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Development of Ligno-Polyol for the Production of Polyurethanes

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ABSTRACT

The overall objective of this work is to explore a new non-food renewable supply of industrially viable polyol to make polyurethane products for industrial applications. Lignin has been considered as an industrial waste coming from the paper and textile fiber industries. From a chemical point of view lignin is a natural polyphenol and polyol. Due to its high molecular weight and its complex structure, however, those functional groups are not easily accessible. As a consequence the industrial application of lignin is very limited.

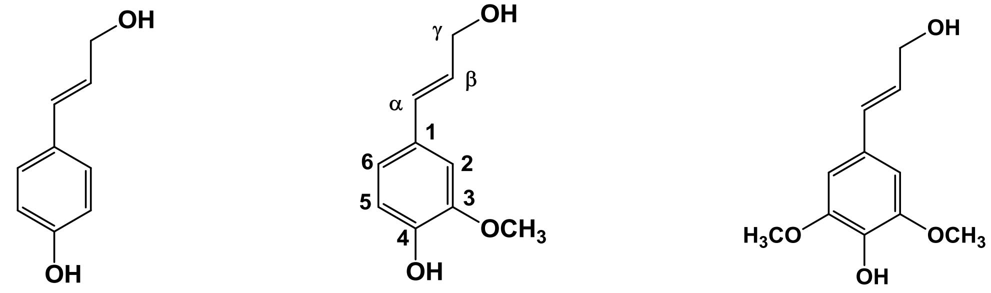
In this work, different approaches have been used for the incorporation of kraft lignin in petroleum-derived commercial polyols for the production of polyurethanes (PU) with minimum energy and chemical used and also minimum negative impact on the environment. The results have demonstrated that, beside lignin chemistry, lignin size and morphology, applied shear force, mixing temperature, and chemistry of polyol have also great influence on the dispersion and interaction of lignin with petroleum based polyols and thus on the PU performance. In this development the studied lignin was well dispersed and interacted with the polyols and they also participated in the PU network structure, resulting in great improvement in the glass transition temperature and the mechanical properties as well.

INTRODUCTION

With ever increasing global concern over rapid depletion of non-renewable fossil fuel based resources, there is a strong interest worldwide in developing suitable technologies that can derive chemicals and materials from renewable biomass. Modification and characterization of bio-based polymers for preparation of higher valued green chemicals and bio-based products favors the future use of lignin biomass components with substantial environmental and economical benefits.

Lignin is the second most naturally abundant biopolymeric substance after cellulose; the natural polymer makes up to 40% of forestry and up to 30% of certain lignocellulosic materials in agricultural residues [1, 2]. Lignin is an amorphous, highly branched polyphenolic macromolecule of complex structure with high molecular weight. The chemical structure of lignin polymer consists primarily of phenyl propanoid units mainly p-coumaryl alcohol (H), coniferyl alcohol (G) and/or sinapyl alcohol (S) (see Figure 1) [3, 4]. These components are cross-linked together in three dimensions via a radical coupling process during its biosynthesis. Its physical and chemical properties are highly dependent on the wood species, botanic region, and the isolation processes [5, 6].

Lignin is available in large quantities as a byproduct of the pulp and paper industries. The most abundant industrial lignins are from kraft and sulfite pulping processes. Lignins obtained under these processes undergo significant structural changes and are no longer identical to their original native structures [7-10]. Due to its complex nature and undefined chemical structure, the industrial applications of lignin are rather limited. Presently lignin is utilized almost exclusively as fuel to power the evaporators of the chemical recovery processes and liquor concentration system of pulp mills [11].



p-Coumaryl alcohol **(H)**

Coniferyl alcohol **(G)**

Sinapyl alcohol **(S)**

Figure 1. *Chemical structure of lignin monomeric units.*

However, based on its interesting functionalities and properties, lignin offers perspective for higher added value applications in renewable products. Lignin contains several important functional groups including phenolic hydroxyl, carboxylic groups carboxylic, and hydroxyl groups that characterize lignin as a versatile reagent in many chemical reactions and the overall reactivity of different lignins depends on their chemical compositions [12]. The particular presence of aliphatic hydroxyl groups makes lignin a typical polyol for PU production. [12]. Different lignin samples contain different types of hydroxyl units depending on the isolation processes.

