# FIBER MORPHOLOGY, CHEMICAL COMPOSITION, AND PULPING OF NINE INTRODUCED POPLAR CLONES GROWN IN BEIJING, CHINA

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**Abstract.** Evaluating the wood properties of poplar clones to be used in fast-growing and high-yield plantation and select superior clones are critical to increase both quantity and quality of wood production. This study determined the fiber morphology, chemical composition, and pulping of nine introduced poplar clones after six growing seasons and assessed their suitability for pulping and papermaking. Results showed that the fiber morphological differences among nine clones were not obvious. Fiber with length less than 1.0 mm accounted for 91.7% of the total fibers which were mainly short. *Taro* had longer and thinner fibers with the largest length–width ratio, followed by *Bellotto*, whereas *Lambro* owned the shortest and thickest fiber with a small length–width ratio. *Neva* had the Runkel ratio much high as 1.0, which was larger than that of other eight clones. From the point of view of chemical composition, *Taro* contained low content of ash, cold/hot water–soluble content, benzene ethanol–soluble content, and lignin, but higher content of holocellulose. 1% sodium hydroxide–soluble and pentosan contents were 20.47% and 22.62%, respectively, on average; thus, *Taro* can be suggested as good-quality industrial material applied in pulping and papermaking. On the contrary, *Bellotto* got imperfect overall performance, which was considered comprehensively before selection.

*Keywords:* Populus  $\times$  canadensis, fiber morphology, chemical composition, pulping properties, clonal evaluation.

#### INTRODUCTION

Poplar (*Populus* L.) is one of the important afforestation trees in China because of its fast growth, ease for clonal propagation, strong heterosis on interspecific hybridization, wide adaptability, and large wood production. Increasing demand of timber, pulp, and wood products is driving the forest industry toward short rotation

cropping of Populus hybrids that can grow on

marginal soils (Law et al 2000; Balatinecz et al 2001). Poplar can be used for different forms of processing in timber and fiber industry (Zhang et al 2003; Balatinecz et al 2014), in which it is considered to be the best hardwood raw material for papermaking and planted through the temperate central area of China, with its plantation more than  $8 \times 10^6$  hm<sup>2</sup> (FAO 2016). Poplar breeding program in China had in the past concentrated on

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selection for such features as growth rate, stem form, antireversibility, parental genetic improvement, and molecular biological breeding. However, recently, there has been renewed interest in selection based on wood quality evaluation such as basic density, fiber characteristics, and chemical composition (Xi et al 2009; Su et al 2010). Therefore, the objective of this study was to investigate the fiber morphology, chemical composition, and pulping properties of nine introduced *Populus* hybrid clones and to evaluate their characteristics as optimal raw material for pulping and papermaking in further study.

The yield and quality of pulp and paper products are largely influenced by the fiber morphology and chemical composition of wood. As the main component of both raw material of pulping, papermaking (Balatinecz et al 2014), and plant cytoskeleton, the morphological structure of fiber directly affects the pulping properties and paper forming strength. Chemical composition is the fundamental basis for determining the advantages and disadvantages of pulping and papermaking, which is closely related to the production and quality of pulp and paper products (Law et al 2000; Balatinecz et al 2001; Sable et al 2017). It was suggested that the wood from different growing seasons of the same poplar clone took little effect on pulping and papermaking, but the intraclonal variation of chemical composition was significant (Dickson et al 1974; Šefc et al 2009).

To the best of our knowledge, less reports available were devoted to the chemical composition, fiber morphology, and pulping of these wood species. Therefore, the purpose of this study was to analyze the chemical compositions and to investigate the fiber morphology properties and pulping of nine introduced *Populus* hybrid clones. From this study, we can assess the potential of these clones in pulping and papermaking.

#### MATERIALS AND METHODS

### Materials

Study site. The experiment was conducted in the western Xinfeng village, Cuicun town, Changping District, Beijing, China (40.34°N, 116.58°E). The climate is temperate continental, with hot, rainy summer and cold, dry winter (average temperature 11.8°C and coldest temperature -19°C). Annual rainfall and sunshine hours were 550.3 mm and 2684 h, respectively. The pH of sandy roam soil was 7.8.

**Plant materials and study design.** Nine clones of P. × *canadensis* introduced from Italy and Portugal were selected for the clonal trial in Beijing, China (Table 1). Trees with 1-yr-old stem and 2-yr-old root of each clone were planted in 2005 with 3 m × 4 m tree spacing. The experimental design of the clonal trial was a complete randomized design with three blocks, which was arranged with 15-30 trees in each plot.

