

Kraft pulping of gayam wood (*Inocarpus fagifer*)

Abstract

Gayam is a tree that grows in community forests with a deeply grooved trunk. This timber is less utilized due to limited information on the quality of gayam wood. This study aims to examine the potential use of utilizing gayam wood as a raw material for kraft pulp. The study was conducted using three individual trees (dbh 28-40 cm). The kraft process used active alkali concentrations of 13, 15, and 17% and sulfidity levels of 21 and 23%, for 2 hours at a maximum temperature of 170°C. Gayam wood had comparatively high average α -cellulose content (58.67%) and low lignin content (23.48%), as well as a fiber percentage of 57.75% and fiber length of 0.89 mm, indicating its potential as a raw material for pulp. The kraft pulp cooking produced screened yield, reject, and kappa number values ranging from 42.00 to 49.38%, 2.46 to 13.33%, and 8.49 to 22.10%, respectively. The optimum yield and properties of gayam wood pulp were obtained using 15% active alkali, yielding a kappa number value of 21.21, a screened yield of 48.86%, and the pulp chemical components (based on pulp weight) of 83.51% α -cellulose, 1.05% ethanol-toluene extractives, and 2.95% Klason lignin. The handsheets range strength at 200-300 CSF ranged from a tensile index of 30.36-44.74 Nm/g, a tear index of 2.43-3.07 mN.m²/g, and a burst index of 1.21-1.67 kPa.m²/g. The active alkali and sulfidity factors did not significantly affect the paper properties. The active alkali concentration significantly affected the kappa number, reject, and the holocellulose and hemicellulose contents of the pulp.

Keywords: community forests, sulfate pulping, pulp properties, kappa number, pulp chemistry

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Introduction

The high increase in the production capacity of the pulp and paper industry in Indonesia needs to be supported by the adequate raw materials availability. So far, raw materials for the pulp industry mainly come from *Acacia mangium*, *Acacia crassiparva*, and *Eucalyptus pellita* woods. The monoculture system applied in Industrial Forest Estate (HTI) offers management convenience. However, it increases the potential for pests and diseases to spread after the second rotation, especially in *A. mangium*.^{1,2} Therefore, exploration of other species abundant in Indonesia is necessary. The main obstacles are the limited data on basic wood properties and limited utilization.

Community forests have the potential to become alternative sources of raw materials due to their area size. One of the species often found is gayam (*Inocarpus fagifer* Forst.), which has only recently been used by the community as firewood. The community considers this species to have low value, be difficult to process, have a deeply grooved trunk (not cylindrical), and be a haunted or sacred tree. This tree is often found in rural areas and is distributed across various regions in Indonesia, including Java, Madura, Bali, Kalimantan, Sulawesi, and Papua.³

Beside wood as the raw material, the pulping process is important for producing the desired paper. The kraft (sulfate) process is a commonly used process in industry due to its advantages of producing high-strength pulp, being efficient for many wood species, tolerant to bark, and efficient in chemical recovery.⁴ In the kraft pulping process, several factors influence the quality of the pulp produced, including the wood species, effective alkali concentration and liquor-to-wood ratio, sulfidity, and the H factor (temperature and cooking time).⁵ Active alkali concentration and sulfidity have been extensively studied due to their influence on pulp yield, kappa number, and paper strength.⁶⁻⁹

Exploration of new raw materials for pulp and paper from community forests has been carried out previously.^{10,11} The emphasis is on identifying raw materials with medium density values. Therefore,

this study aims to determine the basic properties of gayam wood, pulp, and paper produced through the kraft process. To better understand the properties of the paper, a comparison of the chemical properties of the wood and pulp was also evaluated.

Material and methods

Wood samples

Gayam trees used as raw material samples were obtained from Dusun Bodeh, Ambarketawang, Gamping, Sleman, Special Region of Yogyakarta. Three gayam trees were cut (diameter at breast height of 28-40 cm). The trees were felled at the base of the trunk, approximately 50 cm above the ground (Figure 1). The parts used for this study were wood from the felling point to merchantable height. The logs obtained were cut into disks with a thickness of 3 cm. The disks were combined to obtain homogeneous samples. Wood disks were randomly selected from each tree and drilled to obtain sawdust (40-60 mesh) for chemical testing. For pulp cooking, the wood disks were chipped to dimensions of 3 × 3 × 0.3 cm.



