

CHEMICAL COMPOSITION, FIBER MORPHOLOGY, AND PULPING OF *P. BOLLEANA* LAUCHE

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ABSTRACT

The chemical composition and fiber morphology of the *P. bolleana* Lauche was assessed for its suitability for pulping and papermaking. The *P. bolleana* Lauche used in this study contained a low amount of lignin, but higher quantities of cellulose and hemicelluloses. It was found that morphological indices of *P. bolleana* Lauche were adequate for pulping and papermaking. The pulping of this wood was carried out using conventional kraft and soda pulping process under varying conditions. The results indicated that at higher sulfidity (24%–32%) with lower EA (14–16%), higher yields, and viscosity of pulping were obtained with an acceptable kappa number. Kraft pulp of *P. bolleana* Lauche obtained at given conditions (EA 14–16%; sulfidity 24–32%) provided paper sheets with greater tensile strength, burst index and tear index, at the same beating degree.

Keywords: *Populus bolleana* Lauche, kraft pulping, kappa number, fiber morphology, pulp properties.

INTRODUCTION

Paper consumption is continuously increasing across the world in general and in China in par-

ticular (Zhan 2002). The fact that the need of pulp fibers for papermaking is increasing could be enhanced by finding new areas of application of this vegetal fiber, such as composite materials. During the last decade, a series of studies about the possibility of using cellulose into thermoplastic matrices was published (Felix and Gatenholm 1991; Felix et al. 1994; Kim et al.

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1992; Belgacem et al. 1994; Simonsen et al. 1998). In these papers, the fact is that the substitution of classical fibers, e.g. glass fibers, could make materials easy to recycle in terms of energy recovering and with a certain economical viability, since cellulose fibers are cheap, renewable, and have comparable mechanical properties. In addition, these natural fibers do not give any residue when burned.

In many countries, the rising demand for wood will drive prices up because supplies are limited. In China, wood resource is inadequate; therefore, the wood supplied to the fiber and paper industry is also insufficient. With more than 40 years of experience, China is a leader in non-wood fiber pulping and papermaking technology. Nevertheless, due to the differences between wood and non-wood fibrous materials in fiber morphology, microstructure, and chemical components, the grade and quality of the products made by most non-wood fibers are not as good as those made from wood fibers (Zhan 2002). Poplar is considered to be the best hardwood raw material for papermaking. The availability of poplar is estimated to be 107 million m³ in China (Gao et al. 1998). *P. bolleana* Lauche, a potential wood source of poplar fiber plant, indigenous to middle Asia countries, can be grown in the eco-climatic conditions to the northeast and northwest regions of China to prevent wind erosion and control desertification. This biomass not only has a considerable ecological importance for reforestation of deserts and dry steppes, it could also provide wood, fuel, fodder, etc. (Xu et al. 2004). This wood belongs to the poplar family of hardwood and is one of the fast-growing raw materials. Cellulose comprises over 45% of cell walls of *P. bolleana* Lauche. This fast-growing wood could and should find a rational way of utilization, namely as a source of cellulosic material to help offset the growing shortage of wood resources in the pulp and paper production in China.

To the best of our knowledge, no report available was devoted to the chemical composition, fiber morphology, and pulping of this wood species. In this work, the chemical composition and

fiber morphology of *P. bolleana* Lauche was assessed for its suitability for pulping and papermaking comparing it with red pine (softwood), China poplar (*Populus gansuensis*, hardwood), and wheat straw (non-wood). The influence of cooking parameters on kappa number, pulp yield, and pulp viscosity in kraft pulping was investigated. Pulp properties and strength properties at optimal pulping conditions were also studied.

MATERIALS AND METHODS

Materials

The *P. bolleana* Lauche was supplied by Shalin Arboretum in Yikezhao League of Inner Mongolia, China. *P. bolleana* Lauche was 4 years old and was harvested in October 2002 with an average stem height of 5.5 m. The leaves and the bark were removed and only the stalks were used. To evaluate the fiber morphology on pulping behavior, a number of stems were cut into 2.5- × 2.5- × 30-mm strips manually. A small portion of chips was ground, and the 40–60 mesh fractions were selected in order to determine the chemical composition. For pulping experiments, all material was disintegrated manually up to the approximate size of industrial wood chips (~2 cm length; ~1 cm width; ~0.2 cm depth).

