

Study on the Nitric Acid Pulping, Delignification Course, and Waste-Liquid Recovery

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The pulping of birch wood using dilute aqueous nitric acid solution under atmospheric pressure was studied. The pulping conditions, delignification reaction course, and waste-liquid recovery were studied in detail. The optimum cooking conditions were 9.2% nitric acid for 4.3 h at 85 °C. The pulp yield at these conditions was 51.1%, the lignin content was 5.1%, and the brightness was 50%. The delignification course during the pulping involved two phases, namely, bulk and residual phases. The bulk phase was 0 to 1.5 h long, and the delignification level was 92.6% of the total dissolved lignin. The residual phase was 1.5 h to 4.5 h long, and the delignification level reached about 7.4% of the total dissolved lignin. The waste liquid could no longer be used after six rounds of recycling. The contents of nitrogen and organic matter in the organic fertilizer prepared using the final-round of recycled waste liquid were in line with the indicators for the preparation of organic fertilizer.

Keywords: Nitric acid pulping; Delignification course; Waste recycling

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INTRODUCTION

Nitric acid pulping is an established method that is popular for the production of pulp from woody and non-woody resources. Compared with other methods that employ formic acid or acetic acid, the nitric acid pulping uses low concentrations of nitric acid (about 2% to 10%). By contrast, the pulp obtained with formic acid (Jahan *et al.* 2006; Sarwar 2006; Jahan *et al.* 2007) or acetic acid (Mondal *et al.* 2004; Pan and Sano 2005; Pan and Sano 1999) generally requires a concentration not less than 80%, and sometimes it even requires the addition of a sulfuric acid as a catalyst to facilitate cooking at atmospheric pressure. Therefore, the nitric acid pulping is advantageous relative to the other types of pulping in terms of the amount of acid used. Synla and Yohodo (1942a) reported the nitric acid concentration, reaction temperature, and reaction time required for the hydrolysis of cellulose to an average degree of polymerization of ~300, and that the effect of temperature was greater than that of the other two parameters. These researchers also investigated the pulping of beech wood chips (dimensions of 1.5 × 1.5 cm; thickness, 0.2 mm to 0.4 mm) in the presence of a nitric acid concentration of 3% to 4% at a temperature of 95 °C, time of 1 h, and a liquid-solid ratio of 7.5 to 15; a nitric acid concentration of 2% to 3%, at a temperature of 110 °C, a time of 1 h, and a liquid-solid ratio of 10 to 15 (all pre-treatment conditions). The treatment was carried out after the pulp had been filtered and washed, and the post-treatment conditions included an NaOH concentration of 2%, a temperature of 98 °C, a time of 1 h, and a liquid-solid ratio of 10.

Under conditions of low temperature (at 95 °C), the pulp yield was 35% to 41%, the lignin content was zero to 1%, the pentosan content was 8% to 12%, and the average degree of cellulose polymerization was 900 to 1200. Under conditions of high temperature (about 110 °C), the pulp yield was 33% to 37%, the lignin content was zero to 0.06%, the pentosan content was 2% to 6%, and the average degree of cellulose polymerization was 880 to 950 (Synla and Yohodo 1942b). Benson and Walton (1940) studied the pulping of *Pseudotsuga taxifolia* chips under atmospheric pressure with dilute nitric acid and reported that the optimal pulp yield, which was with 6 h of treatment, was low compared with those from hemlock and spruce; however, the quality of the pulp was very good.

Nitric acid pulping is commonly used for non-wood materials. For instance, reed stalk chips were digested in nitric acid and extracted with ammonium hydroxide. Compared with kraft and mono-sulfite pulping, the nitric acid pulping can remove all white membranous materials lining the inner walls of the reed stalk. Nitric acid pulping at the same permanganate number as kraft pulping showed a higher pulp yield but a lower polymerization degree and brightness value (Ba and Toshio 1977). Other non-wood materials, such as rice straw (El-Taraboulsi *et al.* 1983a), bagasse (El-Taraboulsi *et al.* 1983b; Abou-State *et al.* 2010), *Dendrocalamus strictus* (Dhawan 1980), Palmyra palm fruit fibers (Sridach 2010), and cotton have also been investigated (Abou-State and El-Megeid 1972; Abou-State *et al.* 1977).

