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Acid sulfite pulping of *Acacia mangium* and *Eucalyptus pellita* as a pretreatment method for multiproduct biorefineries

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Abstract

Conversion of biomass into saleable biochemicals and fuels requires the use of a pretreatment to enable subsequent processing. Acid sulfite pulping is one of the most cost-effective strategies, because the chemicals are inexpensive and the technology is available on an industrial scale. It also allows the simultaneous production of cellulosic fibers and lignosulfonate. However, too little is known about the feasibility of acid sulfite pulping of tropical hardwoods. The objective of this research was to gain a better understanding of the response of *Acacia mangium* and *Eucalyptus pellita* in acid sulfite pulping. The plantation-grown hardwood chip samples were obtained from Sabah, Malaysia. The sulfite cooking experiments were carried out in autoclaves with temperatures of 130°C and 140°C and varied chemical charges. The results revealed that a cooking temperature of 140°C was needed to reach kappa numbers below 30, but this also resulted in much reduced fiber length and higher fines content than 130°C, probably due to the intensified acid hydrolysis. To reach kappa numbers below 20, more severe cooking conditions are needed. These results demonstrate that using *A. mangium* and *E. pellita* as feedstocks allows feasible production of chemical pulp and sulfonated lignin, which are intermediate products for biorefineries.

KEYWORDS

chemical pulp, fermentable sugars, lignocellulosic biomass, spent sulfite liquor, sulfite process

1 | INTRODUCTION

The importance of many acacia and eucalyptus species as feedstock for pulping has been growing in the past decades. Both *Acacia mangium* and *Eucalyptus pellita* have been planted for pulpwood and timber in many

areas of the subtropics and tropics, including parts of South East Asia.¹ These feedstocks are sometimes underutilized and thus available as relatively inexpensive raw material for nonconventional industrial processes, as well. On the other hand, the technologies for converting biomass into saleable products are in constant need of

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development to meet techno-economic criteria required for investment.

Sulfite pulping starts with the feeding of wood chips and cooking liquor to a digester. The sulfite cooking liquor is a reaction product of sulfur dioxide, a base cation, and water; it also contains an excess of SO_2 . Base cations employed in industrial sulfite pulping processes are ammonium (NH_4^+), calcium (Ca^{2+}), magnesium (Mg^{2+}), or sodium (Na^+). There are several modifications of sulfite pulping, which are designated according to the pH of the cooking liquor.² This makes sulfite pulping very flexible and allows the production of many pulp grades which can be used for a broad range of applications. Currently, acid sulfite pulping process (pH range 1–2) is almost exclusively employed for dissolving pulp production.^{2,3} Dissolving pulp has typically high cellulose content while lignin, hemicellulose, and extractives contents are very low; the desired contents depend on the dissolving pulp grade. Industrial experience on sulfite pulping with mainly softwood as the feedstock has proved that sulfite process can be successfully incorporated with hydrolysis of sugars from hemicellulose and cellulose.⁴ The latter creates opportunities for various biorefinery concepts, which are based on sulfite pulping technology, where the main products are biochemicals or bio-based transportation fuels instead of papermaking fibers or dissolving pulp. Moreover, this type of biorefinery can also be designed to “swing” between different products or their combinations. It is apparent that product yield distribution and the overall productivity of the plant vary significantly^{5,6}—as the feedstock availability and product prices fluctuate with market conditions.

The sulfite pulping does not degrade lignin to the same extent as the kraft cooking process. The generated lignosulfonates are useful by-products that have a steadily growing demand and a relatively stable market. In 2010, the total global production of lignosulfonates was approximately 1.2 million tons which accounted for about 10% of the lignin in the biomass that was pulped.⁷ Major applications include concrete additives (plasticizers), dispersing agents, and industrial binders.^{8,9} These modified natural polymers are nontoxic and non-hazardous, which is why they are increasingly finding new uses, such as water treatment applications, or in the food industry.¹⁰ Moreover, significant research efforts are allocated to the continuous development of new lignosulfonate products, such as those made of hardwood with well-engineered properties. By employing posttreatments, hardwood lignosulfonate properties may be tailored to specific applications.⁷ The latter allows a major improvement in the product profitability of hardwood-based lignosulfonates, which are often considered of less value than those produced from softwoods.