Chemical analysis

The analysis of the chemical composition including cellulose, hemicelluloses, lignin, extractives and ash was performed on debarked material according to the China standard methods for the papermaking industry (GB/T 2677).

Light microscopy (LM)

The dimensions of cells were measured in samples treated with acetic acid:30% hydrogen peroxide 1:1 (v/v) at 60°C for 24 h for cell separation. When the samples turned to a 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 diameter, lumen diameter, and cell-wall thickness determination, cross-sections were obtained from the same height/length as above. The optical microscopy observations were made in transverse section (TS), radial section (RLS), and tangential section (TLS) of the stalks. The sections were cut to 25- μm thickness with a sliding microtome. They were stained with Safranin O and mounted in Glycerol.

Kraft pulping process

Kraft pulp from each entry was prepared by pulping 100 g (dry weight after oven-drying at 105°C) *P. bolleana* Lauche using an 18% effective alkali (EA) and 25% sulfidity with a 4:1 liquid to solid ratio, excluding moisture. The cooking was carried out in 1-L rotating bomb digesters at a cooking temperature of 165°C, allowing 120 min to reach temperature, for 60, 90, or 120 min at the temperature given. The cooking conditions are shown in Table 4. After collecting about 150 mL of black liquor for analysis, the cooked material was separated from black liquor, followed by screening on a vibrating flat screen with 0.2-mm-wide slots. The rejects (material that was retained on the flat screen) were dried to constant weight at 105°C and expressed as percentage relative to the initial *P. bolleana* Lauche charge. The screened pulp was collected in a cotton sack. The pulp was concentrated by a centrifuge to approximately 30% solids and weighed. The accepted pulp was

collected in a plastic bag and stored in a refrigerator. The total weight of the screened pulp was recorded and a sub-sample was dried to constant weight at 105°C to determine the moisture content and calculate the screen yield. Pulp yield was calculated on the basis of o.d. feedstock and pulp. The kappa number, viscosity, and other pulp properties were measured according to China standard of GB/T 1546-1989 and GB/T 1548-1989. The pulp obtained was processed on a PFI beater in order to obtain 45°SR drainability levels (Zhan 2002).

RESULTS AND DISCUSSION

Chemical composition

As compared with wheat straw, red pine, and poplar, the chemical composition of *P. bolleana* Lauche is shown in Table 1. As expected, cellulose and hemicelluloses were the major constituents of the samples. The results of the chemical analysis showed cellulose content (48.65%) of *P. bolleana* Lauche is higher than that of both the poplar (43.24%) and wheat straw (40.40%), but lower than that of the red pine (53.12%). However, the content of hemicelluloses (21.93%) is very similar to that of poplar (22.61%), but much higher than of red pine (10.46%), and slightly lower than of wheat straw (25.56%), which ranged in that of hardwood (19–25%). Furthermore, the ratio of hemicelluloses to cellulose obtained during this study for *P. bolleana* Lauche is about 1:2, which is common to other vegetal species. This ratio is an

TABLE 1. *The chemical composition of P. bolleana Lauche as compared to poplar, wheat straw, and red pine (%)*.

| Content | <i>P. bolleana</i> Lauche | Poplar ^a | Wheat straw ^a | Red pine ^a |
|-------------------------|---------------------------|---------------------|--------------------------|-----------------------|
| Ash | 0.67 | 0.32 | 6.04 | 0.42 |
| Extractives | | | | |
| Hot-water solubles | 3.40 | 2.46 | 23.15 | 4.15 |
| Cold-water solubles | 1.53 | 1.38 | 5.36 | 2.69 |
| 1%NaOH solubles | 17.78 | 15.61 | 44.56 | 17.55 |
| Benzene-ethanol soluble | 3.62 | n/a ^b | 0.51 | n/a |
| Cellulose | 48.65 | 43.24 | 40.40 | 53.12 |
| Hemicelluloses | 21.93 | 22.61 | 25.56 | 10.46 |
| Lignin | 18.12 | 17.10 | 22.34 | 27.69 |