The reaction between lignin and nitric acid depends largely on the temperature and acid concentration. Low nitric acid concentrations cause lignin nitration and phenolic ether bond hydrolysis, whereas higher nitric acid concentrations and temperatures favor the nitration and oxidation of lignin to carboxylated products (El-Taraboulsi *et al.* 1983b). These nitration and oxidation products can be dissolved by alkali solutions. Ba *et al.* (1982, 1984) investigated the behavior of lignin using guaiacylglycerol- β -guaiacyl ether (GG) and veratrylglycerol- β -guaiacyl ether (VG) as dimeric model compounds containing phenolic and non-phenolic lignin during nitric acid pulping at 65 °C, respectively. These authors found a large amount of α -O-(2-methoxyphenyl) glyceraldehyde in the products, indicating that the side-chain displacement reaction was the most important step in the delignifying reaction. In addition, the identification of lignin nitration products, such as 4-nitroguaiacol and 4,6-dinitroguaiacol, indicated that lignin undergoes nitration. The identification of 2-methoxy-*p*-benzoquinone, as well as vanillic, glycolic, glyceric, tartronic, and veratric acids, revealed that oxidation also occurred. Interestingly, both of the aforementioned reactions and the hydrolysis of β -aryl ether bonds occurred at 50 °C.

Birch is widely distributed, predominantly within the cold temperate and temperate regions. Birch is found throughout Europe, especially in northern Europe (Beck *et al.* 2016). In Russia, birch ranks third in stand volume, accounting for 16.8% of the total stocks (Yao *et al.* 2018). Birch is also an important type of forest vegetation in China. According to the data of the Eighth Forest Resource Inventory, the forest area possessing the dominant species of birch vegetation in China is 1.423×10^7 hm², which accounts for 6.84% of the total forest area (Du *et al.* 2019). Therefore, birch provides the most commercially important source of hardwood (Beck *et al.* 2016), and it is also the main raw material within the chemical pulping industry (Bergelin and Holmbom 2008).

However, in previously published nitric acid pulping studies, little attention has been given to the pulping delignification course and the pulping waste liquid recovery routes. Therefore, birch was used in the present study as the raw material to analyze the delignification course and waste liquid recovery during nitric acid pulping.

EXPERIMENTAL

Raw Materials

Birch (*Betula platyphylla* Suk.) wood was obtained from the Liangshui experimental forest of Northeast Forestry University. The sample was air-dried, cut into wood chips (dimensions 30 × 30 × 2 mm), and used for pulping. A chemical component analysis of the sample indicated that it was comprised of 83.8% holocellulose, 22.8% lignin (the sum of Klason lignin and acid-soluble lignin), 15.6% pentosan, 0.5% ash, and 3.5% ethanol-benzene extractives. These values were based on dried wood chips. Nitric acid (specific gravity, 1.42) and ammonium hydroxide (conceded pass (CP) grade) were used as the pulping agents.

Nitric Acid Pulping of Birch at Atmospheric Pressure

Pulping process

Pulping was carried out in a round-bottom flask fitted with a reflux condenser. A fixed amount of wood chips (100 g) and various concentrations (6.7 to 13.5%) of an aqueous nitric acid solution were combined and subjected to cooking in a water bath at atmospheric pressure using various temperatures. After cooking, the residue was neutralized by washing with water. The pulp was then passed through a 0.40-mm slit flat screen to remove the unprocessed fragments. Thereafter, the pulp was analyzed.