In relation to the dominant kraft pulping process, sulfite pulping has a rather comparable environmental impact today.¹¹ For example, the sulfur dioxide remaining in the combustion gases is recycled, and the elimination of all chlorine bleaching chemicals is easier to achieve with sulfite pulps, which are known to have better bleachability than kraft pulp. Moreover, as the spent sulfite liquor (SSL) contains a significant amount of lignocellulosic material, and it is currently being converted to various value-added products instead of being discharged with effluents.

This paper summarizes a laboratory research on *A. mangium* and *E. pellita* and their feasibility as feedstocks for biorefinery. Our research included chemical analyses of the hardwood samples, as well as laboratory cooking experiments and the characterization of the obtained pulp samples for kappa number and fiber quality. Fiber length and fines content may not appear to be important attributes in this research since the resulting pulp is not intended for papermaking. However, too short fibers, fiber fragments, and fines are difficult to retain in the pulp in the subsequent washing and screening operations, which results in decreased yield. It is, therefore, the processability of pulp that makes the fiber length and the fines content important parameters in a feasibility assessment for a biorefinery process. In this investigation, we also analyzed lignosulfonate samples isolated from SSL for their molecular weight (MW), which is among the most important characteristics that contribute to lignosulfonate functionality.

2 | EXPERIMENTAL

2.1 | Materials

The plantation-grown *A. mangium* and *E. pellita* chips were obtained from a company that operates man-made plantations and a chip mill, all of which are located in Sabah, Malaysia (the island of Borneo). Samples of the chips are shown in Figure 1. The sapwood of *A. mangium* is white and clearly defined against the darker brown heartwood. *E. pellita* chips have more uniform reddish-brown color, making it more difficult to distinguish sapwood from heartwood. The chips had been dried before shipping, and both samples had a moisture content of approximately 6%. The low moisture content had to be taken into account by ensuring sufficient impregnation of the cooking liquor. According to previous experience published elsewhere, sulfate (kraft) cooking of eucalyptus chips with a very low moisture content appeared to be manageable with only minor process-related challenges.¹²



FIGURE 1 *Eucalyptus pellita* (left) and *Acacia mangium* chips (right) from East Malaysia

The hardwood chips were screened with a laboratory-scale classifier according to SCAN-CM 40:94 test method, and only the accept fraction was used for acid sulfite cooking experiments.

The Natural Resources Institute Finland (LUKE) analyzed screened hardwood chip samples (the accept fraction) for carbohydrates, lignin, and extractives. Hemicelluloses were determined using method based on acid methanolysis¹³ and cellulose with acid hydrolysis.¹⁴ The total lignin content of the samples was determined according to KCL method N:o 115b:82. The KCL method included also a correction due to the ultraviolet (UV) absorption of carbohydrate degradation products.¹⁵ More information about this analytical procedure and how it relates to some alternative lignin analysis methods has been published elsewhere.¹⁶ Prior to the Klason lignin analyses, the lipophilic extractives were first extracted with hexane and thereafter the hydrophilic extractives with an acetone:water (95:5 v/v) mixture with accelerated solvent extractor ASE-350 (Dionex, Sunnyvale, CA). A more detailed description of the extractives analysis has been published previously.¹⁷ The chemical constituents of the wood samples are summarized in Table 1.

The cooking tests were carried out in 2L autoclaves. Acid sulfite cooking liquor contains higher free SO₂ content compared with bisulfite cooking liquors. In this research, acid sulfite chemical charges were free SO₂ of 21%, 24%, and 28% while the corresponding combined SO₂ charges were 4.2%, 4.8%, and 5.6%, respectively. The base cation was sodium. In addition to the varied chemical charges, varied temperatures at 130°C and 140°C were also included in the tests.

Finally, the resulting pulp samples were screened, and the reject content of each pulp sample was determined gravimetrically. Kappa numbers of the pulp samples were measured in accordance to ISO 302:2004 test method. Length-weighted average fiber length and fines content of the pulp samples were measured with a Kajaani

TABLE 1 Chemical composition of *A. mangium* and *E. pellita* wood samples

Chemical constituent	<i>A. mangium</i>	<i>E. pellita</i>
Extractives, %	2.6	1.4
Lignin, %	32.9	37.3
Cellulose, %	43.2	42.5
Hemicellulose, %	20.7	26.8
Sum of components, %	99.4	108.0

FS-200 fiber length analyzer. It measures fiber length via an indirect optical method. One of its strongest points is the large number of fibers sized in a short time.¹⁸

2.2 | Chemical characterization of the acacia and eucalyptus samples

According to the lignin analyses, the wood of *A. mangium* contained 32.8% lignin while the lignin content of *E. pellita* was 37.3%.

