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ORIGINAL ARTICLE

Production of kraft pulp from *Ochroma pyramidale* wood

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ABSTRACT

Brazil stands out in the international scenario in the production of short-fiber pulp. Despite the great Brazilian biodiversity, that production is based on exotic *Eucalyptus* clones. In this sense, there may be great potential in the assessment of new sources of fibers from the Brazilian flora, including the Amazon. The present study aimed to assess the technical potential of the wood of *Ochroma pyramidale* (Malvaceae) for the production of kraft pulp. Four-year-old trees were harvested from a commercial forest for lumber production in Mato Grosso state (Brazil). We determined the wood's chemical compositions (holocellulose, Klason lignin, soluble lignin, extractives and ash contents), physical properties (density and porosity), and fiber morphology (fiber length, width and thickness, lumen diameter, wall fraction, coefficient of flexibility, and slenderness and Runkel ratios). The wood was subjected to pulping with an effective alkali charge ranging from 10 to 24%, with intervals of 2%. *Ochroma pyramidale* wood presented characteristics favorable to the production of cellulosic pulp, such as appropriate fiber dimensions and low lignin, extractives, and ash content. The amount of residual active alkali and pH of the black liquor were positively related to the increase of the alkali charge employed in the pulping process. The increase of alkali charge decreased the pulp yield, kappa number and waste content, and increased the hexenuronic acid concentration.

KEYWORDS: balsa wood, Amazon, tree, alkali charge, kappa number

Produção de celulose kraft a partir da madeira de *Ochroma pyramidale*

RESUMO

O Brasil se destaca no cenário internacional em relação a produção de celulose de fibra curta. Apesar da grande biodiversidade brasileira, essa produção está baseada em clones de espécies exóticas de *Eucalyptus*. Nesse contexto, pode haver grande potencial na avaliação de novas fontes de fibras da flora brasileira, incluindo a região amazônica. O presente estudo teve como objetivo avaliar o potencial tecnológico da madeira de *Ochroma pyramidale* (Malvaceae) para produção de celulose kraft. Indivíduos de quatro anos de idade foram colhidos em um plantio comercial para produção de serrados no Estado do Mato Grosso. Determinamos as propriedades químicas (teores de holocelulose, lignina Klason, lignina solúvel, extrativos e cinzas), físicas (massa específica e porosidade) e morfologia de fibras (comprimento e largura de fibras, diâmetro do lume, espessura e fração de parede, coeficiente de flexibilidade, índice de enfeltramento e de Runkel) da madeira. Na polpação kraft a carga alcalina aplicada variou entre 10 e 24%, com intervalo de 2%. A madeira de *Ochroma pyramidale* apresentou características favoráveis à produção de polpa celulósica, como dimensões apropriadas das fibras e baixos teores de lignina, extrativos e cinzas. Os teores de álcali ativo residual e pH do licor negro apresentaram relação crescente com a carga alcalina aplicada nos processos de polpação. O acréscimo da carga alcalina reduziu o rendimento em polpação, número kappa, teor de rejeitos e aumentou o teor de ácidos hexenurônicos.

PALAVRAS-CHAVE: pau de balsa, Amazônia, árvore, cargas alcalinas, número kappa

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INTRODUCTION

Brazil stands out as one of the largest world producers of cellulosic pulp, especially short-fiber cellulose produced through kraft pulping of wood of *Eucalyptus* (IBÁ 2020). The Brazilian pulp and paper sector has expanded markedly in recent decades, both due to the increased capacity of traditional companies and to new installed production capacity and planted forests (Sperotto 2014; IBÁ 2020).

The growth and development of the cellulose sector create the demand for research aiming to optimize pulping processes and wood quality to improve production efficiency and quality of cellulosic pulp (Cardoso *et al*. 2011). Among the methods of chemical transformation of wood into cellulosic pulp, the kraft process is by far the most commonly used. This process is based on the reaction of wood with a solution of sodium hydroxide and sodium sulfide under controlled conditions of time, temperature, and pressure. The kraft process produces pulp with good technological quality. The possibility of using different sources of lignocellulosic raw materials and the recovery of the pulping reagents are other advantages of the process (Gomide *et al*. 1980).

