Neutral Sulfite Semi-Chemical Pulping of Moso-Bamboo

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Abstract

This research investigates the potential of neutral sulfite semi-chemical (NSSC) pulping method using moso-bamboo (*Phyllostachys pubescens*) as raw material. NSSC treatment took place in laboratory cooking system. Cooking liquor contained a mixture of sodium sulfite (Na_2SO_3) and sodium carbonate (Na_2CO_3) with a weight ratio of 2 to 1. Total chemical charge was 12% based on oven-dried weight of bamboo chips. Liquor to solid ratio was 4 to 1. Two-stage refining was used to fabricate fibers from cooked chips. The first stage was at 0.2 MPa and 120°C and, the second stage was at normal pressure. A control experiment was involved under the same conditions with no charge of chemicals. NSSC pulping of bamboo resulted in providing a low brightness pulp but high pulp properties (tensile and tear indices) compared to the control pulp. NSSC method could help to reduce the contents of un-separated fiber bundles and shives. NSSC method provided a high aspect ratio of fibers compared to the control. After fibers development and fibrillation during refining, NSSC pulp contained a lot of ribbon-like fibrils which then contributed to a good fiber-to-fiber contact. In addition, the internal bonding between fiber-to-fiber of NSSC pulp may be increased reasonably by a significant amount of parenchyma cells appearing as fines (100 mesh pass) fraction, which were revealed by SEM images evaluation.

Keywords : neutral sulfite semi-chemical, thermo-mechanical pulping, bamboo, aspect ratio, fibrillation

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1. Introduction

Neutral sulfite semi-chemical (NSSC) pulping is a two-stage process in which wood chips are previously treated under mild chemical conditions at elevated temperature to soften, and the treatment partially removes the lignocellulosic interfiber bonding material. This is followed by a mechanical refining to complete the fiber separation. The cooking liquor in the early development of the process contained sodium sulfite and was buffered with sodium carbonate to reduce corrosion, and thus the process became known as the NSSC process. The history of this process should be dated back to the year 1870's when mechanical pulping processes were quickly followed by chemical pulping processes. In 1874, Mitscherlich proposed treating chips with a solution of sulfur dioxide or of bisulfite followed by grinding or rubbing to make pulp. Six years later in 1880, Cross described the advantage of cooking chips in a neutral or slightly alkaline solution of sodium sulfite. For years of lacking adequate attention, researchers at the Forest Products Laboratory (USA) later found the advantage to add a small amount of an alkaline agent to the sodium sulfite solution to control the pH for reducing corrosion on the equipment. They also found that cooking to the fiber liberation point was not necessary, but partially pulped chips at 60–75% yield could be defibered mechanically without damage to the fibers. In 1925, the first NSSC mill started operations for manufacturing corrugating medium¹⁾.

The semi-chemical wood pulping method yields more as compared to the wholly chemical wood pulping method. In the former process there is approximately 50% lignin of retention. Both these factors make semi-chemical wood pulping more commercially attractive than chemical wood pulping. Semi-chemical wood pulp finds utility in important manufacturing processes, such as in the manufacturing of high-quality corrugated medium. In NSSC pulping, the sulfonation mainly introduced at middle lamella lignin causes a partial dissolution of lignin so that the resistance of fibers are weakened for the subsequent mechanical defibration. NSSC pulp remains a significant amount of lignin content (more than 15%) and therefore, is mostly used for unbleached products where good strength and stiffness are particularly important²⁾. To date, this is the most widespread semi-chemical pulping process, and the pulp from hardwood chips is mainly used for corrugating medium, but the trend now is to use it as partial kraft pulp replacement in liner-board and bag grades^{3,4)}.

