Effects of Tree Age and Bole Section on Pulpwood of Korean Spruce (*Picea koraiensis* Nakai)

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Korean spruce (*Picea koraiensis* Nakai) is one of the most important wood resources for pulp and paper industry. However, a long production cycle is required for spruce to obtain mature timber, which may negatively influence the biomass production. In this study, fiber morphology, chemical composition, and pulping properties of Korean spruce with different tree ages and vertical locations were investigated. The results show that, along with the increase of tree ages, the contents of extractives, lignin, and pentosan increase to different extents. There are also some differences of pulping properties with different tree ages. As a pulpwood, the Korean spruce with tree ages of around 22 to 32 years is more suitable for kraft pulping, compared to that with 32-46 years. In addition, the middle segment of the bole had better pulp strength properties than the upper or lower bole segments.

Keywords: Korean spruce; Chemical compositions; Fiber characteristics; Kraft pulping

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INTRODUCTION

Spruce (*Picea*) is one of the world's major industrial timber species, but it is mainly distributed in sub-frigid and frigid zones, with only a small number distributed in temperate and sub-tropical damp mountains (Ma 1993; Wang *et al.* 1997). There are more than 40 named species of spruce in the world, and Korean spruce (*Picea koraiensis* Nakai) is typical of this species (Wang *et al.* 2005a). Spruce is widely used for construction materials, furniture, medicine, *etc.* (Crellin 2004; Stubbs 2003). In addition, it can be used as a good pulpwood because of its superior wood fiber characteristics. Spruce is one of the most important wood resources for the pulp and paper industry, as it has long wood fibers that bind together to make strong paper (Shi and He 2003). The fibers are thin-walled and easily collapse to thin bands upon drying. However, a long production cycle is required for spruce to become mature timber, which may negatively influence the biomass production. Therefore, the rotation time of spruce used as pulpwood has received much attention in recent years.

The dependence on plant fiber resources is high in the Chinese pulp and paper industry. China has a lack of forest resources, and the shortage of wood fiber sources, especially softwood fibers, is one of the main problems for the Chinese pulp and paper industry. In the planted softwood species of China, spruce occupies an important position (Wang *et al.* 2005a). Therefore, it is very important to determine the appropriate rotation of spruce as a pulpwood for the further efficient utilization of spruce fiber resources.

Many previous studies regarding Korean spruce genetic improvement have been conducted (Wang *et al.* 1991, 2001; Xu *et al.* 1997; Yang *et al.* 1992). However, studies of the directive cultivation and breeding of Korean spruce with respect to its use as a pulpwood are scarce. Rational utilization of this high-potential species not only has a vital significance on the development of the pulp and paper industry in China, but can also improve the forestry-pulp-paper integration process.

The chemical compositions and fiber characteristics may be quite different for spruces with different tree ages and bole sections. The pulping properties could decrease when the rotation time is too long. On the other hand, if the wood is cut too early, the wood fiber will be short and wood resources may be wasted. Therefore, it is important to determine the suitable rotation time for each kind of wood resources by statistical methods, which is good for the forestry cultivation of pulpwoods. In this study, the fiber characteristic analysis and the conventional kraft pulp process of Korean spruce were chosen as evaluation criterion for spruce rotation time. The conclusions were done by a statistical method. In this way, a suitable rotation cycle can be demonstrated and the results can be used to guide the rotation time optimization of spruce used as pulpwood.

EXPERIMENTAL

Raw Materials

Korean spruce trees at three different ages (22, 32, and 46 years), Northeast larch, Japanese larch, and Masson pine were provided by the Research Institute of Forestry of the Chinese Academy of Forestry. These samples were obtained from standard planting areas in man-made forests of the Great Khingan mountains (latitude 50°10’N-53°33’N and longitude 121°12’E-127°00’E), in Heilongjiang province, China. There were three whole sample trees of Korean spruce with the same age. The wood logs were cut into three two-meter long segments (top, middle, and root segments of the whole trunk). They were then peeled and chipped for cooking experiments. In addition, three or more parallel wood samples were prepared to ensure the accuracy of experimental data. In total, there were more than 81 spruce samples and 9 samples of other kinds of softwood.