Figure 1 Gayam (*Inocarpus fagifer* Forst.) trees and the wood cross section.

Density, fiber morphology, and cell wood proportion

The wood density was measured using a 1×1×1 cm specimen. The density value was obtained from the constant kiln-dry wood weight divided by its wet volume (water displacement method). For fiber

morphology, matchstick-like pieces of wood ($3 \times 0.2 \times 0.2$ cm) were macerated with a solution of glacial acetic acid and hydrogen peroxide in a ratio of 1:20. The resulting fibers were collected with a pipette and placed on a glass slide, with xylol and Canada balsam added to remove air bubbles and to brighten the color. Fiber measurements included fiber length (L), fiber diameter (D), lumen diameter (d), and cell wall thickness (W) using an Olympus BX51 polarizing microscope connected to a computer and saved in digital format for image analysis (Image Pro Plus software). Fiber length measurements used macerated samples with a $10\times$ objective magnification, while other dimensions used $40\times$. The derived values of fiber dimensions measured consisted of: Runkel ratio ($2W/L$), slenderness ratio (L/D), Luce's shape factor $[(D^2-d^2)/(D^2+d^2)]$, and solids factor $(D^2-d^2) \times L$.¹² Measurement of wood cell proportion was concurred in block specimens ($1 \times 1 \times 1$ cm) prepared by microtome cutting. Samples were taken from each tree with three replications. Prior to testing, the samples were soaked in a 30% alcohol solution. Calculation of cell proportions used microtome slice images at $40\times$ magnification.

Wood chemical properties

Determination of extractive content used sawdust equivalent to 2 g of dry weight. Extraction with an ethanol-toluene solvent (2:1, v/v) was conducted using a soxhlet apparatus for 8 hours (ASTM D1107-96, 2002), and separately extracted with hot water for 3 hours (ASTM D 1110-80, 2002). The extractive-free sawdust from the ethanol-toluene extraction was then measured for holocellulose content using the modified chlorous acid method,¹³ followed by cellulose content testing through 17.5% NaOH dissolution.¹⁴ Hemicellulose content was determined by reducing holocellulose content with α -cellulose content. Separately, Klason lignin content was measured on the extractive-free sawdust through 72% sulfuric acid hydrolysis (SNI 0492, 2008). Acid-soluble lignin content was measured using the clear solution obtained from the previous Klason-lignin test. The H_2SO_4 concentration of the solution was adjusted to 3%, and its absorbance was measured using a spectrophotometer at a wavelength of 205 nm.¹⁵ The H_2SO_4 solution was used as the test blank.

Kraft pulping

The cooking amount was 300 g chips (eq. oven-dried wood) with three replications. The active alkali concentrations used were 13%, 15%, and 17% (as Na_2O) while the sulfidity levels were 21% and 23%. The wood-to-liquor ratio was 1:4. Cooking of the wood chips was conducted using a laboratory-scale rotary electric autoclave (5 l) with a maximum temperature of $170^\circ C$, held for 2 hours, with 45 minutes required to reach the maximum temperature. After cooking, the pulp was washed and screened (100 mesh).

Pulp properties

The screened and reject yields were determined based on the initial dry weight of the chips. The chemical properties of the pulp tested included the kappa number (SNI ISO 302:2014) and its chemical components included ethanol-toluene extractives, holocellulose, and α -cellulose contents, using the same method as the wood samples. The lignin content was calculated using the kappa number, with the formula $\text{kappa number} \times 0.15$.¹⁶ The calculation of each chemical component of the pulp (%) was based on wood weight divided by the weight of the cooked chips (300 g). The weight of each chemical component in the pulp was obtained by multiplying the chip weight, the percentage of the component in the pulp, and the percentage of screened yield. The weight of holocellulose in the pulp was obtained by multiplying the percentage of screened yield by the chip weight and then subtracting the weight of lignin in the pulp.

Pulp beating and handsheets making

Pulp beating was conducted using a Niagara beater (SNI ISO 5264-1-2011). Measurement of the degree of beating (200-300 ml CSF) referred to SNI ISO 5267-2-2010. The beaten pulp was made into handsheets with a diameter of 15.9 cm and a grammage of 80 g/m² (SNI ISO 5269-1:2012).