Cellulose contents of both hardwood species, measured as the concentration of galactose, were found to be quite similar. *A. mangium* and *E. pellita* had cellulose contents of 43.2% and 42.5%, respectively.

The hemicellulose analysis results revealed that *A. mangium* had a lower total concentration of hemicellulose than *E. pellita*, 207 and 268 mg/g, respectively. These values can also be expressed as 20.7% and 26.8%, respectively. The breakdown of total hemicellulose contents to individual hemicellulose components is presented in Figure 2.

All individual hemicellulose components analyzed in this research exhibited a higher content for *E. pellita* than *A. mangium*. Xylose was the most dominant hemicellulose component for both wood species. The xylose content of *E. pellita* was 137 mg/g while that of *A. mangium* was 107 mg/g. The most prominent difference between these two wood samples was the content of galactose: 24.7 mg/g for *E. pellita* while *A. mangium* only had 9.5 mg/g.

In addition, *A. mangium* and *E. pellita* contained significant amounts of extractives, 2.6% and 1.4%, respectively. The extractive content for *A. mangium* wood in this research was much higher than extractive analysis results published elsewhere.¹⁹ Moreover, virtually all remaining knots or “knotwood” in the original chip lot had been separated in screening from the accept fraction; they mostly ended up in the overthick fraction. Hardwood knots typically have a higher content of extractives than the corresponding heartwood.²⁰ However, according to the results of a study on *A. mangium* grown in

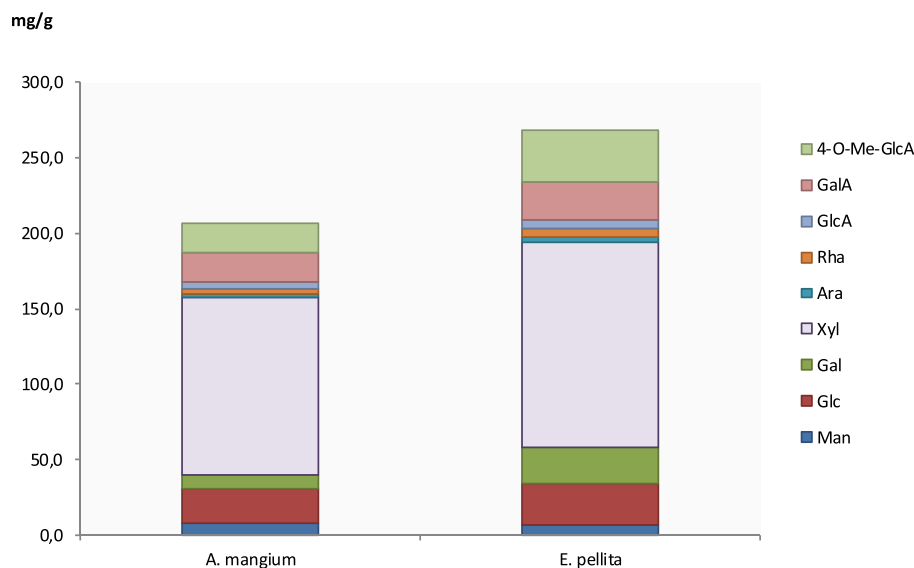


FIGURE 2 Hemicellulose analysis results of *A. mangium* and *E. pellita*. The list of hemicellulosic components (top to bottom) 4-*O*-methyl glucuronic acid, galacturonic acid, glucuronic acid, rhamnose, arabinose, xylose, galactose, glucose, and mannose

Indonesia, knots contained smaller amounts of lipophilic extractives than heartwood or sapwood.¹⁹

The extractive content of *E. pellita* wood in this research was relatively low when compared to the information published elsewhere.²¹

Table 1 summarizes all analyzed chemical constituents and their concentrations (%).

The acetyl groups of hemicellulose were not quantified in this research. They would probably have added “a couple of percentage points” to the sum of the chemical components. In addition, it is obvious that wood contains small amounts of inorganics, typically some 1–2% for debarked wood.²² The inorganics are usually measured as residual constituents or the ash content of wood.

