



Sequential Synergy of Alkaline Peroxide Treatment and Refining in Co-generating Filler for Pulp Web Augmentation

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Abstract: Desired pulp-based product properties can be achieved by addition of filler in the pulp network. In exploring this, fines co-generated upon refining the alkaline peroxide treated oil palm empty fruit bunches (EFB) were collected based on their passage and retention capacities when subjected to varying mesh-sizes stainless-steel square mesh wires. Pulp network incorporating fines produced from the synergy of low alkaline peroxide (AP) and low energy refining effects shows that blending 12% of the 400-mesh fines (P300/R400) with the normal 200-mesh pulp fraction enhanced paper tensile strength by 100% due to their favourable dimensions. This defines the usefulness of fibrillar particles whose cell wall collapsibility increases the web density by increasing bonding ability and thus, strength of pulp-based products. Fines produced from more extreme synergy between alkaline peroxide and degree of refining, exhibit unique submicron fibrils and 'nano-CGF' also responsible for further augmentation of EFB alkaline peroxide pulp network. Whether from the simple (low-AP and low energy refining) or the extreme synergy of AP and refining, the co-generated fines are apparently suitable materials for use as natural filler for augmentation of pulp network. Particularly for the simple AP and refining synergy, the introduced recovery and utilization of the co-generated filler (CGF) was found to reduce 74% turbidity and this improvement will help reduce the complexity of whitewater generation in the pulping system.

Key words: CGF; Empty fruit bunch (EFB); Fines; Nano-fibrils; Refining.

INTRODUCTION

Persistent reliance on timber for pulping and papermaking intensifies concerns over depletion of world carbon storage from forest biomass. Global biomass consumption for 2010, for instance, reveals the needs to gradually reduce the 70%-to-30% portion of wood-to-nonwood fibre supply as the principal measure for resolving the issue [1]. Besides opting to wood alternatives, pulp and paper products consumption pattern and contingency can be improved. Adaptation of existing technology and related innovations in non-wood pulping and processing, which can be extra demanding in comparison to wood pulping [2], also needs extensive research. Efforts in non-wood utilization, in addition, need a proper alignment with the concept of sustainability to arrive at waste minimisation and benign carbon emission.

Asia is a rich source of non-wood pulp produced from annual fiber crop, agricultural residue (also known as agro-waste) and non-plant fibrous mass with Malaysia, known for its 19.3 million tonnes of fibrous empty fruit bunches, EFB, [3] generated from its

palm oil milling sector. Traditionally, the EFB are left to rot in the environment or burnt in open air. While open burning pumps in carbon dioxide into the atmosphere (an excess of which can lead to global warming), leaving the residual fruit bunch to rot in the environment attracts pests and creates a source for foul odour. Utilisation of the industrial-cum-agricultural residue, therefore, offers productive management of the waste from the country's major cash crop. Per hectare oil palm plantation could generate at least double the per annum quantity of pulp harvested from the local rainforest. This corresponds to over 88 million trees-saving, on the assumption that all EFB could be converted to pulp [3].

To date, research on utilization of EFB continues to grow. With minimal processing, EFB could be engineered as an efficient pollutant sorbents [4-6]. It is also a type of biomass currently researched for the practical possibility of biofuel production, despite the often-said snags. Beyond research, today, a small amount of EFB is used as medium-density fibreboard, mats, mattresses, cushions and light furniture. Also proven practical is its use as briquettes, subsequently incinerated for electricity generation [3].

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Similarly, destruction of EFB lignocellulose matrix by high temperature incineration allows production of melt siliceous glaze as alternative coating material for ceramics and pottery [7]. On the contrasting consideration, EFB, having predominance of cellulose, lower lignin in comparison to most local wood, and having unique fibre characteristics, render the residue more viability as raw material for pulping and conversion to paper-based products [8–10] as compared to bio-energy [10] and glazing applications. Production of paper-based products requires huge amount of fibrous materials in the form of pulp and EFB serves the purpose in desirable ways. As the first hurdle, pulp extraction from EFB was attempted by applying an environmentally benign [7] process concept of the alkaline peroxide mechanical pulping, APMP. As the name describes, APMP involves the use of alkaline peroxide at the stage of biomass pre-treatment, prior to refining process. An APMP system, therefore, is a sulfur-free and chlorine-free pulping process. With AP in the liquor formulation and retained within the biomass during refining, pulping and bleaching effects are achieved in a single process, yielding a bright coloured pulp. Under certain circumstances, a separate bleach plant is not required and this taxes less of the ensuing operation and maintenance costs. Apart from the acclaimed [11–16] simplicity, various possible adjustments that can be made to the refining parameters, alkaline peroxide level, alkaline peroxide impregnation stages and processing temperature, to suit to the choice of biomass and the resultant pulp quality. The demonstrated process offers flexibility and high adaptability to a wide spectrum of biomass was first reported by Cort and Bohn [17] based on the success of APMP™ of wood species such as aspen. Subsequent works documented successful application of the system to birch, maple and poplar [18, 19]. Through certain upgrading measures, Xu and co-workers reported success of adapting a modified APMP system [11] to kenaf, straw, bagasse, and jute [14–16], as well as to such tropical hardwood as acacia mangium [20].

