

# Characterization of Asoka (*Saraca Indica* L.) branch wood for pulp and paper manufacturing

## Abstract

Asoka (*Saraca indica* L.) branch wood was evaluated for its chemical composition, morphological features, kraft pulping behavior, and physical properties. The Asoka used in this study contained low amounts of cellulose and hemicellulose, but higher amounts of lignin and ash. It was found that the morphological indices of Asoka branch wood were mostly adequate for pulping and papermaking. The pulping of this wood was performed using chemi-mechanical pulping (CMP), conventional kraft pulping, and soda pulping processes under different conditions, i.e., active alkali and anthraquinone (AQ) addition. The results showed that alkaline pulping and CMP provided low screened yield values. Acceptable kappa numbers and reject levels were obtained at 22% active alkali of soda and kraft-AQ pulpings. CMP pulp showed higher brightness and screened yield than that of alkaline pulps. Alkaline pulpings of Asoka branch wood provided slightly different strength properties of paper sheets than those of CMP pulp. Based on the findings, it can be concluded that Asoka branch wood is more suitable as a raw material for mechanical pulping than it is for alkaline pulping.

**Keywords:** chemi-mechanical pulp, kraft pulping, kappa number, fibre morphology, brightness

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## Introduction

Tree species are the major source of virgin cellulose fiber for pulp production. In particular, eucalyptus and acacia species have been used as a primary raw material in Indonesia because of its fast growth. However, the scarcity of forest resources is forcing the Indonesian pulp and paper industry to turn to other raw materials for pulp and paper production. In order to solve this problem, wood-based pulp and paper mills have initiated lesser-used programs that allow the use of various alternative fiber resources from smallholders and community forests. Asoka or Asoka (*Saraca indica* L.) wood has been considered as a potential hardwood for pulp and paper production due to its availability.

Asoka tree is a member of the Fabaceae family. This flowering and evergreen tree has with a natural range extending across Vietnam, Thailand, Malaysia and Indonesia. The trees are easily to be found as an ornamental. The numerous economic uses of Asoka have raised interest nationally and internationally for the utilization of this tree. The bark is strongly astringent and a uterine sedative.<sup>1</sup> It contains tannins and catechol.<sup>2</sup> The light reddish-brown wood is soft.<sup>3</sup> However, few studies are known to have investigated its physical and chemical properties to ascertain its suitability for pulp and paper production.

Due to the growing global demand for pulp and paper, there has been an interest in developing alternative sources that offer sustainable environmental benefits. The use of biological waste from various sources can be an important source of raw materials. Waste from forest pruning and clearing, as well as residues from primary wood processing, are currently not effectively used because of technical and economic difficulties in their extraction. Most of the branches, especially branch reaction wood, have been wasted. Reaction wood is undesirable in any structural application because it can twist, cup or warp considerably during machining. Therefore, efficient ways to utilize the branch wood need exploring.

Few studies have discussed the difference in wood properties between branch wood and stem wood in different tree species or pulp and in papermaking.<sup>4-6</sup> Furthermore, some experiments had

been conducted to find the optimum pulping condition (kraft/soda-AQ) for branch wood material from pruning activities.<sup>7,8</sup> Shmulsky and Jones<sup>9</sup> summarized that low pulp strength, nonuniformity of chip sizes, and high proportions of bark are reasons why branch wood is not preferred by papermakers. However, that comparatively good - quality mechanical or chemi-mechanical pulp can be obtained from this branch wood. Therefore, in the present study, mature Asoka branch wood with a larger diameter and lower proportion of bark was evaluated with respect to its chemical composition, morphological characteristics, followed by alkaline with anthraquinone (AQ) addition and chemi-mechanical pulping.

