The Influence of Stock Consistency on the Pollution Load in Washing Process

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The influences of stock consistency on the kappa number, residual alkali of pulp, flow direction of lignin, chemical oxygen demand, oxidation with dichromate (COD_{Cr}), and five-day biochemical oxygen demand (BOD_5) in effluents were investigated in a washing process. Compared with 15% stock consistency, 35% stock consistency could lead to a decrease of at least 25% in the consumption of washing water while cutting 75.27% and 46.29% of COD_{Cr} and BOD_5 in washing effluents, respectively. Moreover, the residual lignin in effluents were reduced greatly from 39.35 $g \cdot t^{-1}$ to 12.76 $g \cdot t^{-1}$, which will be beneficial to treatment of washing and bleaching effluent. All the results showed that a higher stock consistency in the washing process could decrease the consumption of washing water, the generation of pollution, and the toxicity in bleaching effluent.

Keywords: Stock consistency; Washing Process; Bleaching process; Pollution load; Lignin

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INTRODUCTION

Wastewater emissions from pulping and paper–making industry account for approximately one sixth of the total amount of industrial wastewater in China, of which COD_{Cr} from pulp mills equals 1/4 of the total load from all sources in the China. The severe pollution caused by industrial wastewater has become the bottleneck for sustainable development and technological innovation in the pulp and paper industry in China. Up to this point much research has been conducted on the development of new technologies for wastewater treatment, such as coagulation (Semerjian and Ayoub 2003; Seki *et al.* 2010), adsorption (Cong *et al.* 2015), membrane separation (Mänttäri *et al.* 2008; Kazuaki *et al.* 2013), and biochemistry (Sridhar *et al.* 2011; Sari *et al.* 2014). But high operating costs and economic burdens have limited the application of these technologies. Therefore, reduction of source pollution emissions in the production process has become urgent for the pulping and paper-making industry in China.

Kraft and soda are the two major alkaline processes being used to produce chemical pulps. However, in both processes, cellulose fibers are disassociated from lignin by chemical reactions. The products resulting from the digester reactions are the cellulose pulp and the black liquor (Quezada *et al.* 2015). At present, soda recovery units (Licursi *et al.* 2015) mainly have been applied for the treatment of black liquor with 95% to 98% recovery ratio for wood material, which means that less than 5% of the black liquor would flow into the washing process. However, in China the fact that non-wood fibers, such as bagasse, straw, grass, and bamboo, are used as the pulp materials and the technological limitations of equipment lead to only about 85% recovery ratio for black liquor. This means that more than 15% of the black liquor remains in the pulp will be unfortunately brought into the washing process subsequently.

Black liquor is well known to be constituted with plenty of lignin detached from raw materials, as well as considerable fibers and other organics with huge COD_{Cr}, BOD_5, and color pollution. Thus, the remaining black liquor in pulp should be cleaned out as much as possible to decrease the bleaching load and effluent toxicity before the bleaching process. The washing process is very necessary and important for the whole production because washing efficiency and paper pulp cleanliness not only directly affects the design and operation of the subsequent bleaching process, but also are directly related to the production of various toxic organic pollutants, such as (AOX) (Vepsäläinen *et al.* 2011; Shukla *et al.* 2013; Zhang and Pagilla 2013). In current industrial production, deacetylations by interval washing and pressing combined with dilution are mainly used in the washing process. Studies of the washing process have been largely confined to the modification of equipment and the combination of pulp washing systems (Chew *et al.* 2013; Zeng *et al.* 2014; Frigieri *et al.* 2015), while few studies have been focused on the effects of stock consistency on the washing process. Based on the view of source pollution control, the influences of stock consistencies in washing process on water consumption, the amounts of various organic pollutants, and the load of pollution are discussed in this paper, which is supposed to bring about fundamental changes in pollution control and treatments for pulping and paper-making industry in China.