Handsheet mechanical properties

Testing of handsheets mechanical properties included tear (SNI ISO 1974:2012), tensile (SNI 14-0437-1998), and burst (SNI ISO 2758-2011) indices. Handsheets were first conditioned at a temperature of $23 \pm 1^\circ C$ and relative humidity of $50 \pm 2\%$ (SNI 14-0402-1999).

Data analysis

Wood properties data were presented descriptively. Pulp and paper properties data obtained were then arranged and analyzed using analysis of variance (ANOVA) with a 95% confidence level. Post-hoc testing was conducted using Tukey's HSD. All calculations were performed using SPSS software version 16.

Results and discussion

Wood properties

The cross-section (x) taken from a microtome slice is presented in Figure 2, while the macerated fibers are shown in Figure 3. The results of cell proportions and fiber dimensions measurements are summarized in Table 1. It can be found that gayam wood fibers are straight, tubular, and have few tails. The comparison shows that the percentage of gayam wood fiber cells is lower when compared to *Acacia mangium*.¹⁷ However, the fiber cell proportion of gayam wood exceeds 50%, which aligns with the general proportion found in broadleaf woods. The proportions of axial and ray parenchyma cells in gayam wood are higher, whereas the proportion of gayam wood vessel cells is lower than in *A. mangium*.

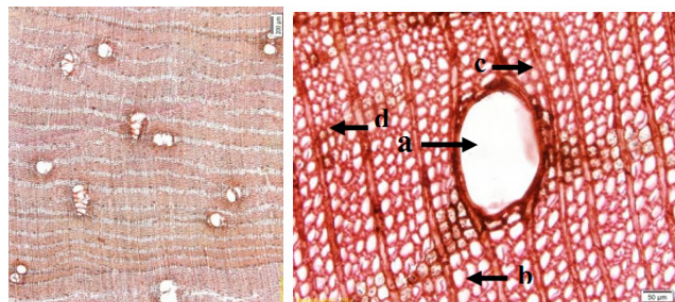


Figure 2 Microscopic cross-section (x) ($10\times$ dan $40\times$ magnification) of gayam wood. a: vessel, b: axial parenchyma, c: ray parenchyma, d: fiber.



Figure 3 Macerated fiber of gayam wood.

Wood density affects the yield and physical properties of paper. Gayam density value is suitable for pulp because it is classified as medium density (0.54 g/cm³) and is slightly higher than *A. mangium* (0.46 g/cm³). Technically, longer fibers provide better tear strength.^{18,19} The dimensions of gayam wood fibers compared to *A. mangium* show that gayam wood fibers are shorter, but the fiber and lumen diameters are wider (Table 1). The fiber wall of gayam wood is relatively thick, similar to that of *A. mangium*. Based on the derived value of the fiber dimensions, the Runkel ratio improves due to its lower value, whereas its slenderness ratio is lower compared to the *A. mangium*. A lower Runkel ratio improves bonding strength because fibers flatten more easily, while higher slenderness ratio is positively correlated with tear strength.^{20–22} High Luce's shape value and solid factor values are less desirable due to negative correlation with paper strength.¹² Those values for gayam fiber are lower than those of *Eucalyptus pellita*,²³ suggesting a potential positive effect on paper strength.

The levels of ethanol-toluene soluble extractives in gayam wood were lower, whereas the content of hot water soluble extractives was higher (9.03%) than in *A. mangium* species.¹⁷ This is likely due to the high proportion of parenchymal cells, which are the storage places for hot water soluble extractives: starch, high molecular weight phenolics, dyes, tannins, and gums. The holocellulose and α -cellulose content of gayam wood were higher, but its lignin and hemicellulose levels were lower compared to *A. mangium*.

Pulp chemical properties

In addition to the chemical properties of the wood, the chemical properties of the pulp were also observed based on the pulp weight (Table 2). When compared with the chemical values of the wood (Table 1), holocellulose and cellulose content increased, while extractive, hemicellulose, and lignin content decreased. The analysis of variance showed no significant interaction between the two factors, but a significant effect was observed for the active alkali factor between 13% and 17% on hemicellulose and holocellulose contents. An increase in these values occurred with the higher active alkali concentration, while a different pattern was observed in the lignin content. However, extractive and cellulose contents were not significantly affected. The sulfidity factor had no significant effect on any chemical components. As a comparison, jenitri (*Elaeocarpus ganitrus*) wood kraft pulp¹⁰ has higher extractive, holocellulose, and hemicellulose content but lower cellulose and lignin content than gayam pulp.