Delignification course

Fifty grams of dried wood chips were placed in the above reactor operating at a temperature of 85 °C in the presence of nitric acid concentration of 9.2% and a liquid/birch ratio of 10:1. After different cooking times, the residue was washed, filtered, air-dried, and weighed. Afterwards, the resulting residue was pulverized, and the obtained powder (20 to 60 mesh) was used for subsequent analyses. A portion of the powder was used to determine the moisture content and sample yield. Another portion of the powder was used for benzene-alcohol extraction to prepare an extract-free sample, and the content of benzene alcohol extract was also determined. The contents of Klason lignin and acid-soluble lignin were quantified using the extract-free wood powder. The total lignin content in the pulp was the sum of Klason lignin and acid-soluble lignin. The carbohydrate content was calculated by subtracting the sum of the lignin and the benzene-ethanol extract from the corresponding total amount.

$$\text{Residual lignin (\%)} = (\text{Klason lignin} + \text{acid-soluble lignin}) (\%) \times \text{residue yield (\%)} \times \text{original wood chip weight.} \quad (1)$$

$$\text{Delignification (\%)} \text{ of raw material} = (1 - (\text{residual lignin/lignin content in original wood})) \times 100\%. \quad (2)$$

Evaluation of the Chemical Properties of the Samples

The starting material and end product were characterized according to the guidelines of Chinese national standards. Holocellulose was determined in accordance with GB/T 2677.10 (1995), Klason lignin was determined in accordance with GB/T 2677.8 (1994), acid-soluble lignin was determined in accordance with GB/T 10337 (2008), pentosan was determined in accordance with GB/T 2677.9 (1994), benzene-ethanol extract was determined in accordance with GB/T 2677.6 (1994), and ash content was determined in accordance with GB/T 2677.3 (1993). The content of carbohydrate was calculated by subtracting the sum of lignin and the benzene-ethanol extract contents from the corresponding total amount of pulp. The brightness of the pulp was measured using a brightness tester (Model YQ-2-48A, Qingtong Instrument Co. Ltd., Hangzhou, China). The size of the fibers was measured using an optical microscope (Model XSP-7CP, Optical Instrument Co. Ltd., Shanghai, China).

Recovery and Utilization of Cooking Waste Liquor

Recycling of waste liquor

Since the cooking waste liquor contained unreacted nitric acid, a fixed amount of fresh nitric acid was added to recycle the waste liquor. The possible number of recycling cycles was monitored from the pulp yield and the lignin content.

Preparation of fertilizer

Fertilizer was prepared using the waste liquid from the final-round of recycling. The waste liquor was neutralized using ammonia, and the excess moisture was evaporated. The remaining viscous material was dried in an oven at 50 °C. The content of total nitrogen in the fertilizer was determined using an automatic nitrogen instrument (Qianjian Instrument Co. Ltd., China). The content of organic matter was determined using the potassium dichromate solution through the volumetric method NY-525 (2012).

RESULTS AND DISCUSSION

Pulping Process

Effect of the cooking temperature on the properties of pulp

The reaction temperature is one of the most sensitive factors affecting the chemical reaction rate. Similarly, the cooking temperature is an important parameter in the pulping process. The properties of pulp at various cooking temperatures are listed in Table 1. With an increase in the cooking temperature, the pulp yield first increased and then decreased, and the lignin content decreased gradually. At cooking temperatures lower than 75 °C, birch was not sufficiently delignified due to the small amount of removed lignin, and the pulp yield was extremely low. Increasing the pulping temperature accelerated the dissolution of lignin and cellulose (Ogunsile and Quintana 2010). At cooking temperatures ranging from 75 °C to 85 °C, the pulp yield improved to 51.1%, and the lignin content decreased to 4.3%. At cooking temperatures higher than 85 °C, the pulp yield decreased. These results indicated that the extent of carbohydrate degradation was greater than that of delignification when the cooking temperature was too high. Therefore, the optimal cooking temperature for the nitric acid pulping of birch was 85 °C.

