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Soda Lignin from Pre-treated Birch Residue and Its Possible Application in Biocomposite

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***Abstract*—In pulping, the pretreatment of wood has advantages over untreated wood due to enhanced pulp yield and reduced chemical and energy consumption. This study examined the effect of alkaline treatment of birch sawdust on the chemical composition of the sawdust and soda lignin separated from black liquor obtained by soda pulping of the treated birch sawdust. It was found that the alkaline pre-treatment of birch sawdust led to a remarkable removal of hemicelluloses and reduced its mechanical strength. A lower content of acidic and methoxyl groups in the chemical composition of the alkaline lignin obtained by soda pulping of the pretreated sawdust showed the advantage of polycondensation reactions that proceeded during the formation of its primary structure over destruction. The possibility of using the modified soda lignin in a biocomposite was demonstrated. The composite material was fabricated from a blend of recycled polypropylene and polylactic acid, reinforced with ammoxidized wheat straw. The conducted mechanical tests demonstrated the potential of the modified soda lignin to replace more than 30% of the synthetic compatibilizer in the composite formulation without deteriorating the properties of the biocomposite.**

Keywords*—*sawdust pretreatment, modified soda lignin, composite, wheat straw, mechanical properties.

I. INTRODUCTION

The integration of advanced biomass conversion processes and state-of-the-art equipment in biorefineries allows for the simultaneous production of multiple outputs, maximizing resource efficiency and economic viability. The biorefinery approach is particularly significant for modern wood chemistry and technology, as it promotes the utilization of lignocellulosic materials, unlocking their potential through innovative chemical pathways.

There are many ways to pretreat wood biomass, such as steam explosion, autohydrolysis, acid hydrolysis, ammonia treatment, chemical pulping, solvent extraction, etc. These methods are directed to an increase of the reactivity of the biomass components, making the extraction process easier and more efficient [1]-[3]. For an increase in the intensity and selectivity of the delignification process during wood pulping, and for a decrease in the consumption of cooking reagents, water, and heat consumption, the pretreatment of wood biomass with alkaline solutions (NaOH, $Ca(OH)_2$, NH_4OH) along or together with other additives is cost effective and promising [4]-[6]. The main aim of the initial stage of the alkaline pre-treatment of wood biomass is the partial destruction of the lignocellulosic matrix to facilitate the removal of hemicelluloses and lignin during pulping.

Technical lignin extraction from wood occurs during different pulping processes used for cellulose production. It is known that approximately 50–70 million tons of technical lignins as a by-product are produced at pulp and paper mills per year. However, the main amount of technical lignins is used for power generation at the mills, but the formed wastewater as a sequence of pulping can negatively affect the environment. Only 1–2% of the produced lignin is utilized for obtaining value-added products [7]. Given lignin's significant economic and industrial potential, extracting high-purity lignin from lignocellulosic biomass is essential for its effective subsequent usage. Technical lignins are used as binders,

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plasticizing and dispersing agents, phenol substitutes in phenol-formaldehyde resins, bio-additives to plastics and composites, carbon fiber precursors, etc. [8]-[12].

Technical lignins obtained by alkaline pulping can be classified depending on the presence of sulfur, namely, the sulfur-containing lignin – Kraft and sulfur-free lignin – soda [13]. Soda lignin is a by-product of the cooking of hardwood and herbaceous plants [5]-[14]. Compared with Kraft pulping, the major advantage of soda pulping is the absence of sulfur, which is a reason for the unpleasant odor during the production of cellulose as well as the closeness of the chemical composition and the structure of soda lignin to that of native lignin. The molecular weight of the soda lignin is lower than that of kraft lignin and lignosulfonates [1]-[6].

This work studied how alkaline pretreatment affects the chemical composition of birch sawdust and the lignin extracted from it via soda pulping. It also investigated the potential usage of the modified soda lignin in a wood-plastic composite filled with wheat straw.

II. MATERIALS AND METHODS

Birch sawdust was supplied by a company Latvijas Finieris Ltd. The fractionated sawdust with particles ⩽ 250 μm was characterized by elemental analysis (Elementar Analysensysteme, Langenselbold, Germany) and wood composition (lignin, cellulose and hemicelluloses) according to standard methods (TAPPI T222 om-11 standard, [15], TAPPI T203 cm-99 standard [16], and TAPPI T264 om-97 [17] standard, respectively). The elemental analysis of the birch wood sawdust revealed the following composition: 48.1% carbon (C), 5.9% hydrogen (H), 45.7% oxygen (O), 2% nitrogen (N), and 0.1% sulfur (S).

