# STEAM EXPLOSION PULPING OF OIL PALM EMPTY FRUIT BUNCH FIBER

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Steam explosion pulping was evaluated for oil palm empty fruit bunches fiber. The fiber morphology was observed by SEM and TEM. Results indicated that lignin was molten and the cell wall damaged after the steam explosion pulping and that the fiber was partly separated at the same time. The results of handsheet tests showed that the steam exploded pulp had a high yield (78.2%), good physical properties (especially for ring crush 8.6 N·m/g), and low effluent load (SS=910 mg/L; BOD<sub>5</sub>=3952 mg/L; COD<sub>Cr</sub>=8140 mg/L). The SEP pulp from oil palm EFB fiber was very suitable for packaging paper when combined with American OCC pulp.

Keywords: Oil palm EFB fiber; Steam explosion pulping; Morphology observation

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#### INTRODUCTION

Lignocellulosic materials are widely used in the papermaking and biomass industries. With the increasing demand, it is urgent to search for new raw material sources, such as non-wood fibers and tropical plant fibers. Among those fibers, oil palm fiber has the potential for application in the papermaking industry. Oil palm is an agricultural plant that is cultivated for its oil-producing fruit in Southeast Asian countries and Africa. Besides oil, the oil palm industry also produces many byproducts, including fiber residues such as trunks, fronds, and the empty fruit bunches (EFB). These residues have been applied in biomass and energy industries (Sulaiman et al. 2011). Several chemical pulping methods have been used for oil palm trunks and EFB (Law and Jiang 2001; Jiménez et al. 2009a, 2009b; Ferrer et al. 2011a, 2011b, 2011c; Muthurajah et al. 1977; Aziz et al. 2002; Wan Rosli et al. 1998; Rodríguez et al. 2008), but it appears that no work has been done concerning high yield pulping methods.

Steam explosion pulping (SEP) is one of the high yield pulping technologies that emerged in the 1990s (Kokta et al. 1990; Vignon et al. 1996; Kallavus and Gravitis 1999). Since 1995, South China University of Technology has done a lot of work in SEP pulping of non-wood fiber, such as wheat straw, rice straw, and bagasse (Chinese patent: CN02115318.3). Steam explosion is also a good pretreatment for biobleaching (Wang et al. 2009).

In the present study, SEP pulping of oil palm EFB fiber was compared to chemical pulping in terms of yield, pulp properties, and effluent load. The morphological changes of fiber during the explosion course were observed with scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The properties of packing paper made with SEP EFB pulp and American old corrugated container (OCC) pulp were also studied in this paper.

#### EXPERIMENTAL

#### Materials

Oil palm EFB fibers were supplied in bulk by Ecopalm Paper Sdn Bhd, Malaysia. EFB fibers were obtained from the palm oil mill after pressing on the machine. Then the fibers were air dried (moisture 10.5%) and cut to 5-10 cm length with a cutter (Keyang, SC-5, made in China).

#### Analysis of Raw Material

The EFB fiber was analyzed for holocellulose content using the Wise et al. method (1946), as well as a-cellulose, lignin, ash, hot water extraction, 1% NaOH extraction, and ethanol-benzene extractives according to TAPPI Standards T 203, T 22, T 211, T 207, T 212, and T 204, respectively (2007).

#### **Steam Explosion Pulping**

The SEP pulping was carried out on pilot scale equipment, which consisted of one steam generator, one 30 L pressurized reactor fitted with a quick-opening ball valve, and a receiver. 1.5 kg of raw materials were immersed in 2% NaOH (odp) solution for 24 h before explosion. The material was placed in the reactor manually, and the liquid-to-wood ratio was adjusted to 2:1 with distilled water. High-temperature steam was then applied to reach the predetermined pressure of 15 kg/m<sup>2</sup>. When the retention time (8 min) was reached, the ball valve was opened to release the pressure, and the exploded samples were blown out at the same time. Then, the exploded samples were passed through the refiner (KRK-NO.2500-1, Japan) twice; the first stage was with a disk gap of 0.3 mm, and the second stage was with a disk gap of 0.15 mm.

#### **SEM Observation**

Scanning electron microscopy (SEM) was used to investigate the morphology of the different type of fibers with a LEO 1530VP instrument (Germany). The specimens for the fibers were coated with gold/palladium and observed using an applied potential of 30 kV.