^a Obtained from Qiu (2001)

^b n/a, not available.

important parameter if one considers the costs (Cordeiro et al. 2004). The value of cellulose was also found higher than those obtained from other hardwoods like fast-growing poplar (*Populus maximowiczii* Henry, 43.80%) (Sun et al. 2000) and *Caragana Korshinskii* (45.12%), two new raw materials used for pulping in China (Qiu 2001).

As expected, the acid-insoluble lignin content (18.12%) was comparable to that of poplar (17.10%), whereas lignin content of wheat straw (22.34%) and red pine (27.69%) were considerably higher. The cellulose to lignin ratio obtained from *P. bolleana* Lauche samples studied was about 2.7. Ash content (0.67%) of *P. bolleana* Lauche was slightly higher than that of both poplar (0.32%) and red pine (0.42%), but much lower than of wheat straw (6.44%). In general, ash consists mainly of inorganic salts and oxide of silicon. Lower lignin and ash content meant normal alkali consumption and fewer problems at spent liquor recovery. There was also a difference in the content of extractives between the materials compared. The quantities of extractives in benzene-ethanol and water were similar to those of poplar and red pine, but much lower than those of wheat straw. Overall, the chemical analysis showed that *P. bolleana* Lauche is suitable for pulp manufacture mainly because of its relatively lower lignin and higher cellulose and hemicelluloses.

Fiber morphology

As observed under light microscopy (Figs. 1 and 2), several cell types could be distinguished. In addition to the fibers, the other cell types, such as axial parenchyma cells, ray parenchyma cells, and vessel elements, were clearly observed. Based on the measurement of various cell areas, the fibers of *P. bolleana* Lauche made up 69.6% of its total cells. The vessel content was slightly lower than of other hardwood (e.g., white birch, 25.2%), about 23.5% of the total cells. The parenchyma content was about 6.9%. The vessel elements were usually long, thin-walled and with or without pitting, and with open ends. Occasionally, there was helix vessel

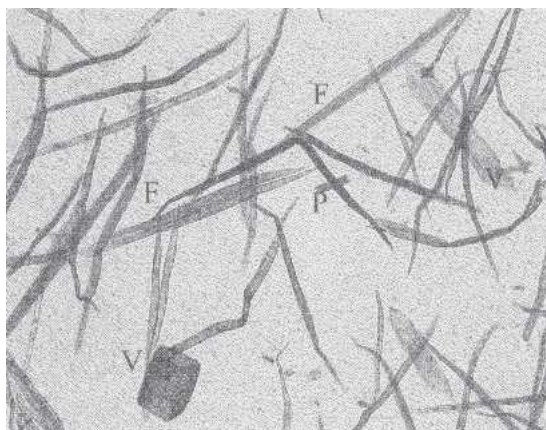


FIG. 1. Cells from the *P. bolleana* Lauche (100 \times). The different cell types identified in the pulps are F: fiber; V: vessel element; P: parenchyma.

present. The parenchyma cells were fairly uniform when compared to those of common hardwood and were similar in dimensions with most of them being claviform-shaped.

It is well known that wood with longer fiber length, higher flexibility coefficient, and/or lower wall to lumen ratio is essential in pulping and papermaking. The fiber dimensions and their derived indices are shown in Tables 2 and 3. In general, fiber dimensions are: length from 0.87 mm to 1.31 mm, and diameter from 15.3 μm to 22.3 μm . As can be seen from Table 2, *P. bolleana* Lauche retained semilong-fibered fibrous material, and the mean fiber length was 1.23 mm. The fiber cell-wall thickness (4.2 μm) was higher than that of poplar (2.4 μm), wheat straw (3.3 μm), and red pine (3.9 μm).