Wheat straw from a Latvian peasant farm was processed by drying, crushing, and sieving to obtain lignocellulosic fibers ⩽ 250 μm. Then, the fibers were ammoxidized by the mechanochemical treatment in planetary ball mill (Retsch, Germany) with zirconia balls at the rotational speed of 300 rpm for 30 min. The ammoxidation was performed with the reaction mixture of $NH_4OH/(NH_4)_2S_2O_8$ at an O/NH_3 mass ratio of 0.2. After the treatment, the ammoxidized wheat straw fibers were washed to a neutral medium, dried and milled for obtaining the particles less than 100 μm.

The pretreatment of birch sawdust was carried out by alkali hydrolysis as it was shown early [18], using 0.5-5.0 g/L NaOH solution at 60 °C for 5 h with a sawdust-to-water mass ratio of 1:20. After the procedure, the isolated pretreated sawdust was washed, dried and was ready for pulping.

Soda lignin samples were obtained from initial and the pretreated birch sawdust by pulping in a laboratory reactor under the following conditions: NaOH concentration – 4.5%, hydromodulus – 1/7, duration – 90 min, temperature – 165 °C and pressure 0.6 MPa. The purification of the precipitated lignin samples was done by three repeated dissolutions with 0.1M NaOH, and subsequent precipitation with concentrated sulfuric acid. Each time, the lignin precipitate was carefully washed on the filter with diluted sulphuric acid (1%) and then with hot water. After triple dissolution/precipitation, the content of Klason lignin in both the lignin samples was more than 90%. The content of Klason lignin was determined according to TAPPI T222 om-02 standard [15].

The chemical composition of the lignin samples was determined by elemental (Elementar Analysensysteme GmbH, Langenselbold, Germany) and functional analysis, according to Zakis [19].

The content of acid groups in the lignin samples was found from the curves of conductometric titration obtained with a Radiometer Analytical Titrolab 90 with autoburette ABV 901 (Radiometer Analytical SAS, Villeurbanne Cedex Lyon, France) at 25 °C.

Fourier Transform Infrared (FT-IR) spectroscopy was performed using a Perkin Elmer Spectrum One apparatus (Perkin Elmer, Waltham, MA, USA) at the range of wavenumbers from 4000 to 450 cm^{-1} (30 scans), at a resolution of 4 cm^{-1}. The tablets were prepared by mixing 20 mg of a sample with 200 mg of KBr, and then the tablet compression method was used.

Ultraviolet (UV) spectroscopy of lignin samples was carried out using a Genesys 10UVUV/VIS spectrophotometer (Termo Fisher Scientific, San Jose, CA, USA).

The composite samples reinforced with the ammoxidized wheat straw were obtained by the extrusion and molding methods using HAAKE MiniLab II and MiniJet II (Thermo Fisher Scientific, Germany) at a temperature of 175 °C, a circulation time in the two-screw extruder of 5 min, the screws rotational speed of 130 rpm, and a molding pressure of 60 MPa at a temperature of 120 °C. Maleated polypropylene (Licocene PP MA7452, Clariant) was used as a compatibilizer (CB).

A binary polymer blend of recycled polypropylene (rPP) and polylactic acid (PA) at a mass ratio of 95/5 was served by a polymer matrix in a new wood-plastic composite. rPP had a density - 0.9 t/m^3, a melt flow index - 5.2 g/10 min at 230 °C and 2.16 kg (Nordic Plast Ltd., Olaine, Latvia). Polylactic acid 2002D was purchased from NatureWorks LLC (USA) and had a density of 1.25 t/m^3 and a melt flow index of 5–7 g/10 min at 210 °C and 2.16 kg.

The milling of birch sawdust was carried out with a planetary ball mill (Retsch, Haan, Germany), but wheat straw with a knife mill and sieved using a mill "Pulverisette 0" (Frisch GmbH, Idar-Oberstein, Germany).

III. RESULTS AND DISCUSSION

A. *Pretreatment*

The hydrolytic destruction of wood residue due to the alkali treatment was evaluated by analysing the changes in the composition of the sawdust, specifically in cellulose, lignin, and hemicellulose content. Their content before and after the alkaline treatment of the sawdust is presented in

Fig. 1. It has been seen that, with increasing the NaOH concentration, the relative content of cellulose in the pretreated sawdust increases by 25.5 %, but the content of hemicellulose decreases almost twice relative to their content in the initial sawdust. The change in the relative content of lignin in the pretreated sawdust is expressed to a lesser extent than cellulose and hemicellulose that may be associated with the resorption of lignin on the cellulosic surface.