Together with temperature and cooking time, the alkali charge strongly influences the delignification ratio of the fibers and consequently the pulp quality from the kraft process. High alkali charges employed in the pulping process are associated with more intense delignification, yielding pulp with low kappa numbers, and an increase in the delignification negatively influences both the yield and strength of the cellulosic pulp due to the partial degradation of the holocellulose (Cardoso *et al*. 2011).

Although the Brazilian market for short-fiber pulp is restricted basically to *Eucalyptus* wood, virtually any lignocellulosic raw material can be used to produce pulp and paper, as long as it has economically suitable silvicultural performance and favorable wood quality (Silva *et al*. 2016). Thus, the study of alternative sources for pulp production among native forest species can contribute to the diversification of raw materials in the sector. In the Amazon region, among a large diversity of wood species, there are several species with potential to be employed in planted forests for the production of pulp and paper. However, there is a lack in studies focused on the assessment of Amazonian wood species, mainly in comparison with the large number of published works involving *Eucalyptus* species currently planted in Brazil (Silva *et al*. 2013; Heckler *et al*. 2014; Silva *et al*. 2016). The analysis of the physical, anatomical, and chemical characteristics of the wood is fundamental to assessing the quality of the material for pulping.

Among the tropical species with potential for production of pulp and paper, is *Ochroma pyramidale* (Cav. ex Lam.) Urbam (Malvaceae), commonly known in Brazil as *pau-de-balsa*. This species is characterized by fast growth and high wood productivity, with mean annual increments of up to 50 m^3 ha^{-1} $year^1$ (Reis and Paludzyszyn Filho 2011), as well as low lignin and extractives contents (Lobão *et al*. 2011). The present study aimed to assess the potential of *O. pyramidale* wood from four-year-old plantations to produce cellulosic pulp through the kraft process.

MATERIAL AND METHODS

Ochroma pyramidale trees were sampled in a plantation located in the municipality of Sinop, state of Mato Grosso, Brazil (11º52.119'S, 55º27.746'W). The local climate is classified as Aw type (tropical savanna climate), characterized by two well-defined seasons, a rainy (October to April) and a dry season (May to September), with low annual thermal amplitude (monthly averages from 24 to 27 °C) and rainfall around 1,974 mm (Souza *et al*. 2013).

Three four-year-old trees were randomly selected, harvested and sawn at a bandsaw mill. From each tree, a central board 2 cm thick x 30 cm wide x 215 cm long was obtained. In the positions at 0, 25, 50, 75 and 100% of the length of each board, samples measuring 2 cm x 2 cm x 10 cm (thickness x width x length) were cut out. The remaining wood was chipped in a semi-industrial equipment to yield chips ranging from 4 to 6 mm in width. The chips were dried outdoors and stored in plastic bags for later analyses of chemical properties, fiber morphology and kraft pulping performance. All analyses were carried out individually for each tree and are reported as the mean and standard deviation of three trees.

The basic density was determined from the five 2 x 2 x 10-cm samples as the ratio between the dry weight (by drying at 103 °C until reaching constant weight) and green volume (after water saturation), according to the procedures described in the standard NBR 11941 from the Brazilian Association of Technical Standards (ABNT 2003a). The porosity was calculated as the ratio between the basic density and the specific density of the cell wall, considering the theoretical value of 1.53 g cm^{-3}, according the Equation 1.

$$P(\%) = \left[\frac{(1/\rho b) - (1/1.53)}{1/\rho b}\right] x\,100 \qquad \text{Equation1}$$

Where:

P = porosity (%)

ρb = basic density (g cm^{-3})

The chemical characterization was carried out in triplicate using sawdust obtained after milling the wood chips. The sawdust was sieved to collect the fraction with granulometry between 40 and 60 mesh. Then the extractives, Klason lignin and soluble lignin, and ash contents were determined following the procedures described in the standards NBR 14853, 7989, and 13999 (ABNT 2010a; 2010b; 2017), respectively. The content of holocelullose was calculated using Equation 2.

$$H\ (\%) = 100 - (E + TL) \quad \text{Equation 2}$$

Where:

H = holocelullose content (%)

E (%) = extractives content

TL = total lignin content (Klason lignin + soluble lignin)