Trials on adapting APMP to EFB [7, 8] witnessed the wide possibility of pulp quality by adjustments of experimental parameters and machinery [21]. The usefulness of fines co-generated by the mimicked APMP system (hereby denoted APP for alkaline peroxide pulping) was also noticed to contradict the reported undesirable effects of wood fines and short fiber [22]. Usefulness of wood and non-wood market pulp fines has been in wide coverage [23–28] till the present time. Lukko and Paulapuro, for instance, found that desirable fines were co-generated with intensified refining [29]. While flakes were found of less value [26, 29], fibrillar structures were found to render paper density, reducing solid-air extension and therefore improved optical qualities.

Early attempts of APP of EFB not only observed encouraging effects of fines on paper strength [21] but also an unquantified improvement in refining discharge clarity, which is important in reduction of total suspended solids (TSS) in used process water. At the present level of knowledge, TSS reduction is

achieved by backwash filtration technology, adopted in the whitewater closed-loop systems where reuse of water in the water-intensive industry is made more efficient [30]. This paper describes the fines elements in EFB APP pulp and identifies the strength-reinforcing potential of the pulping by-products. As EFB pulp is gaining worldwide acceptance as blended pulp in many industrial paper and packaging products, the knowledge on the potential use of the waste material as co-generated filler (CGF) is especially important in maximization of process yield and minimization of waste.

METHOD

Materials: The fibrous strands of EFB were obtained from Sabutek (Malaysia) 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 Universiti Sains Malaysia (USM) laboratory.

Pulp Preparation in Simple and Extreme Sequential Synergies of Alkaline Peroxide and Mechanical Refining: Alkaline peroxide pulping (APP) of EFB was carried out by simplification of the previously adopted method [31]. The named pulping system works on the principle of alkaline peroxide – pretreatment of the biomass and a subsequent refining of the AP treated biomass.

On a partially extractive-free EFB that was obtained by soaking the biomass in distilled water at 70°C for 30 minutes, 15 psi or 103 kPa pressure was applied using a fabricated impregnation device. Upon release of pressure, the alkaline peroxide (AP) was allowed to impregnate into the biomass at a consistency of 10-to-1 liquor to EFB mass ratio. The alkaline peroxide containing 2% sodium hydroxide (NaOH) and 2.5% hydrogen peroxide (H_2O_2) was reacted with EFB, and this cooking process was allowed to stand for 30 minutes to soften and brighten the biomass. After cooking, the fibrous mass was again pressed at 15 or 103 kPa until reaching a dewatering rate of three drops per minute. For the simple AP and refining sequential synergy, EFB was refined using Sprout-Bauer 12” single disc refiner with resultant specific refining energy of 54.95 kWh/t corresponding to 4% pulp consistency and refining temperature of 33.5°C, whereas for extreme AP and high energy refining sequential synergy, refining was performed in PFI mill. This is related to the preparation of samples P and Q.