#### **TEM Observation**

Ultrathin films for transmission electron microscopy (JEM-2010 FEF (UHR), JEOL, Japan) were prepared by cutting from an epoxy block with different fibers at room temperature using an LKB-8800 ultratome. Samples were dyed with 1% KMnO<sub>4</sub> solution and put onto a 200 mesh copper grid covered with a carbon film. TEM observation was conducted with an accelerating voltage of 80 kV.

#### Pulp Characterization

The exploded pulp samples were characterized by beating degree and brightness according to ISO 5267.1 and ISO 3688, respectively (ISO standard, 2003).

#### Handsheet Formation and Properties

Handsheets were prepared with an ENJO-F-39.71 sheet machine. The breaking length, burst index, and ring crush of paper sheets were determined according to TAPPI methods T 494 om-96, T 494, T 403 om-97, and T 822 om-2007, respectively (TAPPI standards 2007).

#### **Determination of Pulping Effluent**

 $BOD_5$  (5 day biochemical oxygen demand),  $COD_{cr}$  (chemical oxygen demand with dichromate), and suspended solids (ss) of samples were determined according to the American Public Health Association (APHA) method (Greenberg et al. 1995). The pH value of effluent was tested with a PHS-3C pH tester (Weiyi, China).

## **RESULTS AND DISCUSSION**

#### **Chemical Composition of EFB Fiber**

The chemical composition of oil EFB fiber is shown in Table 1.

Content		EFB fiber	
Extraction (%)	Hot water	10.53	
	1% NaOH	24.23	
	Benzene-ethanol	4.07	
Hemicellulose (%)		21.36	
Cellulo	41.07		
Klason lignin (%)		16.15	
Ash (%)		3.83	

**Table 1.** Chemical Composition of Oil EFB Fiber

#### **Steam Explosion Pulping**

The properties of different pulps after passing through the refiner are listed in Table 2. It is shown in the table that the SEP pulp had a much higher yield than that of the soda-AQ pulp. The yield of SEP pulp after the second refining stage was 78.2%, while the yield of soda-AQ pulp in the reference (Wanrosli et al. 2004; Jiménez et al. 2009) was only about 39 to 41%. The higher yield indicated that most of the lignin and holocellulose were kept in the SEP pulp. Therefore, the brightness of the SEP pulp was very low. The beating degree increased very fast after the second beating with a disk gap of 0.15 mm, which may be caused by the non-fiber elements in the pulp.

Treatment	Yield on Raw materials (%)	Brightness (%ISO)	CSF (Canadian standard freeness, mL)	Beating degree (ºSR)	Reference
Steam explosion	89.2				
First refining	82.1	22.8	460	28.1	
Second refining	78.2	23.4	250	45.4	
EFB (Soda-AQ)	41				Wanrosli et al. 2004
EFB (Soda-AQ)	39			47.5-	Jiménez et al. 2009a

#### Table 2. Properties of EFB Pulps with Different Treatments

First beating at a 0.3 mm distance between two discs;

Second beating at a 0.15 mm distance between two discs.

#### **SEM Observations**





**Fig. 1.** Morphological change of fiber after steam explosion treatment (A before explosion; B, C, D after explosion)

The results of SEM observation of fibers are shown in Fig. 1. The surface of fiber before explosion (Fig.1-A) was even and covered with very few folds. The surface of fiber after explosion (Fig.1-B) became uneven and covered with more folds and holes, as well as experiencing fiber separation. Most of the fiber bundles were partly individualized. Some fragments (Fig.1-C, D) were also observed on the separated fiber at high magnification. Microfibrils were also observed on the fiber surface (Fig.1-C). Fragments observed in these images should be the melted lignin and primary cell wall peeling from the main body of the fiber (Fig.1-D). Lignin melts at a high temperature and high pressure. After instant cooling and decompression, lignin and fibers may explode from the cell wall of the fibers. The work of Soltes (1983) also demonstrated that the glass transition temperature of the lignin after explosion is lower than in the original material, and this makes it possible to separate. The physical effect is bigger than the chemical effect on lignin during steam explosion. The steam explosion could make the lignin soften (Vignon et al. 1996). But only a little of lignin dissolved and degradated during the explosion. Most of the lignin still remained in the pulp. Fibers were separated partly at the same time. When the exploded samples passed through the refiner, the fibers were separated.

#### **TEM Observations**

The results of TEM observation of fibers are shown in Fig. 2.