Determination of Carbohydrate Composition and Chemical Components

Chips of Korean spruce, Northeast larch, Japanese larch and Masson pine were milled using a Wiley mill in the laboratory, and the wood powder was passed through a 40-mesh screen and retained at 60 mesh; it was then collected for the measurements of carbohydrate composition and chemical components. The carbohydrate compositions were determined using gas-liquid chromatography according to the Chinese standard GB/T 12032-1989. All the indices, including extractive contents (cold-water extractives, hot-water extractives, 1% NaOH extractives, and alcohol-benzene extractives), lignin contents (Klason lignin and acid-soluble lignin), cellulose content, holocellulose content and pentosan content, were determined according to Chinese standard methods (Shi and He 2003).

Fiber Morphology

The fiber length, width, and coarseness were measured using a fiber tester (Fiber Test 912, L&W Inc., Sweden) following the TAPPI method T271pm-91. The weighted average data were obtained from the fiber tester, which is commonly used to evaluate the softwood fiber quality. For each measurement, there were about 10,000 fibers used.

Kraft Pulping Properties

The kraft cooking was done in an air bath cooker (NJKR-10 with a volume of 8×1.25 liters, made in China). The kraft pulping properties of the five spruce types were compared under the same cooking conditions. The cooking conditions were fixed as follows: 18% alkali dosage (based on Na_2O), sulfidity of 25%, wood to liquid ratio of 1:4, max. temp. of 170 ºC, and time at max. temp. of 2.5 h. When the cooking was done, the residual alkali and pH value of the effluent were measured. After washing, some other indices were determined, such as unscreened pulp yield, reject ratio, and kappa number.

Afterwards, the kraft pulps were beaten to 45°SR with a PFI mill (made by HAM-JERN, Norway). Then, handsheets with a basis weight of 60 g/m^2 were made in a standard sheet former (made by Labtech Inc., Canada). The handsheets were placed in a standard room with a temperature of 23 ± 1 °C and relative humidity of 50 ± 2% RH, and the physical properties of the handsheets were determined. All the procedures mentioned above strictly followed the Chinese standard methods (Shi and He 2003).

RESULTS AND DISCUSSION

Chemical Composition Analysis

The quality of pulp fibers and paper products largely depends on the micro-structure and chemical composition of wood materials. The chemical composition of fibrous materials is an important parameter for evaluating the performance of pulp and paper raw materials. The chemical components at different vertical locations in spruce of various ages are shown in Table 1.

The extractives content of the same segment of Korean spruce increased as the tree age increased, whether for water extractives, organic solvent extractives, or 1% NaOH extractives; and as for the 32-year-old whole log, the extractive contents decreased as the vertical location height increased. This may be due to the increased heartwood with increasing tree age. The 1% NaOH extractives, including some resin and tannin that dissolved in NaOH, affected the pulp yield and the fiber quality. However, it was relatively lower than that of other kind of softwoods (*e.g.*, larch).

The lignin content of spruce also increased as the tree age increased. This trend was similar to the trend of extractives content, indicating that an older tree age and higher percentage of heartwood will lead to a higher lignin content. Table 1 also shows that there were no differences in cellulose content (or holocellulose content) in trees with different ages. These results indicated that older Korean spruce was not always suitable for papermaking as a pulpwood. Especially considering the sustainable development of biomass resources, it is helpful to reduce the rotation period to improve the planting efficiency. Taking the 46-year-old spruce as an example, compared with the 22-year-old tree, the lignin content increased by 14.23%, and the 1% NaOH extractives content increased by 15.87%, but the holocellulose content decreased by 1.40%. It is obvious that the younger tree had better characteristics and a more reasonable rotation cycle for the end use of pulping. The results in Table 1 also show that spruce is a good pulpwood, containing lower extractives contents and higher cellulose and pentose contents.