The aim of this investigation was to evaluate the potential of a forestry lignin sample for substituting polyols in polyurethane production. The influence of lignin on the formation and properties of PU was investigated.

EXPERIMENTAL

Materials

Kraft lignin Indulin AT from MeadWestwaco was used in this study. Polycaprolactone diol, CAPA 2043, (Mw = 400g/mol) and polyethyleneglycol, PEG 400 (Mw = 400g/mol) were obtained from Perstorp and Acros Organics, respectively. A methylene diphenyl 4,4'-diisocyanate based isocyanate (MDI) was provided by Enerlab 2000 Inc.

Characterization of Lignin

Lignin moisture was determined gravimetrically after heating in an oven at 105 ± 5 °C for 24 h. Lignin ash content was determined gravimetrically after calcination at 600 °C for 3.5 h. Elemental analysis was performed by Galbraith Laboratories, Inc. (Knoxville, TN, USA).

Morphology of lignin particles was observed by scanning electronic microscope (SEM) (JEOL JSM-6100) at a voltage of 15 kV. Before SEM observation, the samples were coated with Au/Pd to ensure good conductivity.

QUANTITATIVE ^{31}P NMR SPECTROSCOPY

Quantitative ^{31}P NMR spectra of lignin were obtained using published procedures [13-15]. Lignin was treated with either 2-chloro-1,3,2-dioxaphospholane (reagent I) or 2-chloro-4,4,5,5-tetramethyl-1,3,2-dioxaphospholane (reagent II) to first form the phosphorous derivatives. All NMR experiments were carried out at 298 K on a Bruker Avance 500 NMR spectrometer (Bruker BioSpin Ltd, Milton, ON) operated at ^{1}H frequency of 500.13 MHz and equipped with a 5 mm broadband inverse probe. The spectra were recorded with 32768 data points and a spectral width of 60606 Hz. A relaxation delay of 5 s was used and the number of scans was 512. The ^{31}P chemical shifts were referenced with respect to water signal at 132.2 and 121.1 ppm corresponding to reagent II and reagent I, respectively. Spectra were processed and analyzed using Bruker TOPSPIN1.3 software package. The content of hydroxyl groups for reagent I was obtained by integration of the following spectral regions: *erythro* (136.7-134.5 ppm), *threo* (134.5-133.8 ppm), primary hydroxyl units (133.8-132.3 ppm), guaiacyl phenolic hydroxyls (130.6-128.8 ppm), p-hydroxyphenyl phenolic hydroxyls (128.3-127.8 ppm), carboxylic acids (127.8-127.1 ppm). The content of hydroxyl groups for reagent II was obtained by integration of the following spectral regions: aliphatic hydroxyls (149.0-145.4 ppm), condensed phenolic units (144.4-140.3 ppm), guaiacyl phenolic hydroxyls (140.3-138.6 ppm), p-hydroxyphenyl phenolic hydroxyls (138.6-137.0 ppm), carboxylic acids (135.7-133.4 ppm).

FOURIER TRANSFORM INFRARED (FT-IR)

Fourier transform infrared (FT-IR) spectra of powdered solids were obtained using a Bruker Tensor Series FT-IR Spectrometer in the Attenuated Total Reflectance (ATR) analysis mode. Spectra were collected from 4000 to 500 cm^{-1} for 64 scans and with 4 cm^{-1} resolution using a zinc selenide (ZnSe) crystal.

GEL PERMEATION CHROMATOGRAPHY (GPC) OF ACETYLATED LIGNIN

In order to allow solubilization of Indulin AT lignin in THF, lignin was acetylated according to Lundquist's procedure [16] with slight modifications. After acetylation in pyridine/acetic anhydride (1/1) and ethanol washings, the isolated materials were washed and centrifuged three times with water, dissolved in a mixture of dioxane/water (15:4 v/v) and freeze dried under reduced pressure.

GPC analysis was performed using a multi-detection system from Viscotek (Houston, TX) consisting of a Model 302 Multiple Detector Platform, including a PDA detector, refractive index detector, a four capillary viscometer and a 2 angles laser light scattering detector (GPCmax Integrated pump, autosampler and degasser). The light scattering detector was not equipped with fluorescence filters and was only used for qualitative monitoring. The software OmniSEC from Viscotek was used for data collection and calibration. Separation was performed in tetrahydrofuran (THF) by injecting 100 μL of 1.5-2 mg/mL solutions of acetylated lignin in THF into thermostatically controlled SuperRes columns (35 ºC; PAS-102, PAS102.5, PAS-103 L, each 300 mm x 8 mm; PolyAnalytik (London, ON). The flow rate was of 1 mL/min. Molar masses were determined with universal calibration with the refractive index and the viscosity detector. The universal calibration curve was based on polystyrene standard from Polymer Laboratories (Amherst, MA) and Sigma-Aldrich (Oakville, ON) using the molar masses determined by the manufacturer and the intrinsic viscosities measured by the apparatus.

