

## ALKALINE PEROXIDE MECHANICAL PULPING OF FAST-GROWTH PAULOWNIA WOOD

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Alkaline peroxide mechanical pulping of paulownia wood harvested from exotic tree plantations in northern Iran was investigated. The fiber length, width, and cell wall thickness of this wood were measured as 0.82 mm, 40.3  $\mu\text{m}$ , and 7.1  $\mu\text{m}$ , respectively. The chemical composition including cellulose, lignin, and extractives soluble in ethanol-acetone, 1% NaOH, hot and cold water was determined as 49.5%, 25%, 12.1%, 26.9%, 11.4%, and 8.1% respectively. The ash content of this wood was 0.45%. Pre-washed chips were chemically treated at 70°C for 120 minutes with different combinations of three dosages (1.5, 3, and 4.5%) of hydrogen peroxide and three dosages (1.5, 3, and 4.5%) of sodium hydroxide prior to defibration. Other chemicals including DTPA, sodium silicate, and  $\text{MgSO}_4$  were constant at 0.5%, 3%, and 0.5%, respectively. The results showed that using a 1.5% hydrogen peroxide and 4.5% sodium hydroxide charge, the brightness of APMP pulp reached 68.7% ISO and higher chemical dosages did not improve the brightness; however, to produce APMP pulp with higher strength, a sodium hydroxide charge of 4.5% was needed. The tensile strength, tear strength, burst strength indices, and bulk density of the APMP pulp produced from 1.5% hydrogen peroxide and 4.5% sodium hydroxide were measured as 15.5Nm/g, 6.54mN.m<sup>2</sup>/g, 0.56kPa.m<sup>2</sup>/g, and 3.47cm<sup>3</sup>/g, respectively. The resulting pulp was bulky and is suitable for use in the middle layer of boxboard to provide the desired stiffness with a lower basis weight.

*Keywords; Paulownia wood; Brightness; Bulk; Strength; Hydrogen peroxide; Sodium hydroxide*

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### INTRODUCTION

Among different high yield pulping processes, chemimechanical pulping and the more recently developed alkaline peroxide mechanical pulping process (APMP) have attracted interest and have been the center point of research and development. Research has been done in the improvement of CMP pulp bleaching (Zeinaly et al. 2009), chemimechanical pulping of cotton stalks (Ali et al. 2002), and applications of poplar wood CMP pulp in the production of packaging papers as a substitute for expensive softwood or hardwood pulps (Boeva-Spiridonova et al. 2006; Boeva-Spiridonova and Petkova 2007).

APMP pulping offers various advantages, including good pulp quality, elimination of the need for a bleach plant, and energy savings (Pan and Leary 2000). Combining peroxide bleaching, chemical impregnation, and refining not only eliminates

the alkaline darkening of wood chips during chemical treatment, but it also brightens the chips to the required brightness levels suitable for writing and printing papers as well as the middle layer of white paperboard. Because pulp brightness and yield are highly dependent on the hydrogen peroxide and sodium peroxide dosage levels, a compromise should be reached to obtain a balance between brightness gain and yield loss. APMP has been developed for low-density, bright hardwoods such as aspen, and its application on other low-density hardwoods has also been successful (Cort and Bohn 1991; Zhao et al. 2004). APMP pulping has also shown success with non-wood species due to their relatively open and easy-to-disintegrate structure. Such pulping usually generates a lower volume of effluent, thus reducing the environmental impact relative to wood pulping (Zhao et al. 2004; Pan and Leary 2000). The APMP pulping potential of various non-wood raw materials including wheat straw (Zhao et al. 2004; Pan and Leary 2000; Mustajoki et al 2010), jute (Xu 2001), and oil palm empty fruit bunches (Rosnah et al. 2010) has been studied. Even though the APMP process is desired for its low energy consumption and production of quality pulp, research attempts also focus on refining energy reduction and pulp quality improvement through enzyme treatment of the raw material (Hart et al. 2009; Sigoillot et al. 2001; Zhao et al. 2004).

