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EVIDENCE FOR PAPER STRENGTH IMPROVEMENT BY INCLUSION OF FINES GENERATED FROM APMP OF EFB

NURUL HASANAH KAMALUDIN, ARNIZA GHAZALI* AND WAN ROSLI WAN DAUD

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

**Corresponding author: arniza@usm.my*

Abstract: The oil-palm empty fruit bunch (EFB) was subjected to alkaline peroxide mechanical pulping (APMP) treatment concept for pulp production and paper-making purposes. Analysis showed that tensile strength of the pulp network increased twice as much by blending the pulp with 12% of the 400-mesh fines fraction recovered from the refining and screening streams. It was also observed that the recovered 400-mesh fines carried many forms, some being pitted vessel elements with long tube-like shapes of varying diameters as a result of vessel elongation. The 'split' vessels, however, were resemblances of narrow fibres and this arose from the splitting that occurred along the perforation of the cell wall. In the paper formed by the EFB APMP pulp network, the various forms of vessel element inter-locked with each other, filling the micro-voids, which were initially present as a result of partial bonding between the incompletely-fibrillated fibre bundles. It was the filling effects of these fines elements that were responsible for improving the tensile strength of the produced handsheet by 100%.

KEYWORDS: APMP, EFB, lignocellulosic, fines, filler, vessel elements

Introduction

Oil palm has the largest planted area of 4.69 million hectares of land in Malaysia and palm oil contributes the major revenue to the country's economy (MPOB, 2010). Ensuing from the milling activity, however, is the issue of waste management pertinent to the 17 million tonnes of EFB annually generated (Anon., 2011) as the fruits are pruned for palm-oil extraction. Traditionally, the empty fruit bunches are left to rot in the environment or burnt in open air, which is a severe source of carbon emission. While open burning causes air pollution and excessive of this can lead to global warming, leaving the residual fruit bunch to rot in the environment invites pests besides triggering the source of fouling odour. In the effort of improving the present scenario, research on utilisation of EFB has become mandatory and this was realised as early as the 80's (Ghazali, 2006).

To date, research on utilisation of EFB continues to grow, be it for pulp and paper production, in the making of panel products like medium-density fibreboard (MDF), creative application as glazing material, as briquette for biofuel production or as animal feed (Ghazali *et al.* 2009). Its ideal composition and fibre characteristics make the residue an economically potential raw material in preferred applications such as papermaking (Zainon, 2011).

Only an environmentally sound and efficient process for pulp production can ensure sustainable production of pulp from EFB and, to date, APMP or the alkaline peroxide mechanical pulping is the only system in closest-to-perfect match to these criteria. APMP is a benign pulping technique that incorporates both pulping and chlorine-and sulfur-free bleaching in a single process. For offering a 2-in-1 effects, APMP, therefore, is cost-saving for eliminating the need for a separate bleach plant and for being simple in operation and maintenance (Burkhart, 2001). Besides that, APMP system is also reported as a very flexible system and is highly adaptable to a wide spectrum of biomass. Xu and co-workers reported success of adapting a modified APMP system onto kenaf, straw, baggase and jute (Xu *et al.*, 2001a), (Xu *et al.*, 2001b), (Xu *et al.*,

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2001c). The pioneer of the native APMP, Cort and Bohn (1991) of Andritz Sprout-Bauer, U. S. A., however, reported success for pulping of wood species such as aspen, while later, others reported successful application of the system onto birch, maple and poplar (Blodgett, *et al*., 1997), (Francis *et al.*, 2001). Wood, however, is scarce and logging is to be prevented to control deforestation and to ensure a healthier eco-system.

In line with the nation's focus on sustainability development, wood alternative lignocellulosic material such as non-wood and agro-wastes are to be considered for utilisation for pulp production in Malaysia. This paper provides an insight into the application of APMP on EFB – an abundant oil-palm milling waste in Malaysia, with emphasis on the strength-enhancing role of the fines from its downstream process.

Experimental

Material

The fibrous strands of EFB were the chosen lignocellulosic material obtained from Sabutek (M) Sdn. Bhd. in bales of dried long fibrous strands. These consisted of vascular bundles that were washed and air-dried upon receipt and the strands were then cut into 2±0.5 cm segments at USM laboratory.