Table 1. Chemical Components of Different Tree-Age Korean Spruce *(P. koraiensis* Nakai*)* Sampling from Different Vertical Segments of Whole Logs (%)

Tree age	22 years (middle)	Std. Dev.	32 years (top)	Std. Dev.	32 years (middle)	Std. Dev.	32 years (root)	Std. Dev.	46 years (middle)	Std. Dev.
Cold-water extractives	1.24	0.148	1.70	0.761	1.32	0.071	2.70	0.345	1.50	0.084
Hot-water extractives	2.84	0.154	2.32	0.566	2.91	0.250	4.36	0.180	3.91	0.106
Alcohol-benzene extractives	12.60	0.177	12.90	1.375	13.00	0.457	15.40	0.556	14.60	0.394
1% NaOH extractives	2.54	0.100	2.29	0.843	2.55	0.144	4.35	0.226	3.01	0.192
Klason lignin	24.60	0.537	25.00	0.115	25.20	0.012	26.60	0.053	28.10	0.194
Acid-soluble lignin	0.40	0.230	0.37	0.173	0.38	0.035	0.39	0.061	0.42	0.070
Total lignin	25.00	0.016	25.37	0.065	25.58	0.002	26.99	0.039	28.52	0.085
Cellulose	50.7	0.149	52.8	0.553	52.9	1.149	51.2	0.873	52.3	0.204
Holocellulose	78.3	0.231	78.6	1.146	78.3	0.699	78.1	1.003	79.4	0.168
Pentosan	11.6	0.715	13.5	0.262	12.8	0.151	10.7	0.324	14.4	0.192

In addition, the standard deviations (Std. Dev.) for each parameter could be obtained (Table 1), and these were used to evaluate the variability. As shown in the table, the standard deviations for the chemical compositions of spruce with different tree ages were small, indicating that there was a high confidence level. However, the standard deviations of cellulose and holocellulose for spruce with 32 years old were relatively large. There was smaller confidence interval due to the fact that there were more samples. The explanation here is that the fibers of 32-year-old spruce had been produced during the period of rapid growth, and consequently its deviation span was relatively large. Further, the deviation of the middle section was a little greater than the other two for 32-year-old spruce. This might be attributed to the relatively bigger growth rate difference of middle section fiber.

The monosaccharide composition gradually changed as the tree age increased. Hence, by analyzing the sugar composition of Korean spruce at different ages, the difference in each monosaccharide component can be obtained during the process of tree growth. The comparison of carbohydrate composition between Korean spruce and the other three softwoods is shown in Table 2.

Table 2. Carbohydrate Composition of Four Extractive-Free Softwoods (Absolute content unit: mg/g; relative content unit: %)

Monosaccharide	Northeast larch	Japanese larch	Masson pine	Korean spruce
Glucose	421.7 (61.88)	392.1 (54.75)	488.9 (61.08)	456.2 (60.52)
Xylose	57.72 (8.47)	129.7 (18.11)	79.92 (9.99)	79.15 (10.50)
Mannose	160.0 (23.48)	121.7 (16.99)	162.0 (20.24)	160.1 (21.24)
Arabinose	30.93 (4.54)	44.15 (6.16)	40.33 (5.04)	29.32 (3.89)
Galactose	11.13 (1.63)	28.55 (3.99)	29.25 (3.65)	29.05 (3.85)
Total sugar	681.5 (100)	716.2 (100)	800.4 (100)	753.9 (100)

It can be seen that, in the middle segment of 46-year-old Korean spruce, the glucose content (from cellulose and hemicellulose) was the highest (60.52%), and the content of mannose was the next highest (21.24%). Moreover, the total carbohydrate content of spruce was higher than that of larch and a little lower than that of Masson pine, leading to a higher pulp yield during the chemical pulping process.