The expected shortfall in the supply of wood and the ever-increasing demand for paper has motivated the utilization of low quality wood residues and agricultural-based non-woods such as wheat straw and bagasse. To satisfy the quality wood demand, however, plantation-grown wood species such as poplar and eucalyptus have been able to fulfill the needs. Among the fast growth species, paulownia wood, a native of China and Southeast Asia, has attracted recent attention. Even though the biomass generated from these species has been considered as a source of energy, its modest water requirement has generated an interest for its use as an industrial raw material (Jimenez et al. 2005; Olson and Carpenter 1985; Garcia et al. 2011). The genus includes nine different species, with most of them exhibiting extremely fast growth; thus valuable timber can be harvested just 15 years after planting. Lower quality timber can easily be collected from 6 to 7 year-old trees, producing up to 5 tons/ha./year of biomass, which is among the highest reported growth volume even comparable to annual crops (Kalaycioglu et al. 2005; Ates et al 2008; Sanchez 2003).

Paulownia has been introduced and adapted in a number of countries for industrial wood production, and there have been few references to industrial applications, including possible application in veneer and plywood, furniture, tools, musical instruments, and particleboard (Curly 1993; Ashori and Nourbakhsh 2009). The application of paulownia wood in pulpwood has been studied using the soda-AQ (Garcia et al. 2011; Ates et al. 2008), kraft, and ethanol pulping processes (Ates et al. 2008), and promising results have been achieved.

To determine the suitability of paulownia wood for pulping, this research investigated the APMP pulping of paulownia wood in an attempt to find better utilization of this fast growth, low-density wood. The application of different dosages of hydrogen peroxide and sodium hydroxide was studied.

## EXPERIMENTAL

### Materials

Wood samples were harvested from an exotic species adaptation experimental project located in the city of Sari, Iran. Two paulownia trees in a 15 year-old plantation were randomly selected. One bolt at breast height was cut from each tree and transferred to the wood and paper laboratory workshop at the college of Agriculture and Natural Resources, Islamic Azad University, Karaj Branch.

The bolts were chipped using a Pallmann drum chipper, PHT100x400 and then screened to remove the fine particles. Chips dimensions were further reduced manually to produce chips suitable for laboratory-scale alkaline peroxide mechanical pulping. The average dimension of the chips was measured to be 27.6 mm x 9 mm x 4.3 mm.

### Fiber Dimensions and Chemical Analysis

Small chips were macerated using a technique developed by Franklin (1954) and then the dimensions of 100 randomly selected fibers were measured using a light microscope equipped with scaled eyepiece and the average of the measurements was recorded.

Relevant TAPPI standard test methods were used for chemical analysis as follows: Sample preparation, T257 om-08; Ash, T211-om 07; Extractive soluble in alcohol-acetone, T204-om 07; Cold and hot water solubility, T 207-om 08; 1% NaOH solubility, T212-cm 07; Extractive free wood, T264 cm-07; Cellulose, Kurschner-Hoffer, and Klason lignin, T222 om-07.

### Chemical Treatment

Prior to chemical treatment, the chips were pre-washed in hot water at 70 °C with a water-to-wood ratio of 19:1 using de-ionized water. The hot water treated chips were discharged onto a washing screen and the dark, extractive-containing filtrate was removed followed by washing with hot de-ionized water. These chips were then used for alkaline peroxide treatment.

Alkaline peroxide treatment was carried out by applying one of three levels of sodium hydroxide (1.5, 3, and 4.5%, based on the oven-dry weight of the wood) and one of three levels of hydrogen peroxide (1.5, 3, and 4.5%, based on the oven-dry weight of the wood). Other chemicals including sodium silicate, MgSO<sub>4</sub>, and DTPA were constant for all treatments at 3%, 0.5%, and 0.5%, respectively. Chelating treatment with DTPA was combined with alkaline peroxide treatment. Chemical treatment with a liquor-to-wood ratio of 9:1 was carried out in polyethylene bags submerged in a water bath at 70°C for two hours.