Collection of Fines for Use as Co-generated Filler (CGF): Pulp refining discharge consisting of variable pulp sizes was fractionated by allowing flow of the suspension through fabricated sieves of 200-, 250-, 300- and 400- mesh screens. These were stainless steel square-opening mesh sieves with $76\ \mu\text{m} \times 76\ \mu\text{m}$, $63\ \mu\text{m} \times 63\ \mu\text{m}$, $53\ \mu\text{m} \times 53\ \mu\text{m}$ and $37\ \mu\text{m} \times 37\ \mu\text{m}$ square apertures corresponding to the 200-, 250-, 300- and 400-mesh screens, respectively. CGF were

retained on specific mesh sieves due to their inability to escape the 76 μm aperture of the 200-mesh screens trapping the pulp mass called the ‘accepts’. With respect to sieves mesh sizes, the collected CGF are labeled P200/R250, P250/R300 and P300/R400 with P denoting ‘pass’ and R denoting ‘retain’. Both the accepts and CGF constitute 95% yield of the attempted APMP of EFB with loss having association with 1.5% extractives from dewaxing [8] stage, about 1% minerals and 2.5% fines and other organics escaping 400-mesh sieve. Based on the 12% to 20% range of CGF by the 3 mesh fractions, the R200-to-CGF proportion is approximately 60:40 mass ratio.

Making of Handsheet: Handsheets were prepared in accordance to TAPPI procedure [32]. Where CGF were incorporated, the P200/R250, P250/R300 and P300/R400 fractions were added before mixing the slurry in a Toyoseiki disintegrator. Handsheets prepared with the unscreened pulp is labeled L while those prepared with R200 were labeled M. Samples containing R200 blended with 12% CGF are consequently: R200+P200/R250, R200+P250/R300 and R200+P300/R400, labeled as M250, M300 and M400, respectively. The selected percentage of CGF was based on the 10-30% range of fillers used in commercial papers. The selected lower level was also to bootstrap: ensure utilization of fines generated per batch of pulping and to rule out the needs to run pulping specifically to generate fines. Five different sets of handsheet with 10 to 15 sheets per set were prepared and tested for their mechanical properties in accordance to TAPPI Test Method T 511 for folding endurance, T 414 for tearing resistance, T 403 for burst index, and T 494 for tensile index. Optical properties were examined by TAPPI test method T452. All density values are measured as sheet weight per volume of handsheet, where volume is the multiplication product of sheet area and sheet thickness.

Microscopy and Fiber Analysis: Fines, hereby abbreviated CGF or co-generated filler were examined qualitatively using light microscope with four lenses fixed at the rotating nosepiece. All slides were labeled accordingly and analysis of pulp mass was performed without staining. Transmission light microscope interfaced with an image analyzer was then run to analyse the fibres.

SEM or scanning electron microscopy was run on the 30 nm gold-coated handsheet samples using a Carl Zeiss Leo Supra 50VP to check for evidence of fines as CGF entrapped in the pulp network. To obtain dimensions of fiber and CGF, suspension of fines materials were dried prior to coating and analysis.

Fibre dimensional characteristics were acquired from Sherwood FAS-3000 Fibre Analysis System (USA), and this analysis was performed on pulp suspensions (pulp and the co-generated filler, denoted as CGF) as recommended by the instrument manufacturer. Quantitative proportion of the fines was also obtained from this analysis.

RESULTS AND DISCUSSION

Effects of CGF on Paper Network: Examination of pulp and fines suspension under light microscope (Fig. 1) reveals segments of vessel elements amongst the fibre and incompletely fibrillated fibre bundles, which were predominant in the R200 pulp fraction. While single fibers are desirable, fiber bundles are signs of rigidity of biomass arising from inadequate softening or reaction between alkaline peroxide and EFB during the pre-treatment stage. As indicated in Table 1, the R200 pulp fractions mostly long and medium length pulp mass, suggesting also that besides inadequate contact with alkaline peroxide, these are least affected by the subsequent mechanical actions from refining.

Escaping the 200-mesh screens was the split vessel element, originating from the unwinding of the vessel structure along the perforation lines. These are known as the fibrillated vessels, whose extent of fibrillation suggests the severity of the shearing forces experienced by the EFB vascular bundles as they pass through the friction between the refining plates. In comparison to the R200 fractions, it is plausible that these correspond to the portions of EFB that had reacted with alkaline peroxide more effectively and hence, better softened and easily sheared by the refining forces. Further segmentation of these split vessels accounted for the fines in the P300/R400 fraction. The P300/R400 fractions resemble the fibrils and small pieces of fibrils with massive broom-end appearance as indicated by the hollow arrow in Fig 1. Size wise, both of the said fractions added to the count of fines and short fibers in the pulp fractions (Table 1).