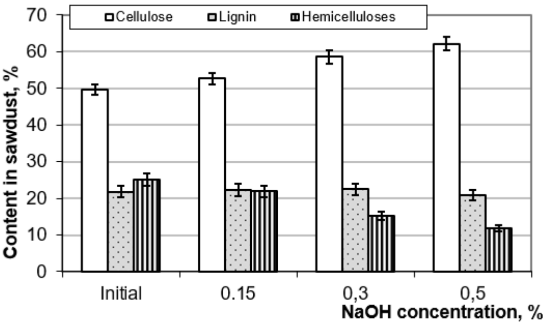

Fig. 1. Content of cellulose, hemicellulose and lignin in birch sawdust before and after the alkaline treatment

The effect of hydrolytic degradation of the lignocellulosic matrix on its mechanical strength was assessed by analyzing the fractional composition of both the initial sawdust and the sawdust treated with a 0.5% NaOH solution after milling in a planetary mill. The changes in the lignocellulosic matrix composition of the treated sawdust led to the reduction of its mechanical strength (Fig.2).

According to Fig. 2, as the alkali concentration increases from 0.15% to 0.50%, the amount of fine particles less than 100 µm in the hydrolyzed sawdust rises from 8 to 12 times when compared to the corresponding particle content in the initial milled sawdust. It is assumed that the pronounced growth of the finest particle content in the pretreated sawdust fractional composition, particularly at the highest alkali concentration, is associated with the essential removal of hemicelluloses from the lignocellulosic matrix of the treated sawdust.

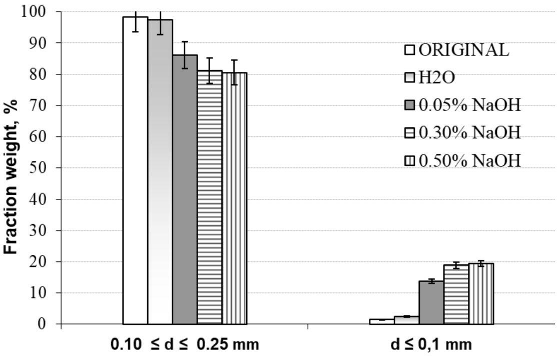

Fig. 2. Content of fractions in initial and pretreated sawdust depending on NaOH concentration after milling

Therefore, based on the generalization of the results obtained, it can be concluded that the highest extent of the hydrolytic destruction of the lignocellulose matrix occurs when birch sawdust was treated with a 0.5% NaOH aqueous solution. The initial and alkali-treated birch sawdust samples are shown in Fig. 3.

B. Characteristics of soda lignin samples

Table 1 shows the chemical composition of the lignin samples obtained by the soda pulping of the initial sawdust and sawdust treated with 0.5% NaOH solution as well as the yield of biorefined products – soda lignin and pulp. The obtained results indicate that the yield of both lignin and pulp is higher for the pretreated sawdust than for the initial one. According to Table 1, the soda delignification of the alkali-treated sawdust, compared to the pulping of the initial sawdust, leads to a decrease in the content of both phenolic hydroxyl and carboxyl groups. A decrease in the content of the acidic groups in soda lignin from the pretreated sawdust is accompanied by its notable demethoxylation.

Fig. 3. Initial and pretreated birch sawdust

TABLE 1 CHEMICAL CHARACTERISTICS OF SODA LIGNIN AND PULP

Sawdust	**Pulp**		**Soda lignin**						**Yield, % from wood**	
	Yield, %	*Lignin content in pulp, %*	*Yield, %*	*OCH_3, %*	*Acid (OH) groups, %*			*Klason lignin, %*	*Pulp + lignin*	*Water-soluble destruction products*
					OH_{all}	*OH_{fen}*	*OH_{COOH}*			
SLI	51.7	3.2	19.6	15.9	6.2	2.7	3.5	91.9	71.3	28.7
SLT	54.5	2.5	22.5	13.4	4.1	1.7	2.4	92.8	77.0	23.0

The content of methoxyl groups in the soda lignin from the treated sawdust (SLT) is 15.1% less than in the lignin from the initial sawdust (SLI). The total yield of the obtained biorefined products derived from the treated sawdust - pulp and lignin - is 5.7% higher than in the case of cooking SLI, while the pollution of the environment with the water-soluble destructive products obtained during the pulping of the treated sawdust is lower. A decrease in the content of phenolic hydroxyl and carboxyl groups along with the enhanced demethoxylation, may indicate prevalence of polycondensation reactions over depolymerization processes, proceeded between the released lignin fragments during the delignification of the pretreated sawdust.