*Sampling.* At the end of the 6th growing season, the sample tree was collected of each clone based on the mean of diameter at breast height (DBH) and total tree height in the clonal trial. The single trees close to the mean of DBH and height and with good form and vigor were selected for each block of each clone before

Table 1. Nine clones of P.  $\times$  canadensis for the study.

No.	Clone name	Species name	Male/Female	Country of origin
1	Brenta	$(P. \times canadensis \text{ cv. 'Brenta'})$	Ŷ	Italy
2	Lambro	$(P. \times canadensis \text{ cv. 'Lambro'})$	ð	Italy
3	Mella	$(P. \times canadensis \text{ cv. 'Mella'})$	Ŷ	Italy
4	Por	$(P. \times canadensis \text{ cv. 'Por'})$	Ŷ	Portugal
5	Taro	$(P. \times canadensis \text{ cv. 'Taro'})$	ð	Italy
6	Timavo	$(P. \times canadensis \text{ cv. 'Timavo'})$	ð	Italy
7	Neva	$(P. \times canadensis \text{ cv. 'Neva', cl. '74/76'})$	Ŷ	Italy
8	Guariento	$(P. \times canadensis \text{ cv. 'Guariento'})$	Ŷ	Italy
9	Bellotto	$(P. \times canadensis \text{ cv. 'Bellotto'})$	Ŷ	Italy

sampling. Three trees were sampled for each clone and 27 trees in total were destructively sampled in the study.

Each sample tree was cut at the ground level, and discs (about 50 mm thick) were collected from DBH in height for all sample trees after felling. The discs were marked with the north–south orientation, the cortex was removed, and transported to the laboratory for measurement.

### Methods

Measurement of fiber morphology. To evaluate the fiber morphology on pulping property, the test strips through the pith were split and cut into a match stick size (30 mm  $\times$  2.5 mm  $\times$ 2.5 mm) manually. The dimensions of cells were measured in samples treated with acetic acid: 10% hydrogen peroxide 1:1 (v/v) at 60°C for 24 h with the intention of cell separation. When the samples turned to white color, the separated fibers were taken off the reactor and thoroughly washed with water. Then, the dissociated fiber was stained with Herzberg reagent on the slides. For fiber lumen diameter and cell wall thickness determination, cross-sections were obtained from the same height/length as described earlier. The optical microscopy observations were made in 30 samples each transverse section, radial section, and tangential section of the stalks. The sections were cut to 20 µm thickness with a sliding microtome. They were stained with Safranin O and mounted in glycerol. Fiber shape and inclusions were observed under a light microscope. A sample of 30 fibers was evaluated to determine the number of replicates it would be necessary to obtain and measure the fiber length, width, and lumen diameter.

*Chemical composition analysis.* A small portion of chips was ground, and the 40-60 mesh fractions were selected to determine the chemical composition. Chemical composition in these samples such as the content of lignin, holocellulose, 1% sodium hydroxide–soluble content, cold/hot water–soluble content, pentosan, and ash were determined in accordance with respective China standard method for the papermaking industry, GB/T 2677-1993 (Table 2); 2 (dryness), 3 (ash), 4 (cold/hot water soluble), 5 (1% sodium hydroxide soluble), 6 (phenyl ethanol soluble), 8 (lignin), 9 (pentosan), and 10 (holocellulose).

**Pulping process.** For pulping experiments, all materials were disintegrated manually up to the approximate size of industrial wood chips (2 cm length; 1 cm width; 0.2 cm depth). The pulp was measured as whiteness according to the Chinese standard method for the papermaking industry, adopted Diff-Geometry Method, GB/T 7974-2002 (Table 2), using a WSB-3A brightness meter (Xinrui Instrument Co., LTD, Shanghai, China).

### **RESULTS AND DISCUSSION**

#### Fiber Morphology

*Fiber length and width.* The mean fiber length and width of nine clones are shown in Table 3.

Table 2. Analysis of inspection contents and standards.