The NSSC pulping process is often integrated into a kraft pulp mill to facilitate chemical recovery by a so-called cross-recovery, where the sulfite spent liquor is

processed together with the kraft black liquor. The sulfite spent liquor then provides the necessary makeup (Na, S) for the kraft process. However, with the greatly improving recovery efficiency of modern kraft mills, the NSSC makeup is no longer needed. In addition, high-yield kraft pulping develops as a serious alternative to NSSC pulping. Semi-chemical pulp is still an important product category, but it accounts for 4% of all fresh pulp fiber materials⁵⁾. To the recent interest of biorefinery, it is noted that the sulfite spent liquor of a neutral sulfite semi-chemical pulping process contains a considerable amount of lignocelluloses and can be used for producing value-added products^{6,7)}.

The conditions of semi-chemical pulping vary depending to the raw material as well as the bleached or unbleached pulp is desired⁸⁾. Research on NSSC pulping of three eucalypt species showed substantially high yields of pulp with an acceptable strength for corrugating medium⁹⁾. A process suitable to manufacturing the pulp for corrugating medium from New England hardwoods had been applied under following conditions: sodium sulfite ratio to sodium bicarbonate: 5 to 1, chemicals on the basis of wood: 14%, temperature: 170°C, pressure: 0.7 MPa, time at maximum temperature: 2–3 hours, and the pulp yield was about 70%¹⁾. Pulping hybrid poplar (*Populus deltoides*) wood with varied NSSC conditions to obtain pulp yield between 72–80% showed that temperature presented the most important effects, and that sodium carbonate effects on the yield were important only at low sulfite charges or high temperatures, whereas sodium sulfite charge had the highest influence on energy consumption for refining³⁾.

Recently, as the rising demand of using non-wood fiber sources, NSSC pulping of wood alternative materials also takes interests. Study of NSSC pulping for bagasse at 165°C with different sodium sulfite charges showed that the influence of cooking time was greater than that of chemical charge on the pulp yield and strength¹⁰⁾, and this conclusion agreed with results of research on NSSC pulping of eucalypt wood⁴⁾. Pulping sunflower stalk for producing corrugated medium grade pulp revealed that the NSSC delignification rate was quite slow even at high chemical charges and cooking temperatures, and that the pulping process for obtaining good fibers should be done under relatively intensive conditions with low yields¹¹⁾. Anatomical structure of a non-wood material such as silvergrass with a little amount of fibers on unit mass has anatomical characteristics of thin cell wall structure of fibrous elements and a high content of parenchyma cells, and they would make the strength of NSSC non-wood pulp poorer than that of wood pulp¹²⁾. In addition, as important previous studies related to

NSSC pulping, studies on fabricating thermomechanical non-wood pulp from oil palm empty fruit bunch and bagasse by pressurized refining process using a single disc refiner have been reported^{13,14,15}.

Bamboo is also expected to be a feasible material for producing the NSSC pulp. Bamboo is widely distributed in subtropical and tropical areas, and about 80% of world bamboo forest are in the Asia and Pacific regions. Especially, China and India together account for approximately 70% of the bamboo forest in Asia¹⁶. The amount of bamboo resources in Asia regions is significant, and thus it can be utilized for mass production of pulp and paper. In fact, bamboo has been utilized for producing a chemical pulp in Chinese papermaking industry.

To the best of our knowledge, research on pulping bamboo with NSSC method is limited, and thus the objective of this research is to provide necessary information for using NSSC method on pulping moso-bamboo (*Phyllostachys pubescens*) in terms of producing corrugating medium grade pulp.

2. Material and methods

2.1 Material

In this research, 2-year-old moso-bamboo (*Phyllostachys pubescens*) harvested in Hitachi-Omiya city, Ibaraki was used as raw material. After harvesting from the forest, the green bamboo was dried naturally to moisture content of 10% with nodes removal, and then, all the internodes (stems) were cut into chips with 5 mm in thickness and 20 mm in length.

