# Fiber Characteristics and Chemical Composition of Three *Taxodium* 'Zhongshansa' Woods and Pulping Evaluation

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Taxodium 'Zhongshansa' (T. 'Zhongshansa') is a hybrid cultivar of the Taxodium Rich. genus. The wood and fiber characteristics and wood chemical compositions of three T. 'Zhongshansa' wood specimens were analyzed and compared with their parents, Taxodium mucronatum (T. mucronatum) and Taxodium distichum (T. distichum). It was found that the wood density of the three T. 'Zhongshansa' hybrids were slightly lower than the densities of their parents. The average fiber length and length to width ratio of the three samples were similar and greater than their parents. Runkel ratios of the three T. 'Zhongshansa' hybrids was smaller than their patents. Compared to the parents, the extractives contents of the three T. 'Zhongshansa' hybrids-hardly varied, while the lignin content was slightly lower. The result of the kraft cooking and bleaching of T.Z.502 indicated that with a cooking yield of 44.9%, the kappa number of the kraft pulp was 41.8. The T.Z.502 kraft pulp was easier to delignify via oxygen with alkali, and the brightness reached up to 76% ISO after an elemental chlorine free bleaching process. The results provide a reference for the application of *T*. 'Zhongshansa' wood in pulp and paper making in future.

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### INTRODUCTION

During the breeding process, the techniques in hybrid breeding and backcrossing are often used to enhance the good growth performance of new species, *e.g.*, *Eucalyptus* and *Populus* (Zhang *et al.* 2006; Yang *et al.* 2010; Yu *et al.* 2011). *Taxodium* 'Zhongshansa' (*T.* 'Zhongshansa') is a hybrid cultivar of *Taxodium* Rich. (Lu *et al.* 2006; Yu *et al.* 2008). There are more than 20 varieties of *T.* 'Zhongshansa', which have the following advantages: fast growth, a tall and straight tree trunk, and resistance to waterlogging, saline-alkaline, disease, pest, and storms, as well as high productivity and good wood quality, *etc.* (Yin *et al.* 2019). So far, *T.* 'Zhongshansa' trees have been planted in more than 10 provinces in China and are primarily used for urban greening construction and wetland restoration, which commonly plant *T.* 'Zhongshansa' *302*, *T.* 'Zhongshansa' *118*, and *T.* 'Zhongshansa' *401* (Yu *et al.* 2008).

*Taxodium* 'Zhongshansa' trees shapes are tall and straight, and its wood has potential value for processing and utilization in various industries. However, the study and production practice in terms of the processing and utilization of *T*. 'Zhongshansa' wood is

not very well reported. A few previous studies have indicated that the moisture content and adhesive strength of T. 'Zhongshansa' preservative plywood all meet the requirements for Class I outdoor plywood, but the smooth grain compressive strength, bending strength, bending elastic modulus, and wood hardness are lower, and the wood easily dries and cracks, causing warped deformation (Zhang *et al.* 2012a,b). Researchers are already working on improving the wood processing performance of T. 'Zhongshansa'. For example, Zhu *et al.* (2019) used a log-core veneer lathe to obtain peeled veneers of T. 'Zhongshansa', whose thickness deviation met the requirements for industrial applications (Chen *et al* 2019).

There is a shortage of fiber resources for pulp and paper making in China because of the lack of forest resources. Given the fast growth of *T*. 'Zhongshansa' trees, the shortage of these fiber resources can be alleviated in China if they are suitable for pulp and paper making. The work by Chi *et al.* (2014) showed that under effective alkali conditions of 22% NaOH, a sulfidity of 27%, and a maximum temperature of 170 °C for 2 h, the cooking yield of *T*. 'Zhongshansa' chips was 47.6%, while the kappa number was 42 for the kraft pulp and the pulp brightness reached up to 60% ISO after bleaching. Obviously, as a chemical pulp, this brightness is not attractive enough. The research group of the authors has investigated the kraft pulping performance of two *T*. 'Zhongshansa' species wood; the brightness of the bleached pulp was improved to approximately 76% ISO by using oxygen delignification and an elemental chlorine free (ECF) bleaching process under cooking conditions similar to Chi *et al.* (2014) (Xu *et al.* 2021).