Fiber Morphologies

As shown in Table 3, the fiber length of spruce was relatively long. The weighted average fiber length varied from 1.931 mm to 2.491 mm. As the tree age increased, the fiber length, fiber width, and fiber coarseness increased. For the 32-year-old whole spruce log, the fiber length was the longest for the middle segment. It also was found that the average growth rate of the spruce fiber length from 22 years to 32 years was 0.0186 mm per year, while from 32 years to 46 years, the growth rate only was 0.0129 mm per year. Furthermore, the average growth rate of fiber coarseness from 22 years to 32 years was 0.0320 $\mu g \bullet m^{-1}$ per year; while from 32 years to 46 years, the growth rate only was 0.0243 $\mu g \bullet m^{-1}$ per year. Moreover, the fiber length of 22-year-old spruce was relatively long (more than 1.9 mm), and its length-width ratio was similar to that of mature wood, such as 32-year-old spruce. Thus, the fiber longitudinal growth rate decreased with increasing tree age, especially after 32 years. Therefore, the tree age was not the most important factor for spruce selection as a pulpwood, which was in agreement with the prior conclusion from the chemical component analysis.

Table 3. Fiber Morphologies of Different Tree-Age Korean Spruce *(P. koraiensis* Nakai*)* Sampling from Different Vertical Segments of Whole Logs

Tree age	Mean fiber length (mm)	Std. Dev	Mean fiber width (μm)	Std. Dev.	Length-width	Std. Dev.	Coarseness (μg/m)	Std. Dev.
22 years (middle)	1.931	0.118	28.1	0.346	68.7	4.23	165.2	0.081
32 years (top)	2.095	0.112	28.4	2.155	73.8	6.25	171.9	0.232
32 years (middle)	2.117	0.050	28.9	2.950	73.3	3.78	182.2	0.126
32 years (root)	1.966	0.052	28.7	1.150	68.5	0.30	157.1	0.148
46 years(middle)	2.491	0.098	30.3	1.735	82.2	6.78	216.2	0.344

The distribution frequencies of the fiber-weighted average lengths from different vertical locations and from different tree ages are respectively shown in Fig. 1. As shown in Fig. 1(a), 22-year-old spruce had a relatively narrow distribution of fiber average lengths, and most fiber lengths were between 1 and 3 mm. The highest frequency was around 2 mm. As the tree age increased, the fiber length distribution ranges became wider and the maximum amplitude decreased. (Kleinschmit 1974; Skrppa 1981; Wang *et al.* 2004; Xie *et a*l. 2004; Zhao *et al.* 2002).

As shown in Fig. 1 (b), for the 32-year-old whole spruce log, there was no significant difference in the distribution ranges of the fiber average length for different bole sections. Additionally, the distribution range of the middle segment of spruce was concentrated in the range of 1.5 to 3.5 mm, indicating that there were more long fibers in this segment than there were in the others.

