Environmentally Friendly Bleaching on Bamboo (*Neosinocalamus*) Kraft Pulp Cooked by Displacement Digester System

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The bleaching of pulp prepared by a displacement digester system (DDS) for displacement cooking of bamboo (*Neosinocalamus*) was established by comparing the results from elemental chlorine-free (ECF) bleaching and totally chlorine-free (TCF) bleaching. This process produced the optimal performance of obtained pulp *via* O-D_0-E_{OP}-D_1 bleaching, where the Kappa number of DDS pulp was 18 to 22, and the Kappa number of oxygen delignified pulp was 10 to 12. The brightness of the obtained pulp was over 86% ISO, the yield was up to 46%, and the viscosity was approximately 800 $mL \cdot g^{-1}$. In contrast, O-Q-P_1-P_2 bleaching was advantageous for pulp with kappa number less than 5 after oxygen delignification. The brightness of obtained pulp was up to 81% ISO, the yield was over 40%, and the viscosity was about 650 $mL \cdot g^{-1}$. In TCF bleaching, the water consumption was 20 $m^3 \cdot t^{-1}$, the chemical oxygen demand (COD) content was 30 $kg \cdot t^{-1}$, and the absorbable organic halogen (AOX) content was zero. The water consumption of ECF bleaching was 4 times that of TCF bleaching, while the COD content was 16 $kg \cdot t^{-1}$ and the AOX content was 2 $kg \cdot t^{-1}$.

Keywords: Bamboo; Neosinocalamus; DDS displacement cooking; TCF bleaching; ECF bleaching

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INTRODUCTION

Due to global trends and environmental pressures for cleaner bleaching processes, elemental chlorine-free (ECF) bleaching and totally chlorine-free (TCF) bleaching are being used more often to reduce the production of chlorinated organic compounds during pulp manufacturing (Jahan *et al*. 2013; Nie *et al*. 2015). Although the substitution of chlorine dioxide (ClO_2) for elemental chlorine in the bleaching process reduces the production of absorbable organic halogens (AOX) substantially, AOX are still generated during the ECF bleaching process (Nakamata *et al*. 2003, 2004, 2010). While the TCF bleaching does not produce AOX, the properties of TCF pulp generally is not as good as that of ECF pulp. Bamboo, a non-wood fiber, is widely distributed throughout China and is a viable alternative source for the paper industry (Moradbak *et al*. 2015). The average fiber length of bamboo is between 1.5 mm and 2.0 mm, and the width is 15 μm to 18 μm. The cellulose content of bamboo is high (40% to 60%) (Li *et al*. 2012b). The pulping of bamboo is mainly done using continuous kraft pulping to a Kappa number of 16 to 18.

Depending on the bleaching requirements, bamboo pulps are often bleached with ECF processes, such as an initial chlorine dioxide stage (D_0), an oxygen and hydrogen peroxide reinforced alkali extraction stage (E_{OP}), a next chlorine dioxide stage (D), and an oxygen reinforced hydrogen peroxide bleaching stage (P_O), with a sequence of D_0-E_{OP}-D or using a chlorine dioxide stage and chelating stage (DQ) and hydrogen peroxide stage and oxygen stage (PO). Alternatively, TCF sequences can be selected with a chelating stage (Q), such as Q-(E_{OP})-Q-(PO), and Q-(PO) (Chang *et al*. 2013). When the D_0ED_1 sequence was applied to unbleached soda-AQ pulp from moso bamboo, the resulting bleached pulp had 85.3% ISO brightness (Lee *et al*. 2016).

The displacement digester system (DDS) is an energy-efficient and environmentally-friendly pulping method. Compared with traditional cooking techniques, it delivers low kappa number, high yield, good strength, and stable quality characteristic while the stems can be collected (Bianchini *et al.* 2007). Therefore, the development of DDS technology of *Neosinocalamus* not only helps to alleviate the shortage of raw material in China, but also is accordance with the trend of green, environment-friendliness and low carbon production in paper industry. Displacement digester system (DDS) cooking can produce bamboo pulp of a relatively lower Kappa number, between 10 and 14 in comparison to conventional bamboo kraft pulp, with a yield from 40% to 45% and a viscosity is less than 1100 $mL \cdot g^{-1}$ (Li *et al*. 2012a). Considering the practical benefits for pulping industry, pulps with a lower kappa number, can be bleached more easily, and the consumption of bleaching agents and the pollutions caused by bleaching can be reduced. However, there is a common understanding that lower kappa number will definitely lower the pulp yield, and the production cost per ton of pulp can increase sharply. Consider a bamboo pulp mill with an annual output of 180,000 tons, for example. If the unbleached pulp Kappa number declines from 22 to 14, the yield of bleached pulp may decrease by 6% or 7%, and the average per-ton cost of bleached pulp will increase. As a result, the pulp mill will lose 12,000 ton of the bamboo pulp per year. Therefore, it is important to properly select the endpoint of cooking at different Kappa numbers. This practice is not only conducive to improving pulp performance, but it also provides the most economical technical solutions for pulp mills.