The changes in the chemical compositions of the lignin samples were consistent with the results of the obtained FTIR spectra analysis presented in Fig. 4. In both FTIR spectra, the bands at 1323 cm^{-1} and 835 cm^{-1} are assigned to syringyl rings and are characteristic bands for hardwood lignin. The significantly lower absorbance band observed at 3410 cm^{-1}, which is associated with hydrogen-bonded hydroxyl groups found in phenolic and phenyl propane aliphatic structures, along with the bands at 2933 cm-^{1} and 2837 cm-^{1} due to C-H stretching vibrations in methoxyl groups in the FTIR spectrum of SLT, indicates a reduced presence of these functional groups in this sample compared to SLI.

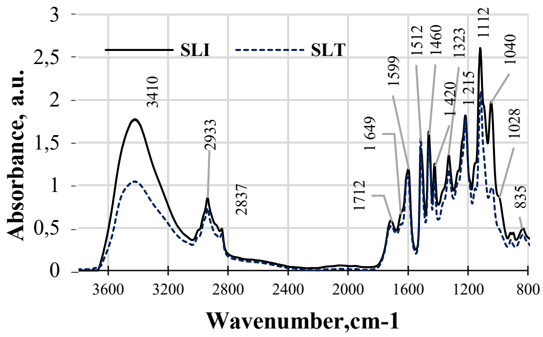

Fig. 4. FTIR spectrum of soda lignin samples obtained from initial and pretreated sawdust

This observation further confirms that the structure of SLT is more condensed than that of SLI. The FTIR peaks in the range of 1712-1595 cm^{-1} are assigned to the presence of unconjugated and conjugated carbonyl and carboxyl groups in lignin. Within this range, SLI has a higher absorbance than SLT which may indicate a lower content of these functional groups in the lignin sample obtained from the hydrolysed sawdust, which is consistent with the results presented in Table 1. The bands at 1596 cm^{-1}, 1512 cm^{-1}, and 1420 cm^{-1} are assigned to skeletal vibrations and C=O group stretching in the syringyl and guaiacyl aromatic rings. The relatively higher intensity of the band at 1512 cm^{-1} compared with that of the band at 1595 in both spectra is gained by the features of hardwood lignin structure. The bands at 1323 and 1215 cm^{-1} correspond to the syringyl structures of lignin. The most significant difference in the FTIR spectra of the lignin samples is observed in the range of absorbance bands of 1112-800 cm^{-1}. These bands are attributed to C-C ring vibrations, overlapped with the absorbance of C-O vibrations in aliphatic hydroxyl and ether C-O-C groups. The intensity of the absorbance bands in this band range is remarkably higher for SLI than for SLT. A decrease in the intensity of the absorbance bands which corresponds to the presence of aliphatic and phenolic hydroxyl groups, and ether bonds in SLT in comparison with SLI may also indicate its more cross-linked chemical structure.

Fig. 5 shows the UV spectra of both soda lignin samples dissolved in an alkaline solution. The absorbance in the UV region results from the ionization of phenolic hydroxyl groups of phenylpropane units, as well as structures containing carboxyl groups, hydroxystilbenes, quinones, and charge transfer complexes. The absorbance of SLI is higher than the absorbance of SLT, which may be due to a more cross-linked polymer structure of SLT decreasing the phenolic hydroxyl group content.

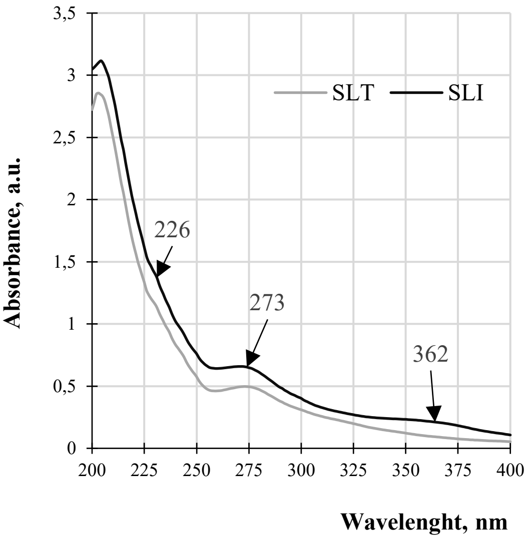

Fig. 5. UV spectrum of soda lignin obtained from original and pretreated sawdust

The pronounced peaks at 205 nm, 273 nm, and a less pronounced shoulder at 227 nm are presented in both UV spectra of the lignin samples. At the same time, a maximum at 362 nm assigned to carboxyl groups is absent in the UV spectra of SLT.