Methods

Pulp Preparation via APMP Concept

APMP of EFB was carried out in accordance to the method adopted by Ghazali (2006). On a partially extractive-free EFB segments that were obtained by soaking the biomass in distilled water at 70°C for 30 minutes in water bath, 15 psi pressure was applied on the decanted EFB using an impregnation device. After pressing, the alkaline peroxide chemicals were allowed to impregnate into the biomass at a resultant consistency of 10-to-1 liquor-to-fibre ratio. The alkaline peroxide containing 2% (wt/wt) sodium hydroxide (NaOH) and 2.5% (wt/wt) hydrogen peroxide (H_2O_2) was reacted with EFB and this cooking process was allowed for 30 minutes to soften and brighten the biomass. Next, the biomass was once again pressed at 15 psi and the applied pressure was released at a dewatering rate of three drops per minute. The EFB was next refined using Andritz Sprout-Bauer 12" single disc refiner.

Fines Collection

Fines were collected by placing the 250, 300, and 400 mesh screens at the discharge of the 200 mesh sieve that was normally used to collect the 'screened pulp' or 'accepts'. The screens carrying opening sizes of 76 mm, 63 mm, 53 mm and 37 mm for 200, 250, 300 and 400-mesh, respectively, were selected for capturing fines that escaped the 76 mm diameter hole of the 200-mesh screens trapping the pulp mass. Pulp and fines fractions were carefully labelled and stored in refrigeration for handsheets preparation.

Making of Hand Sheet

The 200, 250, 300 and 400 mesh fines were weighed before mixing in due proportions with the 200-mesh pulp mass and each resultant slurry was homogenised using Toyoseiki disintegrator. Handsheets were to constitute 12% fines as given in Table 1, amounting to formation of paper with 12% filler, so that filler proportion falls within the range of filler added in commercial papers. The next steps for handsheet preparation adopted TAPPI standard procedure, T 205. The prepared sheets were then tested for their mechanical properties in accordance to TAPPI Test Method T 511 for folding endurance using Kumagai Riki Instrument, T 414 for tearing resistance by way of Elmendorf Tearing Tester, T 403 for burst index and T 494 for tensile index using Lloyd Instruments.

As is apparent from Table 1, sample A (control) was formed by unscreened OPEFB pulp and sample B is the handsheet of screened pulp (accepts) while C, D and E were the handsheets blended with 250, 300 and 400 mesh fines fractions.

Microscopy

Sample preparation

Handsheets with different mesh fines were examined using light microscope with four

Table 1: Pulp and Fines Compositions in the Six Sets of Handsheet

Sample Description	**Portion of Pulp and Fines (%)**			
	Screened Pulp	**Fines**		
		250 mesh	**300 mesh**	**400 mesh**
Unscreened pulp (A)	-	-	-	-
Screened pulp (B)	100	-	-	-
B + 250 mesh fines (C)	88	12	-	-
B + 300 mesh fines (D)	88	-	12	-
B + 400 mesh fines (E)	88	-	-	12

lenses fixed at the rotating nosepiece. All slides were labelled accordingly. Light-transmission microscope interfaced with an image analyser was then run to analyse the suspension of fibres and fines.

SEM or scanning electron microscopy was run on the 30 nm gold-coated samples using Carl Zeiss Leo Supra 50VP to check for evidence of fines entrapped in the pulp network. For low magnification viewing of the intact sheet surface-fibre network, Dino Lite Pro portable microscope was used.