To determine the fiber dimensions, 50 g of wood chips were employed in triplicate. The wood chips were comminuted into small fragments and subjected to maceration, carried out with samples weighing 1 g in a test tube containing a mixture of hydrogen peroxide and acetic acid (1:1 weight ratio) at 60 °C for 48 h. After maceration, the individualized fibers were washed with distilled water until all reagents were removed. Then the fibers were counterstained with a mixture of safranin and glycerin (Franklin 1945). The fibers were then placed on histological slides and observed using a trinocular microscope (Zeiss, USA) with reflected light with an attached camera. Ten fibers per slide were measured with 10 replicates, for a total of 100 measurements. Fiber length, width, lumen diameter and wall thickness was measured. The images of the fibers were evaluated by the Image-Pro Plus software (OSB Software, São Paulo, SP, Brazil). The following relationships between the fibers' dimensions were calculated: Runkel index (Equation 3), wall fraction (Equation 4), coefficient of flexibility (Equation 5), and slenderness index (Equation 6).

$$H\ (\%) = 100 - (E + TL) \quad \text{Equation 3}$$

$$WF = \left(\frac{2 \times T}{W}\right) \times 100 \quad \text{Equation 4}$$

$$CF = \left(\frac{DL}{W}\right) \times 100 \quad \text{Equation 5}$$

$$SI = \frac{L}{W} \quad \text{Equation 6}$$

Where:

RI = Runkel index

WF = wall fraction (%)

CF = coefficient of flexibility (%)

SI = slenderness index

T = wall thickness (µm)

LD = lumen diameter (µm)

W = fiber width (µm)

L = fiber length (µm).

For the kraft pulping process, an electrically heated rotary digester was employed. The equipment had eight individual vessels. The process was performed in triplicate. In each vessel, 45 g of wood chips were placed with 270 mL of white liquor (cooking liquor) in a proportion of 6:1 (liquor to wood – weight to weight). The cooking runs were carried out in triplicate for 60 min with a heating program of 90 min from room temperature to 166 °C. In each cooking process, eight levels of active alkali were assessed, varying from 10 to 24% (NaOH based), with intervals of 2% and sulfidity of 25%. At the end of the cooking, the residual black liquor was poured into a glass beaker equipped with a 400-mesh metallic screen to collect the pulp separately. The residual active alkali and pH of the black liquor were determined according to the standard T625 cm-14 (TAPPI 2014). The resulting pulps were washed with running water, processed in a hydrapulper, screened in a laboratory depurator with a 0.2 slot screen, centrifuged, and disaggregated. The residues from the depuration process were collected and oven-dried at 103 °C under forced ventilation until constant weight. For each experimental treatment, the total pulp yield, screened pulp yield, and screened rejects were determined along with the hexenuronic acid content and the kappa number, according to the procedures described by Chai *et al.* (2001) and the standard T236 cm-13 (TAPPI 2013).

The data on physical and chemical properties of the wood, and on fiber morphology were only analyzed descriptively. The variables for black liquor and cellulosic pulp were submitted to polynomial regression modeling. Before the analyses, the distribution of the variables was evaluated for normality (Shapiro-Wilk test), homoscedasticity (White test), and data independence (Durbin-Watson test).

RESULTS

The samples presented low basic density (average 0.296 g cm^{-3}) and high porosity (80.68%) (Table 1).

Regarding the analysis of the black liquor from the pulping process, the residual active alkali amount and pH increased with the alkaline charge in the cooking liquor (Table 2).

Total and screened pulp yield, residue content and kappa number decreased, and the hexenuronic acids increased with the increase in alkaline charge in the cooking liquor (Table 3).

The regression models to predict of the values of quality parameters of the black liquor and cellulosic pulp as a function of the alkaline charge were statistically significant at 1% probability (Table 4).

Table 1. Physical and anatomical properties and chemical composition of *Ochroma pyramidale* wood.