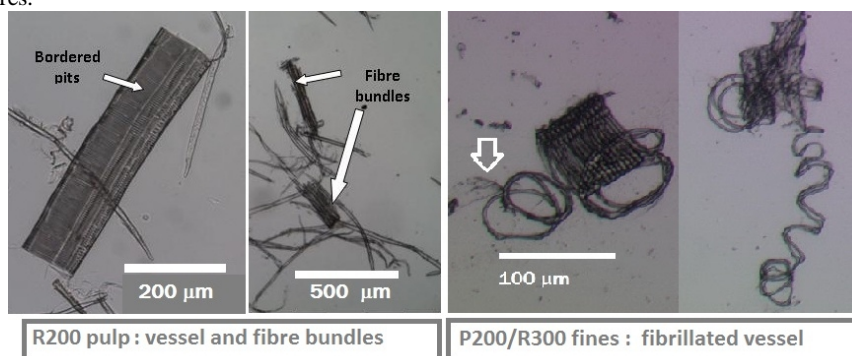


Fig. 1: Pulp mass amongst the R200 and the P200/R300 fractions [Reproduced from Kamaluddin *et al.*, 2012]

Examination of the dry CGF shows a slight but noticeable compactness. Owing to the relative size and conformability differences, in their dry forms, the P300/R400 [Fig. 2(a)] appeared denser than the more protruded raffling structures of the P200/R300 mass [Fig 2(b)]. The better density of the former fines fraction may be directly linked to their relatively reduced length (Table 1) and thus, better ability to fit into the pulp micro-voids as apparent in Fig. 2(d). Without fines, consequently, the R200 handsheets seem to show massive micro-voids analogous to Fig. 2(c). Incorporation of CGF, exhibits a layer of dried agglomerates of fibrils atop the R200 layer [Fig. 2(d)]. These were plausibly corresponding to the severely

mechanically sheared fibrils and ruptured vessel elements, typically encountered in the P300/R400 mass. This portion of the CGF better fits the fibrous web of pulp due to its higher collapsibility as also portrayed in the more clumped manifestation of the dried isolated P300/R400 fibrillar fines. The better swelling capacity associated to this CGF mass might have been the principal factor imparting higher density, in comparison to the 300-mesh fines (Fig. 1). With less gaps in between the pulp, CGF-added pulp network is also likely to manifest an enhanced overall sheets strength.

Table 1: Pulp fractions

Pulp Fractions (%) and Dimensions Defined by FQA	M	M300	M400	P
Fines (width 3-60 μm; length < 0.112 mm)	16.4	18.5	22.9	33.7
Short (3-60 μm; length 0.112-0.448 mm)	21.8	26.2	29.2	35.1
Medium (width 3-60 μm; length 0.560-1.456 mm)	50.8	46.8	42.9	30.5
Long (width 3-60 μm; length 1.568-7.168 mm)	7.7	5.6	3.7	0.6
Canadian Standard Freenes, CSF (ml)	580	550	530	57

L ≡ whole pulp;
M ≡ screened pulp;

M250 ≡ M + 12% P200/R250 fines fraction
M300 ≡ M + 12% P250/R300 fines fraction
M400 ≡ M + 12% P300/R400 fines fraction