Results and Discussion

Microscopic Observation of Fines and Pulp

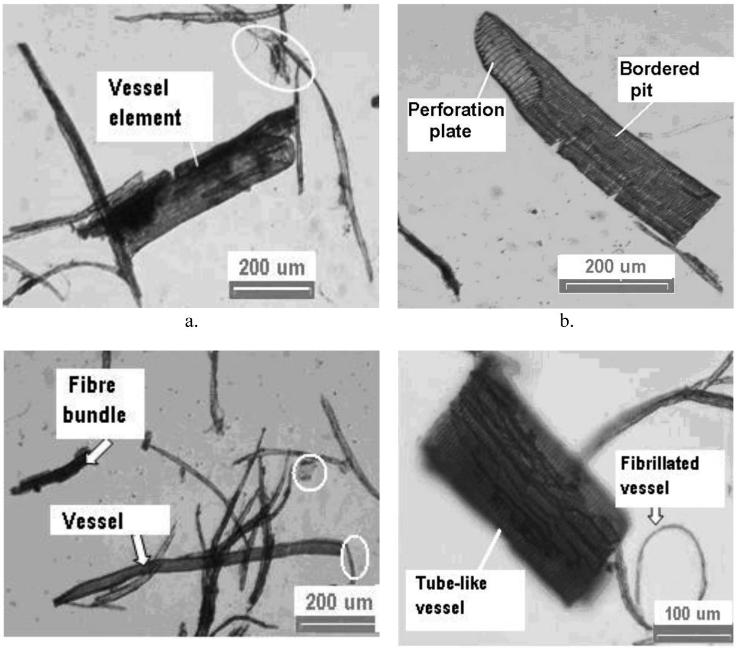
Microscopic inspections of the unscreened pulp collected as 200-mesh pulp fraction show the presence of common vessel element in the form of bordered pits of length, 573.1 µm (Fig. 1a). Also spotted were fibre bundles (629.5 µm in length, Fig. 1b), from unfibrillated biomass fragments making up the accepts hereby referred to as the pulp mass.

Fig. 1c presents the evidence of further shearing of the said fragments, resulting in formation of fibre bundles. At this magnification, fines elements are visible as debris, indicated in circles. Although these vessel elements appear similar to fibres, they differ, somewhat, in structural properties (Gorres *et al*., 1996), with fines defined as the fraction passing through a 200 mesh-size screen that consists of pieces of fibre, influential in giving better sheet density (Gorres and *et al*., 1996). This stems from the higher swelling capacity of fibrillar such as fibrils and thin lamella.

The aforementioned fibrillar fines are more prevalent of the higher mesh-fractions. Fines, according to Peel (1999) are believed to be associated with the refining action, which only allows fibres and fibre bundles to pass between the plates. Of these, nothing like flake structures, which are stiffer and hygroscopically less susceptible, were encountered but the presence and amount of the found unfibrillated materials reflected the rigidity of the lignin-carbohydrate matrix, suggesting that the adopted EFB treatment condition was insufficient to soften the lignocellulosic material, typically derived from the specified APMP concept.

The long tube-like and tapered vessel with 792.4 µm length (Fig. 1d) was also found in the 200-mesh pulp fraction (Fig. 1c). These indicate that vessel and fibre fraction failed to fibrillate extensively and the 200-mesh and 250-mesh fractions shared an extent of similarity. In the 250-mesh fraction of the examined pulp, a shorter tube-like vessel element of 272.6 µm length (Fig. 1d) occurred as a result of fibrillation at the end of vessel element. The main problem associated to the abundance of these structures was poor inter-fibre bonding in the whole pulp network because the fibres tend to form networks that on-trap the vessels. Having added non-uniformity to the size and shape of the pulp mass, these fragments are likely to interrupt inter-fibre bonding.

Both Fig. 2a and Fig. 2b show fines characteristic of the extensively fibrillated vessel



a. b.

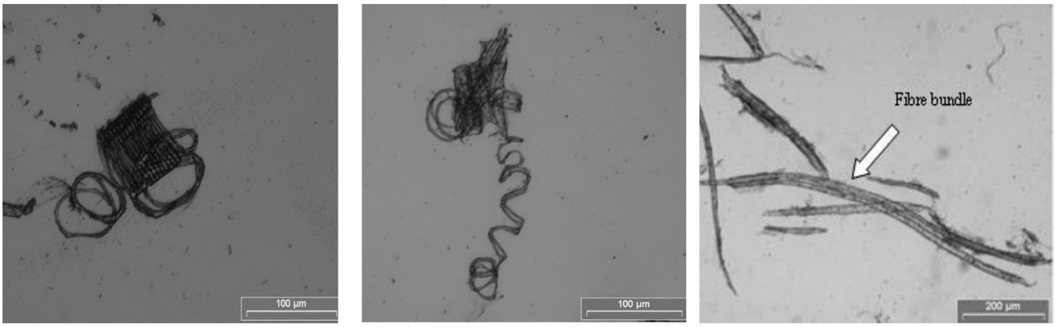
Figure 1: Fragments of EFB collected in the 200-mesh and 250-mesh fractions a) vessel element and fibres, b) Vessel element within the perforation plate c) Fibres and vessel elements that were incompletely fibrillated d) tapered vessel recovered on 250-mesh fractions (x10). NB: circled are debris found in the pulp mass.