## Materials and methods

### Raw material

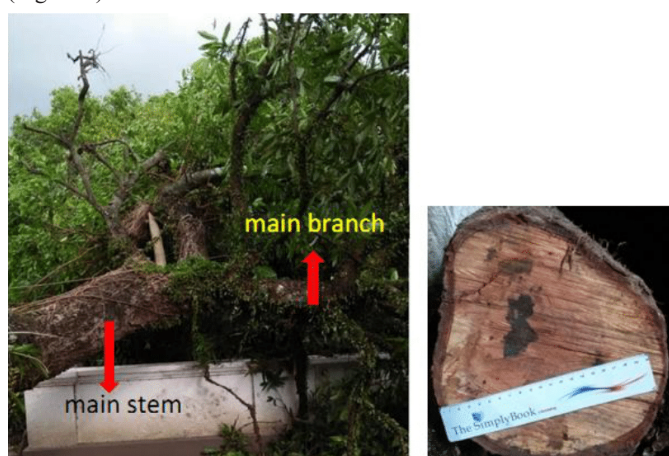
The sample of Asoka wood (diameter of  $\pm 38$  cm) was collected from the main branch wood of a mature fallen-tree (age unknown) in Yogyakarta City (Figure 1). After bark removal, the wood was manually chipped (3 cm  $\times$  2 cm  $\times$  2-3 mm). The chip samples were thoroughly mixed and air-dried to obtain uniform moisture. The moisture content of the chip samples was determined according to standard procedures prior to the pulping experiments.

### Proximate chemical analysis

To determine the chemical composition of Asoka branch wood, the chip sample was milled to powder and the fraction passing through the 40 mesh, but retained on 60 mesh, was collected. The chemical composition was determined according to the respective standard procedures. The extractive content was determined by ethanol-toluene (ASTM D1107 - 96)<sup>10</sup> and hot-water (ASTM D1110 - 84)<sup>11</sup> extraction. Holocellulose content was determined by the Wise's chlorite method.<sup>12</sup> For the determination of alpha-cellulose in wood, NaOH (17.5%) was applied to holocellulose to dissolve hemicelluloses.<sup>13</sup> Hemicellulose content was determined by the difference between holocellulose and alpha-cellulose content. Klason lignin was determined by hydrolysis of the carbohydrates with 72% sulfuric acid.<sup>14</sup> Ash content was determined according to ASTM D 1102 - 84 procedure.<sup>15</sup>

## Basic density and morphology of the fibers

Preparation and measurement of basic density were performed according to ASTM D2395.<sup>16</sup> Detailed morphological features, such as fiber length and fiber width of macerated fibers (by hydrogen peroxide), were evaluated using a with image-analysis (Image pro Plus). All the other characteristics, such as cell wall thickness, lumen diameter, were determined using a light microscope (Olympus BX 51; Olympus Corporation; Japan) with a digital camera (Olympus DP 70). The proportion of wood cell types, which are vessels, fibers, ray parenchyma and axial parenchyma, was measured in percentage. For fibers, the following parameters were calculated: Runkel ratio =  $2 \times (\text{fiber cell wall thickness})/(\text{lumen diameter})$ ; slenderness ratio =  $\text{fiber length}/\text{fiber diameter}$ ; Luce's shape factor =  $(\text{fiber diameter}^2 - \text{fiber lumen diameter}^2)/(\text{fiber diameter}^2 + \text{fiber lumen diameter}^2)$ ; Solids factor =  $(\text{fiber diameter}^2 - \text{fiber lumen diameter}^2) \times \text{fiber length}$  (Figure 1).<sup>17</sup>



**Figure 1** The Asoka mature tree and cross-section of its branch wood.

## Alkaline pulping

Alkaline pulping of Asoka branch wood (150 g, oven-dried chip) was performed in a laboratory digester of 5 L capacity, rotating in an electrically heated autoclave. The pulping conditions of kraft, kraft-AQ, soda, and soda-anthraquinone are as follows:

- Active alkali was 20%, and 22% on o.d. raw material as  $\text{Na}_2\text{O}$ , cooking time was 2 h at maximum temperature ( $170^\circ\text{C}$ ), and 45 min were required to reach the maximum temperature ( $170^\circ\text{C}$ ) from room temperature.
- Liquor to material ratio was 4.
- AQ charge was 0.1% on oven-dry raw material in kraft-AQ and soda-AQ processes (active alkali 22% only).
- Sulfidity was 25% for the kraft process.