Physical properties	Mean ± SD	CV (%)
Basic density (g cm^{-3})	0.296 ± 0.007	2.51
Porosity (%)	80.68 ± 0.49	0.60
Anatomical properties		
Fiber length (mm)	1.280 ± 0.291	22.76
Fiber width (µm)	28.62 ± 5.35	18.71
Lumen diameter (µm)	19.27 ± 4.94	25.63
Cell wall thickness (µm)	4.68 ± 1.10	23.44
Wall fraction (%)	33 ± 8.10	24.30
Coefficient of flexibility (%)	67 ± 8.10	12.15
Slenderness index (%)	49 ± 17.07	35.10
Runkel index	0.524 ± 0.20	38.09
Chemical composition (%)		
Holocellulose	71.57 ± 0.33	0.47
Klason lignin	19.09 ± 0.12	0.62
Soluble lignin	3.56 ± 0.16	4.53
Total lignin	22.65 ± 0.20	0.90
Extractives	5.78 ± 0.35	6.06
Ash	1.03 ± 0.00	0.00

Mean ± standard deviation. CV = coefficient of variation.

Table 2. Residual active alkali content and pH of the black liquor from pulping of *Ochroma pyramidale* wood with increasing active alkali levels.

Active alkali (%)	Residual active alkali (g L^{-1})	pH
10	0.00 ± 0.00	8.53 ± 0.38
12	0.00 ± 0.00	9.17 ± 0.15
14	0.16 ± 0.27	9.77 ± 0.25
16	2.41 ± 0.38	10.70 ± 0.20
18	4.29 ± 0.42	11.17 ± 0.06
20	5.77 ± 0.91	11.87 ± 0.12
22	9.00 ± 0.76	12.13 ± 0.15
24	11.27 ± 0.97	12.23 ± 0.15

Mean ± standard deviation.

DISCUSSION

Our result for basic density corroborates that reported by Lobão *et al*. (2011) (0.250 g cm^{-3}) for mature trees of the same species, collected in Rio Branco, Acre, Brazil. Values of basic density ranging from 0.080 to 0.250 g cm^{-3}, and of porosity from 86 to 95% were reported for *Ochroma lagopus* Sw. wood (Follrich *et al*. 2010). The variation in these parameters may be owed to several factors, such as age, growing site and sample position in the trunk, among others (Borrega and Gibson 2015). The high porosity observed for *O. pyramidale* demands an increase in the white liquor/wood ratio to ensure enough free volume of liquor and an effective mass transfer during the pulping reactions. However, high white liquor/wood ratios would imply higher energy consumption for concentration of the black liquor on an industrial scale.

Low-density wood, as is the case of *O. pyramidale*, when directed to the production of pulp, can present high specific consumption of wood and low industrial productivity considering the volumetric limit of digesters (Queiroz *et al.* 2004; Mokfienski *et al.* 2008). These factors negatively impact the potential of *O. pyramidale* for the production of cellulosic pulp through the kraft process. Regarding the anatomical characteristics of the wood, the values of length, width, lumen diameter, and wall thickness of the fibers were higher than those reported for 10 eucalyptus clones (Gomide *et al*. 2005). The comparison with eucalyptus is relevant as a reference of the main raw material employed in Brazil for the production of short-fiber kraft pulp. However, the analysis of the dimensions of the fibers alone indicates low practical applicability when assessing the potential to produce different types of pulp and paper (Gomide *et al*. 2005).

Fibers with Runkel index lower than 1.0, as was the case of the *O. pyramidale* wood in this study (0.524) are considered as having good quality to produce paper (Foelkel *et al.* 1978). Likewise, the coefficient of flexibility of the fibers of our samples (67%) was also appropriate for pulp production, as values from 50 to 75% indicate a good contact surface among fibers and their consequent interconnection for making paper,

Table 3. Comparison of total (TY) and screened yield (SY), residue content (RC), kappa number (KN), and hexenuronic acids (HexAc) obtained from the pulping process of *Ochroma pyramidale* wood with increasing active alkali levels.

Active alkali (%)	TY (%)	SY (%)	RC (%)	KN	HexAc (µmol g^{-1})
10	72.96 ± 1.12	65.68 ± 3.03	7.28 ± 1.93	153.59 ± 5.33	1.81 ± 1.88
12	69.53 ± 1.56	67.31 ± 1.29	2.21 ± 0.28	145.41 ± 6.61	1.23 ±1.00
14	66.96 ± 1.73	64.71 ± 0.30	2.25 ± 1.45	132.27 ± 6.76	2.00 ± 1.69
16	60.97 ± 2.78	58.81 ± 1.51	2.16 ± 1.31	90.46 ± 8.56	10.12 ± 0.42
18	55.11 ± 1,59	54.41 ± 0,59	0.70 ± 1,05	52.02 ± 5.97	25.00 ± 5.98
20	51.70 ± 1.06	51.60 ± 0.95	0.00 ± 1.06	33.96 ± 3.06	34.44 ± 10.12
22	49.75 ± 0.32	49.72 ± 0.33	0.10 ± 0.12	19.18 ± 1.32	49.95 ± 6.76
24	48.28 (± 0.95	48.26 ± 0.94	0.03 ± 0.01	15.73 ± 1.34	51.67 ± 5.90

Mean ± standard deviation.