*Taxodium* 'Zhongshansa' 302 (T.Z.302) is a hybrid species of *Taxodium distichum* (*T. distichum*) and *Taxodium mucronatum* (*T. mucronatum*). It has been cultivated since 1973. Previous studies indicated that under soil conditions with a pH of 8.0 to 9.5 and a salinity less than 0.2%, *T.Z.302* has an excellent synthetic growing appearance (Yin and Chen 1997; Yin *et al.* 2019). Although *T.Z.302* has advantages, with the increasing age of the tree, various problems, *e.g.*, low rooting rates and breeding difficulties, occurred. To address these problems, *T.Z.302* were backcrossed with its parents, *T. distichum* and some breeding varieties are cultivated, with *T.Z.118* being one of them. Previous investigation indicated that the growth characteristics and stress resistance of *T.Z.118* was better than *T.Z.302* (Yu *et al.* 2010). *Taxodium* 'Zhongshansa' *502* (*T.Z.502*) is also a new variety, which was obtained after years of artificial hybrid investigation from *T. distichum* and *T. mucronatum*; *T.Z.502* not only maintains the excellent characteristics of being semi-evergreen with high landscape value, but also shows remarkable superior characteristics compared to its parents in terms of growth. In addition, it is more resistant to red blight than *T. mucronatum* (Yu *et al.* 2011; Yin *et al.* 2019).

In order to understand the characteristic differences between the varieties and their feasibility as a pulp and paper making raw material, the chemical composition, wood properties, and fiber properties of the above three *T*. 'Zhongshansa' woods were analyzed and compared with their parents. In addition, the pulping and bleaching performance were evaluated for *T.Z.502* wood, in order to provide a reference for the use of *T*. 'Zhongshansa' in the pulp and papermaking industries.

### EXPERIMENTAL

### Materials

The T.Z.502, T.Z.302, and T.Z.118 wood as well as their parents, T. mucronatum and T. distichum were all plantation trees with  $3 \text{ m} \times 2 \text{ m}$  spacing. They were supplied by

the Institute of Botany, Chinese Academy of Sciences. All samples were 11 years old, and were harvested in Yancheng, Jiangsu province, China. The sampling and preparation of the wood for analysis was performed according to TAPPI standard T257 sp-14 (2014).

## Methods

## Chemical composition analysis of the wood

For the determination of the ash content, water extract, and benzene-alcohol extractive, the procedures outlined in TAPPI standards T211 om-12 (2012), T207 cm-08 (2008), and T 204 cm-07 (2007) were used, respectively.

The lignin and polysaccharides contents were analyzed according to the Laboratory Analytical Procedure from the National Renewable Energy Laboratory (Sluiter *et al.* 2010). The hydrolysate from the acid hydrolysis was used to analyze the sugars and acid-soluble lignins (ASL). The sugar contents were measured *via* a high-performance liquid chromatography (HPLC) system equipped with an Aminex HPX-87H column (300 mm  $\times$  7.8 mm) and a refractive index (RI) detector; a 5 mM H<sub>2</sub>SO<sub>4</sub> solution was used as the eluent at a flow rate of 0.6 mL/min. The acid soluble lignin content was determined *via* absorbance at 205 nm with a UV-Vis spectrometer (TU-1810, Puxi, Beijing, China). The Klason lignin (KL) content was taken as the ash residue after acid hydrolysis. All experiments were tested using three replications.

## Fiber morphology analysis

The wood sticks (1 mm  $\times$  2 mm  $\times$  30 mm) were obtained by cutting small wood block with a sharp blade and were soaked in an acetic acid and hydrogen peroxide (30%  $\pm$  0.1%) solution (1 to 1 by vol) at a temperature of 60 °C for 48 h. After thoroughly washing, 40 mg of oven-dried fibers were dispersed in 1000 mL of water. The fiber length, width, coarseness, and fines content were measured *via* a Morfi Compact fiber morphology analyzer.

### Determination of the wood density

The wood samples were cut into 20 mm segments. The water-saturated volume and oven dry mass of the segments were measured. The basic density was defined as the ratio of the oven-dry mass to the water-saturated volume.

### Cooking

A digester with an oil bath was used with a batch capacity of oven dry wood chips (20 mm  $\times$  15 mm  $\times$ 2 mm) of 80 g. The wood chips were directly subjected to cooking using sodium hydroxide and sodium sulfide with total titratable alkali (TTA) charge of 19% (as Na<sub>2</sub>O) and sulfidity of 25% at a temperature of 170 °C for 90 min. After cooking, the pulp was thoroughly washed and dewatered *via* centrifugation, then refrigerated for usage.

## Oxygen delignification

Oxygen delignification was carried out in an electric rotary digester with a water bath. The conditions were as follows: an oxygen pressure of 0.7 mPa, a NaOH dosage of 2.5%, a MgSO<sub>4</sub> dosage of 0.1%, and a pulp consistency of 10%, at a temperature of 100  $^{\circ}$ C for 60 min.