2.3 | Characterization of lignosulfonates in spent liquor samples

Among other important characteristics, lignosulfonate functionality is defined by its MW. In order to perform an accurate determination of the weight-averaged MW of lignosulfonates, M_w , we employed size-exclusion chromatography (SEC) in combination with light-scattering techniques. More detailed descriptions of the method are available elsewhere.^{23,24}

3 | RESULTS AND DISCUSSION

3.1 | Acid sulfite cooking experiments

For both acacia and eucalyptus, kappa number decreased substantially with increased cooking temperature. That

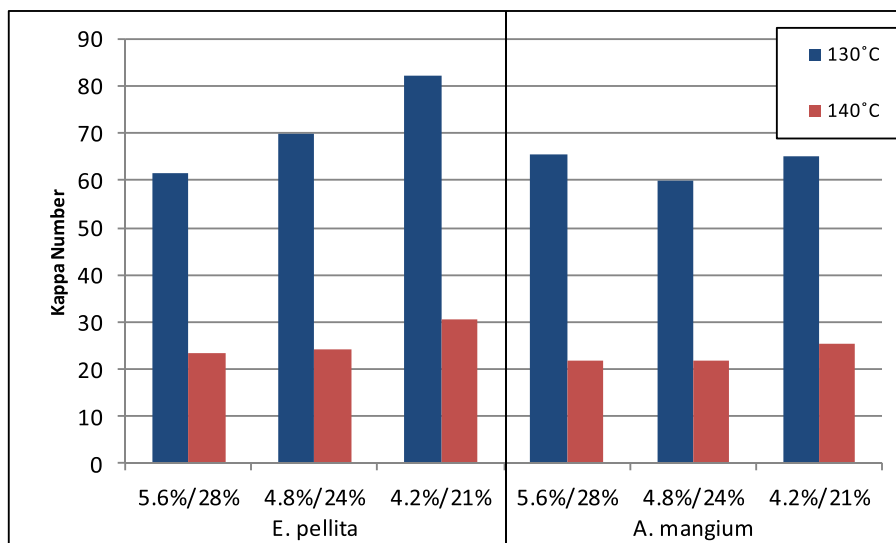
also indicated that at 130°C, the pulp was not properly digested. All kappa numbers from the cooking at 130°C were very high, 60 or higher. Eucalyptus has somewhat higher kappa numbers than acacia with the same cooking conditions, but the difference is not consistent. The cooking temperature of 140°C resulted in kappa numbers between 23 and 31 for eucalyptus and between 22 and 26 for acacia. This confirms that in terms of kappa number, the response of *A. mangium* to acid sulfite cooking was a little better than that of *E. pellita*. Kappa number results of the acid sulfite cooking experiments are presented in Figure 3.

The limited extent of delignification with *A. mangium* and *E. pellita* wood may be attributed to their dense wood structures. The exact densities of our hardwood chip samples were not known, but it is apparent that an intensively managed industrial tree plantation aims at maximizing the performance of trees, and thus, the highest possible wood density is also an important objective. For example, Ismaili et al. reported a basic density of 540 kg/m³ for *A. mangium* grown in Malaysian Borneo.²⁵ Other studies have reported basic densities of 464 and 494 kg/m³ for *A. mangium* grown in Malaysia.^{26,27} The density of *E. pellita* grown at a plantation in Indonesian Borneo has been reported to vary from 556 to 652 kg/m³.²⁸ For comparison, another two genera, namely, *Macaranga spp.* and *Endospermum spp.*, also fast-growing timber trees grown in Malaysia, only have basic densities of 371 and 389 kg/m³, respectively.²⁷ Moreover, in the case of *A. mangium*, the relatively high content of extractives may further retard the delignification by generating cross-links with lignin during acid sulfite pulping.

The visual appearance of the pulp samples after the acid sulfite pretreatment shows no apparent difference



FIGURE 3 Kappa numbers after sulfite cooking. The chemical charges of combined and total SO_2 are presented between the x -axis and wood species



between the two species. However, both soft and hard particles of undercooked wood material can be observed. It seems that the cooking temperature of 130°C resulted in higher reject content than the cooking temperature of 140°C. Pulp reject contents, calculated as % on oven dry pulp, are presented in Table 2.

As can be seen in Table 2, the lower cooking temperature of 130°C generated more reject than 140°C. Cooking with acacia resulted in much higher reject than with eucalyptus. Especially with the lower cooking temperature (130°C), the reject content of *A. mangium* was unusually high, up to 21% on oven dry pulp.