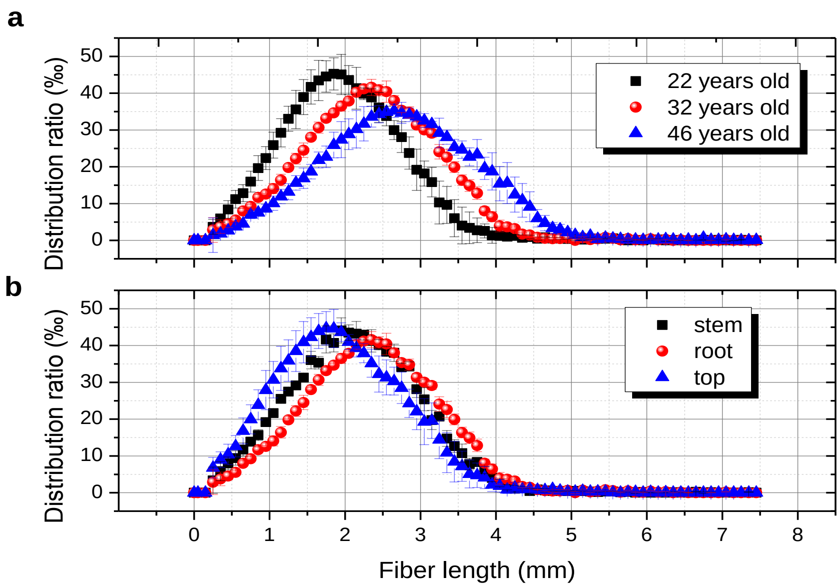

Fig. 1. Fiber length distribution of spruce at different ages and vertical locations; (a): different tree ages; (b): different vertical locations of 32-year-old tree

Kraft Pulping Properties

A comparison of the kraft pulping properties of spruce at different ages under the same cooking conditions is shown in Table 4. It can be seen that spruce was a good pulpwood, with a relatively high kraft pulp yield and low reject ratio (under 1%). With increasing tree age, both the unscreened and screened kraft pulp yield increased. For example, the unscreened pulp yield of 32-year-old spruce was 4.73% higher than that of 22-year-old spruce; and the unscreened pulp yield of 46-year-old spruce was only 2.63% higher than that of 32-year-old spruce. For different vertical location segments of the 32-year-old spruce, the pulp yield, residual alkali, and kappa number all increased from the top segment to the root segment.

Table 4. Kraft Pulping Properties of Korean Spruce Samples from Different Locations and of Various Ages

Tree age	22 (middle)	32 (top)	32 (middle)	32 (root)	46 (middle)
Unscreened pulp yield (%)	44.62	46.41	46.73	47.34	47.96
Std. Dev.	0.872	0.286	0.598	0.333	0.764
Screened pulp yield (%)	42.15	45.94	46.62	46.94	47.34
Std. Dev.	0.996	0.806	0.985	1.061	0.677
Reject ratio (%)	0.15	0.11	0.1	0.24	0.28
Residual alkali (g/L)	10.81	10.89	11.09	11.33	11.78
Std. Dev.	0.193	0.205	0.621	0.313	0.336
Kappa number	25.57	26.7	27.94	29.64	30.72
Std. Dev.	0.51	1.34	1.51	2.74	1.95
pH value of effluent	13.12	13.13	13.15	13.12	13.15

Comparing Table 1 with Table 4, it is clear that there is a close relationship between the kraft pulping properties and the chemical components of spruce with different ages. The relationship between lignin content and kappa number is shown in Fig. 2. The kappa number of spruce kraft pulp increased with total lignin content, and both the kappa number and lignin content increased with increasing tree age. However, the kappa number for 46-year-old spruce was higher than that obtained from 32-year-old trees under the same kraft pulping conditions, which affects the final pulp quality and results in a relatively higher request for pulp bleaching. Therefore, Korean spruce with a long rotation age, such as the 46-year-old sample, is not a good choice for pulpwood. The relevant parameters of three fitlines, shown in Fig. 2, are listed in Table 5.