At the end of the treatment period, the contents of polyethylene bags were discharged on a 200 mesh screen, the spent liquor was collected, and its volume was measured. 100 mL samples of the spent liquors were taken for residual chemical measurements, and the remaining liquor was combined with the treated chips ready for defibration. Residual chemicals in the spent liquor were measured in accordance with the procedure outlined by Strunk (1993).

### Chip Defibration

Treated chips were defibrated at low consistency (about 2%) using a 25-centimeter single disc refiner (Faravari Ghomes Wood and Paper Equipment Manufacturing Co.) in three passes to complete the defibration. The clearance of the discs was reduced gradually in each pass to produce suitable APMP pulp. The pulp was screened using a set of two screens: a 14 mesh screen above a 200 mesh screen. The material that remained on the 14 mesh screen was rejected. Fibers that passed through the 14 mesh screen and were retained on 200 mesh screen were considered as acceptable pulp and used for pulp evaluation.

### Pulp Evaluation

Before handsheet making, each pulp was refined in a PFI mill as defined by the TAPPI standard procedure T248 sp-08 to reduce the freeness to between 320 and 350 mL CSF. Handsheets were then formed according to TAPPI standard test method T205 om-08, and the strength and optical properties of each pulp were measured on five replicated hand sheets according to the relevant TAPPI standard methods as follows: Basis weight, T410 om-08; Caliper, T411 om-97; Tear strength, T414 om-06; Tensile strength, T494 om-04; Burst strength, T403 om-02; Brightness, T452 om-08; and opacity, T425 om-08.

## RESULTS AND DISCUSSION

### Fiber Dimensions and Chemical Compositions

The fiber dimensions and chemical compositions of paulownia wood are listed in Table 1 along with similar data for other fiber materials for comparison. As seen, the fiber dimensions used in this study are in agreement with Ates et al. (2008). In comparison with wood and non-wood fiber sources, the fiber length of paulownia wood is shorter than eucalyptus fibers and non-wood fibers. The cellulose content of paulownia wood is either comparable or greater than other wood and non-woods, but its Klason lignin content and 1% NaOH solubility are higher. The low alcohol-acetone soluble extractives and ash content makes paulownia wood suitable for mechanical pulping.

### Pulping

APMP pulping exhibits outstanding characteristics, as it is a flexible pulping process adaptable to a variety of lignocellulosic raw materials. APMP has proved to be a good technique for producing mechanical pulp with improved strength and brightness (Korpela 2008; Pan and Yuan 2004).

The results of the APMP pulping on fast growth paulownia wood are summarized in Table 2 and Figs. 1 through 4. The pulping yield ranged between 87.8% using the lowest chemical charge and 81.6% yield with the highest sodium hydroxide dosage. The pulp freeness was improved at higher chemical dosages, meaning better beating behavior and less refining energy to reach the suitable freeness for strength development. The lignin content of the wood showed no change with the chemical treatment. The alkaline peroxide treatment yield of wheat straw has been reported to be 75% (Pan and Leary 2002) and 68.1% (Zhao et al. 2004). Ali et al (2002) measured the chemimechanical pulp

yield of cotton stalks to be between 54.7% and 56.5%. Ates et al (2008) studied the chemical pulping of *Paulownia enlongata* wood grown in Turkey using kraft-AQ, soda-AQ, and ethanol pulping processes and measured the pulping yields to be 38.3%, 37.8% and 38.4%, respectively.