2.2 Chemical compositions analysis

The contents of acid-insoluble lignin, acid-soluble lignin, extractives, and ash were determined using TAPPI Test Methods T 222 om-15, T 204 cm-07, and T 211 om-02, respectively. The amount of glucose, xylose, and others sugars in the acid hydrolysate of materials and obtained pulp fibers in each fraction were determined by ion chromatography. The fibers in each fraction were subjected to nitrobenzene oxidation (NBO) to understand the chemical characteristics of lignin in the pulp fractions, according to a modified NBO method¹⁷.

2.3 NSSC cooking and refining

NSSC cooking treatment was done by using an oil-bath. Cooking liquor contained a mixture of sodium sulfite (Na_2SO_3) and sodium carbonate (Na_2CO_3) with a weight ratio of 2 to 1. Total chemical charge was 12% based on oven-dried weight of bamboo chips. Liquor to solid ratio was 4 to 1. Cooking temperature was set constantly at 160°C, cooking time was 80 and 120 min. After the cooking, without removal of cooking liquor and washing materials, the cooked materials were subjected to the next refining step.

A laboratory pressurized single disc refiner (Model BRP45-300SS manufactured by Kumagai Riki Kogyo Co., LTD., Nerima, Tokyo) with a refiner plate (Type J, 305 mm in diameter), which connected to the pressure gauge and temperature sensor module in order to control and observe conditions, was used to refine the cooked chips. The first-stage refining was done at 0.2 MPa pressure and 120°C, and the second-stage refining was done at atmospheric pressure. A gap of the disc was 1.0 mm for the first refining and 0.1 mm for the second refining. Control experiment was taken under the same condition with no chemicals in the cooking liquor.

2.4 Pulp properties

Obtained pulp was fractionated into five fractions using a Bauer McNett Fiber Classifier No.2593 with wire opening 1180, 600, 300 and 150 μm (mesh number 14, 28, 48, and 100, respectively). Fiber length and width distribution were determined using an auto fiber-length tester (Alva F5800 Fiber Tester, Alva Instruments, Jinan, China). The fiber aspect ratio was calculated by the following equation:

$$\text{Fiber aspect ratio} = \text{Fiber length } (\mu\text{m}) / \text{Fiber width } (\mu\text{m})$$

Scanning electron microscopy (SEM) equipment (SU1510 Hitachi) was used to take SEM photo of the fibers in order to identify the shape and surface of fiber characteristic of each fraction.

To characterize pulp strength, pulp was firstly treated with a PFI mill at 10,000 revolutions according to ISO 5264-2:2011 standard. Forming handsheets for physical tests of the pulp followed the ISO 5269-1:2005 standard. The physical properties, as well as the tensile and tear indices of the handsheets were determined according to ISO 5270:2012, 1924-2:2008, 1974:2012 standards, respectively.

3. Results and discussion

3.1 Chemical components of moso-bamboo

The glucan (cellulose) and lignin contents of 2-year-old moso-bamboo were determined as 36.2% and 28.8%, respectively (Table 1). Comparing with woods, this lignin content of bamboo was similar to that of softwoods (in the range of 26–34%), but the cellulose content is lower than both hardwoods and softwoods (in the range of 38–49%)¹⁸.

3.2 Fiber classification and pulp properties

Table 2 shows the response of NSSC treatment on pulp properties in comparison with the control experiment. Effects of cooking liquor containing sodium sulfite and sodium carbonate were obvious on the cooked yield as well as the CSF value and physical strength of the pulp. The NSSC treatment provided much better pulp

Table 1 Chemical compositions of moso-bamboo raw material

Components	Content (%)
Acid-insoluble lignin	26.5 ± 0.2
Acid-soluble lignin	2.30 ± 0.04
Subtotal	28.8 ± 0.2
Glucan	36.2 ± 0.4 ^a
Xylan	16.0 ± 0.3 ^b
Other sugars	1.43 ± 0.02 ^c
Ash	1.2 ± 0.2
Subtotal	54.8 ± 0.9
Unknown	16.4

^a As polymer of glucose.^b As polymer of xylose.^c As polymer of arabinose, galactose and mannose.**Table 2** Pulp properties