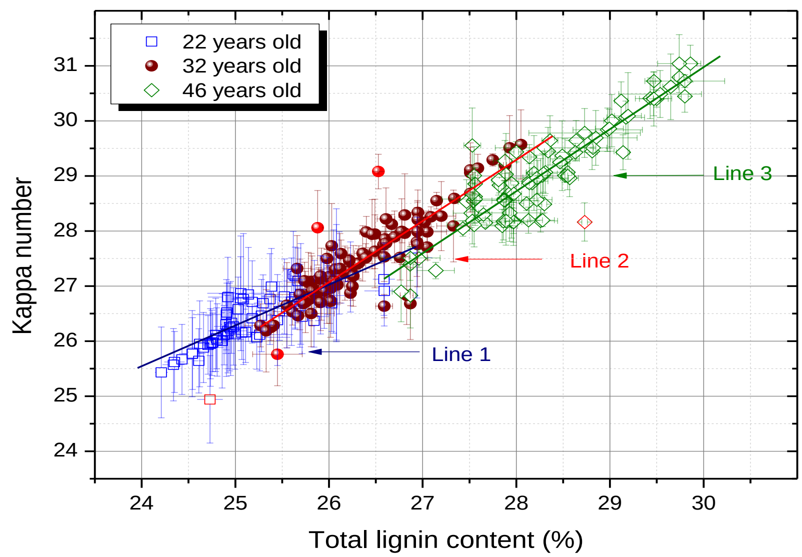

Fig. 2. The relationship between total lignin content and kappa number of Korean spruces with different ages

Table 5. Fitline Parameters of Relationship between Total Lignin Content and Kappa Number of Korean Spruces with Different Ages

Equation	y = a + b*x		
Weight	No Weighting		
Fitline number	1	2	3
Residual Sum of Squares	4.485	9.490	13.247
Pearson's r	0.868	0.911	0.910
Adj. R-Square	0.749	0.827	0.827
		Value	Standard Error
Line 1 (22 years old)	Intercept	7.959	1.235
	Slope	0.733	0.049
Line 2 (32 years old)	Intercept	-1.845	1.381
	Slope	1.111	0.052
Line 3 (46 years old)	Intercept	-2.411	1.474
	Slope	1.112	0.052

It can be found that three fitted curves for spruces with different tree ages were linear, as is apparent from the r values (Pearson's r in Table 5) close to 1 (0.868, 0.911, 0.910). This indicated a good correlation between total lignin content and Kappa number. Furthermore, the slopes of three fitted curves were similar to each other, and there was a slight increase with the increase of tree age. It can be concluded that for pulpwood with identical total lignin content, higher tree-age leads to higher cooking Kappa number.

Table 6 shows the physical properties of unbleached kraft pulps of spruce with different vertical locations and tree ages. The tensile index, tearing index, and folding strength of the middle segment were higher than those of the other two segments. The strength properties of samples at different tree ages were also different. In Table 6, all strength properties of 46-year-old spruce were higher than those of 32-year-old spruce; the 22-year-old spruce sample had the worst strength properties of these three samples. For the middle segment of spruce, the tree age was an important factor in determining the strength properties (He 1988; Wang *et al.* 2004, 2005b).

Table 6. Physical Properties of Unbleached Spruce Kraft Pulp

Type	22 (Middle)	32 (Top)	32 (Middle)	32 (Root)	46 (Middle)
Density(g/cm^3)	0.68	0.66	0.66	0.65	0.66
Burst index (KPa·m^2/g)	7.54	7.19	6.81	7.05	7.18
Std. Dev.	0.097	0.288	0.400	0.167	0.223
Folding endurance(time)	1252	1212	1443	1330	1755
Std. Dev.	125	145	127	215	136
Tensile index(N·m/g)	79.3	84.0	89.8	83.3	94.0
Std. Dev.	3.16	1.41	2.53	5.10	6.14
Tearing index(mN·m^2/g)	2.10	2.29	2.48	2.40	2.60
Std. Dev.	0.131	0.171	0.233	0.096	0.120