This study applies the technologies of more environmentally friendly bleaching of DDS-cooked *Neosinocalamus* pulp. The correlation between DDS cooking and both DCF and TCF bleaching of *Neosinocalamus* was established. The ECF sequence $OD_0E_{OP}D_1$ and the TCF sequence OQP_1P_2 were investigated to bleach the *Neosinocalamus* pulp. Kappa number, brightness, yield, and viscosity were measured on these pulps to identify the most appropriate bleaching conditions. Then, the pilot of the pulping and bleaching conditions was verified in a bamboo mill in China.

EXPERIMENTAL

Materials

Bamboo (*Neosinocalamus*) culms from 3-year-old bamboo plants were provided by Yongfeng pulp and paper company in Sichuan province of China. Before cooking, the culms were washed with water to remove residues and were converted into chips. After screening, they were cut into pieces with the size of approximately 50 mm × 10 mm. When the size of the chips was homogenized, they were stored in polyethylene bags to achieve constant moisture. After air drying, the moisture of the raw material was 9.5%.

Methods

DDS cooking process

The cooking process was performed in a laboratory-made digester (Fig. 1) that was designed to fulfill the requirements of displacement cooking and was manufactured by Shaanxi University of Science and Technology, Xi'an, China. The cooking system consisted of a digester, a chemical liquid spray tank, a controller, and a cooking liquor circulating pump. All the items of equipment were made from stainless steel. The cooking temperature and circulation speed were controlled accordingly throughout the process. The limitations for the equipment were included a maximum pressure of 5.0 MPa, a maximum temperature of 220 °C, and a maximum volume of 1.5 L.

Fig. 1. The DDS cooking digester

The DDS cooking process was as follows: For initial cooking (IC), the pre-preg liquid (mixture of NaOH and Na_2S) was heated to 80 °C, and 150 g of oven-dried bamboo was placed into the cooking digester and treated with pre-preg liquid. The mixture was then heated to 80 °C for 30 min. Second, in initial middle cooking (IMC), the displacement liquid (NaOH and Na_2S), which was heated to 110 °C, was pumped into the cooking digester and cooked for 30 min at 110 °C. Third, in final middle cooking (FMC), the displacement liquid (NaOH and Na_2S) was heated to 150 °C and was pumped into the cooking digester and cooked for 30 min at 150 °C. Fourth, in heating, the bamboo and displacement liquid were heated from 155 °C to 165 °C in 30 min as shown in Table 1.

Table 1. Performance of *Neosinocalamus* DDS Cooking Pulp

Sample	Alkali charge (%)			Sulfidity (%)	Maximum temp (°C)	Retention time (min)	Kappa number	Screened yield (%)	Viscosity 1 (mL·g^{-1})	Brightness 1 (% ISO)
	IC	IMC	FMC							
1	1.4	4.1	8.2	20	165	118	9.4	40.8	877	32.8
2	0.5	4	10	21	156	90	12.3	44.8	1083	32.5
3	2.5	4	8	21	157	89	15.2	45.1	1102	31.5
4	2.5	4.2	8	21	155	78	16.6	45.3	1055	31.2
5	2.5	4	8	20	155	64	18.7	46.5	1113	30.9
6	2.4	7.6	6	15	159	73	19.8	49.8	1125	30.2
7	2.5	4.1	6	16	155	90	22.4	50.3	1213	29.3
8	1.5	5.2	6.1	15	155	116	25.3	51.1	1201	31.3

Fifth, in retention, the bamboo and displacement liquid were kept at a temperature of 155 °C to 165 °C for 90 min to 120 min. When the IMC liquid was charged, the IC liquid was drained into the spray tank. When the FMC liquid was charged, the IMC liquid was also drained into the spray tank. The spray tank had two layers. IC liquid and IMC liquid, which were drained from digester, were placed in the outer layer. New IMC liquid and new MFC liquid were placed in the inner layer.