Table 3 gives the derived indices for *P. bolleana* Lauche and poplar, wheat straw, and red pine. Obviously, fibers of *P. bolleana* Lauche had very good derived values (especially slenderness ratio) compared to those of some softwoods and certainly to most hardwoods. The flexibility coefficient (64.1) was much higher than that of wheat straw (19.4), which is enough for pulping and papermaking. Its high flexibility is expected to have a positive effect on tensile and bursting strengths as well as on folding endurance. Furthermore, Runkel ratio (0.66) was fairly similar to poplar (0.37) and red pine

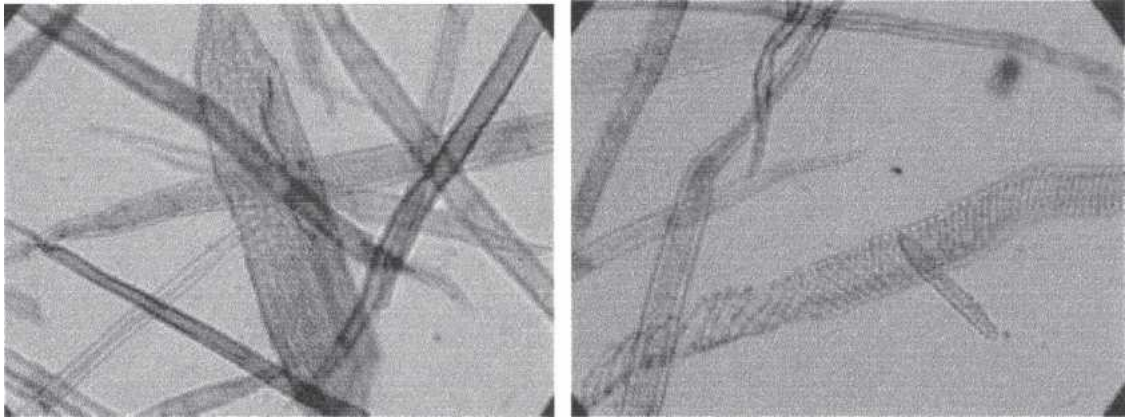


FIG. 2. Vessel types from the *P. bolleana* Lauche (400 \times). Left: pitted vessel; Right: helix vessel.

TABLE 2. *The fiber morphological characteristics of P. bolleana Lauche as compared to poplar, wheat straw, and red pine.*

| Plant material | Length (mm) | Diameter (μm) | Lumen diameter (μm) | Wall thickness (μm) |
|---------------------------|-------------------|----------------------------|----------------------------------|----------------------------------|
| <i>P. bolleana</i> Lauche | 1.23 ± 0.08^c | 19.8 ± 2.5 | 12.7 ± 2.1 | 4.2 ± 0.7 |
| Poplar ^a | 1.18 | 21.0 | n/a ^b | 2.4 |
| Wheat straw ^a | 1.32 | 12.9 | 2.5 | 3.3 |
| Red pine ^a | 3.62 | 54.3 | n/a | 3.9 |

^a Obtained from Qiu (2001).

^b n/a, not available.

^c Represent the standard deviation.

TABLE 3. *Derived values (indices) of P. bolleana Lauche as compared to poplar, wheat straw, red pine, softwood, and hardwood.*

| Plant material | Slenderness ratio | Flexibility coefficient | Runkel ratio |
|---------------------------|-------------------|-------------------------|--------------|
| <i>P. bolleana</i> Lauche | 62.1 | 64.1 | 0.66 |
| Poplar ^a | 56.2 | n/a | 0.37 |
| Wheat straw ^a | 102.3 | 19.4 | 4.4 |
| Red pine ^a | 66.7 | n/a | 0.37 |
| Softwoods ^b | 95–120 | 75 | 0.35 |
| Hardwoods ^b | 55–75 | 55–70 | 0.4–0.7 |

^a Obtained from Qiu (2001).

^b Obtained from Saikia et al. (1997).