EXPERIMENTAL

Cooking

Softwoods are one of the most important feedstocks for pulp and paper production in China (Liers *et al.* 2011), *Pinus koraiensis* was used as material with alkaline cooking, followed by washing and bleaching processes. The active alkali, liquor-to-wood ratios, cooking temperature and cooking time were 28%, 20%, 170 °C, and 3 h, respectively. These production conditions are typical for a bleached soda pulp mill in China. The pulp yield was 44.07% in our batch experiments. After cooking, pulp consistencies of 15%, 19%, 23%, 30%, and 35% were obtained through dewatering on squeezer (Changsha China) . Each pulp quantity applied in the washing process was 0.4 kg for absolute dry mass, which means that the pulp quantities of consistencies of 15%, 19%, 23%, 30%, and 35% were 2.67 kg, 2.11 kg, 1.7 kg, 1.33 kg, and 1.13 kg, respectively. A three-step washing procedure, with tap water consumptions of 4 L, 3 L, and 2 L, was adopted to determine the effects on washing with different stock consistencies. After each washing step, the original consistency was restored through dewatering and then passed into next washing with 0.1 kg of sample taken for analysis.

Other Analytical Methods

Prior to experiments, all liquid effluent samples were filtered with a membrane filter (pore size 0.45 μm) to remove suspended matter and particles. The determination of the COD_{Cr} was carried out using a Hach spectrophotometer (DR2800, Hach, Loveland, CO, USA), according to standard methods (ISO 1983). The BOD_5 was measured using samples incubated for five days at 20 °C on a Hach BOD detector (Hach, Loveland, CO, USA) following standard methods (Liu *et al.* 2008). The pH was measured with a

Sartorious PB-10 (Sartorius, Germany) pH meter. The UV spectra were obtained on a Scinco S-3100 UV spectrophotometer (Hach, Loveland, CO, USA).

Methods employed for chemical composition of *Pinus koraiensis* are shown in Table 1.

Table 1. Methods Used to Determine Chemical Composition of *Pinus koraiensis*

Chemical compositions	references
Ash in wood	TAPPI Test T15wd-80
Cellulose in wood	TAPPI Test T17wd-70
Pentosans in wood	TAPPI Test T 223cm-01
Holocellulose in wood	TAPPI Test T 9wd-75
Acid –soluble lignin in wood	GB/T 2677.8-1994
Acid –insoluble lignin in wood	TAPPI Test T18wd-76
Hot water extractives in wood	GB/T 2677.4-1993

RESULTS AND DISCUSSION

Main Chemical Composition of *Pinus koraiensis*

In this work, only the most important chemical compositions are presented, as shown in Table 2. Such analyses were performed according to the Technical Association of the Pulp and Paper Industry (TAPPI) and recommended national standards of China (GB/T). Results of these analyses supply data for predicting the properties of *Pinus koraiensis*. Ash and cellulose of *Pinus koraiensis* is 0.45% and 42.21%, respectively. This means that it is suitable to be used as material. Moreover, hot water extractives of *Pinus koraiensis* are greatly high.

Table 2. Main Chemical Composition of *Pinus koraiensis*

Chemical compositions	Content （%）
Ash in wood	0.45
Cellulose in wood	42.21
Pentosans in wood	4.29
Holocellulose in wood	75.73
Acid –soluble lignin in wood	31.23
Acid –insoluble lignin in wood	0.12
Hot water extractives in wood	1.42

The Influence of Stock Consistency on Kappa Number of Pulp in the Washing Process

The kappa number of pulp is used to indicate the degree of delignification occurring during cooking, which can affect the chemical requirements for bleaching. The influence of the stock consistency on the kappa number of pulp and kappa number removal extent is presented in Table 3. A certain amount of pulp was taken and washed thoroughly with enough water, and the end kappa number of the pulp was obtained as 50.2, which can be used to calculate kappa number removal extent.