At the end of the pulping, the material obtained was dispersed with a pulp disintegrator, and washed with cold water to remove the black liquor and dissolved substances. Subsequently, the pulp was screened using a 100 mesh screen to separate the rejects. Two replicates were made and the average reading was taken.

## Chemi-mechanical pulp (CMP)

CMP of Asoka branch wood (150 g, eq. oven-dried chip) was prepared with cooking time of 120 min, a temperature of  $170^\circ\text{C}$ , a liquor to material ratio of 6, and sodium sulfite percentage of 20 %

based on the dry weight of Asoka branch wood. After treatment with sodium sulfite chemicals, the fibers were refined through a two-step high consistency refinery (KRK) at scale 8.

## Pulp and handsheet characterization

Kappa number was determined in the unbleached pulp samples according to SNI ISO 302:2014.<sup>18</sup> The degree of beating of the Niagara beater was determined for Canadian Standard Freeness (CSF) of 200-300 ml.<sup>19</sup> The production of 60 g/cm<sup>2</sup> handsheets under different pulping methods was in accordance with SNI ISO 5269-1-2012.<sup>20</sup> Finally, the handsheets were evaluated for tensile (SNI 14-0437-1989-A),<sup>21</sup> tear (SNI ISO 1974:2012),<sup>22</sup> and burst (SNI ISO 2758-2011)<sup>23</sup> indices, while brightness was measured according to SNI ISO 2470-1-2014.<sup>24</sup>

## Results and discussion

### Proximate chemical analysis

According to the results of proximate chemical analysis results tabulated in Table 1, the amount of holocellulose indicated a total carbohydrate content of 67.3%. In general, a high content of cellulose in wood indicates a higher pulp yield. The acceptable  $\alpha$ -cellulose content in the lignocellulosic raw materials for pulp production is greater than 40%.<sup>25,26</sup> Accordingly, the materials in this study had cellulose content lower than 40%, which predicts low pulp yield and low tensile strength for the paper produced.<sup>27</sup> Furthermore, a high percentage of hemicellulose retained in the pulp after the cooking process improves the swelling and strength characteristics of the pulp and paper and reduces the energy consumed during beating.<sup>28,29</sup> Table 1 reveals the value of the branch wood hemicellulose (27.75%), which predicts that the paper produced would have low strength properties.

**Table 1** Chemical composition of Asoka branch wood

Chemical composition (%)	Asoka branch wood
Ethanol-toluene extractive content	1.17
Hot-water solubility	4.12
Holocellulose	67.73
$\alpha$ -Cellulose	39.98
Hemicellulose	27.75
Lignin	33.5
Ash	4.5

Generally, lignin is an undesirable polymer and its removal during pulping requires high amounts of energy and chemicals.<sup>30</sup> The recommended percentage of acid-insoluble lignin for common pulp raw materials is less than 30%.<sup>25</sup> The lignin content influences the pulping time and the chemical charge; therefore, the high lignin content in this study (33.50%) corresponds to a longer pulping time and a higher chemical charge. The high ash content (4.5%) corresponds to more minerals in the pulp, which increases cooking time and alkali consumption and causes problems during the black liquor recovery.<sup>28,31</sup> The higher quantity of solubility in water (4.12%) can cause many problems such as reduced yield and increased alkali consumption.<sup>31</sup> Previously, the branch wood exhibited lower cellulose and higher hemicellulose content than the trunk wood of *Betula platyphylla*<sup>32</sup> or *Trema orientalis*.<sup>5</sup> Furthermore, branch wood contained a larger amount in lignin, total extractives than those determined in stem wood of *E. camaldulensis*, *Acacia gerardii*, and *Tamarix aphylla*.<sup>33</sup>