**Table 1.** Fiber Dimensions and Chemical Composition of Paulownia Wood

| Fiber source               | Dimension   |                         |                                       | Chemical composition (%) |               |                 |                   |                    |                         |      | Reference                     |
|----------------------------|-------------|-------------------------|---------------------------------------|--------------------------|---------------|-----------------|-------------------|--------------------|-------------------------|------|-------------------------------|
|                            | Length (mm) | Width ( $\mu\text{m}$ ) | Cell wall thickness ( $\mu\text{m}$ ) | Cellulose                | Klason lignin | 1% NaOH soluble | Hot water soluble | Cold water soluble | Ethanol-acetone soluble | Ash  |                               |
| <i>Paulownia</i> wood      | 0.82        | 40.3                    | 7.1                                   | 49.5                     | 25            | 26.9            | 11.4              | 8.1                | 12.1                    | 0.45 | Measured                      |
| <i>Paulownia enlongata</i> | 0.82        | 36.3                    | 8.6                                   | 43.6*                    | 20.5          | 24.5            | 10.05             | 6.5                | 3.76                    | 0.21 | Ates et al. 2008              |
| <i>Eucalyptus</i>          | 1.28        | 18                      | 7                                     | 43.3*                    | 24.5          | 23.56           | 9.91              | 5.62               | 3.29                    | 1.35 | Ayata 2008                    |
| Canola straw               | 1.31        | 31                      | 5.75                                  | 48.5                     | 20            | 50.3            | 15                | 13.8               | 6.6                     | 6.6  | Hosseinpour et al. 2010       |
| Wheat straw                | 0.74        | 13.2                    | 4.6                                   | 38.2                     | 15.3          | 49.5            | 16.24             | 10.6               | 7.8                     | 4.7  | Deniz et al. 2004             |
| Bagasse                    | 1.59        | 20.9                    | 5.6                                   | 55.8                     | 20.3          | -               | -                 | -                  | 3.15                    | 1.74 | Samariha and Khakifirooz 2011 |
| Kenaf                      | 2.6         | 2                       | -                                     | 37.4*                    | 14.5          | 34.9            | 12.8              | 11.7               | 5                       | 4.1  | Atchison 1993                 |
| Corn stalks                | 1.03        | 23.6                    | 3.5                                   | 51.2                     | 23            | 45.9            | -                 | -                  | 15                      | 2.1  | Jahan Latibari et al. 2011    |

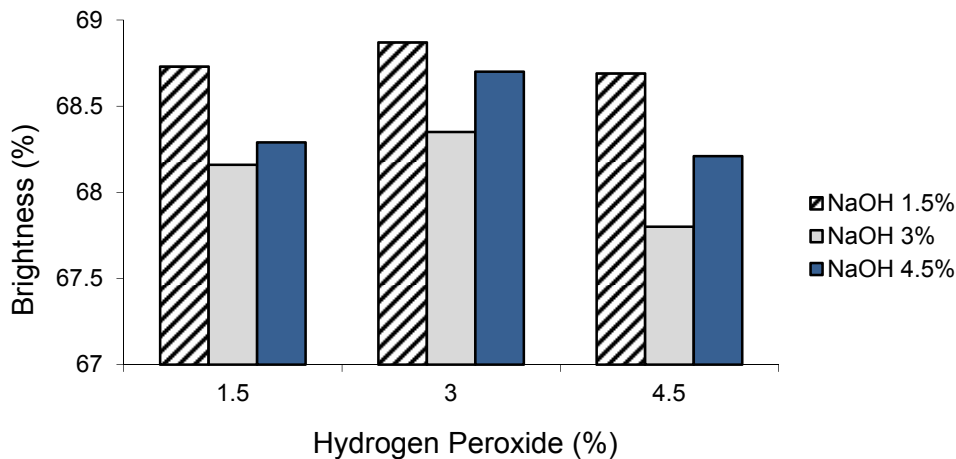
**Table 2.** Pulping Conditions and Properties of APMP Pulp from Paulownia Wood