## Bleaching

Each stage of the  $D_0ED_1$  bleaching process were carried out in plastic bag with a water bath. The conditions of each stage are shown in Table 1.

	Pulp Consistency (%)	Temperature (°C)	Time (min)	NaOH Dosage (%)	ClO <sub>2</sub> Dosage (%)	pН
D <sub>0</sub>	8	70	90	-	1.5	2.5 to 3.5
E	8	70	90	1.25	-	-
D <sub>1</sub>	8	70	120	-	2.0	3.5 to 4.5

Table 1. Bleaching Conditions of the T.Z.502 Oxygen Delignified Pulp

# **RESULTS AND DISCUSSION**

## **Density of the Wood Samples**

The tree shapes were measured, and the wood densities of the T.Z.502, T.Z.302, and T.Z.118 samples, as well as their parents, T. distichum and T. mucronatum, were determined and listed in Table 2.

Although the five trees were basically the same age and had the same growth environment, the apparent size of the individuals varied. The tree height and diameter at breast height of the *T.Z.502* trees were similar to its parents, while the *T.Z.302* and *T.Z.118* trees had greater tree heights and diameters compared to *T.Z.502*. The basic density of the three *T*. 'Zhongshansa' ranged from 0.28 kg/m<sup>3</sup> to 0.30 kg/m<sup>3</sup>, which was slightly lower than the basic density of their parents. All the three *T*. 'Zhongshansa' species are hybrids of *T. distichum* and *T. mucronatum*, and the differences in appearance between the trees might be due to the different microenvironments in which they grew. Based on the average of the three trees it can be concluded that *T*. 'Zhongshansa' wood hybrids have certain advantages over than their parents, as with other hybrid trees, *e.g.*, *Liriodendron* (Rehfeldt 1977) and Douglas-fir (Pan *et al.* 2014).

Table 2. Appearance and Wood Density of <i>T. 'Zhongshansa</i> ' and their Parent
Woods

Species Height (m)		Diameter at Breast Height (cm)	Basic Density (kg/m <sup>3</sup> )				
T.Z.302	11.3	30.2	0.29 ± 0.01				
T.Z.118	10.7	24.8	0.28 ± 0.01				
T.Z.502	9.4	19.5	0.30 ± 0.01				
T. distichum	9.0	17.5	0.34 ± 0.01				
T. mucronatum 9.8		21.8	0.33 ± 0.01				

# Fiber Morphology

Previous studies have indicated that the fiber length values measured *via* a fiber quality analysis (FQA) were lower than the values measured *via* microscopy, either the

arithmetic average length  $(L_n)$  or the weighted average length  $(L_w)$ . The  $L_n$  especially was considerably lower due to the influence of fines content, whereas the weight weighted average length  $(L_{ww})$  was closer to the value measured *via* microscopy. Table 3 shows the fiber morphology of the three *T*. 'Zhongshansa' trees and their parents.

The  $L_{ww}$  values of the three *T*. 'Zhongshansa' trees were longer the  $L_{ww}$  of the *T*. *mucronatum* and *T*. *distichum* trees, with *T.Z.118* having the longest (2.02 mm). The fiber width of the three *T*. 'Zhongshansa' trees were similar, and all were close to the fiber width of *T*. *distichum* and slightly larger than the fiber width of *T*. *mucronatum*. However, the length to width ratios of the three hybrids were greater than the length to width ratios of *T*. *mucronatum* and *T*. *distichum*. Especially *T.Z.118*, which had the highest length to width ratio (46), was higher than its parent, either *T.Z.302* or *T*. *mucronatum*. The fiber width of the three *T*. 'Zhongshansa' trees were greater than the fiber width of their parents, but the cell wall thickness (Table 3) did not increase proportionally; all three hybrids had a lower cell wall thickness compared to *T*. *mucronatum* and *T*. *distichum*. This indicated that the *T*. 'Zhongshansa' species had a smaller Runkel ratios (Table 3), which is why the *T*. 'Zhongshansa' species have lower wood densities (Table 2). Similar phenomena have occurred in other tree species (Xu *et al.* 2002). However, a smaller Runkel ratios for fiber is conducive to the physical strength when they are used for papermaking.