Sample	Yield ^a (%)	Brightness (%ISO)	CSF ^b (ml)	Density (g/cm ³)	Tensile index (Nm/g)	Tear index (mNm ² /g)
NSSC (1) ^c	71.3	26.6	130	0.46	17.8	5.90
NSSC (2) ^d	87.6	27.4	150	0.44	14.5	5.05
Control	96.3	31.6	210	0.39	7.20	3.10

^a Yield after cooking^b PFI at 10000 revolutions^c NSSC (1): cooking for 120 min^d NSSC (2): cooking for 80 min

strength, but the brightness of the pulp was lower than the control experiment. Comparing between two NSSC treatments, pulp cooked for 120 min provided a high pulp strength with a slightly low pulp brightness. For the next pulp characteristics analysis, we use NSSC (1) pulp cooked for 120 min, as the representation of NSSC treatment.

Fiber fractionation results showed that NSSC pulp had smaller ratios of 14–28 mesh, 28–48 mesh, and 48–100 mesh three fractions than the control pulp, but it contained a larger amount of the 100 mesh pass fraction (Fig. 1). Furthermore, fiber aspect ratios of NSSC pulp were higher than those of control pulp (The fraction which did not pass 14 mesh was immeasurable) in all fractions (Fig. 2). Fiber aspect ratio as an important parameter of fiber has been proved to have positive effects on mechanical properties, especially the tear strength of pulp¹⁹⁾. NSSC pulp had the fiber aspect ratio as 53.5 on average, and it was comparable to those of hardwood pulp which were reported to be around 40 to 60²⁰⁾.

In addition, it is well known that the fraction of wood mechanical pulp which passes through 100 mesh contains practically fines and ribbon fragments of the cell wall S1 layer. The fines as flakes of the middle lamella or ribbon fragments from the cell wall should be rich in lignin content, and these should significantly affect properties related to light absorption coefficient of the pulp. Mechanical pulp fines are believed to have a light absorption coefficient which is about three to four-times high as that of the long fiber fraction²¹⁾. In this case, the high content of fines in NSSC pulp may take responsibility for the overall pulp brightness. However, the elements of fines fraction still need further assessment. The contents of glucan, xylan and lignin in NSSC pulp fractions were shown in Table 3. There was a downward trend from the contents of lignin and glucan when the size of fiber diminished. However, the syringyl aldehyde molar ratio to vanillin (S/V molar ratio) by NBO of NSSC pulp showed an upward tendency when the size of fiber become shorter.

Fig. 1 Fibers fractionation

Fig. 2 Fiber aspect ratio of each fractions

Note : The fraction which did not pass 14 mesh was immeasurable.

Table 3 Lignin and carbohydrate contents and syringyl aldehyde molar ratio to vanillin (S/V molar ratio) by NBO of NSSC pulp

Content (%)	Pulp fraction				
	>1180 μm (14 mesh remain)	600–1180 μm (14–28 mesh)	300–600 μm (28–48 mesh)	150–300 μm (48–100 mesh)	<150 μm (100 mesh pass)
Acid-insoluble lignin	26.5 ± 0.5	24.1 ± 1.4	24.4 ± 0.3	26.0 ± 0.5	22.5 ± 0.9
Acid-soluble lignin	1.53 ± 0.01	2.01 ± 0.01	1.76 ± 0.06	2.01 ± 0.03	2.75 ± 0.02
Subtotal	28.0 ± 0.5	26.1 ± 1.4	26.2 ± 0.4	28.0 ± 0.5	25.3 ± 0.9
Glucan	51.5 ± 3.1	52.2 ± 0.9	49.2 ± 0.5	47.7 ± 1.5	44.3 ± 0.2
Xylan	20.6 ± 1.2	20.7 ± 0.2	20.5 ± 0.1	22.1 ± 0.6	25.4 ± 0.5
Arabinan	1.10 ± 0.12	1.21 ± 0.01	1.27 ± 0.06	1.25 ± 0.02	1.30 ± 0.05
Subtotal	73.2 ± 4.4	74.1 ± 1.1	71.0 ± 0.7	71.1 ± 2.1	71.0 ± 0.8
Unknown	-	-	2.8 ± 1.1	0.9 ± 2.6	3.7 ± 1.7
S/V molar ratio	0.75	0.83	0.83	0.88	1.87