elements. This occurred as a result of 'splitting' along the perforation line, amounting to pulp mass in the form of fibre or single strand, not thin enough (Length= 66.8, width= 76.5) to escape the 53 mm x 53 mm holes of the 300-mesh screen. It is also this dimensional characteristic that allows these fines to serve as filler of the screened pulp network, offering an increase in pulp overall bonding network by way of fibre-to-fibrillar and fibrillar-to-fibrillar, in addition to the existing fibre-to-fibre bonding.

Amidst the 300-mesh fines fractions, however, incompletely fibrillated fines originating from fibre bundle were still detected as the most common cell types besides other fragments such as vessel elements, which are being better fibrillated. These are about 70 mm (Fig. 2) in comparison to 272.6 mm vessel elements in the 250-mesh fraction. Less fibre bundles were observed as occurring amongst the 300-mesh fines.

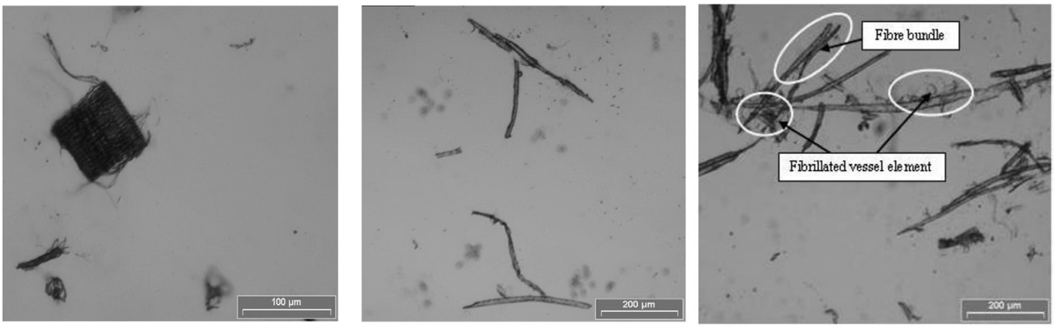
As expected, fibrous mass of reduced dimensions (vessel fragments: length = 74 mm, width = 76 mm; fibre length = 261.3 mm) were most probable in the 400-mesh fines fractions (Fig. 3) and this slandered the possibility of trapping the bigger fibre bundles. It was also discovered that on top of structure variability, completely fibrillated xylem vessel were predominant amongst the 400-mesh fines fractions, suggesting the tremendous shearing effect experienced by EFB, plausibly associated with the lignocellulosic sites receiving adequate alkaline peroxide in the preceding treatment.

Also noteworthy is the possibility of excessive shortening of the biomass and this is evident from Fig. 3c showing 200 μm fragments of fibre bundles amid individual fibres. With the tremendous size reduction (11.35% for vessels and 37.5% for fibre bundles in 300-mesh fines fraction), these fines are to give more compactness to fibre network (Fig. 4a and Fig 4b).



a. b. c.

Figure 2: Fines collected amongst 300-mesh fines fractions a. Xylem vessel ‘splitting’ to a single coiling strand (x10) b. Split xylem vessel resembling long, spiral fibres (x10) c. Fibrillated structure representing the most common cell types (x4).



a. b. c.

Figure 3: Fines collected as 400-mesh fractions a) Shorter vessel (x10) having lost the dangling split strand b) Fibre fragments (x4) c) Mixture of completely ‘split’ xylem vessel and fibre bundle (x4).