Table 1. Effect of the Cooking Temperature on the Properties of Pulp

Cooking temperature (°C)	65	75	85	95
Pulp Yield (%)	4.4	5.4	51.1	47.4
Lignin Content (%)	14.2	10.6	4.3	2.8
ISO Brightness (%)	28.5	30.2	49.3	55.6
Nitric acid concentration - 9.2%; Cooking time - 4.3 h; Liquid/birch ratio - 10:1.				

Effects of the nitric acid concentration and cooking time on the properties of pulp

When the cooking temperature was constant, the chemical reaction rate was determined by the nitric acid concentration. Therefore, at this stage, the concentration of nitric acid determined the extent of lignin removal and carbohydrate degradation. In addition, increasing the cooking time ensured that the cooking liquor completely permeated the material, thus increasing the efficiency of lignin removal. However, the prolonged cooking time increased the degradation of carbohydrates, which inadvertently affected the pulp yield and fiber strength. The effects of the nitric acid concentration and cooking time on the properties of pulp are listed in Table 2. With an increase in the nitric acid concentration, the pulp yield increased first and then decreased. Compared with nitric acid concentrations of 6.7% and 13.5%, the pulp obtained with 9.2% nitric acid had a higher yield. This was attributed to the low concentration of nitric acid, which contributed to the poor solubility of lignin. Though the cooking time was extended to 5.3 h, the pulp yield was still not significantly increased. However, the concentration of nitric acid was too high, resulting in too many activated molecules per unit volume, as well as the increase in reaction rate with carbohydrates and lignin.

Table 2. Effects of the Nitric Acid Concentration and the Cooking Time on the Properties of Pulp

Nitric Acid Process (%)	Pulp Parameter	Cooking Time (h)				
		3.3	3.8	4.3	4.8	5.3
13.5	Pulp Yield (%)	48.4	48.4	49.6	46.5	43.2
	Lignin Content (%)	5.5	5.3	3.2	2.8	1.6
	ISO Brightness (%)	49.0	49.2	49.5	50.1	50.6
9.2	Pulp Yield (%)	54.6	53.1	51.1	47.6	46.5
	Lignin Content (%)	6.2	5.9	4.3	3.8	2.0
	ISO Brightness (%)	50.5	49.0	49.3	47.2	50.1
6.7	Pulp Yield (%)	4.6	9.8	24.7	28.8	28.7
	Lignin Content (%)	15.5	13.2	9.6	8.8	8.7
	ISO Brightness (%)	37.7	38.8	37.0	37.6	36.0
*Other Pulping Process	Pulp Parameter	Time at Maximum Temperature (min)				
		20	30	45	60	90
Soda-AQ	Pulp Yield (%)	27.0	35.7	47.3	52.7	50.4
	Lignin Content (%)	9.3	8.0	7.4	6.8	5.2
Kraft	Pulp Yield (%)	7.2	26.2	46.4	45.6	51.8
	Lignin Content (%)	12.5	10.9	8.34	5.6	4.4
For nitric acid pulping: Cooking temperature, 85 °C; liquid/birch ratio, 10:1.						
* Processing conditions from reference Wang (2003): For soda-AQ: Alkali concentration 16% (based on Na ₂ O) and AQ concentration 0.032%. For kraft: Alkali concentration 16% (based on Na ₂ O) and sulfidity 15% (based on Na ₂ O). The other parameters were the same: a solid/liquid ratio of 1:3.5, maximum temperature of 165 °C, and a temperature-increasing time of 90 min.						

When lignin was completely removed, the remaining nitric acid continued to react with the carbohydrates, resulting in a decrease in pulp yield. With an increase in the cooking time, the pulp yield and lignin content gradually decreased. When the cooking time was 4.3 h, the pulp yield was 51.1% and the lignin content was 4.3%. With prolonged cooking time, the pulp yield dropped significantly. Therefore, the suitable cooking conditions of the nitric acid process were 9.2% nitric acid for 4.3 h at 85 °C. In terms of pulp yield and lignin content, the conditions were equivalent to those of the soda-AQ and kraft pulping of birch (Wang 2003).