Analysis and determination item	Determination of character	Standard method		
Fiber morphology	Fiber composition	GB/T4688-2002		
1 00	Fiber length	GB/T10336-2002		
Chemical composition	Dryness	GB/T2677.2-1993		
-	Ash	GB/T2677.3-1993		
	Cold/hot water soluble	GB/T2677.4-1993		
	1% sodium hydroxide soluble	GB/T2677.5-1993		
	Phenyl ethanol soluble	GB/T2677.6-1994		
	Lignin	GB/T2677.8-1994		
	Pentosan	GB/T2677.9-1994		
	Holocellulose	GB/T2677.10-1995		
Pulping	Pulp whiteness	GB/T7974-2002		

Clone name	Average length (mm)	Average width (µm)	Length-width ratio	Maximum length (mm)	Minimum length (mm)
Taro	0.603	21.3	28.3	2.2461	0.2514
Mella	0.633	24.4	25.9	2.4730	0.2525
Brenta	0.585	22.7	25.8	2.1757	0.2522
Timavo	0.591	22.6	26.2	2.0037	0.2502
Neva	0.536	21.0	25.5	2.2270	0.2533
Guariento	0.552	21.4	25.8	2.2218	0.2529
Lambro	0.486	21.0	23.1	1.7739	0.2531
Por	0.578	23.7	24.4	2.1012	0.2517
Bellotto	0.587	21.7	27.1	2.2370	0.2529
Average	0.572	22.2	25.8	2.1620	0.2520

Table 3. Fiber morphology of nine clones of P. × canadensis

The average fiber length, width, and ratio of fiber length to width are 0.572 mm,  $22.2 \mu \text{m}$ , and 25.8. The average maximum and minimum fiber length are 2.162 mm and 0.252 mm, respectively.

In the pulping and papermaking process, fiber length plays a decisive role in paper tearing, tensile strength, breakage resistance, and folding resistance, which directly affects the quality of pulp and paper products. Short fibers, such as average length under 0.4 mm, are not suitable for papermaking. In this study, the average fiber length and width of nine clones range from 0.486 mm to 0.633 mm and 21.0 mm to 24.4 mm, respectively. The maximum fiber length appears in the *Mella* (2.4730 mm), followed by *Bellotto* (2.2461 mm). The average fiber width of both *Lambro* and *Neva* is the smallest (21.0  $\mu$ m). The difference among other clones is not significant.

It is comprehensive to consider both the fiber length and the ratio of fiber length to width. However, the most suitable fiber length–width ratio gave different views. In general, the fibers with large length–width ratio are interwoven and connected with each other closely, which can make high paper strength as well as improve the folding and tearing resistance. On the contrary, the fibers with small length–width ratio are not suitable enough for pulp beating and finally get the paper products of poor quality (Balatinecz et al 2001). The results of this study show that *Taro* has the largest length–width ratio (28.3), which is the most conducive clone for pulp and papermaking, followed by *Bellotto* (27.1), whereas the smallest is *Lambro*, 18.4% smaller than that of *Taro*.

In Table 4, the frequency distribution of fiber length showed that fiber length concentrates under 0.5 mm and 0.5 mm to 1.0 mm, accounting for 91.65%. In addition, there is a frequency distribution in the range from 1.0 mm to 1.5 mm, among which *Mella* has the largest frequency (12.56%), whereas *Lambro* has no distribution in

Table 4. Fiber length distribution of nine clones of P. × canadensis.

	Frequency of fiber length distribution (%)								
Clone name	<0.5 mm	0.5-1.0 mm	1.0-1.5 mm	1.5-2.0 mm	>2.0 mm				
Taro	44.91	43.38	10.85	0.76	0.10				
Mella	42.33	44.33	12.56	0.66	0.12				
Brenta	49.20	40.3	9.89	0.57	0.05				
Timavo	45.20	45.79	8.72	0.27	0.02				
Neva	53.86	40.24	5.76	0.72	0.07				
Guariento	49.53	43.95	6.32	0.17	0.02				
Lambro	59.96	37.32	2.57	0.15	0.00				
Por	45.39	47.26	7.12	0.20	0.03				
Bellotto	43.72	48.24	7.77	0.22	0.05				
Average	48.23	43.42	7.95	0.41	0.05				

the range of more than 2.0 mm. According to the relevant regulations of the International Society of Wood Anatomy, the fibers with length less than 0.90 mm are defined as short fibers, those with length more than 1.60 mm are long fibers, those between them become intermediate fibers, and the fibers with length more than 3.00 mm reach the level of extremely long fibers (Wu et al 2011). Therefore, short fibers are dominant among the nine clones in this study.

*Fiber lumen diameter and double wall thickness.* In Table 5, the mean of lumen diameter, double wall thickness, and Runkel ratio of nine clones are 14.74 µm, 9.68 µm, and 0.67, respectively.