(0.37), but significantly lower than wheat straw (4.4)(Qiu 2001). The lower Runkel ratio also indicated that fiber of *P. bolleana* Lauche should have the ability to collapse easily and form good fiber-to-fiber bonding, which is important for paper strength.

P. bolleana Lauche fibers can be considered as a semilong-fibered material with a good slenderness ratio (close to that of some hardwoods) and an acceptable Runkel ratio (0.66). Therefore, papers made from *P. bolleana* Lauche fibers are expected to have increased mechanical

strength and be suitable for writing, printing, wrapping, and packaging purposes (Saikia et al. 1997). From the result obtained, we can deduce that the morphological indices of *P. bolleana* Lauche are adequate for pulping and papermaking.

Effect of pulping conditions on pulp yield, kappa number, and viscosity

Figure 3 gives the effects of effective alkali and sulfidity on yield of the pulps, kappa num-

TABLE 4. The sulphate pulping conditions of *P. bolleana* Lauche

| | |
|--|--------------------|
| EA, on o.d. <i>P. bolleana</i> Lauche (as NaOH, %) | 14, 16, 18, and 20 |
| Sulfidity (%) | 0, 16, 24, and 32 |
| Liquor-to-solid ratio (L/kg) | 4:1 |
| Pulping temperature (°C) | 165 |
| Time from 50°C to maximum temperature (min) | 120 |
| Pulping time (min) | 60, 90, and 120 |

ber, and viscosity at the same pulping time (90 min). When Fig. 3 is examined, it can be seen that EA and sulfidity had a significant influence on kappa number. A high alkaline concentration is required to break down lignin. This was confirmed by the fact that the kappa number was lower during cooking at alkaline dosage of 20% than when cooking was performed at lower alkaline dosage of 14%. On the other hand, an increase in sulfidity at a constant EA resulted in a clear reduction in kappa number. In soda pulping (without sulfidity), it was difficult to arrive at a kappa number of 30 even though high EA (20%) were used. In addition, as indicated in Fig. 3, the screened yield of kraft pulp decreased as either EA or sulfidity increased. Screened pulp yield could be considered to be dependent on unscreened yield, rejects, and lost pulp material during screening.

Carbohydrate degradation during pulping was determined in terms of pulp viscosity. It was found that increases in EA and sulfidity within the range studied decreased the pulp viscosity (Fig. 3). During wood pulping, the viscosity of pulp generally decreases as the kappa number decreases, indicating cellulose depolymerization (Johansson et al. 1984; Sjöblom 1996). Similar relationship between viscosity and kappa number has been found in this study (Fig. 4). Alén (2000) reported that during kraft pulping, hydrogen sulfide ions reacted with lignin, and carbohydrate degradation reactions were only affected by hydroxide ions. In the present study, when the kappa number reached was lower than 19, a decrease in viscosity was obviously seen owing to enhanced delignification and carbohydrate degradation. At lower EA (14–16%) and higher sulfidity (24–32%), higher pulp viscosity and