## **Chemical Composition**

The chemical compositions of the *T*. 'Zhongshansa' hybrid woods and parents are listed in Table 4. The total polysaccharide contents of the three *T*. 'Zhongshansa' woods were similar, all were approximately 60% of the dried weigh of wood, while the lignin contents (31% to 32%) were slightly lower than their parents. The lignin content of *T.Z.302* wood was 31.9%, which was lower than the lignin content of its parentals; the lignin content of *T. mucronatum* and *T. distichum* 34.1% and 33.8%, respectively. However, the lignin content of the *T.Z.118* wood was close to that of its parent *T.Z.302*. The water soluble extractive contents of the three *T*. 'Zhongshansa' woods were similar and all were close to the water soluble extractive contents of *T. distichum* and slightly higher than *T. mucronatum*. However, all the benzene-alcohol extractive contents were lower in the three hybrids compared to their parents. The 1% NaOH extractive contents of *T.Z.302* and *T.Z.118* were all considerably lower than the parents, while the *T.Z. 502* 1% NaOH extractive content was slightly higher, somewhere between the parents.

The ash content of three T. 'Zhongshansa' woods were higher than the ash contents of their parents, more than twice that of T. *distichum*. The three T. 'Zhongshansa' trees used in this work were all grown in the same saline-alkali environment, and there was no yellowing phenomenon, which meant that T. 'Zhongshansa' can assimilate inorganic salt ions from the soil during the growth process, which is the reason T. 'Zhongshansa' had a higher ash content.

The lignin content of the three *T*. 'Zhongshansa' hybrids were similar and close to the lignin contents of *Cryptomeria fortunei* Hooibrenk (34.2%) and *Cunninghamia lanceolata* (33.5%), but higher than the lignin contents of *Picea jezoensis* var. (28.5%) and *Picea koraiensis* Nakai (27.0%), which are also grown in China (An *et al.* 1993; Lu *et al.* 1994). As the primary substance of wood growth, lignins maintain the firmness of the trunk. However, a higher lignin content will lead to lower chemical pulping yields, because lignins are hard to degrade and remove from the raw material during the cooking and bleaching process.

	Fit	per Length (r	nm)			Length to Width Ratio					< 0.2 mm
Species	Arithmetic average	Weighted average	Weight weighted average	Fiber Width (µm)	Coarseness (mg·100m <sup>-1</sup> )	Arithmetic average	Weighted average	Weight weighted average	Cell Wall Thickness (µm)	Runkel ratio	Fines Content (%)
T.Z.302	1.30	1.80	1.64	44.12	16.36	29	41	37	4.59	0.26	0.44
T.Z.118	1.26	1.79	2.02	43.68	20.16	29	41	46	4.22	0.24	0.37
T.Z.502	1.19	1.62	1.72	43.37	17.22	27	37	40	5.26	0.32	0.40
T. distichum	1.51	2.00	1.35	43.30	13.47	35	46	31	5.52	0.34	0.46
T. mucronatum	1.17	1.54	1.31	40.22	13.09	29	38	33	5.39	0.37	0.39

Table 3. Fiber Morphology of T. 'Zhongshansa' and Their Parents Woods	
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\* Runkel ratio = 2 \* cell wall thickness / cell cavity diameter

Table 4. Main Chemical Composition of *T.* 'Zhongshansa' and Their Parents Woods

		Extractive (%)				Lignin (%)			Polysaccharides (%)			Tatal	
Species	Ash (%)	Benzene- alcohol	Cold water	Hot water	1% NaOH	Klason	Acid- soluble	Total	Glucan	Other Polysaccharides	Sum	Total balance (%)	
T.Z.302	0.84 ± 0.04	2.48 ± 0.18	2.63 ± 0.04	4.08 ± 0.14	13.61 ± 0.03	31.24 ± 0.17	0.62 ± 0.03	31.86	38.73 ± 0.36	22.14 ± 0.20	60.86	96.05	
T.Z.118	0.71 ± 0.03	2.13 ± 0.21	2.88 ± 0.11	4.54 ± 0.03	13.27 ± 0.01	31.55 ± 0.11	0.56 ± 0.02	32.11	37.05 ± 0.50	19.79 ± 0.09	56.84	91.79	
T.Z.502	0.82 ± 0.04	2.02 ± 0.25	2.70 ± 0.06	3.94 ± 0.09	16.36 ± 0.01	30.84 ± 0.34	0.51 ± 0.03	31.38	37.98 ± 0.37	20.1 ± 0.20	58.09	92.29	
T. distichum	0.33 ± 0.02	2.76 ± 0.06	2.72 ± 0.11	4.84 ± 0.05	17.45 ± 0.02	33.03 ± 0.19	0.76 ± 0.01	33.79	38.91 ± 0.23	19.63 ± 0.13	58.54	95.42	
T. mucronatum	0.60 ± 0.02	2.56 ± 0.10	2.28 ± 0.07	4.00 ± 0.10	15.02 ± 0.01	33.33 ± 0.10	0.79 ± 0.03	34.12	38.55 ± 0.16	21.57 ± 0.19	60.12	97.40	