Characterization of the fiber dimensions

The dimensions of the pulp fibers affected the physical properties of the pulp. To characterize the fiber size of the pulp obtained after nitric acid pulping, the distribution frequencies of the fiber length and width at various cooking times under identical processing conditions were determined. As shown in Fig. 1a, the fiber length ranged from 0.5 mm to 1.5 mm, the percentage of fibers within this length range was 90% to 95% and the percentage of fibers smaller than 0.5 mm or larger than 1.5 mm was ~5%. As shown in Fig. 1b, the fiber width ranged from 15 μm to 30 μm , which accounted for 86% to 89% of the total number, and the number of fibers smaller than 15 μm or greater than 30 μm in length was small. The effects of various cooking times on the length and width of the pulp fibers are shown in Table 3. As shown in Table 3, the average length and width of the fibers decreased with increasing cooking time, which can be attributed to the increased degradation of the pulp fibers.

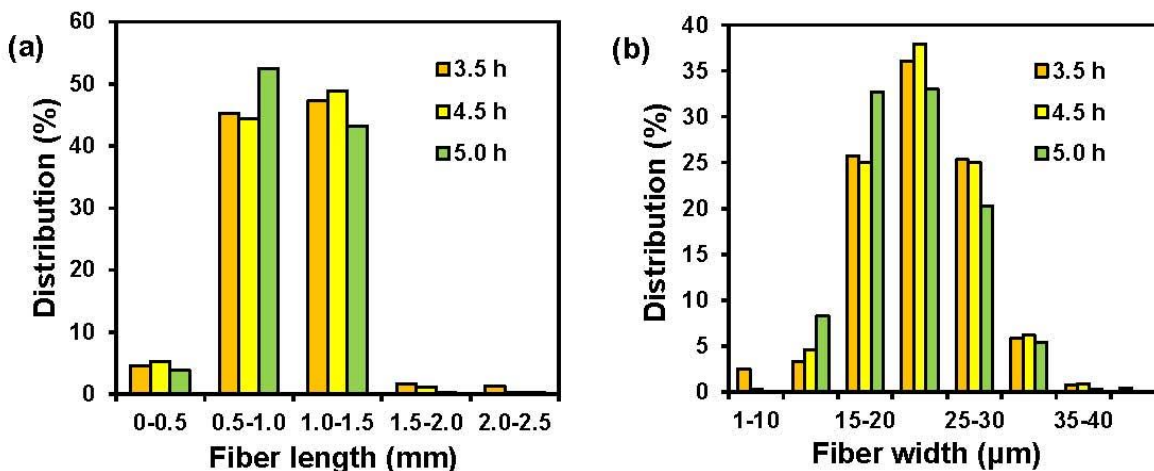


Fig. 1. The distribution of fiber length (a); and fiber width (b) of pulp (cooking temperature, 85 °C; nitric acid concentration, 9.2%; and liquid/birch ratio, 10:1)

Table 3. Effect of the Cooking Time on the Length and Width of Pulp Fibers

Cooking time (h)	Average Fiber Length (mm)			Average Fiber Width (μm)		Length-width Ratio
	L_n	L_w	L_{ww}	W_n	W_w	L_w/W_w
3.5	1.02	1.12	1.24	23.04	24.23	46.22
4.5	0.98	1.08	1.18	22.21	23.63	45.70
5.0	0.96	1.04	1.12	21.60	22.80	45.61

L_n , length-number average; L_w , length-weighted average; L_{ww} , weight-weighted average; W_n , width-number average; and W_w , width-weighted average. The measurement range was >0.10 mm in length.

Delignification Course

The effect of the cooking time on the lignin content in birch pulp from nitric acid is shown in Fig. 2. The delignification from nitric acid cooking can be divided into two stages. The first stage, which spanned 0 to 1.5 h, constituted the bulk of the delignification process. The delignification amount at this stage was 81.5%. The second stage, which spanned 1.5 h to 4.5 h, constituted the remnants of the first delignification process. The lignin removal amount at this stage was low at 6.5%. These two phases were different from the three phases (initial, bulk, and residual) and the delignification of kraft pulping of wood chips (Kubo *et al.* 1983; Shafi *et al.* 1993; Dang and Nguyen 2007). In the bulk delignification stage, the lignin removal from birch was swift, which was probably due to the rapid dissolution of lignin.