| Pulp No. | Chemical charge (%)           |      | Residual chemical (%)         |      | Yield (%) | Freeness (mL CSF) |                |              | Bulk ( $\text{cm}^3/\text{g}$ ) |
|----------|-------------------------------|------|-------------------------------|------|-----------|-------------------|----------------|--------------|---------------------------------|
|          | H <sub>2</sub> O <sub>2</sub> | NaOH | H <sub>2</sub> O <sub>2</sub> | NaOH |           | Initial           | PFI revolution | Post refined |                                 |
| P1       | 1.5                           | 1.5  | 0.09                          | 0.75 | 87.8      | 750               | 15500          | 340          | 2.47                            |
| P2       | 1.5                           | 3    | 0.07                          | 1.51 | 83.4      | 750               | 14500          | 320          | 3.34                            |
| P3       | 1.5                           | 4.5  | 0                             | 1.72 | 84.7      | 750               | 8000           | 320          | 3.47                            |
| P4       | 3                             | 1.5  | 0.15                          | 0.79 | 85.3      | 750               | 13000          | 340          | 2.62                            |
| P5       | 3                             | 3    | 0.09                          | 1.43 | 83        | 721               | 7500           | 350          | 2.65                            |
| P6       | 3                             | 4.5  | 0.07                          | 2.07 | 81.9      | 721               | 7500           | 340          | 3.37                            |
| P7       | 4.5                           | 1.5  | 0.21                          | 0.59 | 87.7      | 721               | 13000          | 360          | 2.65                            |
| P8       | 4.5                           | 3    | 0.07                          | 1.34 | 84.2      | 700               | 8000           | 355          | 3.12                            |
| P9       | 4.5                           | 4.5  | 0.17                          | 2.41 | 81.6      | 700               | 8000           | 355          | 3.03                            |

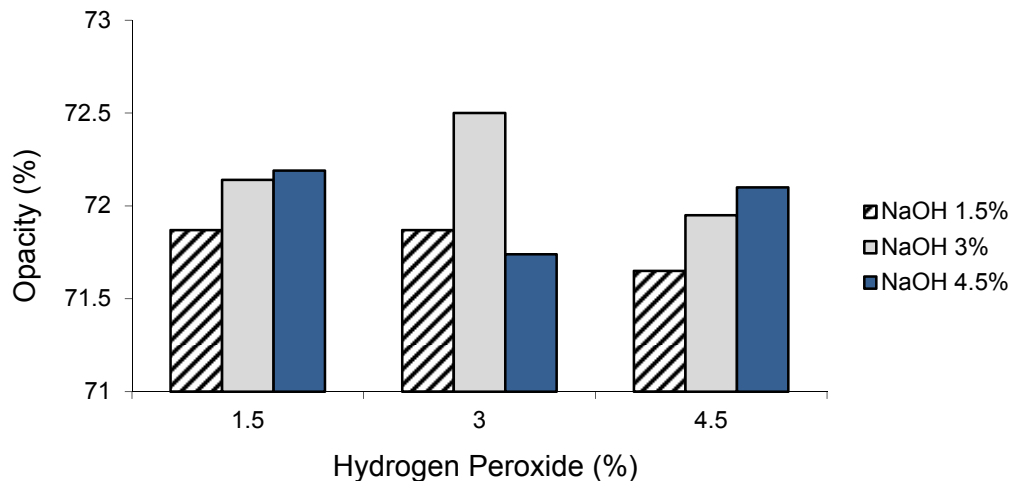
\*DTPA: 0.5%, time: 120 min., temperature: 70 C, sodium silicate: 3%, and MgSO<sub>4</sub>: 0.5%

The favorable brightness of the paulownia APMP pulp indicates that the hydrogen peroxide treatment eliminated the chromophores, but the deterioration of the low molecular weight carbohydrates also was evident. The brightness response of paulownia wood to hydrogen peroxide bleaching was very fast, and even at the low dosage of 1.5%

hydrogen peroxide, the pulp brightness reached 68.7% ISO and remained at almost this value when 3 and 4.5% hydrogen peroxide was used. It was interesting to observe that even 15 minutes after the chemical charge, very bright pulp was produced. Even though other low density hardwoods such as aspen exhibit good response to alkaline peroxide bleaching, the brightness development of non-wood fibers such as straw (Zhao et al. 2004; Pan and Leary 2000; Mustajoki et al. 2010), cotton stalks (Ali et al. 2002), kenaf (Cunningham et al. 1979), jute (Xu 2001), and oil palm empty fruit bunches (Rosnah et al 2010) have not been as successful as with paulownia or poplar wood.