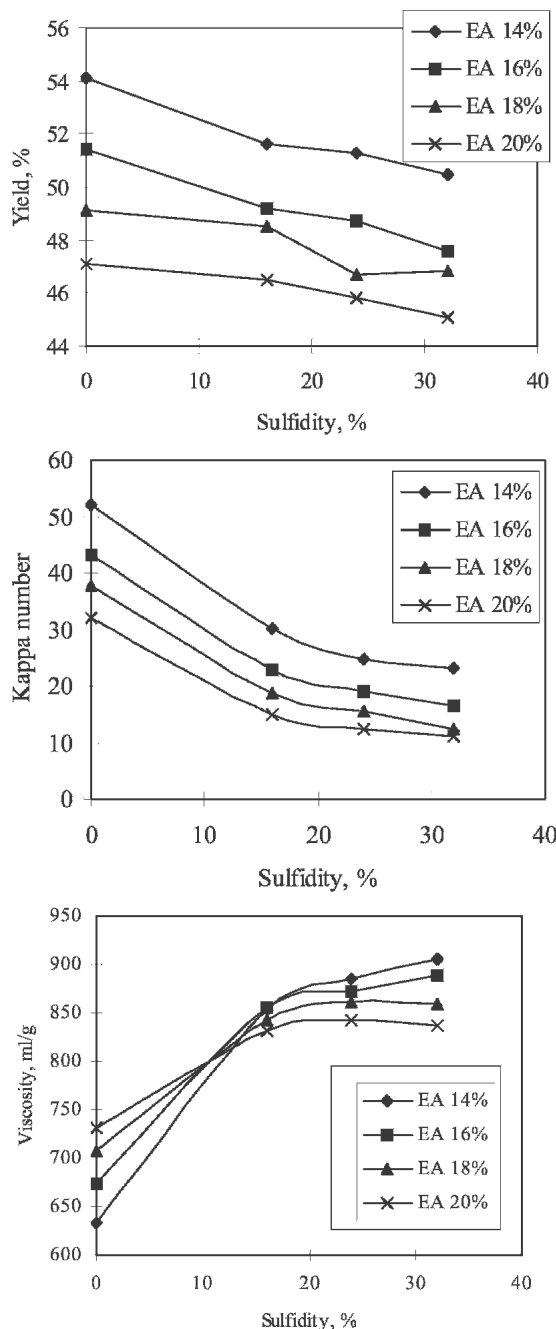


FIG. 3. Effect of EA and sulfidity on screened yield, kappa number, and viscosity of kraft pulp.

yield were obtained than those obtained under the conditions of high EA (18 and 20%) and low sulfidity (0–16%). In short, in order to obtain

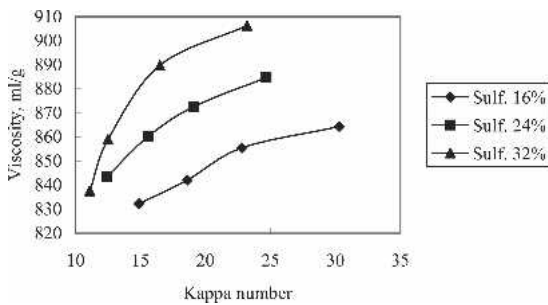


FIG. 4. The relationship between pulp viscosity and kappa number of kraft pulp at different sulfidities.

the acceptable kappa number (17–25) in the kraft pulping of *P. bolleana* Lauche, high sulfidity (24–32%) with lower EA (14–16%) increased both viscosity and yield. On the contrary, when sulfidity was lower than 16%, even at higher EA (18–20%) the viscosity and yield were lower.

Pulp properties of paper sheets produced by kraft pulping

Table 5 shows the cooking results of kraft pulping from *P. bolleana* Lauche obtained at 14–16% EA, pulping time (60–120 min), and sulfidity (24–32%). Increasing the pulping time from 60 to 120 min while keeping other pulping conditions constant had slightly affected total yield, screened yield, and viscosity of kraft pulp. Table 5 also indicates that increasing the reac-

tion time resulted in a modest decrease in the kappa number of the pulps, but at the same time induced polysaccharide degradation, as seen by the decrease of the viscosity.

From the results (Table 5) it follows that obtaining paper sheets of acceptable quality (high breaking length, stretch, burst index, and tear index) entails using effective alkali concentration (14 or 16%) and sulfidity (24% or 32%), in addition to a low liquor/wood ratio (4:1) and a medium-to-long pulping time (90 or 120 min). At the given conditions, breaking length and burst index decreased with an increase in pulping time, therefore resulting in a slight increase in tear index. As the cooking time increased from 60 to 120 min at the same amounts of EA and sulfidity, the brightness of kraft pulp increased slightly. In addition, as EA and pulping time were kept at the same values, such as EA 14% and 90 min pulping time, and when sulfidity increased from 24% to 32%, similar pulp properties and strength properties were observed. Regarding breaking length, tear index, and burst index, those of kraft pulp were higher than those of *Caragana Korshinskii* semithermomechanical pulp (CTMP) (Tian and Lu 2002), and the fast-growing hardwood (the triploid of *populus tomentosa*) APMP (Kong et al. 2003). This suggests that *P. bolleana* Lauche kraft pulp could be a potential reinforcement component in products based on mechanical pulp, such as newsprint.