## Basic density and morphological features

Table 2 shows the basic density and morphological features of Asoka fibers. It reveals that a large amount of parenchyma cells can be observed, justifying the high extractive content of the wood. These findings indicate that Asoka wood contains cellulose fibers along with non-fibrous cellular materials, such as parenchyma tissue and vessel element tissue. These non-fibrous cellular materials do not have any papermaking properties and adversely affect inter-fiber bonding. High density of vessel and parenchyma proportions would limit its potential for commercial uses.<sup>33</sup> In this study, Asoka branch wood exhibited comparatively high vessel (10.46%) and parenchyma (21.15%) proportion.

**Table 2** Basic density and morphological features of Asoka branch wood

No	Characteristics	Branch wood
1	Basic density (kg/m <sup>3</sup> )	590
2	Cell Proportion	
	Fibre (%)	56.68
	Vessel (%)	10.46
	Parenchyma (%)	21.15
	Ray (%)	11.71
3	Fibre dimension	
	Fibre length (µm)	1005.71
	Fibre diameter (µm)	15.45
	Fibre wall thickness (µm)	2.96
	Fibre lumen diameter (µm)	9.54
4	Derived values	
	Runkel ratio	0.62
	Slenderness ratio	65.09
	Luce's shape factor	0.45
	Solid factor	148.83

Wood density is an important parameter for assessing the quality of raw materials for pulping. Wood density influences the fiber morphology and the final paper properties.<sup>34</sup> Raw materials with basic densities between 400 and 600 kg/m<sup>3</sup> were more preferred for paper manufacturing.<sup>35</sup> The high density of Asoka (590 kg/m<sup>3</sup>) may not be desirable for the chemical pulp and paper industry. In general, the majority of previous studies indicated that the density of branch wood is generally higher than that of stem wood, which may be attributed to the slower growth rate of the branches and the presence of reaction wood compared to the stem wood.<sup>36</sup> Moreover, the extractive content may contribute to the high density of branches.

Fiber morphological indices – such as fiber length, and width, and wall thickness – and their derived values are some of the most important parameters used to determine the suitability of wood as a raw material in the cellulosic pulp and paper industry.<sup>37,38</sup> Fiber length is the most important indicator of the suitability of raw materials, as longer fibers are preferred. It is also directly proportional to paper strength.<sup>39</sup> The fiber lengths of Asoka branch wood (1.005 mm) were moderate according to the classification of Metcalfe and Chalk<sup>40</sup> which states that fiber lengths less than 0.9 mm are short and those above 1.6 mm are long fibers. The longer the length of the fiber is, the higher the tearing strength of paper will be. A wider fiber lumen (9.54 µm) provides better pulp fibrillation in a shorter period of time due to easier penetration of liquids into the fiber lumen.

Four associated parameters were calculated from the determined fiber dimensions to evaluate the suitability of Asoka fiber for papermaking: Runkel ratio, slenderness ratio, solid factor, and Luce's shape factor. The Runkel and slenderness ratios of Asoka fiber were 0.62 and 65.09, respectively, while they were 0.37 and 51.29 in the case of *Acacia mangium*.<sup>41</sup> A Runkel ratio value less than 1.0 is preferred to achieve a good conformability and an interfiber contact in the production of paper from hardwoods.<sup>42</sup> The high values of Runkel ratio indicate that the fibers are less elastic and thick-walled with low bonded areas, which negatively affects the fiber-to-fiber bonding.<sup>43</sup> The slenderness ratio is also called the relative fiber length or felting power. It is an indirect indicator of the paper tear index and pulp digestibility.<sup>17</sup> Generally, it is recommended that a slenderness ratio greater than 33 produces paper with acceptable properties.<sup>44</sup> Thus, Asoka can be predicted to produce paper with good mechanical strength, especially tear strength.