Fiber Runkel ratio is the ratio of fiber double wall thickness to lumen diameter. Its role in pulp and papermaking is mainly used to measure the pulp yield (Ona et al 1997; Xu et al 2006), as well as related to paper conformability (Zhou et al 2005; Wu et al 2010). Generally, the fibers with small Runkel ratio are easily interwoven together because of its thin wall and large cavity, and convenient to the rapid forming of the paper, which makes the paper more flexible. The fibers with thick wall and small cavity have great tensile strength and breaking strength, but their tearing index is gradually decreased. The results of this study showed that the lumen diameter and double wall thickness of nine clones range from 12.00 µm to 16.86 µm and 8.1 µm to 11.94 µm, respectively. Neva has the smallest lumen diameter and the largest double wall thickness. As for their ratio (Fig 1), Por has the smallest (0.5), followed by *Bellotto* (0.53). In addition, the general view is that the ratio less than 1 can be classified as superior papermaking materials; medium papermaking materials with a ratio equal to 1; for inferior papermaking materials, the ratio was often greater than 1. According to this, except *Neva* whose ratio is equal to 1, other clones are all excellent superior papermaking materials.

### **Chemical Composition**

The chemical composition of nine clones is shown in Table 6. As expected, holocellulose was the major components of the samples. Based on data, the samples used in this study have no significant difference among their chemical composition. It showed that the mean content of moisture and ash of the nine clones are 91.33% and 0.55%, respectively; the mean content of cold water-, hot water-, 1% sodium hydroxide- and benzene ethanol-soluble contents are 1.91%, 3.30%, 22.15%, and 2.07%, respectively; and the mean contents of lignin, holocellulose, and pentosan are 21.22%, 83.48%, and 23.24%, respectively.

The dryness of nine clones ranges from 90.46% (*Mella*) to 91.92% (*Bellotto*), which indicates that the energy dissipation in the pulping process is relatively high, making the fibers intertwined tightly (Khampan et al 2010). The resistance to the external adverse reaction is great, which makes the final paper excellent.

The ash content ranges from 0.49% (*Taro*) to 0.61% (*Guariento*), all of which are lower than

Clone name	Lumen diameter (µm)	Double wall thickness (µm)	Runkel ratio
Taro	15.27	10.74	0.70
Mella	16.86	10.50	0.62
Brenta	15.22	9.36	0.61
Timavo	14.09	10.23	0.73
Neva	12.00	11.94	1.00
Guariento	15.12	9.60	0.63
Lambro	12.36	8.40	0.68
Por	16.26	8.10	0.50
Bellotto	15.45	8.25	0.53
Average	14.74	9.68	0.67

Table 5. Fiber lumen diameter, double wall thickness, and Runkel ratio of nine clones of P.  $\times$  canadensis.

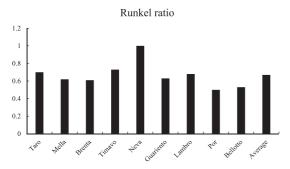


Figure 1. Runkel ratio of nine clones of P. × canadensis.

1%, within the high quality range. The amount of ash content reflects the amount of inorganic substances, miscellaneous cells, and dust contained in wood. Although the ash content is not large, it often has an adverse impact on pulping, bleaching, and other processes, such as silicon interference in the recovery of black liquor. It means that the lower the ash content, the better the quality.

The amount of cold water-, hot water-, and benzene ethanol-soluble contents is low and not significantly different among the nine clones. The soluble content of *Mella* and *Bellotto* are slightly higher than that of other clones, which indicates that its resin, water-soluble sugar, plant alkali, tannin, and pectin, including some organic solvents, are relatively lower. The amount of 1% sodium hydroxide–soluble content is high, among which *Bellotto* has the largest (23.79%), indicates that more holocellulose (pentosan, aldonic acid, etc.) are easy to be degraded and

dissolved in low-alkali solution. High soluble content (cold/hot water, 1% sodium hydroxide, and benzene ethanol) will have negative effects on pulping and papermaking, which makes more consumption of chemical reagents in the industrial process, delays the cooking process, reduces the pulp yield, and is easy to cause resin barrier.

Lignin has an adverse effect on pulp yield, which makes it harder to be cooked and bleached. Therefore, removing the corresponding requirements of lignin according to the different applications of pulp and paper products in the pulping process is necessary. In this study, the lowest lignin content was obtained from *Taro* (20.38%), whereas the highest from *Neva* (21.86%).

Typical hardwood holocellulose content was 74% (Long 1980). According to Table 6, the holocellulose content of all clones are higher than 74%, with the average value of 83.48%. *Taro* has the highest value of 84.48%, 13% higher than the average value, followed by *Por* (83.97%).