TABLE 5. *The pulp properties of P. bolleana* Lauche kraft pulp obtained at optimum EA, pulping time and sulfidity.

| Sample number | EA (%) | S (%) | t | SY (%) | R (%) | TY (%) | KN | V (mL/g) | BL (m) | BI (kPam ² /g) | TI (mNm ² /g) | B (%ISO) |
|---------------|--------|-------|-----|--------|-------|--------|------|----------|--------|---------------------------|--------------------------|----------|
| 17 | 14 | 24 | 60 | 51.6 | 3.0 | 54.6 | 27.2 | 921.8 | 11087 | 7.58 | 7.39 | 27.6 |
| 18 | 14 | 24 | 90 | 51.3 | 2.9 | 54.2 | 24.7 | 884.7 | 11002 | 7.51 | 7.42 | 28.0 |
| 19 | 14 | 24 | 120 | 50.6 | 2.5 | 53.1 | 20.3 | 856.5 | 9783 | 7.43 | 7.47 | 28.2 |
| 20 | 15 | 32 | 60 | 51.7 | 2.7 | 54.4 | 25.9 | 930.4 | 10816 | 7.54 | 7.43 | 27.8 |
| 21 | 14 | 32 | 90 | 50.5 | 3.1 | 53.6 | 23.2 | 906.3 | 10665 | 7.47 | 7.48 | 28.3 |
| 22 | 14 | 32 | 120 | 50.0 | 2.3 | 52.3 | 20.6 | 881.8 | 9987 | 7.40 | 7.50 | 28.6 |
| 23 | 16 | 24 | 60 | 48.8 | 2.9 | 51.7 | 23.5 | 913.8 | 10702 | 7.45 | 7.43 | 27.8 |
| 24 | 16 | 24 | 90 | 48.7 | 2.8 | 51.5 | 19.1 | 872.6 | 9883 | 7.35 | 7.54 | 28.2 |
| 25 | 16 | 24 | 120 | 48.2 | 2.6 | 50.8 | 18.8 | 860.3 | 9801 | 7.27 | 7.58 | 28.8 |
| 26 | 16 | 32 | 60 | 48.8 | 2.4 | 51.2 | 22.4 | 900.7 | 10578 | 7.05 | 7.45 | 28.0 |
| 27 | 16 | 32 | 90 | 48.5 | 2.2 | 50.7 | 19.0 | 856.3 | 9782 | 6.87 | 7.56 | 27.8 |
| 28 | 16 | 32 | 120 | 48.1 | 1.9 | 50.1 | 18.8 | 840.6 | 9678 | 6.72 | 7.60 | 28.4 |

EA = Effective alkali; S = Sulfidity; t = Time; SY = Screened yield; R = Reject; TY = Total yield; KN = Kappa number; V = Viscosity; BL = Breaking length; TI = Tear index; BI = Burst index; B = Brightness.

In summary, the *P. bolleana* Lauche used in this study contained a lower amount of lignin but higher quantities of cellulose and hemicellulose. Although fiber of *P. bolleana* Lauche belongs to semi-long fiber, the morphological indices of *P. bolleana* Lauche are adequate to use it as feedstock in the pulping and papermaking industry. In the pulping procedure, EA and sulfidity had a significant effect on kappa number and viscosity. At a higher content of sulfidity (24%–32%) with a lower amount of EA (14–16%), higher yields and viscosity of pulping were obtained with an acceptable kappa number. It is very likely that the kraft pulp of *P. bolleana* Lauche obtained at the given conditions (EA 14–16%; sulfidity 24–32%) provided paper sheets with greater tensile strength, burst index, and tear index at the same beating degree.

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