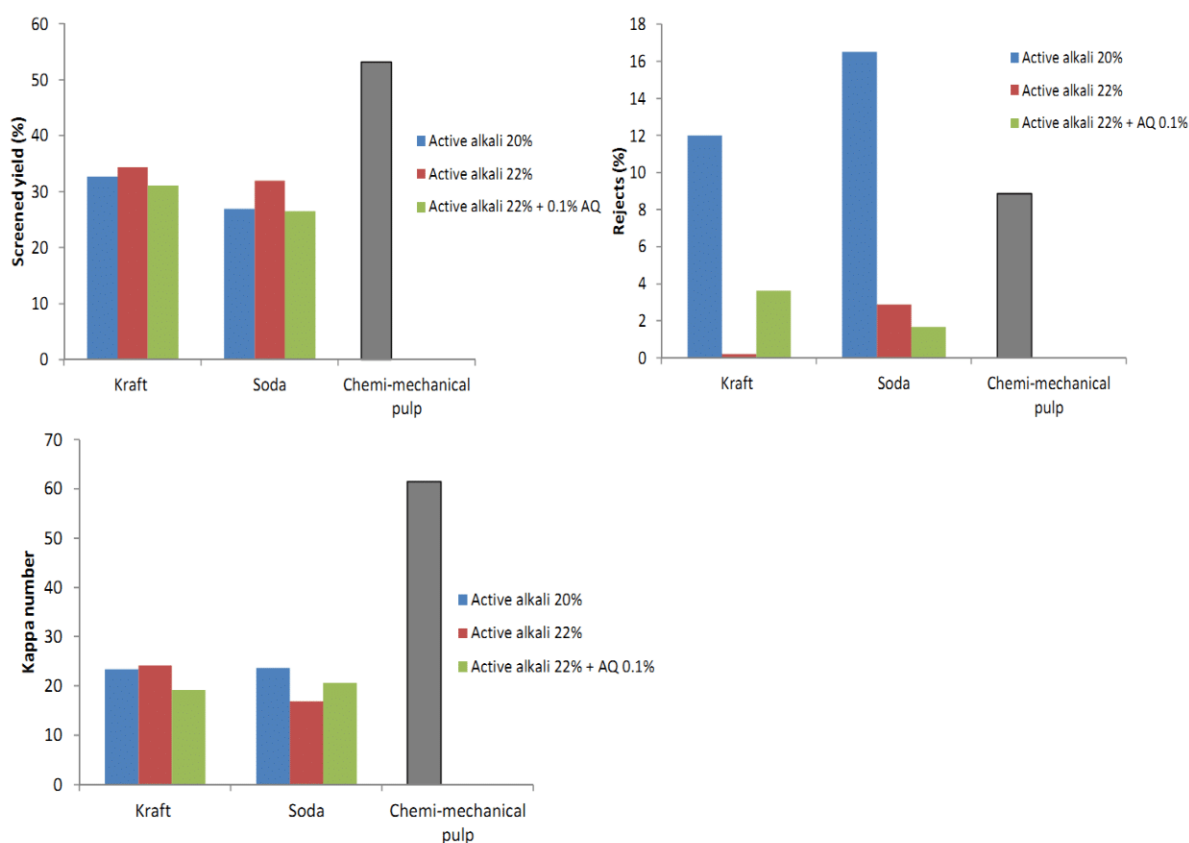
In an earlier report, the branch wood of *Pinus halepensis* and *Eucalyptus camaldulensis* had lower in fiber length, Runkel ratio, Muhlsteph ratio, rigidity coefficient, and Luce's shape factor, and higher in basic density and flexibility coefficient than those in stemwood.<sup>45</sup> In addition, the branch samples have consistently shorter fibers than the stem samples of *Trema orientalis*.<sup>5</sup> The Luce's shape factor and solid factor of Asoka branch wood were 0.45 and  $148.83 \times 10^3$ , respectively. For comparison, the average Luce's shape factor and solid factor of *Eucalyptus pellita* fiber were 0.57 and  $130.91 \times 10^3$ , respectively.<sup>46</sup> Luce's shape factor and solid factor were negatively associated with the breaking length of paper among eucalyptus species.<sup>47</sup> The low value of Luce's shape factor in this study predicts that the handsheet of Asoka branch wood will produce paper with good breaking length.