Based on the wood weight (Table 3), the holocellulose content of wood with an average of 77.54% was reduced to 40.22–47.96%, while cellulose had a smaller reduction (58.67% to 34.81–41.35%). The average hemicellulose content of 18.87% was reduced to 5.41–6.91%. The highest dissolution was observed in lignin, with its average wood content of 23.48% decreasing to around 1.04–1.78%. When compared to the chemical composition of birch pulp (broadleaf wood),²⁴ gayam wood pulp tends to have higher cellulose and extractive content but lower levels of other chemical components.

Cellulose is reduced proportionally in the range of 29–40% while hemicellulose decreases by 63–71%. Although an important component in papermaking, hemicellulose, which is a short polymer sugar, dissolves more easily during the pulping process.²⁴ High cellulose loss during the cooking process will reduce the yield and strength of the paper. In addition, the extractive content of gayam pulp residue, ranging from 0.94–1.42% based on pulp weight or 0.46–0.62% based on wood weight, is interesting to be studied. If the composition comes from lipophilic components that are insoluble in polar solvents, it may cause pitch problems in pulp sheets²⁵ and decrease the bleachability of the pulp.²⁶

Pulp yields and kappa number

The results of pulp yield and kappa number measurements are shown in Figure 4. The yield is classified into screened yield, residual yield (reject), and total yield. The screened yield tended to increase up to an active alkali concentration of 15% before decreasing, while the reject yield, total yield, and kappa number decreased with increasing active alkali concentration. The effect of sulfidity was not clearly observed. The analysis of variance showed that the interaction between sulfidity and active alkali did not have a significant effect on the total yield of gayam pulp. A highly significant effect ($p < 0.01$) was observed for the active alkali concentration on yield and kappa number, where differences based on Tukey test were observed between the 13% and 17% levels for both parameters.

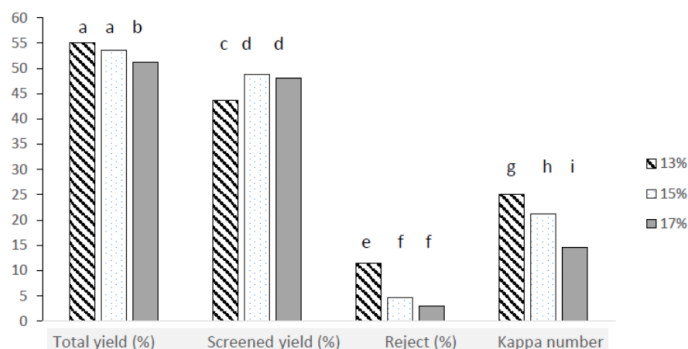


Figure 4 Yield and kappa number based on various active alkali concentrations. The same letters are not statistically different at $P < 0.05$ by Tukey's test.

The highest average screened yield at 15% active alkali concentration was 48.20%, with an average kappa number of 21.21. The yield aligns with the high proportion (57.75%) of gayam wood fiber cells, the high α -cellulose content (58.67%), the low lignin content (23.48%), and the low ethanol-toluene soluble extractive content (3.93%) (Table 1). Technically, kraft pulp yield is positively correlated with the percentage of fiber cells and the α -cellulose content of wood, but negatively correlated with extractive and lignin content.^{25,28–30}

Table 1 Physical and chemical properties of gayam wood

Wood properties	Gayam	<i>Acacia mangium</i> *
Density(g/cm ³)	0.54	0.46
Cell Proportion		
Fiber (%)	57.75±1.46	62.46
Ray (%)	13.73±3.19	9.77
Parenchyma (%)	22.21±4.11	15.66
Vessel (%)	6.31±0.63	12.11
Fiber Dimension		
Fiber length (μm)	890 ± 6.02	982
Fiber diameter (μm)	21.71 ± 3.86	14.29
Lumen diameter (μm)	14.08 ± 3.76	2.55
Cell wall thickness (μm)	3.82 ± 0.56	19.39
Derived values		
Runkel ratio	0.54 ± 0.15	0.37
Slenderness ratio	42.26 ± 9.83	51.29
Luce's shape factor	0.42 ± 0.08	-
Solid factor × 10 ³ (μm ³)	242.6 ± 52.2	-
Chemical properties (%)		
Ethanol-toluene extractives	3.93 ± 1.24	5.38
Hot-water solubility	9.03 ± 1.59	-

Table 1 Continued....