The samples were chosen to produce pulp with a Kappa number of approximately 10 to 24. The parameters and characterization of the pulp obtained are given in Table 1.

Oxygen delignification

For the oxidative delignification process, 50 g of oven-dried pulp and the required chemicals (magnesium sulfate and NaOH) were placed into a GSHA-2 high pressure reactor (manufactured by Xi'an HEB Biotechnology Co., Ltd. Xi'an, China). The established optimum conditions for oxygen delignification (O) were 10% pulp consistency, 0.9 MPa, 100 °C for 60 min, 1% to 2% NaOH, 1% magnesium sulfate, and a final pH of 11.0 to 11.5. At the end of the process, the pulp was washed and then put into polyethylene bags to obtain a constant moisture.

Bleaching process

The ECF bleaching process was conducted in stages, using chlorine dioxide (D_0), oxygen and hydrogen peroxide reinforced alkali extraction stage (E_{OP}), and chlorine dioxide (D_1). The bleaching conditions are listed in Table 2. On the other hand, TCF bleaching process was comprising of QP_1P_2, where Q refers to the EDTA (ethylenediaminetetraacetic acid) stage, P_1 refers to the following hydrogen peroxide stage, and P_2 refers to the final hydrogen peroxide stage. After each step, the washed and dried pulp was analyzed for Kappa number, viscosity, and brightness according to TAPPI standard methods as mentioned earlier.

Table 2. Bleaching Process

Sample	Kappa number after O	ECF						TCF					
		D_0		E_{OP}		D_1		Q		P_1		P_2	
		Active chlorine	Time	NaOH	Time	Active chlorine	Time	EDTA	Time	H_2O_2	Time	H_2O_2	Time
		(%)	(min)	(%)	(min)	(%)	(min)	(%)	(min)	(%)	(min)	(%)	(min)
1	4.2	2	30	1	90	1	120	0.4	60	1	90	2	120
2	5.8	2.5	30	1	90	1.5	120	0.4	60	1	90	2.5	120
3	7.0	2.5	60	1	90	1.5	140	0.4	60	1	90	2.5	120
4	7.7	2.5	60	1	90	1.5	140	0.4	60	1.5	90	2.5	120
5	9.2	3	60	2	90	2	160	0.4	60	1.5	90	2.5	120
6	9.9	3	60	2	90	2	160	0.4	60	1.5	90	3	120
7	11.3	3	90	2	90	2	180	0.4	60	1.5	90	3.5	120
8	13.7	3.5	90	2	90	2.5	180	0.4	60	1.5	90	3.5	120

Design formulae are given as Eqs. 1 to 4 (Shilin *et al*. 2007),

$$Kappa\ number\ reduction\ (\%) = (Kappa_1 - Kappa_2) / Kappa_1 \times 100 \quad (1)$$

$$Reduction\ rate\ of\ pulp\ viscosity\ (\%) = (V_1 - V_2) / V_1 \times 100 \quad (2)$$

$$Delignification = Kappa\ number\ reduction / Reduction\ rate\ of\ pulp\ viscosity \quad (3)$$

$$Rate\ of\ brightness\ change\ (\%) = (B_2 - B_1) / B_1 \times 100 \quad (4)$$

where $Kappa_1$ is the Kappa number of unbleached pulp, $Kappa_2$ is the Kappa number of bleached pulp, V_1 is the viscosity (mL·g^{-1}) of unbleached pulp, V_2 is the viscosity (mL·g^{-1}) of bleached pulp, B_1 is the brightness (% ISO) of unbleached pulp, and B_2 is the brightness (% ISO) of bleached pulp.

Pulp Analysis

The Kappa number, viscosity, and brightness were tested based on T236 om-99, T230 om-13, and T452 om-08, respectively. Pulp yield was measured by drying the wet pulp and determining the mass. Total drying pulp mass was calculated and pulp yield was expressed as a percentage of the original raw material.

Pilot

A bamboo paper mill in China used the process conditions of this study, which comprised DDS replacement cooking, oxygen delignification, TCF bleaching, and ECF bleaching.

RESULTS AND DISCUSSION

Oxygen delignification is an important technology to produce bleached kraft pulp (Chong *et al*. 2013). Most kraft pulp mills use oxygen delignification before bleaching to reduce the generation of chlorinated organic compounds in the bleach plant effluent (Okan *et al*. 2013).