Table 4. Regression models to predict the parameters of black liquor and cellulosic pulp from the pulping process of *Ochroma pyramidale* wood as a function of the active alkali content. All models were statistically significant.

Model	Equation	R^2	Syx	F_{cal}
	Black liquor			
AA x AARes	$AARes = -3.7577 + 0.0253.AA^2$	91.21	0.98	239.77*
AA x pH	$pH = -2.1627 + 4.6017 \cdot log\ (AA)$	96.91	0.19	721.29*
	Celulosic pulp			
AA x RB	$RB = 1/0.078 + 0.0005 \cdot AA$	95.91	0.00	540.76*
AA x TY	$SY = 1/(0.0024 + 0,0037\sqrt{AA})$	92.91	0.00	302.58*
AA x SR	$TR = -5.2063 + 110.5830 \cdot 1/AA$	73.11	0.91	63.55*
AA x KN	$NK = exp^{(10.0390 - 1.4610\sqrt{AA})}$	95.77	0.56	521.41*
AA x AHex	$AHex = \sqrt{-5.0305 + 0.5278 \cdot AA}$	90.69	0.58	225.00*

AA = active alkali; AARes = residual active alkali; TY = total yield; SY = screened yield; SR = screen reject; KN = kappa number; AHex = hexenuronic acids; R^2 = coefficient of determination; Syx= standard error of the estimate; Fcal = calculated F; * = statistically significant at P ≤ 5%.

which can provide higher levels of mechanical resistance (Bektas *et al.* 1999). The wall fraction of the fibers was 33%, a value within the limit of 60% defined by Foelkel *et al*. (1978) as suitable for producing paper. High values of wall fraction are related to more rigid fibers that are more resistant to collapse, resulting in paper with low fiber connection. The slenderness index was 34.93, which is considered low when compared to the values reported for eucalyptus fibers, from 57.39 to 68,96 (Baldin *et al*., 2017). Fibers with low slenderness index usually produce paper with poor mechanical properties, low tearing resistance and bursting strength. Our values for the chemical components of the wood were similar to those reported by Lobão *et al*. (2011) for mature trees of the same species, collected in Rio Branco, Acre, Brazil [holocellulose (69.64%), total lignin (25.61%), and extractives contents (4.75%)]. The ash content in our samples was higher than that reported by Vale *et al*. (2007) for adult urban trees of the same species, collected in Brasília, central Brazil (0.59%).

In eucalyptus clones grown in Brazil for making pulp and paper, reported values of lignin content (23.3 to 31.7%) (Queiroz *et al*. 2004; Gomide *et al*. 2005; Mokfienski *et al*. 2008; Gomide *et al*. 2010; Cardoso *et al*. 2011) were similar to that determined here for *O. pyramidale* wood (25.61%). Hardwood for kraft pulping usually is processed aiming to yield unbleached pulp. To this end, the pulping conditions are adjusted to obtain pulp with a level of delignification (kappa number) compatible with the bleaching processes (Gomide *et al*. 2005).

The regression models for pulping process and black liquor parameters allowed to estimate that an alkali charge of 23.9% (NaOH basis) is required to obtain pulp with a kappa number equal to 18 (bleachable pulp). This value can be considered high and is related to the high hemicellulose content of the wood, as it is more sensitive to the alkaline hydrolysis that generates acids(Gullichsen 1999). These acids increase the pH of the black liquor, consequently decreasing yield (Cardoso and Gonçalez 2016). The alkali charge applied also had the goal to ensure a minimum level of residual alkali of 5 g L^{-1}, keeping the pH of the black liquor above 11, thus preventing reprecipitation of the lignin on the fibers, which can hamper further bleaching (Gullichsen 1999; Cardoso and Gonçalez 2016). Considering an alkali charge of 23.9%, the regression models predict a residual alkali amount of 10.8 g L^{-1} and pH of the black liquor of 12.4.