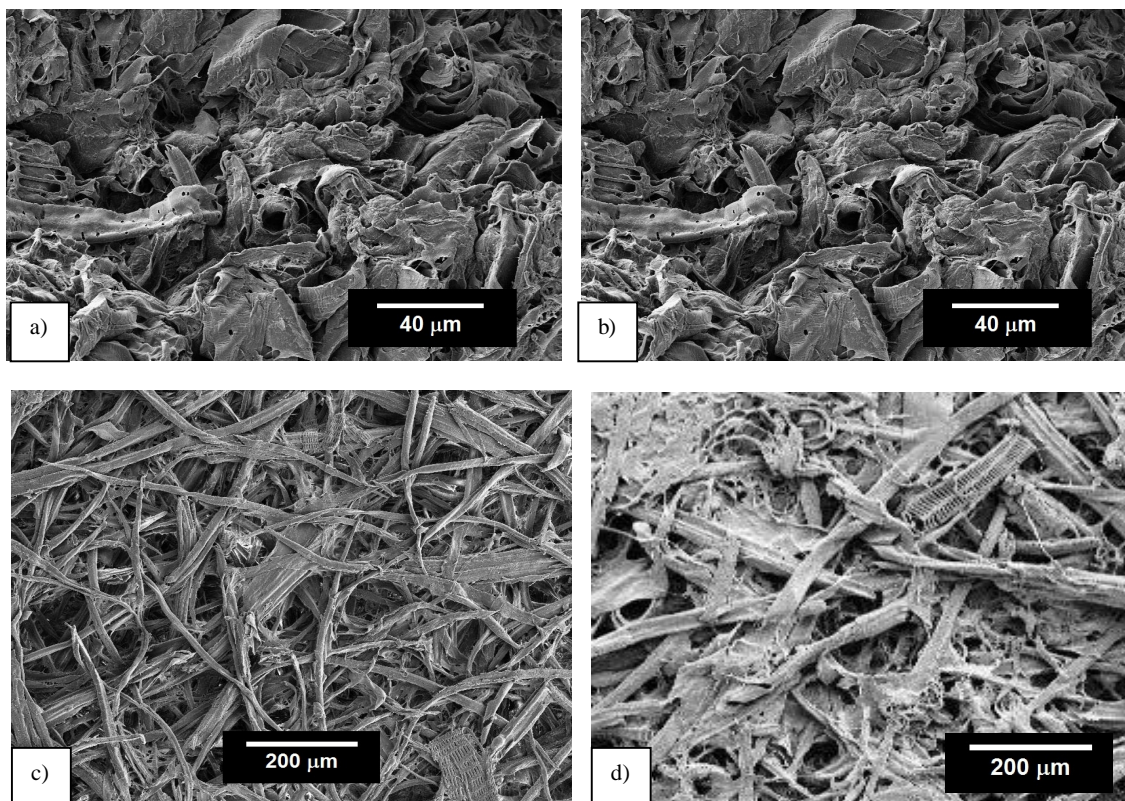


Fig. 2: Micrographs of fines and fines a) the P300/R400 fines b) the more protruded P200/R300 fines c) micro-voids in the R200 handsheet d) CGF in micro-voids.

Table 2 presents the changes in the optical and mechanical properties of the produced handsheets.

The increase in brightness as a result of adding fines is a signal of favourable equilibrium between peroxide

and fines. This suggests the predominance of surface lignin, to which peroxide is prone [28]. The findings, therefore, map surface lignin as residing the aforementioned fibrillated vessel elements and fibrils (Fig. 1). With more gaps accommodated by fines, reduction in sheet porosity and enhancement in opacity indicated in Table 2 unanimously reflects the possible improvement in inter-fiber bond.

Without fines, screened pulp (Sample M), however, offered the lowest tensile and tear indices due to poor inter-fiber bonding. Low extent of fibrillation, which resulted in the

predominance of unfibrillated structures and fibre bundles (Fig. 1), was the key factor and this reflects the rigidity of the lignin-carbohydrate matrix and the inadequacy of biomass softening allowed by the adopted conditions. The principal reason for occurrence of these fiber bundles was the inadequate reaction between alkaline peroxide and EFB that would otherwise soften the biomass and facilitate fibrillation process. One way to improve this is by applying alkaline peroxide at refining process, similar to the concept of PRC-APMP™ [12, 20], the results of which will be discussed elsewhere.

Table 2: Selected properties of handsheet.

	L	M	M250	M300	M400	P
Density (g/cm ³)	0.222	0.230	0.244	0.253	0.254	0.702
Tensile Index (Nm/g)	4.3	3.6	5.8	6.3	7.2	58.2
Tearing Index (mNm ² /g)	3.9	3.3	3.8	3.8	3.8	3.9
Folding Endurance	0.0	0.0	1.4	1.7	1.9	-
Brightness (%ISO)	44.9	44.4	46.4	46.5	46.9	33.6
Print Opacity	88.2	89.8	93.6	93.6	94.7	72.1
Tappi Opacity	81.1	83.0	89.3	90.7	90.6	73.8

As far as mechanical strengths are concerned, incorporation of fines tear index improved by 15% and this is only 0.1 point inferior to the whole (unscreened) pulp network of Sample L, suggesting that the individual fibres were more difficult to pull from the network of native fibrous mass blended with size-specific fines fractions. The better strengths as a result of enhancement in inter-fiber bonding and reduction of sheet porosity is also implied in the higher density of handsheets prepared with fines as co-generated filler, CGF. Similar behaviour was also portrayed by the highly fibrillated fines of bleached softwood kraft pulp reported by Subramanian and team [26].

Incorporation of the P200/R250 fines fraction in M250 led to 61% (relative to Sample M) enhancement of tensile index, quantitatively suggesting positive effect of fines although difficult to delineate from qualitative microscopic observations. This was also demonstrated by the initiation of folding resistance.

