner plate 1

Pulp derived from refining EFB with refiner plate 12716 shows a 6% reduction in the Canadian Standard for Freeness (CSF) by increasing the impregnation stages from one to four. The reduction suggests an increase in the percentage of lower-size fibrous mass, suggesting also the reduced number of fibre bundles. This correlates well with the aforementioned better softening effect by application of 4-stage AP impregnation, which had potentially allowed detachment of cell walls for liberation of individual fibres. The improvements in mechanical properties include:

- Tensile index enhancement by 134%, indicating an improved fibre bonding;
- Folding endurance improvement by 571%, suggesting improvement in flexibility of hand sheet;
- Tear index enhancement by 39%, implying better portion of individual fibres. This results in good sheet uniformity arising from good EFB fibrillations;
- Burst index showed no significant changes, plausibly due to the inherent microvoids or gaps in the fibre network [11]. This could probably be

the result of a big scatter in fibre size variation and an insufficient amount of desirable fines and fibres to act as gap fillers, analogous to the observation reported by Kamaluddin and team [10].

b. Refiner plate 2

Canadian standard freeness reduced by 72% with multiple AP impregnation stages, arising from an increment in the number of fines and number of shives. The improvements in other mechanical properties include:

- Tensile index improvement by four-fold,
- Folding endurance enhancement by 850%,
- Tear index increment by 82%,
- Burst index increased by over 400%.

Hand sheet made by four 4-stage AP impregnation with refiner plate pattern D2A505 showed an overall higher strength compared to its 1-stage counterpart and also to hand sheet produced by plate pattern 12716. This was also demonstrated in the image of SEM (Fig. 2c) showing improvement in EFB fibrillations, the extent of fibre coalescence and the intensive bonding, typical of EFB hand sheet of high strength value.

CONCLUSION

SEM results offered important information on hand sheets morphology and described fibre bonding behaviours. These make SEM a useful aid in identification of pulp properties development. In this regard, refiner plate pattern was profound in its effect on pulp and this is particularly true for multiple-stage alkaline peroxide impregnation. The targeted pulp properties can be developed by varying the refiner plate patterns and AP impregnation stages. For certain applications demanding for high strength and flexibility, refiner plate pattern D2A505 would serve the purpose.

ACKNOWLEDGEMENT

The author of this paper is the holder of USM Fellowship (First Year: 1st January – 31st December 2010, and Second Year: 1st January – 31st December 2011). Execution of this research may not be possible without the funding from Research University Grant 1001/PTEKIND/814048.

REFERENCES

- [1] Hai, T. C., (2000Q2). *The palm oil industry in Malaysia: from seed to frying pan*. (WWF Switzerland).
- [2] Cort, C. J., and Bohn, W. L., (1991). Alkaline peroxide mechanical pulping of hardwoods. *Tappi J.*, 74(6):79-84.
- [3] Blodgett, R., Genco, J. M., and Cole, B. J., (1998). Alkaline peroxide mechanical pulping of northeastern hardwoods, in innovative advances in the forest product industries (edited by Brian Brogdon). *AIChE Symp. Series*, No. 319(94):21-31.
- [4] Xu, E. C., (2002). Some of the latest investigations in P-RC APMP pulping of hardwood part 1: LCR at secondary refining. *Proceedings PAPTAC 88th Annual Meeting*. Montreal, Canada. 28 January-1 February, 2002.
- [5] Kong, F., Chen, J., Wang, S., And Zhan, H., (2005). Effect of NaOH charge on fibre characteristics of P-RC APMP Pulp. *Proceedings 59th Appita Annual Conference and Exhibition: Incorporating the 13th ISWFPC*. Auckland, New Zealand. May 16-19, 2005, pp. 197-201.
- [6] Xu, E. C., (2001). APMP pulps from non-wood fibres part 3: Baggase. *Tappi Pulping Conference Seattle*. Nov 4-7. Washington, USA.
- [7] TAPPI (1996). *TAPPI Test Method 1996-1997*. (TAPPI Press. USA).
- [8] Smook, G. A., (1989). *Handbook for Pulp & Paper Technologists*. 2nd edition. (Angus Wilde Publications. Vancouver Bellingham). pp. 197-201.
- [9] Kure, K.-A., Sabourin, M.J., Dahlqvist, G., Helle, T., (2000). Adjusting refining intensity by changing refiner plate design and rotational speed – effects on structural fibre properties. *J. Pulp. Pap. Sci.*, 26(10), p. 346-352.
- [10] Kamaludin, N. H., Ghazali, A., and Wan Rosli, W. D. (2012). Potential of Fines as Reinforcing Fibres in Alkaline Peroxide Pulp of Oil Palm Empty Fruit Bunch. *BioResources* 7(3), 3425-3438.
- [11] Groot, B. D., Kolk, J. C. V. D, Dam, J. E. G. V., and Riet, K. V., (1999). Papermaking Characteristic of alkaline Hemp-Woody-Core Pulps. *Tappi J.*, 82(7).