In recent years, new oxygen delignification technology has developed rapidly (Jafari *et al*. 2014; Rosales-Calderon *et al*. 2016; Wilke *et al*. 2016). When using identical oxygen delignification process conditions, pulps of different properties (Kappa number, yield, and viscosity) would have different bleaching results. In this study, the effect of oxygen delignification technology on eight unbleached *Neosinocalamus* pulp samples with Kappa number from 9.4 to 25.3 were studied.

The properties of the pulp after the oxygen delignification reaction are shown in Table 3. The data showed that the Kappa number and viscosity of the obtained pulp decreased, while the brightness increased under the same technical conditions of oxygen delignification. With increasing Kappa number of unbleached pulp, the kappa number reduction decreased.

The delignification was greatest (4.7 and 4.6) when the Kappa number of unbleached pulp was 16.6 and 18.7, respectively. With further increasing of the initial Kappa number, the oxygen delignification gradually decreased. This demonstrated that the oxygen delignification was noticeably affected.

Table 3. Effect of Oxygen Delignification on DDS Cooking Pulp

Condition	1	2	3	4	5	6	7	8
$Kappa_1$	9.4	12.3	15.2	16.6	18.7	19.8	22.4	25.3
$Kappa_2$	4.2	5.8	7	7.7	9.2	9.9	11.3	13.7
Delignification	3.1	3.8	4.3	4.7	4.6	4.2	3.3	2.7
Kappa Number Reduction	55	52.5	53.7	53.4	51	50.1	49.7	46
$Viscosity_2$ ($mL \cdot g^{-1}$)	720	932	964	936	989	992	1030	998
Reduction Rate of Pulp Viscosity	17.9	13.9	12.5	11.3	11.1	11.8	15.1	16.9
$Brightness_2$ (% ISO)	42.5	41.3	41.2	40.7	40.2	39.8	39.3	38.3
Rate Change of Brightness	19.4	26	26.8	29.3	28.9	31.7	23.3	22.2
Yield (%)	39.9	43.2	43.5	43.6	44.7	47.5	48.0	48.9
Bleaching Loss (%)	2.3	3.4	3.6	3.7	3.8	4.6	4.4	4.3

As shown in Table 3, the Kappa number decreased considerably, and the delignification extent, *i.e.*, the lignin rate of removal, was between 46% and 55%. Overall, the delignification extent was approximately 50%. Notably, when the initial Kappa number was 25.3, the delignification extent was 46%. When the initial Kappa number was in the range of 12 to 22, oxygen delignification remained at a similar degree, with approximately 50% of the lignin removed (Li *et al*. 2016). When the Kappa number reached 24, the oxygen delignification efficiency decreased because of the higher residual lignin content in the pulp. This indicated that effective and efficient oxygen delignification was achieved when the Kappa number of unbleached pulp was 12 to 22. However, to determine the optimal conditions of oxygen delignification, it is necessary to consider the pulp viscosity, brightness, and yield.

With increasing unbleached pulp Kappa number, the viscosity of the pulp gradually decreased during oxygen delignification. The change of viscosity was between 11.1% and 17.9%. When the unbleached pulp Kappa number was between 16 and 20, the viscosity of the pulp decreased after oxygen delignification. Greater viscosity generally corresponds to better strength. The brightness increased after oxygen delignification, while the change of brightness first increased and then decreased. Although pulps with higher initial kappa number retain higher pulp yields (based on original raw material), which also underwent greater bleaching loss after oxygen delignification.

In summary, the unbleached pulp Kappa number for DDS cooking varied between 9.4 and 25.3. Kappa number less than 12 with yield less than 45% was correct only for unbleached pulps (Table 1). To OD pulp with pulp yield 45%, the kappa number of unbleached pulp was 18.7 and above. When the unbleached pulp Kappa number was greater than 22, although the pulp yield was greater, the pulp viscosity decreased considerably, and the change rate of brightness was not obvious. If it were to reach final bleached pulp, the subsequent bleaching chemical dosage should be increased. Overall, it was better to choose the Kappa number between 18 and 20 for DDS cooking. After oxygen delignification, the pulp had excellent comprehensive performance and delignification selectivity. The Kappa number decreased, viscosity decreased, brightness was improved, and the bleached pulp yield was greater.