Pentosan content has a certain relationship with the pulp yield in the cooking process, which indicates the retention or loss of hemicellulose during pulping and bleaching processes. High pentosan content not only leads to high pulp yield but also improves the compactness of the paper because the holocellulose contributes to the strength of paper pulps. In this study, the pentosan content of the nine clones ranges from 21.67% to 24.46%, with the largest from *Lambro* (24.46%) and the smallest from *Neva* (21.67%).

Table 6. Chemical composition of nine clones of P. × canadensis.

				S	oluble contents (%)				
Clone name	Dryness (%)	Ash (%)	Cold water	Hot water	1% sodium hydroxide	Benzene ethanol	Lignin (%)	Holocellulose (%)	Pentosan (%)
Taro	91.44	0.49	1.81	2.91	20.47	1.57	20.38	84.48	22.62
Mella	90.46	0.52	2.27	3.52	22.65	2.39	21.61	82.78	23.48
Brenta	91.87	0.52	1.56	2.89	22.36	2.33	20.77	83.56	23.03
Timavo	91.19	0.56	1.46	2.92	22.34	2.19	21.58	83.86	22.90
Neva	91.68	0.59	1.72	3.17	22.02	1.90	21.86	83.49	21.67
Guariento	91.40	0.61	1.80	3.36	21.91	1.87	21.82	83.46	23.67
Lambro	90.81	0.54	2.37	3.61	22.18	1.81	21.17	83.05	24.46
Por	91.20	0.54	1.78	3.11	21.66	1.97	20.71	83.97	23.89
Bellotto	91.92	0.58	2.42	4.24	23.79	2.61	21.05	82.70	23.42
Average	91.33	0.55	1.91	3.30	22.15	2.07	21.22	83.48	23.24

Table 7. Whiteness of nine clones of P. × *canadensis*.

Clone name	Taro	Mella	Brenta	Timavo	Neva	Guariento	Lambro	Por	Bellotto	Average
Whiteness (%)	57.32	54.16	59.61	55.79	58.53	57.18	56.27	55.13	60.80	57.20

### **Pulp Whiteness**

Pulp whiteness is one criterion of evaluating the properties of pulp, which is the reaction of pulp color. When the pulp whiteness is relatively low, the paper is shown as gray, but not the higher the whiteness value, the better. It is necessary to adjust the whiteness according to the utilization requirements of the paper products.

In Table 7, the average whiteness of nine clones is 57.20%, with the maximum from *Bellotto*, the minimum from *Mella*, and the remaining whiteness value fluctuates within a small range from 54.16% to 60.80%, which indicates the overall effect is good. According to the standard of raw materials for papermaking, the content range is satisfactory for pulping and papermaking, and the nine clones can be used as intermediate pulping and papermaking materials.

#### CONCLUSIONS

#### **Fiber Morphological Characteristics**

The results show that the fiber length–width ratio of *Taro* and *Bellotto* are relatively large, which makes the final paper with high strength, tearing resistance, and folding strength. On the contrary, *Lambro* and *Por* get relatively poor performance. In addition, in terms of Runkel ratio, except for *Neva*, other the eight clones can be used as topgrade papermaking materials.

#### Wood Chemical Composition

The wood ash, soluble, and lignin contents of *Taro* are relatively low, whereas the holocellulose and pentosan contents are the highest. However, *Bellotto* gets poor performance, with the highest cold and hot water-, 1% sodium hydroxide- and benzoyl ethanol-soluble contents, whereas the holocellulose content is not high. Therefore, *Bellotto* should not be chose as papermaking wood.

Integrating fibers morphology and chemical composition, fibers of *Taro* are thin and long, which own a thin wall and large cavity. In addition, it has the highest content of holocellulose and lowest soluble contents, lignin, and ash. Therefore, it can be selected as the high-quality material for papermaking. Other clones have some disadvantages in various characteristics, but the intraclonal difference is not significant. Before choosing, considering combination with other tree species will obtain higher economic benefits.

#### DISCUSSION

This study is limited to the evaluation of nine poplar clones as papermaking materials. However, the applicability and possible problems of its real application in industrial pulping and papermaking cannot completely be implemented now. Therefore, the future research should focus on the pulp and paper process, and the optimization of cooking parameters is needed for each type to obtain fibers of similar best properties. Moreover, some scholars have shown that wood quality is also greatly affected by microfibril angle (MFA). Variation in MFA in the S<sub>2</sub> layer of the cell wall among individual tracheid, and among various clones or from different position of wood stems, has been related to the strength and shrinkage properties of solid wood, as well as to the tearing strength and antiresistance (Fang et al 2004, 2006). The analysis of wood properties of poplar trees involves a wide range of knowledge, including not only biological but also physical and chemical knowledge, to provide certain theoretical basis for further research.

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