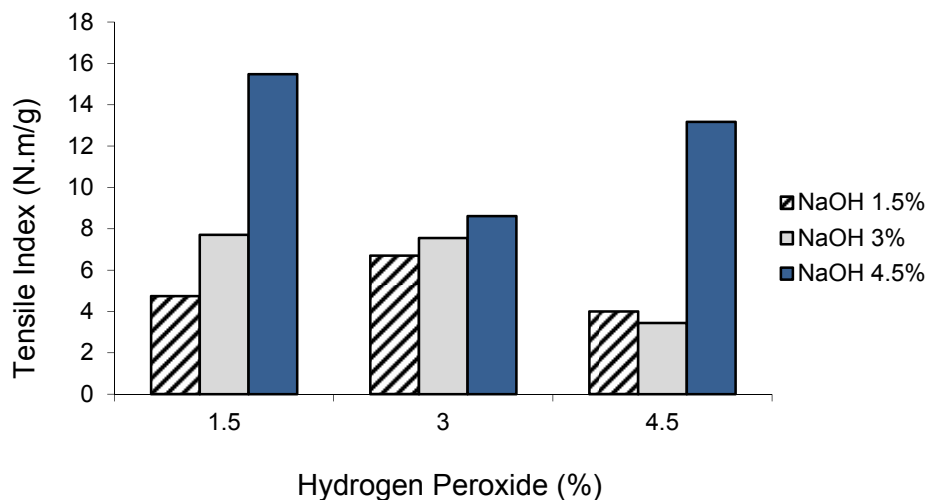
**Fig. 1.** The influence of hydrogen peroxide and sodium hydroxide charge on the brightness of APMP pulp from paulownia wood



**Fig. 2.** The influence of hydrogen peroxide and sodium hydroxide charge on the opacity of APMP pulp from paulownia wood

Even though the addition of more pulping chemicals decreases yield, it is required to achieve adequate strength values. At the low sodium hydroxide charge, the pulp strength was not satisfactory, and the higher sodium hydroxide dosage (4.5%) was needed to produce stronger pulp. Hydrogen peroxide treatment eliminates the chromo-

phores, but under alkaline conditions carboxylic acid groups are generated on the fiber surfaces (Pan 2004). Such carboxylic groups have a strong influence on inter-fiber bonding (Katz et al. 1981; Barzyk et al. 1997). Dissolution of wood components also increases the fiber bonding potential (Pan and Yuen 2004). The removal of extractives enhances the hydrophilicity of fiber surfaces, augmenting fiber bonding capacity. Superior pulp was produced by applying 1.5% hydrogen peroxide and 4.5% sodium hydroxide (pulp no. 3), and only 1.3% sodium hydroxide was consumed, which can be considered as the optimum alkaline peroxide mechanical pulping condition for this wood. Pulps did not exhibit sufficient burst strength, and only two pulps could be measured for burst strength (pulps no. 3 at 0.56 kPa.m<sup>2</sup>/g and no.9 at 0.5 kPa.m<sup>2</sup>/g). APMP pulps from paulownia wood were found to be very bulky, with the bulk varying between 2.47 and 3.37 cm<sup>3</sup>/g (Table 2). Contrary to usual expectation that at higher dosage of the chemicals, the bulk of the pulp should decline, in our work at very low chemical charge, we observed lower bulk. At low chemical charge, the defibration of treated chips generated more fines and the fibers are not fibrillated, but at higher chemical charge and subsequent post-refining, more fibrillation was produced, causing bulkier paper. Mechanical pulps are frequently used in the middle layer of folding boxboard and groundwood-containing paper grades to impart bulk and stiffness to the product (Hart et al. 2009).