Effects of Fines on Paper Network

The forms of fines occupying the micro-voids in the paper network are revealed in the scanning electron microscopic analysis of the produced handsheets (Fig. 4c, 4d, 4e, 4f). Intact vessels were found in the sheet of paper prepared with the 200-mesh pulp blended with 250-mesh fines fraction while more fibrillated structures (split vessels) are shown in Fig 4e amongst the pulp network incorporating 300-mesh fines fraction. Of these, apparently, the fines from 400-mesh fractions fit into micro-voids more snuggly and layering to the top of the paper network, due to the higher collapsibility of the 400-mesh fibrillar type of fines (Fig 4a). These short fibrillar served the said features mainly due to their contribution to the number of substances with swelling capacity and consequently, imparting higher density, in comparison to the 300-mesh fines (Fig. 4b). At the same magnification, less gaps are detected in the handsheet with 300- and 400-mesh fines (Fig. 4e and 4f) in comparison to the network with purely 200-mesh pulp fraction (Fig. 4c) and the 200-mesh fraction blended with 250-mesh fines (Fig. 4d).

As a way of verifying the trend of fines effects on the produced pulp network, handsheets mechanical properties were examined. The trend of improvement in bondability of the pulp network is evident from Table 2. The presence of fines contributing to filling of micro-voids and improved bonding is well illustrated by the vivid improvement in handsheet density, which appears to correlate well with the general effects of fines claimed by Gorres and team (1996).

It is apparent from Table 2 that screening of EFB pulp with 200-mesh screen hampered tensile and tear indices by about 19%. Addition of 250-mesh fines fraction improved tensile

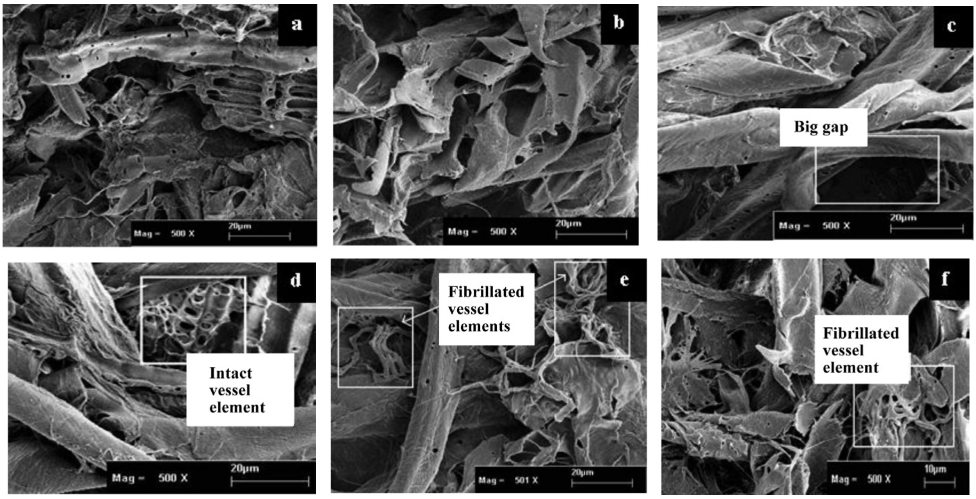


Figure 4: Fines a) 400 mesh fines. b) 300 mesh fines. Fines in the handsheet: c) 200 mesh pulp fraction. d) 200+250 mesh fines. e) 200+300 mesh fines. f) 200+400 mesh fines.

index by 61% while the 300-mesh fines and 400-mesh fines fractions improved tensile index by 75% and 100%, respectively. All pulp and fines blends improved tear index by 15% and this is only 0.1 point inferior to the whole pulp network (Sample A), suggesting that the individual fibres were bound quite tightly to the surrounding fines, making them more difficult to pull out from the network of native fibrous mass comprising the mixture of more complex fines. Even without strength of fibre imparting better sheet tear resistance, these complex fines contributed well to better fibre bonding, as partial fulfillment for acclaimed findings (Page and MacLeod, 1992; Seth and Page, 1988) establishing that tear strength is a function of both fibre strength and fibre bonding.

Table 2: Properties of Handsheets by Varying Blend of Pulp and Fines.