Table 3. Kappa Number and Kappa Number Removal Extent

Stock consistency (%)	15	19	23	30	35
Kappa number of different pulp without washing	62.8	62.5	62.3	61.7	61.2
Kappa number of pulp after first-step washing	59.1	56.8	56.2	53.8	53.4
Kappa number removal extent of first-step washing (%)	29.37	46.34	50.41	68.70	70.90
Kappa number of pulp after second-steps washing	55.2	54.6	53.7	51.8	50.8
Kappa number removal extent of second-steps washing (%)	60.31	64.23	71.07	86.09	94.55
Kappa number of pulp after third-steps washing	54.3	53.5	51.9	50.9	50.3
Kappa number removal extent of third-steps washing (%)	67.46	73.17	85.95	93.91	99.09

The kappa numbers of different pulp consistencies without washing were basically uniform for the same materials and cooking condition. Kappa numbers of different pulp consistencies all showed a clear diminishing trend with the washing proceeding, which means that some of the lignin remaining in the surface of solid fiber or in the liquor can be cleaned out effectively with washing. An obvious enhancement of kappa number removal ratio with the increase of pulp consistency should be noticed, which may have resulted from the fact that higher pulp consistencies will lead to less liquor remaining in the pulp before washing, and then more lignin in liquid phase were separated from the pulp. Better washing effects could be obtained with higher consistency (Table 3) that will be beneficial for the bleaching process, because less lignin will result in lower bleaching agent consumption, less generation of AOX, COD, BOD, and other pollution, as well as the feasibility of less-pollution technologies such as TCF or ECF application.

As the pulp consistency was increased, kappa number removal ratio of the first-step washing apparently increased. The kappa number removal ratio of the first-step washing for 15% pulp consistency was only 29.37%, while for 35% pulp consistency was 70.79%. The end kappa number for 15% pulp consistency after 3 steps washing was 54.3, which was even greater than the value of 53.4 for 35% pulp consistency with only first-step washing. This suggested that one could obtain a better effect using the pulp consistency of 35% after first-step washing than the pulp consistency of 15% after 3 steps washing. There could be a considerable reduction in the amount of water consumption with higher consistency which leads to the decrease of wastewater emission.

The Influence of Stock Consistency on Cleanliness in the Washing Process

Residual alkali of liquid after washing is often used to express the cleanliness of pulp. The influence of the stock consistency on the residual alkali and alkali removal extent are presented in Table 4 and Fig. 1, respectively. Wood pulp with residual alkali less than 0.05 g/L can be regarded as clean enough to be bleached, so 0.05 g/L residual alkali was applied to calculate residual alkali removal ratio.

Table 4. Residual Alkali of Black Liquor and Washing Effluent

Stock consistency (%)	15	19	23	30	35
Black liquor (g/L)	8.50	8.98	9.56	10.60	11.68
The first-steps washing effluent (g/L)	4.60	3.04	2.60	1.44	1.58
The second-steps washing effluent (g/L)	0.72	0.52	0.27	0.12	0.10
The third-steps washing effluent (g/L)	0.16	0.10	0.10	0.08	0.04

As pulp consistency was increased, the residual alkali of the black liquor at the beginning showed an increasing trend, which may have resulted from the fact that higher pulp consistency leads to less liquid between fibers, and then to higher concentration of sodium ion in black liquor. Higher pulp consistency implies less liquid between fibers and then more sodium ion in liquid phase was separated from the pulp. Therefore, the residual alkali of effluent for different pulp consistencies all showed a clear diminishing trend, especially in 35% pulp consistency with the washing proceeding.

After the two-steps washing, the residual alkali removal ratio for 15% pulp consistency was only 92%, while for 35% and 30% pulp consistency was 99%. The end residual alkali for 15% pulp consistency after 3 steps washing was 0.16, which was even greater than the value of 0.12(0.1) for 30% (35%) pulp consistency with 2 steps washing. As wood pulp with residual alkali less than 0.05 g/L can be regarded as sufficiently clean to be bleached, pulp consistencies of 15%, 19%, 23%, 30% after 3 steps washing did not meet the requirements of industrial production, and a fourth or even a fifth step of washing will need to be carried out. Compared with lower stock consistency, 35% stock consistency could lead to an at least 25% decrease in the consumption of washing water.