With a Kappa number of 23 for bamboo, the pulp still contained approximately 3% lignin (Runge *et al*. 2012). The oxygen stage was restricted to approximately 50% delignification to avoid severe carbohydrate degradation. Therefore, to achieve the full benefit of oxygen delignification, with respect to economic as well as environmental aspects, it was necessary to continue the lignin removal to as great an extent as possible in order to improve the selectivity of this process.

ECF Bleaching

The bleaching sequence was conducted on kraft pulp after undergoing the oxygen delignification mentioned above. The aim of the bamboo ECF bleaching was to acquire bleached pulp with a brightness degree above 85% ISO. The ECF started with different dosages of bleaching agents and bleaching duration times at the same temperature for each stage. The optimum conditions of bleaching and the bleaching results are summarized in Fig. 2. The results showed that the brightness of bleached pulp was greater than 85% ISO.

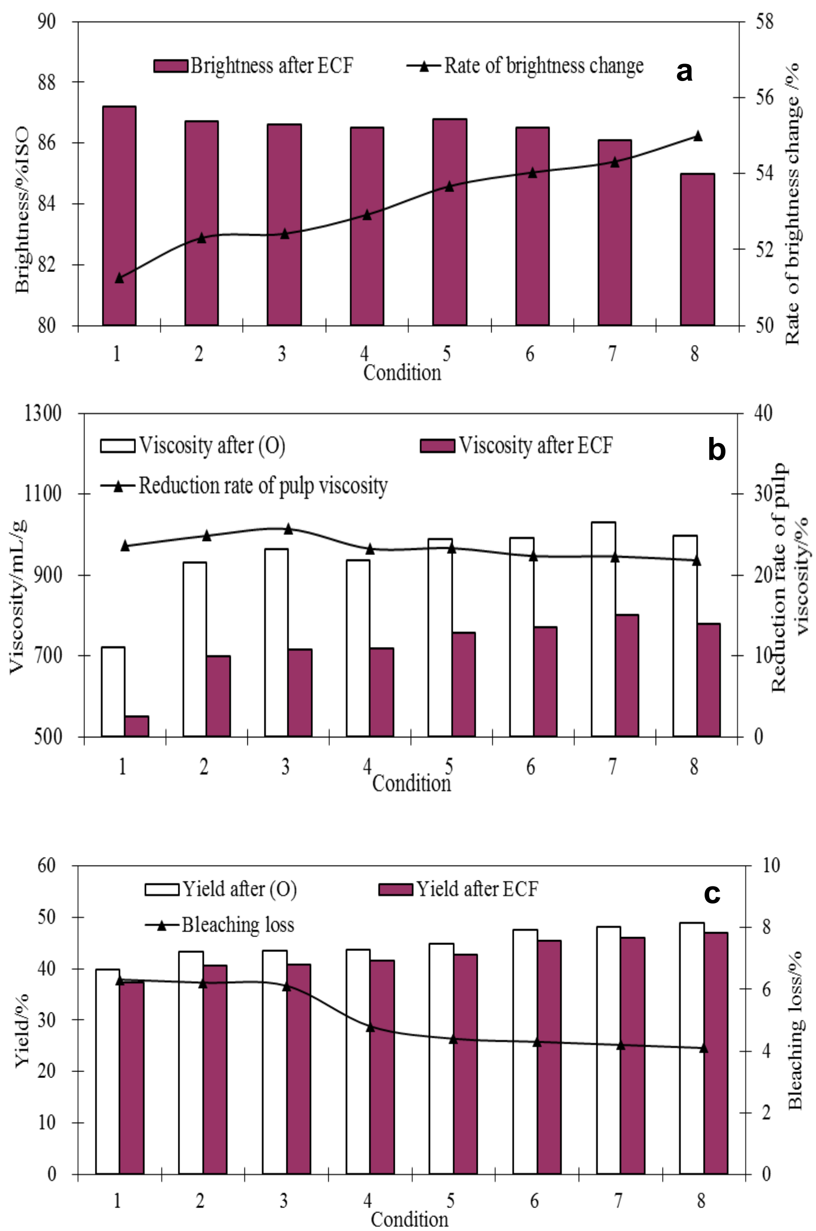

Fig. 2. Change in brightness (a), viscosity (b), and yield (c) of ECF bleached pulp

As shown in Fig. 2b, the viscosity of bleached pulp increased as the Kappa number of unbleached pulp increased, but the collective reduction in pulp viscosity showed little difference, ranging from 21.8% to 25.7%. However, it was clear from Fig. 2b that the percent loss in viscosity decreased as the Kappa number of unbleached pulp increased. As shown in Fig. 2c, an increase in the Kappa number of unbleached pulp was accompanied by a continuous increase in the yield of bleached pulp. Production cost is explicitly affected by the yield, and with greater Kappa numbers of unbleached pulp come greater bleached pulp yields. The results also showed that the bleaching loss decreased as the Kappa number of unbleached pulp increased.