**Fig. 2.** Morphology of cell wall with TEM (A before explosion; B, C, D after explosion)

In Fig. 2, it is shown that the cell wall (Fig. 2-A) of the fiber before explosion was thin and unbroken and the lumen of the fiber cell was very small, but the fiber cell wall was damaged (Fig. 2-B, C, D) after steam explosion. At the same time, the lumen of the fiber cell had expanded greatly because of the pressure difference between inside and outside of the fiber cell. Some of the lumen was broken during explosion, such as in Fig. 2-C. Some lignin fragments were observed in the cell corner (Fig. 2-D). The presence of high amounts of lignin in these corner features is consistent with an observation of a much darker color (Xu et al. 2005).

#### **Properties of Handsheets**

The properties of the handsheets from SEP pulp and OCC with different ratios are shown in Table 3. From this Table, it is clear that the ring crush was very high for the 100% SEP pulp handsheet. The ring crush reached 8.6 N·m/g. The fact that most of the lignin remained in the pulp would lead to a high ring crush value for paper. This is a distinct characteristic of SEP pulp. However, the breaking length and burst index of this handsheet was lower than that the handsheets made with OCC. With an increasing OCC content, the breaking length and burst index of the paper became better and the ring crush decreased. These effects may be caused by the fiber length of the corresponding pulp; the fiber length of SEP pulp from oil palm EFB fiber is shorter than OCC pulp. The bond provided by SEP pulp was therefore weaker than that of OCC. The Chinese standard for corrugated medium grade A packaging paper are as follows: base weight 112  $\pm$  6 g/m<sup>2</sup>, ring crush > 7.1 N·m/g, and breaking length > 4.3 km. SEP pulp from oil palm EFB fiber combined with long fiber pulp could achieve this standard and therefore be suitable for packaging paper.

SEP pulp:OCC	Base Weight (g/m <sup>2</sup> )	Breaking Length (Km)	Ring Crush (N⋅m/g)	Burst Index (kPa.m <sup>2</sup> /g)	
4:1	115.1	4.4	8.2	2.2	
3:1	115.4	4.6	7.8	2.4	
2:1	115.6	4.8	7.5	2.5	
1:1	115.7	5.4	7.2	2.7	
1:0	115.3	3.2	8.6	2.2	

SEP pulp: exploded samples after second beating; OCC: American old magazine container after hydrapulper with 37 °SR .

#### **Characteristics of SEP Pulping Effluent**

The characteristics of the pulping effluent from SEP and Soda-AQ pulping of oil palm EFB fiber are listed in Table 4. The pH value of SEP pulping was 6.5. But pH value of the traditional soda-AQ pulping was 13.5. The SS, BOD<sub>5</sub>, and COD<sub>cr</sub> of SEP pulp were much lower than the corresponding values for the Soda-AQ pulp. This indicated that the SEP pulping is a more environmentally friendly pulping method.

# **Table 4.** Comparison of the Pollution of Waste Water from SEP and Soda-AQPulp Processes

Pulping Method	pH Value	SS(mg/L)	BOD <sub>5</sub> (mg/L)	COD <sub>Cr</sub> (mg/L)
SEP	6.5	910	3952	8140
Soda-AQ	13.5	2107	15485	48050

Soda-AQ pulping conditions: NaOH 18% (odp), AQ 0.05% (odp), wood to liquid ratio 1:4, yield 41.8%, time to time 60 min, time at time 120 min.

# CONCLUSIONS

- 1. SEP pulp from oil palm EFB fiber has a higher yield (78.2%) than soda-AQ chemical pulping. Most of the lignin and holocellulose were kept in the pulp.
- 2. SEM and TEM observations showed that lignin was molten and that the cell wall was damaged during the steam explosion pulping. Fibers were separated and microfibers were exposed after discharge.
- 3. SEP pulp from oil palm EFB fiber combined with long fiber pulp is very suitable for packaging paper.
- 4. The effluent loadings of SEP pulping were much less than those of chemical pulping Soda-AQ pulping. The pH value of SEP pulping effluent is nearly neutral.