**Fig. 3.** Illustration of the tensile strength index development of APMP pulp from paulownia wood

The tensile index of jute APMP with 2.6% hydrogen peroxide and 3.4% sodium hydroxide was measured to be 38.4 Nm/g. Aspen pulped with 1.2% hydrogen peroxide and 2% sodium hydroxide had a tensile index of 30.6 Nm/g (Xu 2001). Zhao et al. (2004) reported the burst strength index and tear strength index of wheat straw APMP pulp from 8% sodium hydroxide and 4% hydrogen peroxide to be 1.3k Pa.m<sup>2</sup>/g and 4.4 mN.m<sup>2</sup>/g, respectively. The burst strength index and tensile strength index of APMP pulp from oil palm empty fruit bunches were reported to be 5.35kPa.m<sup>2</sup>/g and 18Nm/g, respectively (Rosnah et al. 2010). The tear strength indices of paulownia kraft-AQ, soda-AQ, and ethanol pulps were measured to be 2.10, 1.96, and 3.36 mN.m<sup>2</sup>/g, respectively (Ates et al. 2008). The tensile strength, burst strength, and tear strength indices of *Paulownia fortune*

*x tormentosa x elongata* wood soda-AQ pulp at 52.3% yield were measured to be 21.5 Nm/g, 0.8 kPa.m<sup>2</sup>/g, and 1.53 mN.m<sup>2</sup>/g, respectively (Garcia et al. 2011).

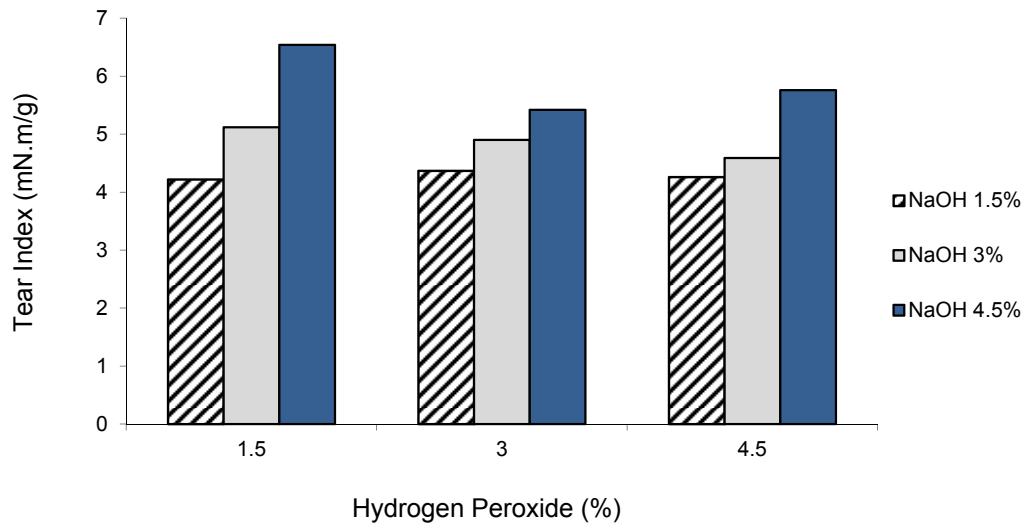


Fig. 4. Illustration of the tear index development of APMP pulp from paulownia wood

## CONCLUSIONS

1. Paulownia wood, having a fast growth rate, exhibits good potential as an industrial feedstock.
2. APMP pulping of paulownia wood demonstrated the possibility of pulp production with suitable properties for use as filler-grade pulp, e.g. for use in the center ply of folding boxboard. Applying 1.5% hydrogen peroxide and 4.5% sodium hydroxide, it was possible to obtain pulp with a 87.8% yield, 68.7% ISO brightness, 15.5 mN/g tensile index, 6.54 mN.m<sup>2</sup>/g tear index, and 0.56 kPa.m<sup>2</sup>/g burst index. The bulk of this pulp was found to be 3.47cm<sup>3</sup>/g.
3. Paulownia APMP pulp can be utilized in the middle layer of multi-layer paperboard to provide stiffness, as it is needed for its frequent use as boxboard.

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