The experimental results obtained in this bleaching process indicated that at the Kappa number of oxygen delignification of 4.2, the yield, viscosity, bleaching time, and consumption of active chlorine of obtained pulp were 37.4%, 550 mL/g, 240 min, and 3%, respectively. The yield and viscosity of obtained pulp increased at Kappa numbers of oxygen delignification of 5.8 to 9.9, where the obtained yield, viscosity, bleaching time, and consumption of active chlorine were 40.6% to 45.4%, 700 mL/g to 770 mL/g, 240 min to 310 min, and 4% to 5%, respectively. When the kappa number of oxygen delignification pulp was 13.7 (greater than 11.3), the viscosity of bleached pulp decreased compared to Sample 7, and the yield slightly increased, but bleaching time and consumption of active chlorine increased greatly. The obtained yield, viscosity, bleaching time, and consumption of active chlorine were 46% to 46.9%, 800 mL/g to 780 mL/g, 360 min, and 5% to 6%, respectively.

Consequently, it is reasonable to keep the Kappa number of oxygen delignification pulp between 9.88 and 11.27, for which the active chlorine consumption was 5%, the bleaching time was 310 min to 360 min, and the yield and viscosity were high. Therefore, the optimal performance of obtained pulp with high yield and superior viscosity by ECF bleaching was observed when the Kappa number of unbleached pulp was 18 to 20. This was the most suitable condition for commercial production and the least costly to manufacture, considering the consumption of chemical reagents and the characteristics of the obtained pulp.

Comparison between ECF Bleaching and TCF Bleaching

Figure 3 shows that the kappa number of unbleached pulps was greatly affecting the performance of the subsequent bleaching processes. Although different bleaching processes can attain a certain brightness, ECF bleaching was more suitable for achieving pulp with higher brightness, yield, and viscosity. The improvement of brightness was limited by TCF bleaching and its low yield. The cooking system for the pulp must be capable of cooking to at least 18 to 22 kappa for the ECF case and less than 5 kappa for the TCF case. These values were in agreement with the literature (Steffes and Germgård 1995). Due to the low selectivity of the bleaching reactions, there was little impact on the lignin structure when hydrogen peroxide was used, which led to lower final brightness at a higher cost than that of ECF bleaching (Rencoret et al. 2013; Perrin *et al*. 2015).

The causes for the low adoption of TCF bleaching have been diverse, including the poorer efficiency of the bleaching reactions when combinations of oxygen and hydrogen peroxide were used, which can lead to lower final brightness at a higher cost.

The flexibility to cook as low as possible for the TCF case is important when bleaching to full brightness. For this reason, an extended batch cooking system (SuperBatch) has been used, which is capable of operating to as low as 10 kappa.

TCF bleaching hardly affected the lignin structure, and important amounts of β−O−4′ linkages still were present in the TCF-bleached pulps

The actual production showed that the cost of per ton of TCF pulp was higher by 200 yuan to 300 yuan than that of ECF pulp in China.