## Pulp properties

Hassan *et al.*<sup>48</sup> used soda-AQ, which is more environmental friendly process compared to kraft pulping to determine the optimum pulping for oil palm empty fruit bunch. Compared to soda-AQ pulps, kraft pulps generally have lower pulp yields.<sup>49,50</sup> The pulping conditions, unbleached pulp screen accepts and rejects, and kappa numbers for all pulp cooking scenarios used in this study are presented in Figure 2. The unbleached pulp yield ranges from 31.13 to 34.41% for kraft cooking and from 26.51 to 31.96% for soda cooking. Pulp yield increased with the percentage of active alkali from 20 to 22% in each pulping process. The pulp rejects were not all within acceptable limits, ranging from 0.22 to 12.00% for kraft pulping and from 1.68 to 16.52% for soda pulping. In comparison, soda-AQ pulping of the main fraction of orange pruning with alkali active of 10-16% had screened yield of 34-52% and kappa number of 25-89.<sup>7</sup> Kraft pulps from olive tree pruning using an effective alkali concentration between 18% and 24% (sulfidity of 30%) produced yield of 28-32% and kappa number 13-21.<sup>8</sup>

A strong relationship between yield or kappa number and the concentration of active alkali in the pulping was observed in several species.<sup>51-53</sup> In general, rejects decreased as the percentage of active alkali used per charge was increased. The presence of AQ in soda and kraft pulping did not improve delignification and decreased screened yield. In the kraft pulping process, the kappa number of the resulting pulp decreased as the percentage of active alkali in the charge was increased (Figure 2). Furthermore, the kappa number was increased but the reject increased in kraft pulping. In soda pulping, the AQ addition showed an inverse effect. As expected, the screened yield (53.77%) and kappa number (61.53) of the pulp obtained were higher in the CMP process than in the alkaline processes. However, the reject content was high (8.88%).





**Figure 2** Effect of pulping method on screened yield, reject and kappa number, and viscosity of Asoka branch wood pulp.

The screened yield values obtained in this study were lower than the range for alkaline and mechanical pulping.<sup>28</sup> The lower pulp yield in the present study for all processes can be explained by low cellulose and high hot water solubility values, as well as a high proportion of parenchyma and ray cells in the branch wood tissues (Table 2). Another reason was a high wood density (590 kg/m<sup>3</sup>), which causes poor penetration of cooking.<sup>54</sup> Gulsoy *et al.*<sup>6</sup> observed the yield of kraft pulp made from branch wood was lower than that of stem wood due to low  $\alpha$ -cellulose content of the branch wood compared to the stem wood. A similar trend was found in the branches of *Picea glauca*, *Betula papyrifera*, and *Populus tremuloides* by using bisulfite CMP process.<sup>4</sup>