Pulping Performance Index	Results				
Cooking yield (%)	$44.9 \pm 0.0$				
Kappa number	41.8 ± 0.3				
Residual effective alkali (g/L as Na <sub>2</sub> O)	3.6 ± 0.5				
Kappa number of the oxygen delignified pulp	17.0				
Oxygen delignified rate* (%)	59.3				
Yield of oxygen delignification (%)	89.8				
Brightness of ECF bleached pulp (% ISO)	76.8				
Post color number	0.51				
Degree of polymerization of bleached pulp	902				
Note: * Oxygen delignification rate = (Kappa No. of KP- Kappa No. of oxygen delignified pulp) / Kappa No. of KP pulp					

## Table 5. Pulping Performance of T. 'Zhongshansa' 502

## Pulping Performance Evaluation of T.Z.502

Given the superior characteristics in terms of growth and resistance to red blight, *T.Z.502* has high productivity (Yu *et al.* 2011), and therefore was evaluated for pulping performance in this work. Under the conditions of a TTA charge of 19% (as Na<sub>2</sub>O) and a sulfidity of 25% at a temperature of 170°C for 90 min, the cooking results are listed in Table 5.

The pulp yield was 44.9% when Kappa number of the pulp was decreased to 41.8. The kraft pulp was subjected to oxygen delignification, and the kappa number of the pulp decreased to 17.0; the corresponding overall yield was 40.4%, while the extent of oxygen delignification was 59.3%. The oxygen delignified pulp was bleached using the DED process with a chlorine dioxide usage of 3.5% (on oven dried pulp). The resulting pulp brightness reached up to 76.8% ISO, and the brightness stability was a PC (post color) number of 0.51. After bleaching, the DP of the pulp was 902.

The lignin content of T. 'Zhongshansa' wood is similar to the lignin content of *Liushan* wood. *Liushan* is also a species of fir grown in China, whose lignin content is close to that of T. 'Zhongshansa', which has also been evaluated for good pulping performance (An *et al.* 1993). Under the same cooking conditions as above, the pulp kappa number of *T.Z.502* (41.8) was much higher than the pulp kappa number of *Liushan* wood, which was approximately 33. The kraft pulp of *T.Z.502* is easily delignified *via* oxygen with alkali and has a good delignification selectivity, given the high yield of 96%. Since the kappa number of the oxygen delignified pulp was still as high as 17, the brightness only reached 76% after the ECF bleaching process, which is obviously not high enough for usage as a chemical pulp.

Of course, by strengthening the cooking conditions, *e.g.*, increasing the alkali usage, the kappa number of the kraft pulp can be further reduced, which needs to be further studied. However, the wood density of T. 'Zhongshansa' is relatively low, cooking conditions that are too intense may result in a lower cooking yield, which is not economical. Although the kappa number of T.Z.502 kraft pulp is relatively high at the acceptable cooking yield, and the brightness of the bleached pulp is not particularly high, it does not impact the application of the pulp in paperboard and some paper products requiring normal brightness. Based on the characteristics of the relatively low wood density of T. 'Zhongshansa', chemical mechanical pulping may be more suitable for it, which will be studied as the next step.

# CONCLUSIONS

- 1. The morphological characteristics of the wood and fibers of three hybrid *Taxodium* 'Zhongshansa' woods and their parents, *T. mucronatum* and *T. distichum*, were analyzed and compared. The wood densities of the three *T*. 'Zhongshansa' hybrids were slightly lower than the wood densities of *T. mucronatum* and *T. distichum*. The fiber length and length to width ratio of both *T. Z.302* and *T. Z.502* were similar and greater than their parents, while *T.Z.118* was greater than its parent, *T.Z.302* and *T. distichum*. The cell wall thicknesses of the three *T*. 'Zhongshansa' hybrids were thinner than their patents and therefor had a greater cavity to diameter ratio.
- 2. Compared with the parents, the extractives contents of the three *T*. 'Zhongshansa' hybrids were not much different. The lignin contents of the three *T*. 'Zhongshansa' hybrids were slightly lower than their parents.
- 3. At the acceptable cooking yield, the kappa number of the *T.Z.502* kraft pulp was higher than the kappa number of the softwood, which currently used in the pulping industry. The *T.Z.502* kraft pulp was easier to delignify *via* oxygen with alkali, but it was harder to reach a higher brightness *via* the ECF bleaching process. *T.Z.502* can be used for pulping, which is used in the production of paperboard and certain paper products whose high brightness is not a particular requirement.

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