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# **REFERENCES CITED**

- Aziz, A. A., Husin, M., and Mokhtar, A. (2002). "Preparation of cellulose from oil palm empty fruit bunches via ethanol digestion: Effect of acid and alkali catalysts," *Journal* of Oil Palm Research 14(1), 9-14.
- Chinese patent CN02115318.3. "Middle or low pressure steam explosion pulping of hardwood."
- Ferrer, A., Vega, A., Rodríguez, A., Ligero, P., and Jiménez, L. (2011a). "Milox fractionation of empty fruit bunches from *Elaeis guineensis*," *Bioresource Technology* 102, 9755-9762.
- Ferrer, A., Rosal, A., Valls, C., Roncero, B., and Rodríguez, A. (2011b). "Modeling hydrogen peroxide bleaching of soda pulp from oil-palm empty fruit bunches," *BioResources* 6(2), 1298-1307.
- Ferrer, A., Vega, A., Ligero, P., and Rodríguez, A. (2011c). "Pulping of empty fruit bunches (EFB) from the palm oil industry by formic acid," *BioResources* 6(4), 4284-4301.
- Garcia-Jaldon., C., Dupeyre, D., and Vignon, M. R. (1998). "Fibers from semi-retted hemp bundles by steam explosion treatment," *Biomass Bioenergy* 14(3), 251-260.
- International Organisation for Standardization Documentation and Information (ISO).

(2003). ISO Standards Collection. Paper, Board and Pulps, ISO, Geneva.

- Jiménez, L., Serrano, L., Rodríguez, A., and Sánchez, R. (2009a). "Soda-anthraquinone pulping of palm oil empty fruit bunches and beating of the resulting pulp," *Bioresource Technology* 100, 1262-1267.
- Jiménez, L., Serrano, L., Rodríguez, A., and Ferrer, A. (2009b). "TCF bleaching of sodaanthraquinone and diethanolamina pulp from oil palm empty fruit bunches," *Bioresource Technology* 100, 1478-1481.
- Kallavus, U., and Gravitis, J. (1999). "A comparative investigation of the ultrastructure of steam exploded wood with light, scanning and transmission electron microscopy," *Holzforschung* 49, 182-188.
- Kokta, B. V., Ahmed, A., Garceau, J. J., Carrason, F., Zhai, D. M., and Huang, G. Q. (1990). "Steam explosion pulping (SEP) of spruce and aspen. Optimization of the process," *Annual Meeting - Technical Section, Canadian Pulp Paper Assoc.*, 91-105.
- Law, K. N., and Jiang, X. (2001). "Comparative papermaking properties of oil-palm empty fruit bunch," *TAPPI Journal* 84(1), 95.
- Muthurajah, R. N., and Peh, T. B. (1977). "Manufacture of paper pulps from oil palm empty bunch waste," *Malaysian International Symp. Palm Oil Process*, 147-157.
- Rodríguez, A., Serrano, L., Moral, A., Pérez, A., and Jiménez, L. (2008). "Use of highboiling point organic solvents for pulping oil palm empty fruit bunches," *Bioresource Technology* 99, 1743-1749.
- Soltes, E. J. (1983). "Thermochemical routes to chemicals, fuels and energy from forestry and agricultural wastes," *Biomass Utilization*, Cote, W. A. (ed.), Plenum Press, New York.
- Sulaiman, F., Abdullah, N., Gerhauser, H., and Shariff, A. (2011). "An outlook of Malaysian energy, oil palm industry and its utilization of wastes as useful resources," *Biomass and Bioenergy* 35, 3775-3786.
- TAPPI Methods (2007). TAPPI Press, Atlanta, Georgia, USA.
- Vignon, M. R., Dupeyre, D., and Garcia-Jaldon, C. (1996). "Morphological characterization of steam-exploded hemp fibers and their utilization in polypropylene-based composites," *Bioresource Technol.* 20, 203-215.
- Wan Rosli, W. D., Law, K. N., Zainuddin, Z., and Asro, R. (2004). "Effect of pulping variables on the characteristics of oil-palm frond-fiber," *Bioresource Technology* 93, 233-240.
- Wise, L. E., Murphy, M., and D'Addieco, A. A. (1946). "Chlorite holocellulose, its fractionation and bearing on summative wood analysis and on studies on the hemicelluloses," *Paper Trade J.* 122(2), 35-43.
- Wang, K., Jiang, J. X., Xu, F., Sun, R. C. (2009). "Influence of steaming pressure on steam explosion pretreatment of Lespedeza stalks (*Lespedeza crytobotrya*): Part 1. Characteristics of degraded cellulose," *Polymer Degrad. Stability* 94, 1379-1388.
- Xu, F., Zhong, X. C., Sun, R. C., and Gwynn, L. L. J. (2005). "Lignin distribution and infrastructure of *Salix psammophila*," *Transactions of China Pulp and Paper* 20(1), 6-9,

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