Blending the R200 pulp with P250/R300 and P300/R400 fines fractions improved tensile index by 75% and 100%, respectively. This demonstrates the strength enhancing effects of the fines collected from the APMP of EFB, which were evidently vessel elements. The sheared vessel elements splitting along the perforation lines resembles thin, long fibre [cf. EFB fiber: 1000 µm length, 20 µm diameter [33] possessing better flexibility and collapsibility, likely to enhance inter-fiber bonding by filling of microvoids and acting as connecting medium. This

renders better pulp consolidation, enhanced sheet cohesion and thus, an increase in tensile strength. Similar observation was also reported by Sirvio and Nurminen [34] and Rousu and Niinimäki [2] for inter-fiber bond enhancement by wood fibrillar fines and by parenchyma and epidermal cells of the monocot non-wood (common reed and wheat straw) pulp fines, respectively.

Owing to their higher surface areas, other than acting as filler, these elements also provided contact for enhanced fibre-to-fibril and fibril-to-fibril bonding and the resultant overall pulp network strength. Considering CSF of 580 to 530 is acceptable range of pulp drainability, the findings point to the high potential of the aforementioned fines as pulp network augmenting filler with acceptable practicality. The reverse effect may be imposed by higher mesh fines fractions due to the probable challenges with pulp drainability during paper product fabrication.

The resemblance of the fines materials escaping the 400-mesh screen are shown in Fig. 3. These are typical of fibrous mass produced under alkaline peroxide pulping synergizing low alkaline peroxide with extreme refining actions of 20 000 revolutions. In the network of segmented [length of 500 µm to about 1 000 µm Fig. 3(a)] fibrous mass consisting of non-spiral fibres and short fibre bundles, the pulp network is aided by the fine fibrils that rendered hairy appearance of fiber [Fig. 3(c)]. With the hooking and gripping effect of these fine-haired fibers, pulp network of 12 Nm/g tensile index could be achieved.

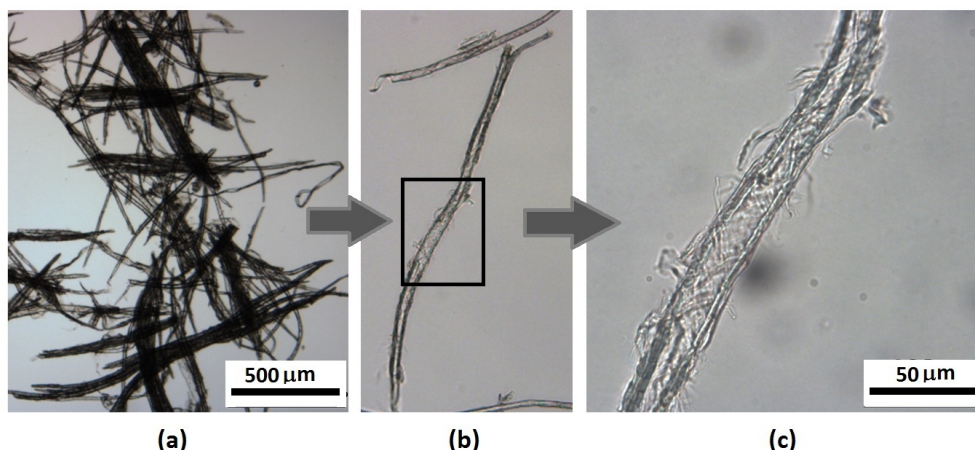


Fig. 3: Pulp produced by synergistic actions of chemical and extreme mechanical forces a) fiber amongst pulp mass b) relatively undersized fiber and c) surface of fiber showing fibrillation.

With higher alkaline peroxide level and refining revolutions of less than 20 000 revolutions (Sample P, Table 2) results portray complete elimination of fiber bundles and extensive fibrillation of segmented fibers (Fig. 4). With these severely sheared structures, a resultant pulp web of 58 Nm/g tensile index was achievable, most attributable to an extraordinary effect of intensive fibrillation of the intensely fibrillated ends dangling, which seem obscure under light microscope (circles in Fig. 4). These are the CGF encountered in the yield of pulp mass from a high-energy process. By increasing AP and refining effects to a certain extreme, pulp network, consisting of almost entirely fines, exhibit superbly improved inter-fiber bonding

and this is reflected by the highest achievable sheet density (Table 2). Perhaps due to thermal effect, sheets formed by fines network show relatively lower brightness. This is associated also to the observed sheet translucence, which in turn, reduces sheet opacity. In this regard, sheets produced from these fines may on its own suit application as chemical-free in-door shading material. As natural pulp web filler, the pronounced augmentation in the pulp network tensile strength is attributable to the nano-cross-sections of the fibril structure (Fig. 4 insert on the left), or the ‘nano-CGF’. Detailed description of this will be discussed elsewhere.