The general objective of the pulping process is to decrease the kappa number, increasing the alkali content and the pH of the black liquor without having significant losses in the yield of cellulosic pulp (Sixta 2006). However, the employment of high alkaline charges, despite favoring the decrease of the kappa number, can negatively influence the degradation of the carbohydrates, promote overload in the recovery boiler, and increase the production costs of the unbleached pulp (Gomide *et al.* 2005; Sixta 2006). In our study, as the alkali charge increased, there was a decrease in the total and screened yields and in the residue content. This can be explained by the increase in the intensity of the pulping reactions, resulting in the decrease of the kappa number, process yield and residue content. Considering a kappa number of 18, the regression models estimate a screened pulp yield of 48.5% without the generation of residue. A study of cellulosic pulp of wood from 10 eucalyptus clones reported mean pulp yield of 52% with a kappa number of 18 ± 0.5 (Gomide *et al.* 2005).

In Brazil, pulp and paper companies establish an average pulp yield of 50% based on the initial weight of dry wood as a minimum value to select wood types for the production of cellulosic pulp (Gomide *et al*. 2005). In this sense, our results for *O. pyramidale* wood can be considered satisfactory, as we tested a strain that was not subjected to genetic enhancement, as is the case with eucalyptus, which has been intensively modified for this use. Furthermore, no steps for optimization of the pulping process were carried out before the wood testing. The estimated parameter values for *O. pyramidale* pulp with a kappa number of 18, especially for residual active alkali, are indicative of the potential for optimization of the processing conditions, especially time and temperature, which can contribute to increase yield at the same kappa number (Gomide *et al*. 2005). The kappa number strongly affects important variables of the pulping process, such as the alkali charge applied, time, temperature, yield and residue content, among others (Sixta 2006).

Low amounts of residues were observed in the kraft pulping, which can be related to the effective impregnation and diffusion of the cooking liquor into the wood chips during the process (Cardoso and Gonçalez 2016). Most likely, the low basic density and high porosity favored the delignification reactions.

The hexenuronic acid amounts present in the *O. pyramidale* wood pulp can be considered low when compared to the values of 49.4 to 61.3 μmol g^{-1} reported for *Eucalyptus grandis* W. Hill ex Maiden. wood pulp (Ventorim *et al.* 2009). Low contents of this component were observed for pulp produced with alkali charges of 22 and 24%. The formation of hexenuronic acids is associated with modifications that occur in the methyl-glucuronic acids present in the xylans, and the concentration of these constituents is influenced directly by the pulping conditions (Ventorim *et al*. 2009). The presence of hexenuronic acids in the cellulosic pulp helps to minimize the terminal depolymerization of the xylans, increasing the pulp yield (Chai *et al*. 2001). However, the presence of these constituents is harmful to the bleaching process, since hexenuronic acids form covalent bonds with lignin, requiring higher consumption of chemical reagents during the process, as well as favoring the reversal of whiteness in pulp and paper (Sixta 2006).

Despite some unfavourable characteristics, the high holocellulose content resulting from the low ash, extractives and lignin contents indicate that *O. pyramidale* wood has potential for the production of pulp and paper through the kraft process.

CONCLUSIONS

Ochroma pyramidale wood presented chemical properties adequate for the production of kraft pulp due to its low contents of lignin, extractives and ash. Regarding anatomical characteristics, the Runkel index, flexibility coefficient and wall fraction favored fiber interconnection. However, the slenderness index was low. Residual active alkali and the pH of the black liquor were positively related with the alkali charge applied in the pulping runs. The decrease in the alkali charge brought about a decrease in the total and screened yields and an increase in the amount of hexenuronic acids. Nevertheless, this cooking condition with decrease in the alkali charge minimized the residue contents and the kappa number. Regression models estimated that, to obtain bleachable kraft pulp from *Ochroma pyramidale* wood with kappa number = 18, the active alkali charge should be 23.9% (NaOH basis), with a screened yield of 48.5%. Our results contribute to the evaluation of the wood quality of native Amazonian forest species for pulping purposes.

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