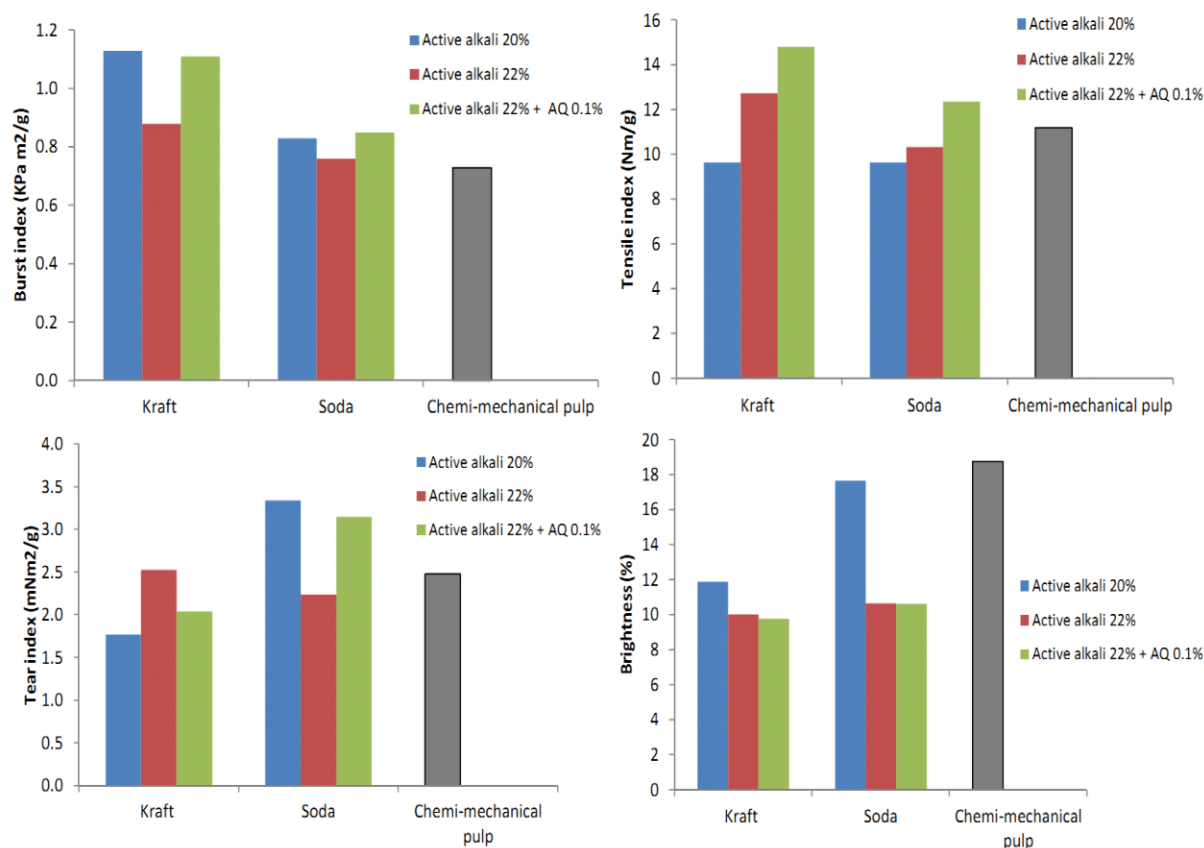
A lower kappa number indicates a lower lignin content in the pulp. For alkaline pulpings, the kappa number within acceptable range for bleaching (ranges from 18 to 20) was found in soda (active alkali 22%) and kraft+AQ pulp samples. In this experiment, delignification was not accelerated by the addition of AQ, indicating that the addition of AQ did not increase the selectivity of the pulping. This trend was different from previous works on AQ addition.<sup>7,54,55</sup> The majority of reducing groups needed for AQ to be effective are provided by alkaline peeling and cellulose chain cleavage reactions.<sup>56</sup> It was assumed that branch wood material would not produce sufficient reducing sugars to generate the anthrahydroquinone that would be needed to react with each  $\beta$ -aryl ether bond.<sup>6</sup>

### Handsheet physical properties

Figure 3 presents the relative strength properties for the unbleached paper sheets. In a previous study, kraft AQ pulping had better handsheet strength properties compared to other soda-AQ processes

in orange tree wood.<sup>7</sup> The tensile, burst factor, and tear indices were found to be not so different in alkaline and CMP processes. This indicates that the cooking conditions in alkaline pulping were not suitable in this experiment. Furthermore, the strength values did not meet the Indonesian National Standard for leaf (hardwood) bleached kraft.<sup>57</sup> It is thought that the relatively high values of Runkel ratio and solid factor, together with the high kappa number of Asoka branch wood, may be the cause of the low strength levels. For comparison, the values of brightness, tear, burst, and tensile indices of willow wood chemi-mechanical pulp for newsprint purposes were 16.33%, 4.40 mN m<sup>2</sup>/g, 1.67 KPa m<sup>2</sup>/g, and 23.84 Nm/g, respectively.<sup>58</sup> It implies that pulp from Asoka branch wood can be utilized as hardwood pulp to a low or moderate extent, especially in writing, newspaper, toilet paper, etc.