All pulp samples were also characterized for fiber length and fines using Kajaani FS-200 fiber length analyzer after sulfite cooking and the removal of reject. Kajaani FS-200 is specifically designed to evaluate fiber-length distributions of cellulosic fibers.¹⁸ Pulp and paper industries frequently discuss fiber length in terms of length-weighted average. Fiber quality results are presented in Figure 4.

According to the results, the longest fibers (measured as length-weighted average length, L_w) were obtained with the lower cooking temperature (130°C), between 0.65 and 0.70 mm for both acacia and eucalyptus pulp samples. With the higher cooking temperature, 140°C, the average fiber length was reduced significantly. The impact of cooking chemical charge was a much less pronounced. *A. mangium* pulp samples had slightly longer fibers than those of *E. pellita*, when compared with the same cooking conditions. The highest fiber length values presented here are lower than those published in the literature, for example, 0.93 mm for *A. mangium*²⁶ and 0.904 mm for *E. pellita*,²⁸ which reflect their full potential as papermaking fibers. With acid sulfite cooking, it is hardly possible to reach the full potential of fiber length

TABLE 2 Pulp reject contents (% on oven dry pulp) after acid sulfite pretreatment

Sample	5.6%/28%	4.8%/24%	4.2%/21%
<i>E. pellita</i> , 130°C	0.7	1.1	1.9
<i>A. mangium</i> , 130°C	15.8	13.8	21.0
<i>E. pellita</i> , 140°C	1.2	0.5	0.9
<i>A. mangium</i> , 140°C	3.5	5.9	7.6

Note: The chemical charges of combined and total SO_2 are shown in the top row.

due to acid hydrolysis of cellulose polymers. *A. mangium* pulp samples had clearly higher fines contents than those of *E. pellita* at both cooking temperatures.

We also characterized lignosulfonates isolated from the SSL samples. By fermentation of the hexoses and by chemical degradation of the pentoses, purified lignosulfonate products are obtained.⁸ Lignosulfonate functionality in various industrial uses is greatly defined by its MW. For various applications such as dispersants, a greater MW is beneficial.²⁹ Figure 5 summarizes weight-averaged MW (M_w) results of all six lignosulfonate samples.

The M_w values of *E. pellita* lignosulfonates were substantially higher than those of *A. mangium* under the same pulping conditions. There appears to be a positive correlation between lignosulfonate M_w and lignin content in wood. The average M_w ranged from 11 600 to 18 900 g/mol for *E. pellita* and 7600 to 10 700 g/mol for *A. mangium*. The lower cooking temperature of 130°C resulted in higher average M_w than 140°C. This suggests that the higher M_w values were not resulting from extensive lignin condensation under severe cooking conditions.³⁰ In all, both *A. mangium* and *E. pellita* yielded

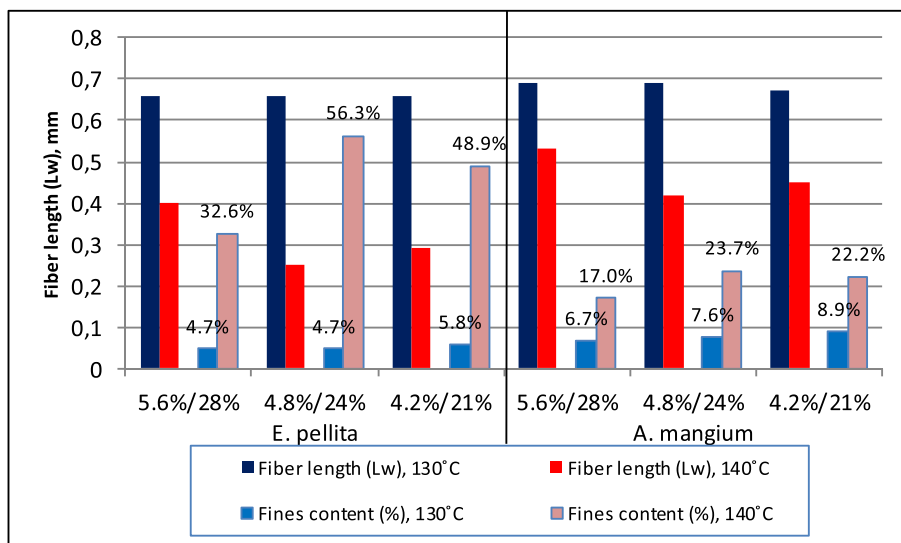


FIGURE 4 Fiber analysis results (Kajaani FS-200). The chemical charges of combined and total SO₂ are shown between the x-axis and wood species. Fines content results (%) are shown numerically on top of each bar