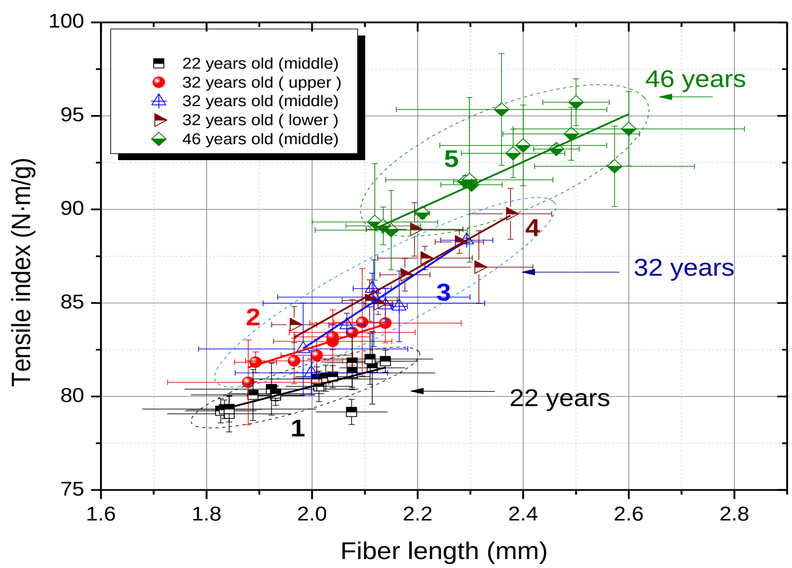

Fig. 3. The relationship between the fiber length and tensile index of Korean spruce at different ages

Figure 3 shows the relationship between the fiber length and tensile index of Korean spruce kraft pulp. With the increase of tree age, both the tensile index and fiber length increased. For spruces of different ages, the correlation was a little different: the tensile index of kraft pulp made from 22-year-old spruce increased relatively slower compared to that from 32- and 46-year-old trees. This means that the strength of a handsheet made from 22-years-old spruce was lower than that from 32- and 46-year-old trees. The tensile index of kraft pulp made from 32-year-old spruce was 13.24% higher than that from 22-year-old and 18.53% lower than that from 46-year-old spruce. However, the tendency of tensile strength to increase became slower when the spruce grew up (For example, spruce more than 32 years old). Therefore, it seems that Korean spruce of around 30 years old is a good choice for pulpwood.

Table 7. Fitline Parameters of Relationship between the Fiber Length and Tensile Index of Korean Spruce at Different Ages

Equation	y = a + b*x				
Weight	Instrumental				
Fitline number	1	2	3	4	5
Residual Sum of Squares	4.318	0.807	5.601	8.245	9.810
Pearson's r	0.988	0.929	0.917	0.967	0.814
Adj. R-Square	0.973	0.843	0.819	0.930	0.641

		Value	Standard Error
Line 1 (22 years old, middle)	Intercept	45.463	2.480
	Slope	18.708	1.097
Line 2 (32 years old, top)	Intercept	64.831	2.664
	Slope	8.897	1.340
Line 3 (32 years old, middle)	Intercept	51.869	5.705
	Slope	15.913	2.610
Line 4 (32 years old, root)	Intercept	61.966	2.182
	Slope	12.742	0.935
Line 5 (46 years old, middle)	Intercept	66.079	2.537
	Slope	7.231	1.290

The relevant parameters of five fitlines, shown in Fig. 3, are listed in Table 7. It can be seen that the r values (Pearson's r in Table 7) were very close to 1, indicating that there was a strong relationship between fiber length and paper strength. However, with the increase of tree age (from 22 years old to 32 years old and 46 years old), the slopes of fitline 1, 3, and 5 decreased significantly from 18.708 to 15.913 and 7.231, respectively. This may be due to the relatively lower fiber growth rate of 46-year-old spruce, which has high total lignin content, shown in Table 1, resulting in a negative effect on interfiber bonding.

CONCLUSIONS

1. The chemical composition, wood fiber characteristics, and kraft pulping properties of five different kinds of spruce were studied. The contents of extractives and lignin

increased slowly and the fiber length developed relatively quickly when the tree age was less than 32 years.

2. The pulping properties of 32-year-old spruce were better than those of 22- and 46-year-old spruce. The middle part of the log (or bole) had better kraft pulp strength properties than those obtained from the lower or upper parts of the logs.
3. The results showed that Korean spruce can be a relatively good pulpwood at certain ages. Korean spruce aged between 22 and 32 years is relatively good material to produce kraft pulps, following the principles of directive cultivation and efficient utilization of spruce resources.

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