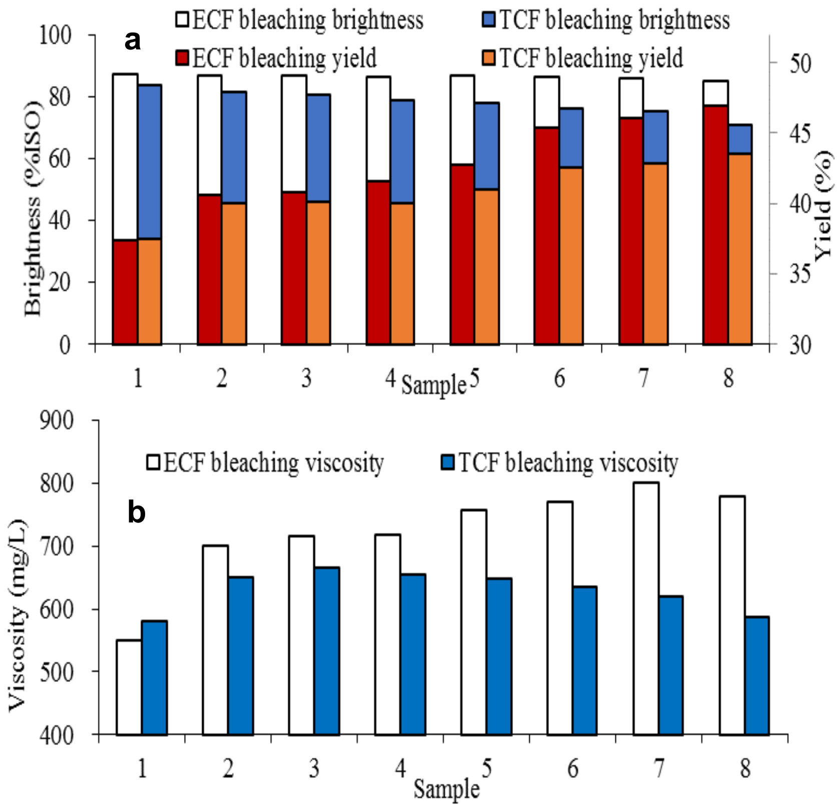

Fig. 3. Comparison between brightness (a), yield (a), and viscosity (b) of ECF bleaching and TCF bleaching

Table 4. Comparison between Environmental Factors of ECF and TCF Bleaching

Environmental Factor	TCF Bleaching	ECF Bleaching
Water consumption ($m^3 \cdot t^{-1}$)	20	80
COD ($kg \cdot t^{-1}$)	30	16
AOX ($kg \cdot t^{-1}$)	0	2

However, ECF bleaching has a disadvantage with regards to environmental pollution. While TCF bleaching would not produce absorbable organic halogens, they were generated during the ECF bleaching process (Nakamata *et al*. 2010; Nie *et al*. 2015). A bamboo paper mill in China used the process conditions of this study, which comprised DDS replacement cooking, oxygen delignification, TCF bleaching, and ECF bleaching. When TCF bleaching was implemented, the brightness was 82% ISO, and the yield was 41%. When ECF bleaching was implemented, the brightness was 86% ISO, and the yield was 46%. As shown in Table 4, for TCF bleaching, the water consumption was 20 $m^3 \cdot t^{-1}$, the chemical oxygen demand (COD) content was 30 $kg \cdot t^{-1}$, and the AOX content was zero. In contrast, the water consumption of ECF bleaching was 4 times that of TCF bleaching, the COD content was 16 $kg \cdot t^{-1}$, and the AOX content was 2 $kg \cdot t^{-1}$. The COD content of

TCF bleaching was greater than that of ECF bleaching; this may have been due to lower TCF bleaching yields because of cellulose degradation.

Therefore, by using TCF bleaching and ECF bleaching after DDS cooking, mill operators also could achieve a high brightness. These results indicated that ECF bleaching had advantages in pulp brightness, yield, and viscosity, while limited improvement in brightness can be obtained by using TCF bleaching with pulp of Kappa number less than 5 after oxygen delignification.

CONCLUSIONS

1. Both ECF bleaching and TCF bleaching were suitable to bleach the *Neosinocalamus* pulp after DDS cooking. However, the optimal performance could be achieved by ECF bleaching or TCF bleaching, with the condition of choosing the endpoint of cooking at different Kappa numbers. This process produced the optimal performance of obtained pulp by ECF bleaching, where the Kappa number of DDS displacement cooking pulp was 18 to 22, and the Kappa number of oxygen delignification pulp was 10 to 12. The brightness of obtained pulp was over 86% ISO, the yield was up to 46%, and the viscosity was approximately 800 $mL \cdot g^{-1}$. In contrast, TCF bleaching gave more favorable results when the pulp had been cooked to a kappa number of less than 5 after oxygen delignification. The brightness of obtained pulp was up to 81% ISO, the yield was over 40%, and the viscosity was about 650 $mL \cdot g^{-1}$.
2. The water consumption of TCF bleaching was 20 $m^3 \cdot t^{-1}$, while the COD content was 30 $kg \cdot t^{-1}$, and the AOX content was zero. The water consumption of ECF bleaching was 4 times that of TCF bleaching, while the COD content was 16 $kg \cdot t^{-1}$, and the AOX content was 2 $kg \cdot t^{-1}$.

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