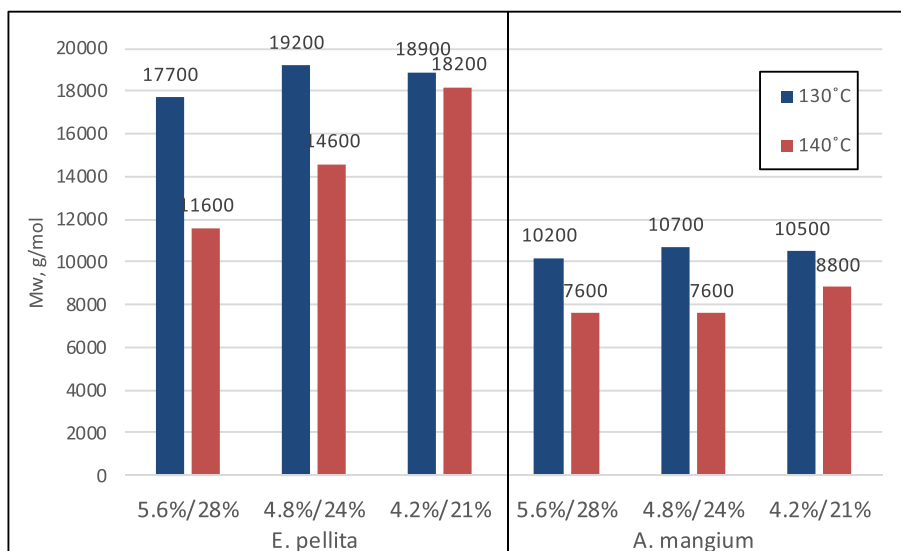


FIGURE 5 Weight-averaged molecular weight (M_w) results of lignosulfonates from SSL samples

quite high M_w values, especially with the cooking temperature of 130°C. For comparison, M_w values of *E. globulus* and *E. grandis* lignosulfonates have been reported to be much lower, 6300 and 5700 g/mol, respectively.²⁴ On the other hand, lignin obtained from laboratory kraft pulping of *E. pellita* was found to have a very large polydispersity, but the M_w values of different fractions averaged 2500–2600 g/mol only.^{31,32}

Dispersants and concrete admixtures probably account for the broadest use of lignosulfonates and sulfonated lignin. This is due to their appropriate MW (10 000–50 000 g/mol).⁹ In this range of M_w , commercial lignosulfonate qualities are typically produced of SSL from softwood pulping. However, according to Ghorbani et al., several commercial spruce lignosulfonate products had M_w values ranging from 5780 to 11 390 g/mol.³³ In

our research, the high M_w of *E. pellita* lignosulfonate provides therefore very interesting opportunities for product engineering, although it is obvious that further research on this subject is necessary. Other important lignosulfonate parameters, such as the degree of sulfonation, the content of methoxyl groups, or polydispersity, must also be investigated.

4 | CONCLUSIONS

According to the results, *E. pellita* chips showed more positive response in sulfite pretreatment than *A. mangium*. After acid sulfite pretreatment of 130°C, kappa numbers and average fiber lengths of the both species were similar, but reject amounts of acacia were much



higher than those of eucalyptus; 140°C cooking temperature was needed to reach kappa numbers below 30, which, in turn, resulted in much reduced fiber length and high fines content, apparently due to the acid hydrolysis of cellulose polymers. Moreover, to reach kappa numbers below 20, even more severe cooking conditions would be needed. Nevertheless, our results demonstrate that it is possible to delignify both *A. mangium* and *E. pellita* by acid sulfite pretreatment to reasonably low kappa number, which allows the further processing of the separated fibers as well as the carbohydrates dissolved in the SSL. The hydrolysis of hemicelluloses into monomer sugars allows their utilization as feedstock for fermentation into biofuels and biochemical products. In addition, our results show that the resulting lignosulfonates had unexpectedly high MW, especially those of *E. pellita*. The production of high MW lignosulfonate may open up very interesting opportunities for lignin product engineering. In all, the pulp, the dissolved carbohydrates, and the resulting lignin may well find their uses as raw materials for multiproduct biorefineries. The data obtained from this research can serve as fundamental information in the selection of suitable wood species to be applied as feedstock in an acid sulfite-based biorefinery process.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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