The effect of the cooking time on the chemical composition of pulp is shown in Fig. 3. As illustrated in Fig. 3, in the bulk delignification stage, the delignification amount was 81.5%, accounting for 92.6% of the total dissolved lignin.

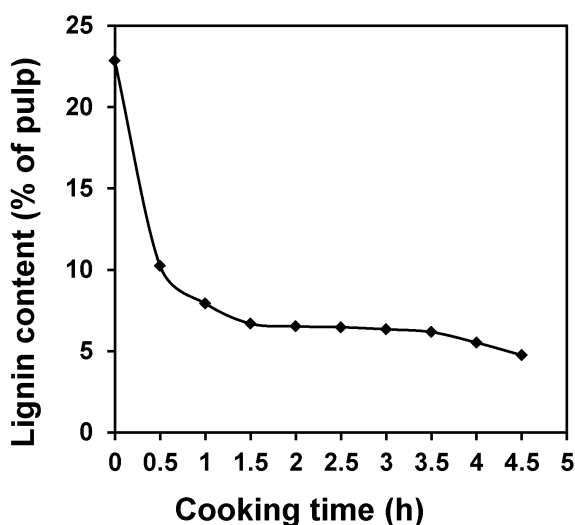


Fig. 2. The content of lignin in pulp with prolonged cooking time (cooking temperature, 85 °C; nitric acid concentration, 9.2%; and liquid/birch ratio, 10:1)

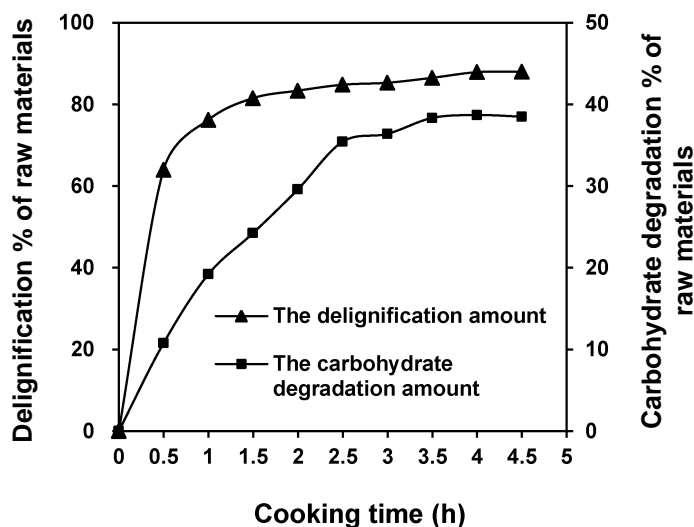


Fig. 3. Changes in the chemical composition of pulp with prolonged cooking time (cooking temperature, 85 °C; nitric acid concentration, 9.2%; and liquid/birch ratio, 10:1)

The carbohydrate degradation amount was 24.2% at this bulk stage, accounting for 63.0% of the total dissolved carbohydrate; and the dissolution ratio of lignin to carbohydrate was $92.6:63.0 \approx 1.5:1$, indicating that the decreased pulp yield was mainly attributable to the dissolution of lignin at this stage. In the residual delignification stage, the delignification amount was 6.5%, accounting for 7.4% of the total dissolved lignin, whereas the carbohydrate degradation amount was 14.2%, accounting for 37.0% of the total dissolved carbohydrate. The dissolution ratio of lignin to carbohydrate at this stage was $7.4:37.0 \approx 1:5.0$, indicating that the decreased pulp yield was mainly caused by the degradation of carbohydrates. This was not conducive to the improvement of the pulp yield. Therefore, the prolonged cooking time was avoided as much as possible at the residual delignification stage.