	Sample A	Sample B	Sample C	Sample D	Sample E
CSF (ml)	710	580	564	550	530
Density (g/cm^3)	0.2	0.2	0.3	0.3	0.3
Tensile Index (Nm/g)	4.3	3.6	5.8	6.3	7.2
Tearing Index (mNm^2/g)	3.9	3.3	3.8	3.8	3.8
Folding Endurance	0.00	0.00	1.4	1.7	1.9

A – Unscreened EFB APMP
B – Screened EFB APMP
C – Screened + 12% 250-mesh fines
D – Screened + 12% 300-mesh fines
E – Screened + 12% 400-mesh fines

The manifold improvement in tensile index of other pulp blends, in addition, reflects the

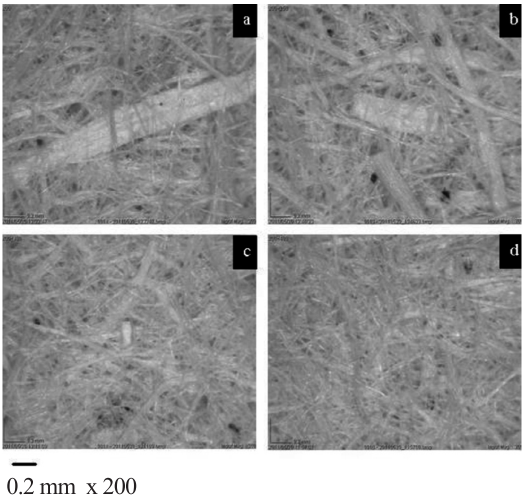


Figure 5: Relative filling effects of fines is seen as disappearance of vessel elements in the handsheet a) made from unscreened pulp b) Handsheet added with 250-mesh fines fraction c) handsheet added with 300-mesh fines fraction d) Handsheet added with 400-mesh fines fraction.

strength-enhancing effects of the fines collected from the APMP of EFB, which were evidently vessel elements. The split vessel elements shown in Figure 3 resemble the tiny versions of slender fibres with increased flexibilibity and collapsibility in comparison to the xylem vessel in Figure 2a, for instance. Fines amongst split vessel elements allowed better bonding of fibres and offered an increase in tensile strength. This correlates with the observation made by Rousu and Niinimaki (2005) who reported strengthening effect of fines in the form of vessel elements from non-wood pulp of monocot category.

The absence of ray cells in all of the studied fines fractions further explains the strength-enhancing effects of fines from EFB pulp. According to Sundberg and team (2003), ray cells are of no aid to pulp network strength improvement. The better strength-enhancing effects of the 400-mesh fines fractions were principally due to the more extensive rupture of the vessel elements into split cells of dimensions small enough (130-360 µm in length and width 5-10 µm – Fig. 3c) to hold higher possibility of getting into the micro-voids (approximately 10 mm x 20 mm) created by the fibre bundles of the screened pulp. Besides the perfect split structures analogous to the mass shown in Fig. 3c, other fragmented mass indicated in Fig. 3 were also responsible in filling the micro-voids in the 200-mesh pulp network. Owing to their higher surface areas, other than acting as filler, these elements also provided contact for enhanced fibre-fines and fines-fines bondability (Fig. 4f), to an extent of masking the rigid mass (Fig. 5a and Fig. 5b) that was glaring in the screened pulp network (Fig. 5a) and pulp blended with 250-mesh fines fraction (Fig. 5b).

The masking effect resulted in paper surface covered with more uniform fibers illustrated in Figure 5c and 5d. The more uniform appearance also arose from the filling effects of the shorter and thinner fines amongst the 400-mesh fractions. As much as forming uniform topping, these fines also accommodate the micro-voids between the 200-mesh pulp fraction, resulting in an overall 100% increase in the network tensile strength.

Conclusions

Handsheets blended with 400-mesh fines fractions show superior improvement in tensile index and folding endurance. Predominance of the strength-promoting cells such as vessel elements and the sheared fragments of these was the key factor for strength enhancement and these were detected in a size suitable to accommodate the micro-voids commonly found in the network of pulp produced by the adopted method. Besides acting as filler, pulp network strength was also improved by the enhanced pulp bondability promoted by the high surface areas of the examined fines. Besides improving pulp network strength, retention of fines for papermaking can also reduce discharge of organics in the pulping downstream process. This helps reduce pollution, ease treatment of pulping discharge and serve as mimicry of a zero-waste system.

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