3.3 SEM observation

Evaluating the pulp fractions by SEM images can reveal interesting information. The 14 mesh remain fraction from the both kinds of pulp (the NSSC and control pulps) contained mostly un-separated fiber bundles (Fig. 3). Fibers from control pulp were still stuck together

er in a block-like shape including parenchyma cells. It indicated that this fraction was not suitable for making paper and should be treated as “screen rejects”.

The 14–28 mesh fraction showed better isolated pulp fibers in the case of NSSC pulp, but still much fiber bundles in the case of control pulps as presented in Fig. 4.

Fig. 3 SEM images from the 14 mesh remain fraction of NSSC pulp (a, b) and control pulp (c, d)

Note : Un-separated fiber bundles (white arrows) and parenchyma cells (black arrows) are clearly shown.

Fig. 4 SEM images from 14–28 mesh fraction of NSSC pulp (a, b) and control pulp (c, d)

Note : NSSC pulp shows a smooth surface of individual fiber by exposing S1 layer of the cell wall (b).

This fraction of NSSC pulp contained a lot of whole fibers with smooth surfaces of S1 layer of the cell wall.

Fig. 5 shows the 28–48 mesh fractions of both pulps. It revealed there were shorter and thinner fibers but still whole fibers. These fibers should remain their intrinsic stiffness, and they should be unable to create well fiber-to-fiber connection. Furthermore, there were some fibers

of NSSC pulp that have been stripped of the cell wall exposing S2 layer similarly in a previous paper²²⁾.

As shown in Fig. 6, fibrils were well observed only from 48–100 mesh fraction, and they were cell wall segments in shape of ribbon-like or flake-like. The NSSC pulp were much fibrillated comparing with control pulp. This kind of fibrillation should contribute to improving

Fig. 5 SEM images from 28–48 mesh fraction of NSSC pulp (a, b) and control pulp (c, d)
Note : This fraction of both pulps contains almost whole fibers, exposing of S2 layer of the cell wall observable only in NSSC pulp (b).

Fig. 6 SEM images from 48–100 mesh fraction of NSSC pulp (a, b) and control pulp (c, d)
Note : Fibrillations (white arrows) in shape of ribbon-like or flake-like segments appear more likely in NSSC pulp.

Fig. 7 SEM images from 100 mesh pass fraction of NSSC pulp (a, b) and control pulp (c, d)
 Note : Parenchyma cell are concentrated in NSSC pulp (black arrows).

pulp strength by providing a high fiber-to-fiber contact.

In the 100 mesh pass fraction, parenchyma cells were concentrated in case of NSSC pulp.

Meanwhile, for the control pulp there were a lot of short fibers and their segments (Fig. 7). It is known that parenchyma cells with a suitable ratio have a positive effect on paper tensile strength²³⁾. Therefore, this evidence can explain for the high tensile strength of NSSC pulp compared with that of the control pulp.

4. Conclusions

NSSC pulping of moso-bamboo by NSSC resulted in providing a low brightness pulp but high pulp properties (tensile and tear indices) compared to the control pulp. NSSC method helped to reduce the amount of un-separated fiber bundles and shives of pulp. NSSC method provides a higher aspect ratio of pulp compared to the control. After fibers development and fibrillation during refining, NSSC pulp contained a lot of ribbon-like fibrils which then contributed to a good fiber-to-fiber contact. In addition, the internal bonding between fiber to fiber of NSSC pulp may be increased reasonably by a significant amount of parenchyma cells appearing in the fine fraction, which were revealed by SEM images evaluation.

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