Holocellulose	77.54 ± 1.80	80.43
α-cellulose	58.67 ± 1.41	45.71
Hemicellulose	18.87 ± 3.18	34.72
Klason lignin	23.48 ± 1.46	31.3
Acid soluble lignin	0.11 ± 0.04	-

Remarks: *Yahya et al.,¹⁷ 7-year-old tree

Based on the chemical properties of gayam pulp (Table 2), the high yield produced aligns with the high α-cellulose content of the

pulp (82.88-84.19% based on pulp weight) and the high degree of delignification (remaining lignin of 2.20-4.23%). The analysis of variance showed a tendency for reject levels to decrease as the concentration of active alkali increased. An increase of 2% active alkali (from 13% to 15%) reduced the reject by 6.64%, while an increase from 15% to 17% reduced the reject by only 0.28%. As a comparison, at an active alkali range of 16-20%, the screened yield value was 49.10-51.37% and the kappa number was 12.73-17.60%, for *A. mangium* pulp, while *E. pellita* pulp showed screened yield values of 46.50-47.85% and kappa numbers of 11.50-16%.⁷

Table 2 Chemical components of gayam pulp based on pulp weight on various active alkali and sulfidity concentration factors

Active Alkali (%)	Sulfidity (%)	Chemical components of pulp (%)				
		Ethanol-toluene extractives	Holocellulose	α-cellulose	Hemicellulose	Lignin
13	21	1.11	87.64	84.19	3.46	3.41
	23	1.42	85.28	82.88	5.54	4.23
	Average	1.26	86.46 a	83.53	4.50 c	3.82 f
15	21	1.17	88.82	83.28	2.45	3.03
	23	0.94	88.92	83.74	5.39	2.88
	Average	1.05	88.87 ab	83.51	3.92 cd	2.95 fg
17	21	1.3	90.87	83.22	7.65	2.2
	23	1.27	90.49	83.4	7.13	2.43
	Average	1.28	90.68 b	83.31	7.39 e	2.31 g
Jenitri *		1.56	92.48	82.21	10.27	2

Remarks *: Laksono and Lukmandaru.¹⁰ The same letters on the same column are not statistically different at $P < 0.05$ by Tukey's test.

Active alkali and sulfidity are the main factors affecting kraft pulp cooking.³¹ This study showed that OH⁻ (hydroxide) has a greater effect on delignification than HS⁻ (hydrogen sulfide). In the kraft process, hydrogen sulfide primarily ions reacts with lignin, while sugar degradation is influenced by hydroxide ions.²⁴ Active alkali is more dominant in breaking down lignin, as reflected in the kappa number measurements (Figure 4). The trend indicates that higher active alkali concentrations lead to greater lignin degradation in the pulp.

Technically, higher active alkali concentration results in lower kappa numbers.³² The kappa number of gayam wood pulp decreased as the active alkali concentration increased. A 2% increase in active alkali led to a drastic reduction in the kappa number. This pattern, along with the decrease in screened yield aligns with trends reported for *A. mangium* and *E. pellita*,⁷ *A. nilotica*,³¹ and *Acacia* hybrid.⁸

In general, the kappa number of gayam pulp is bleachable as the value approaching 15-20.³¹ The kappa number values from cooking using active alkali concentrations of 13% and 15% were >20, while the kappa number value at an active alkali concentration of 17% was below the standard value. The active alkali concentration of 15% is considered to produce the best kappa number because it is close to the lower limit of the standard and not extensively low. The low kappa value at an active alkali concentration of 17% might be due to cellulose degradation by chemicals, as observed in the screened, reject, and total yield patterns in the pulp (Table 3). This supports the use of 15% active alkali to achieve the best yield.