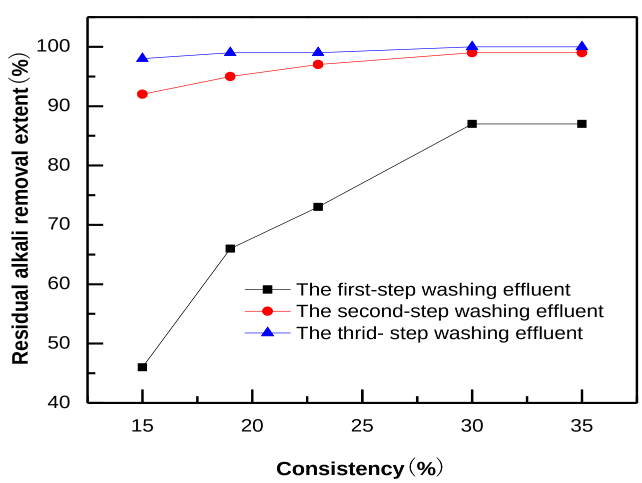

Fig. 1. Residual alkali removal extent of different steps washing effluent

Determination of Lignin in Black Liquor and Washing Effluent

There are two main pollutants in the fiber pulp process flows after cooking: carbohydrates and lignin. The BOD/COD ratio of carbohydrates was more than 0.5 with good biodegradability (Liberatore *et al.* 2012, 2015), which means that carbohydrates can be easily removed by biochemical methods. However, lignin not only contributes to the COD_{Cr}, but also is difficult to biodegrade. What is more, lignin degradation is the source of AOX (Lacorte *et al.* 2003; Dhakhwa *et al.* 2014; Lei *et al.* 2013; Lei and Li 2013). Improving the biodegradability of lignin has been a major issue in the field of lignin research (Bugg *et al.* 2011a; Bugg *et al.* 2011b; Liers *et al.* 2011), but a breakthrough is unlikely at present. Therefore, it is very important to reduce the lignin brought into the bleaching process, which can considerably reduce the pollution load and toxicity of washing and bleaching effluent.

A linear relationship between absorbance and lignin consistency in Fig. 2 has been established as references suggested (Lin and Dence 2012; Zhou *et al.* 2012; Ślesak *et al.* 2015), and lignin content of black liquor and washing effluent is presented in Table 5.

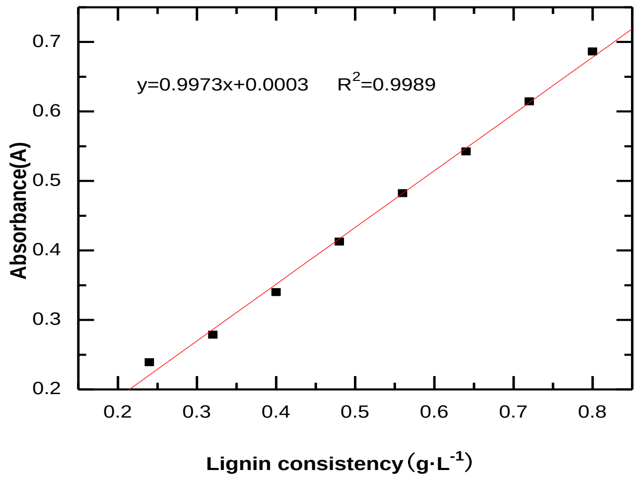

Fig. 2. Linear relationship between absorbance and lignin consistency

Table 5. Lignin Content in Black Liquor and Washing Effluent

Stock consistency (%)	15	19	23	30	35
Black liquor (g/L)	34.98	42.88	56.73	78.50	88.43
The first-step washing effluent (g/L)	31.60	26.38	19.65	14.80	10.53
The second-steps washing effluent (g/L)	6.60	5.10	4.03	2.13	1.60
The third-steps washing effluent (g/L)	1.15	1.05	1.20	0.95	0.63
Washing effluent (g/L)	39.35	32.53	24.88	17.88	12.76