Recycling of Used Pulping Liquor

Effect of the number of cycle times of the waste liquor on the properties of pulp

The effect of the number of used pulping liquor cycles on the pulp yield and lignin content is shown in Fig. 4. As shown in Fig. 4, the pulp yield decreased, and the lignin content increased with the increasing number of cycle times of the used liquor. When the used liquor was recycled six times, the pulp yield decreased from 50.3% to 37.7%, and the lignin content increased from 5.3% to 8.1%. As a consequence of the pulp yield being very low, the used liquor was recycled a maximum of six times and could not be reused afterwards. This was because as the number of cycles increased, the content of organic matter degradation products that accumulated in the waste liquid also gradually increased, though the concentration of nitric acid remained unchanged, causing the delignification reaction to proceed unfavorably.

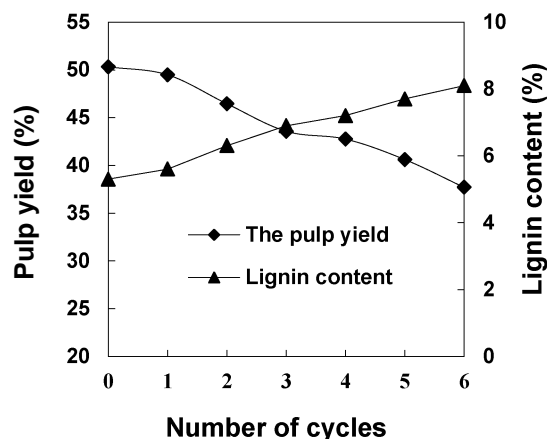


Fig. 4. The effects of the number of used pulping liquor cycles on the pulp yield and the lignin content

Preparation of solid organic fertilizer from the final batch of waste liquid

The final batch of used pulping liquor could be used to prepare organic fertilizer because it contained a large amount of carbohydrate and lignin degradation products, as well as unreacted nitric acid. However, the used pulping liquor was acidic. Therefore, an aqueous solution of ammonia was added to the waste liquid to neutralize it and to introduce more nitrogen content. The dried organic fertilizer was a dark brown solid. As shown in Table 4, the contents of total nitrogen and organic matter in the fertilizer were determined to be 13.3% and 60.3%, respectively. Compared to commercial compost and farm-compost, the total nitrogen content was higher, and the organic matter content was comparable. These results indicate that the nitric acid pulping waste liquid can be used to prepare organic fertilizer. The other components in the fertilizer were not tested.

Table 4. Contents of Various Components of Organic Fertilizer *versus* Standard Organic Fertilizer

*Item	Waste Liquid Fertilizer	#Commercial Composts			#Farm-compost
		A	B	C	D
Moisture Content (%)	11.4	32.2	25.8	30.5	38.5
Total Nitrogen (%)	13.3	5.2	2.6	2.4	2.1
Organic Matter (%)	60.3	71.5	52.1	73.6	46.9
pH	7.0	7.2	5.5	7.3	8.0

* On a dry weight basis except for the moisture content. # This parameter was derived from reference Chang *et al.* (2007): Compost A was composed of pig manure and sawdust; compost B was composed of wool; compost C was composed of soybean meal, peanut hull, and sawdust; and compost D (farm-compost) was composed of the plant residues of the farm.

CONCLUSIONS

1. Using birch wood as the raw material, the pulp yield obtained by nitric acid pulping was similar to that of conventional alkaline pulping.
2. The pulping delignification course involved bulk and residual phases. The bulk phase was 0 to 1.5 h long, and the delignification level of 92.6% of the total dissolved lignin was achieved. The residual phase was 1.5 h to 4.5 h long, and the delignification amount accounted for 7.4% of the total dissolved lignin.
3. The used pulping liquor could not be used after six rounds of recycling. The contents of nitrogen and organic matter in the organic fertilizer prepared using the final-round of recycled waste liquid were in line with the component indicators from other commercial organic fertilizers.

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