Table 3 Chemical components of gayam pulp based on wood weight on various active alkali and sulfidity concentration factors

Active Alkali (%)	Sulfidity (%)	Chemical components of pulp (%)				
		Ethanol-toluene extractives	Holocellulose	α-cellulose	Hemicellulose	Lignin
13	21	0.5	43.66	38.05	5.6	1.54
	23	0.6	40.22	34.81	5.41	1.78
	Average	0.55	41.94	36.43	5.5	1.66
15	21	0.57	46.88	40.26	6.62	1.46
	23	0.46	47.96	41.35	6.61	1.42
	Average	0.51	47.42	40.8	6.61	1.44
17	21	0.62	46.33	39.42	6.91	1.04
	23	0.61	46.98	40.16	6.82	1.17
	Average	0.61	46.65	39.79	6.86	1.1
Birch *		0.5	51 a	34	17 b	2

Remarks *: Gellerstedt,²⁴ a = sum of α-cellulose and hemicellulose contents, b = sum of glucomannan and xilan content.

Sulfidity did not affect any yield parameters, including total, reject, or screened yield. This is likely because the sulfonation reaction produced by the two concentrations had a uniform effect on lignin. In addition, the insensitivity of sulfidity is attributed to the low concentration range used in this experiment. Previously, a sulfidity range of 15-30% produced decreases in screened yield and kappa number in *Acacia* hybrid.⁸ Similarly, the use of 0-32% sulfidity resulted in decreased screened yield and kappa number for *Pinus bolleana* pulp, with the optimum kappa number (17-25) obtained at a high sulfidity range of 24-32% and lower active alkali of 14-16%.⁶ The research could be improved by using a wider sulfidity range than that applied in this study because the reject level (3.03%) remained high at the highest active alkali concentration (Figure 4).

Mechanical properties of handsheets

The mechanical properties of gayam handsheets were evaluated using tensile, burst, and tear indices at a beating degree of 200-300 ml CSF. The tensile, tear, and burst indices ranged from 30.36-44.74 Nm/g, 2.43-3.07 mN m²/g and 1.21-1.67 kPa m²/g, respectively (Table 4). The analysis of variance showed no significant effect of the factors tested. This indicates that the strength properties of gayam paper were not highly affected by differences in pulp kappa numbers. It is assumed that the physical properties of the pulp had a greater influence on handsheets strength. The effect of active alkali on handsheets strength at higher concentrations has previously been observed for asoka (*Saraca indica*),¹¹ *Acacia nilotica*,³³ as well as *A. mangium* and *E. pellita* woods.⁷

Table 4 Mechanical properties of gayam handsheet on various active alkali and sulfidity factors

Sulfidity (%)	Active Alkali (%)	Tensile index (Nm/g)	Tear index (mN m ² /g)	Burst index (kPa m ² /g)
21	13	38.48	2.56	1.49
	15	30.36	2.49	1.24
	17	34.21	3.07	1.43
23	13	33.43	2.72	1.21
	15	44.74	2.55	1.67
	17	39.98	2.43	1.56
SNI 6107-2009		45	5.5	2.5
<i>Acacia mangium</i> *	16-20	22-95	5.6-10.0	0.9-6.5
<i>Eucalyptus pellita</i> *	16-20	13-79	2.1-10.9	0.5-5.6

Remarks * = bleached kraft pulp on various beating degree⁷

Chemical properties, cell proportions, fiber dimension, and derived values of gayam wood are adequate when associated with paper properties. However, the strength value of gayam paper is lower than the Indonesian National Standard (SNI) for bleached broadleaf wood pulp.³⁴ The maximum values of gayam pulp are also lower than those of bleached *A. mangium* and *E. pellita* pulp.⁷ Therefore, gayam pulp is recommended for paper that does not require high strength or as reinforcement in mechanical pulp mixture. The relatively low strength value may be due to the intensive cellulose dissolution during pulping (Table 3). This needs to be confirmed by measuring pulp viscosity in further research. Improvements in cooking conditions by modifying temperature and cooking time should be studied to achieve low reject values without excessive sugar removal. Another factor to consider is optimizing the beating degree to increase paper strength.

Conclusion

The basic properties of gayam wood are generally suitable for pulp and paper production. The advantages of gayam wood include its relatively high screened yield at a low active alkali concentration (15%). In general, the physical properties of gayam wood handsheets are relatively low, which is likely due to the high cellulose dissolution during the process. Improvement of the cooking process should be pursued to determine the optimum conditions for enhancing handsheet strength.

Acknowledgments

None.

Conflicts of interest

The authors declare that there are no conflicts of interest.

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