As pulp consistency increased, lignin brought to the alkali recovery unit increased from 34.98 g/t to 88.43 g/t, which will favor the vapor production (Liu *et al.* 2015) and economical efficiency for soda recovery technique. Furthermore, some new ways using black liquor to get renewable feedstock could be invented (Alonso *et al.* 2010; Serrano-Ruiz *et al.* 2011). One also directly converts black liquor to bio-fuel, which presents both advantages to destroy a waste together with economizing planetary feedstock (Azadi *et al.* 2013; Kleinert and Barth 2008). On the contrary, lignin brought to the washing process is decreased, which is beneficial to the washing process and reduces the pollutant load of washing and bleaching effluent. As pulp consistency increased, the total amount of lignin in washing effluent decreased from 39.35 g/t to 12.76 g/t. The lignin has been reported as mainly responsible for the toxicity of resulting wastewater, mainly due to chloro-organic derivatives (Pereira *et al.* 2009). All in all, less lignin will result in lower bleaching agent consumption, as well as less generation of AOX, COD_{Cr}, BOD_5. and other pollutants.

The Influence of Stock Consistency on pollution loads of Washing Effluent

COD_{Cr} is based on an oxygen depletion test, in which the oxygen demand is measured by direct chemical methods. BOD_5 is the most widely used test for the oxygen demand of an effluent stream, which a sample of effluent is allowed to consume oxygen by the action of microorganisms. Table 6 shows the COD_{Cr} and BOD_5 measured by the respirometric method of the black liquor and washing effluent. The total content of COD_{Cr} and BOD_5 of the washing effluent are shown in Table 7.

Table 6. COD_{Cr} and BOD_5

Stock consistency (%)	Black liquor (mg/L)		The first-step washing effluent (mg/L)		The second-steps washing effluent (mg/L)		The third-steps washing effluent (mg/L)	
	COD_{cr}	BOD_5	COD_{cr}	BOD_5	COD_{cr}	BOD_5	COD_{cr}	BOD_5
15	104844	22134	43832	13708	8634	2458	3306	391
19	106084	25856	36363	12650	7469	1876	1946	282
23	106412	28945	29327	11958	5802	1525	1158	226
30	113236	34908	19709	7895	2938	1295	822	186
35	117064	41100	11184	7600	1939	1080	668	180

Table 7. Total Content of COD_{cr} and BOD_5 in the Washing Effluent

Stock consistency (%)	15	19	23	30	35
COD_{cr} (mg/L)	55772	45778	36287	23469	13791
BOD_5 (mg/L)	16557	14808	13709	9376	8860

From Table 6 it can be found that COD_{Cr} and BOD_5 in the black liquors slightly increased with the increase of stock consistency, which has little influence on the pollution because black liquor will be pumped to the soda recovery units directly. However COD_{Cr} and BOD_5 in the same step (1,2,3) washing effluent showed a significant decrease. The organic compounds contained in the black liquor are mainly constituted of lignin (Table 7.) (Hamaguchi *et al.* 2012). As pulp consistency increased, there was more lignin in the black liquor (Table 5). Therefore, for the same materials and cooking condition, washing effluent had less lignin. It can be concluded that pressing the pulp to high consistency before washing can reduce the content of BOD_5 and COD_{Cr} of the washing effluent. Compared with 15% stock consistency, the COD_{Cr} and BOD_5 of washing effluent of 35% stock consistency had been decreased by 75.27% and 46.49%, respectively.

CONCLUSIONS

1. Under the same cleanliness (<0.05 g/L), compared with 15% stock consistency, 35% stock consistency could lead to an at least 25% decrease in the consumption of washing water. Therefore, the amount of washing and bleaching wastewater and difficulty of dealing with wastewater were reduced.
2. Pressing the pulp to high consistency before washing can reduce the COD_{Cr} of washing effluent from 55,772 $mg\cdot L^{-1}$ to 13,791 $mg\cdot L^{-1}$ and the BOD_5 of washing effluent from 16,557 $mg\cdot L^{-1}$ to 8860 $mg\cdot L^{-1}$, decreases of 75.27% and 46.29%, respectively. Moreover, the residual lignin in effluents had been reduced greatly from 39.35 $g\cdot t^{-1}$ to 12.76 $g\cdot t^{-1}$, which will be beneficial to treatment of washing and bleaching effluent.

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