Fig. 4: Nano-CGF amidst pulp produced by synergistic actions of alkaline peroxide and high energy refining. NB: The dimensions given in the picture on the left refer to the fibers marked with white asterisks.

Within the simple alkaline peroxide and refining synergy, the CGF responsible for the augmented strength properties in samples M300 and M400 also exhibit brightness and opacity enhancement effects (Table 2). This has much connection with the better alkaline peroxide brightening as much as the discussed softening effects. This, in turn, ruled out the general drawback of fines having affinity for leached

extractives [29] but defines further the CGF affinity to materials of high level of yellowness [31] in the colourimetric scale. Whether full-length, segmented, spiral or linear, fines utilization as pulp web augmenting filler is a gateway to (pulp) waste utilization. One of the ensuing benefits is apparent from the spent liquor turbidity improvement illustrated in Fig. 5.

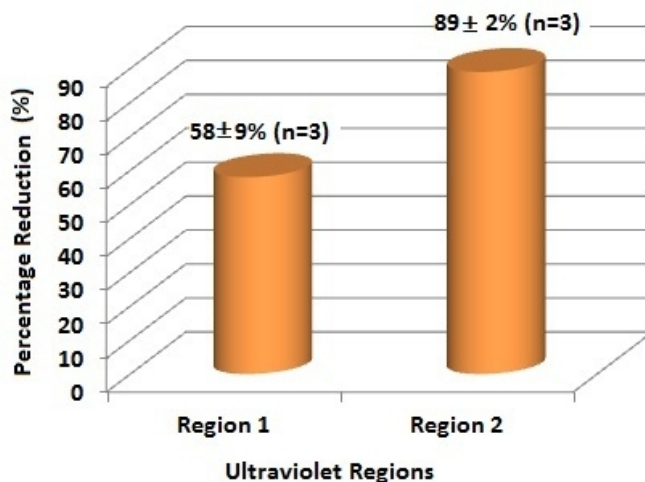


Fig. 5: Percentage reduction in turbidity of refining discharge associated to the red (Region 1) and blue (Region 2) regions in the UV spectrum.

The collection of fines for use as CGF was seen to consistently improve turbidity of spent liquor in the blue region (Region 2, Fig. 5). The lower scatter of date and higher turbidity reduction may be due to the absence of colour and hence, association with a plateau region. Meanwhile, the larger data scatter associated to the red region may be attributable to the said natural affinity of fines to the leached extractives, which are not only auxochromes, but also exhibit colour belonging to the red region (350 nm). The factor behind the inconsistency (high error value, Region 1 in Fig. 5) in turbidity improvement associated to this region, however, lies in the difficulty of controlling the proportion of free leached materials and those bound to the CGF. Averaging the effects, however, turbidity show a glaring improvement of 74% and this reflects the portion of total suspended solids effectively removed by collecting the fine elements in the refining discharge for use as CGF. This not only eases the subsequent ultra-filtration of the refining discharge for generation of process water but also promotes re-use of waste from the downstream process to add value onto other products.

CONCLUSION

Simple synergy between alkaline peroxide and the subsequent refining actions could produce useful fines in the form of full-length, segmented, spiral as well as linear vessel elements. Co-generated from the refiner discharge, these fines serve as pulp web filler, fitting into pulp web micro-voids and improving inter-fiber bonding. Besides improving pulp web strength, utilization of fines as co-generated filler (CGF) in the web of EFB alkaline peroxide pulp can also reduce discharge of organics in the semi-chemical pulping downstream process. This helps reduce pollution,

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eases treatment of pulping discharge and serves as mimicry of a zero-waste system.

ACKNOWLEDGEMENT

The work was initially performed with a seed grant from Universiti Sains Malaysia through Grant 304/PTEKIND/638088 and further scrutinized with funding from Ministry of Science, Technology and Innovation (MOSTI) of Malaysia through RU grant 1001/PTEKIND/814048.

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