It is expected that the paper properties of the stem wood will be superior to those of the branch wood. Unfortunately, the technical data of the stem wood are not available in this current study. In another species, the branch wood of *Betula platyphylla* could be used to produce low-grade paper despite it met the basic requirements of papermaking.<sup>32</sup> By bisulfite CMP process, Law and Lapointe<sup>4</sup> found that the branch pulps had lower strength properties than the corresponding bole pulps. The weaker mechanical properties might be due to the differences in dimension of fibers and vessel elements and the relative proportion of these cells in the wood. On the contrary, branch wood handsheets exhibited higher strength properties and brightness than those of stem wood handsheets in *Punica granatum*.<sup>6</sup> It was assumed that the *Punica granatum* branch wood had higher fiber flexibility, higher bonding ability, higher slenderness ratio, and lower Runkel ratio.



**Figure 3** Effect of pulping method on sheet physical and optical data for the Asoka branch wood.

In the kraft process, the tear and tensile indices increased as the percentage of active alkali in the charge was increased (Figure 3). A similar trend was observed for the tear strength of soda pulp, whereas the burst and tear indices decreased as the percentage of active alkali in the charge was increased. Those trends suggest that each strength parameter behaved differently with regard to the changes of pulp chemical properties indicated by the kappa number. The presence of AQ in soda and kraft pulping improved the strength properties of the pulp sheets except for the tear index in kraft pulp. In a review by Hart and Rudie,<sup>56</sup> the addition of AQ only slightly affected the strength properties of the pulp. No significant difference was found in the strength properties of the AQ-added for the branch wood of *Punica granatum*.<sup>6</sup>

As expected, the CMP process obtained a higher brightness value (18.76) than the alkaline processes. Furthermore, kraft pulp has darker color than soda pulp due to the retained chromophore groups of lignin.<sup>59</sup> In this study, the brightness of the soda pulp content was high (17.66%) at 20% of active alkali concentration. The higher alkali active concentration allows more lignin to be dissolved and reduces kappa number thus increase the brightness of the paper. Technically, lignin causes the paper to appear dark. Therefore, after lignin removal, it stopped the chromophore's ability to absorb light.<sup>48</sup> However, the pattern of kappa number did not follow the brightness value for soda and kraft pulpings. In an earlier report, a significant difference was observed in the paper brightness of the AQ-added in *Punica granatum*.<sup>6</sup> This suggests that some parts of phenolic lignin groups are less converted to colored aromatic quinones such as paraquinone and orthoquinone, which attract the light compared to other pulping conditions.

## Conclusion

From the above results and discussion, it is reasonable to conclude that Asoka branch wood did not have suitable fiber from the standpoint of chemical composition, pulping and papermaking characteristics, and that paper made from its pulp did not have acceptable properties, particularly in alkaline pulping. Pulp from these materials are expected to have relatively low mechanical strength, which is suitable for hardwood pulps in low or moderate proportions for the production of newsprint or tissue paper. Future works should be conducted to increase the yield and physical properties of papers from its mechanical pulps. Apart from overcoming the lack of wood supply, implementing zero waste in logging, and adding value to logging waste, the use of branch wood from lesser used species as pulp's raw material is anticipated to lessen Indonesia's future forest exploitation from an environmental standpoint.

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## Conflicts of interest

The authors declare that there are no conflicts of interest.

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