Thus, the analysis of the results presented in Table 1, FTIR and UV spectra, allows to conclude that SLT had a more condensed structure than SLI.

C. Composite samples

The composite samples reinforced with the wheat straw microparticles (< 100 µm), containing the compatibilizer (CB) and modified soda lignin (MSL) were fabricated by extrusion and moulding methods. For increasing the compatibility of the initial wheat straw with the polymer matrix, it was ammoxidized by introducing amide and imide groups in its functional composition by using a mechanochemical method. The ammoxidized wheat straw had the following elemental composition: 47.52% C, 43,6% O, 6.16% H and 2.72% N. It contained 54.1% cellulose, 22.3% lignin and 18.3 % hemicellulose.

The modified soda lignin (MSL) was used as a functional additive. It was modified by the mechanochemical method in the planetary ball mill at a rotational speed of 600 rpm for 5 h in the presence of $(NH_4)_2S_2O_8$. MSL had the following elemental composition: 57.62% C, 35.97 % O, 5.85% H, and 0.56% included N, S, and trace of metals. Its chemical composition contained 10.75% hydroxyl, 11.18% methoxyl and 3.7% carbonyl groups.

The formulations of the obtained biocomposites are given in Table 2.

Table 2 Formulations of composite samples

Nr.	**Formulations**			
	Polymer matrix, rPP/PA, %	***Ammoxidized wheat straw, %***	***CB, %***	***MSL, %***
1	50	50	-	-
2	50	47	3	-
3	50	47	2	1
4	50	47	1	2

Table 3 and Table 4 show the results of testing the mechanical properties of the composite sample filled with 50% ammoxidized wheat straw. It can be seen that the obtained sample is characterized by low values of mechanical strength in both tension and bending and insufficient impact viscosity.

Table 3 Tensile properties of composite samples

Nr.	**Tensile properties**		
	Tensile straight, MPa	***Tensile modulus, MPa***	***Elongation, %***
1	15.5±1.8	1044±22	3.2±0.4
2	28.9±2.1	920±17	3.8±0.3
3	29.3±1.9	952±18	3.8±0.3
4	25.1±2.2	992±21	3.5±0.5

Table 4 Bending properties and impact viscosity of composite samples

Nr.	**Bending properties and impact viscosity**			
	Bending strength, MPa	***Bending modulus, MPa***	***Deformation, %***	***Impact viscosity, kJ/m^2***
1	21.7±2.0	2337±25	1.4±0.2	13.8±1.5
2	38.9±2.3	2557±22	2.4±0.3	23.7±1.7
3	39.7±2.3	2591±23	2.4±0.3	24.0±1.6
4	34.5±2.5	2479±27	2.6±0.4	22.1±1.8

The comparatively high values of the mechanical moduli and the low values of bending deformation indicated the stiffness of the resulting composite structure. To improve compatibility between the polymer matrix and the amooxidized wheat straw, a compatibilizer - maleated polypropylene in the amount of 3% was introduced into the composite formulation. According to the results of mechanical testing, this leads to an increase of 86% in tensile strength, 79.1% in bending strength and 72.3% in impact toughness. At the same time, the increase in the values of tensile and bending moduli is less significant and amounts to only 9.6% and 10.8%. At the same time, the composite sample becomes less stiff.

Taking into account the fact that the modified soda lignin contained an increased content of hydroxyl and carbonyl groups capable of interacting with maleic anhydride, it was of interest to study the possibility of its use as a compatibilizer in combination with maleated polypropylene at different mass ratios. According to tables 3 and 4, the mixture containing 2% CB and 1% MSL has the same positive effect as 3% CB on the compatibility of the matrix with the filler in the composite. The mechanical properties of the composite sample in the presence of the new compatibilizer are practically the same as in the case of the composite containing maleated polypropylene. This indicates that the modified soda lignin can replace more than 30% of the synthetic compatibilizer in the composite consisting of a rPP/PA polymer matrix and the ammoxidized wheat straw. The substitution of CB with the modified soda lignin in the composite by more than 60% leads to a slight decrease in mechanical properties and impact strength. This may indicate a physicochemical interaction between CB and MSL during the processing of the composite material, for which a CB: MSL ratio of 2:1 may be optimal.

IV. CONCLUSION

The pretreatment of wood residue before pulping resulted in the extraction of soda lignin, which had a lower content of acidic and methoxyl groups, indicating a more cross-linked structure. Mechanical tests of the biocomposite, which was filled with the ammoxidized wheat straw particles, demonstrated that the mechanochemically modified soda lignin could effectively replace over 30% of the synthetic compatibilizer in the biocomposite without deteriorating its mechanical properties.

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