Fabrication of Ligno-PU

Firstly lignin was mixed with polyol by means of different methods and devices. The first method was a "room temperature with low-speed mixing" process (RM), in which the lignin and polyol were stirred at room temperature for 20 min by a conventional mechanical stirrer at 1200 rpm. The second method was a "high temperature with low-speed mixing" process (TM), in which the lignin and polyol were stirred at 90°C for 20 min by a conventional mechanical stirrer at 1200 rpm. The third one was a "high temperature and high-speed mixing" process (TS), in which the lignin and polyol were stirred at 90°C by means of an Ultra-Turax homogenizer at high speed (13,500 rpm) for 10 min. The quality of dispersion of lignin in the studied polyols at various mixing methods was qualitatively evaluated by Nikon Eclipse optical microscope (OM) in the transmission mode.

The obtained ligno-polyol mixture was then cooled down to room temperature prior to mixing with isocyanate using the molar ratio NCO/OH= 1.5. The ligno-PU samples for testing were prepared by casting in a closed mold in a dried atmosphere at room temperature followed by a post-cure at 100 ^{o}C for 4 h. Different concentrations of lignin in polyol have been used, however, only the 10 wt% lignin in polyol is reported in this paper.

Characterization of Ligno-PU

The quality of dispersion of the lignin in the final PU products was observed by OM using reflexion mode. A differential scanning calorimeter (DSC) Perkin-Elmer DSC7 was used to evaluate the glass transition temperature (Tg) and the degree of curing of the PUs.

Tensile properties of the PUs were measured on the MTS Instron machine according to ASTM D638 at ambient atmosphere and room temperature.

RESULTS AND DISCUSSIONS

Lignin Characterization

Moisture and ash contents of Indulin AT lignin were found to be equal to 2.99 and 2.86 %, respectively.

Quantitative ^{31}P NMR analysis allowed quantifying different classes of –OH groups of Indulin AT lignin. Two different phosphitylating reagents were employed to obtain more in-depth information about characterization of different –OH functional groups. Reagent I provides essential information about primary –OH groups along with both *erythro* and *threo* diastereomers of the arylglycerol-β-aryl ether structures at 136.69-134.59 ppm and 134.59-133.72 ppm, respectively (Figure 2, Table 1). In contrast, reagent II allows distinguishing several different phenolic condensed units in lignins while being less specific for the aliphatic OH groups. The quantification of all hydroxyl groups were obtained from the integration of ^{31}P

NMR spectra with reagent II except for the primary –OH groups (Figure 3, Table 1). The primary –OH groups data were obtained from ^{31}P NMR spectra with reagent I and reported instead of aliphatic –OH units for the sole purpose of this study. The elemental analyses and empirical formula of phenylpropane structural unit of Indulin AT lignin are given in Table 2. Finally the molecular weights and molecular weight distribution of the lignin determined by GPC are illustrated in Table 3.

FTIR of the Indulin AT lignin is given in Figure 4 and attribution of its FTIR bands is as follows [17]: **-OH** stretch, 3100-3400 cm^{-1}; **C-H** stretch (Methylene & Methyl), 2800-3000 cm^{-1}; unconjugated **-C=O** stretch, 1705-1740 cm^{-1}; conjugated **-C=O** stretch,1675-1655 cm^{-1}; **Aromatic** skeletal vibrations, 1420-1430, 1505-1515, 1595-1603 cm^{-1}; **Ether C-O**, 1194 cm^{-1}; -**Alcohol** C-O, 1040 cm^{-1} (primary) & 1128 cm^{-1} (secondary); **Guaiacyl C-H** in plane, 1066 cm^{-1}; **Guaiacyl C-H** out of plane 908-831 cm^{-1}.

SEM image shows the lignin includes round particles and also broken ones. They are porous and have different sizes which are mostly less than 100 μm (Figure 5).

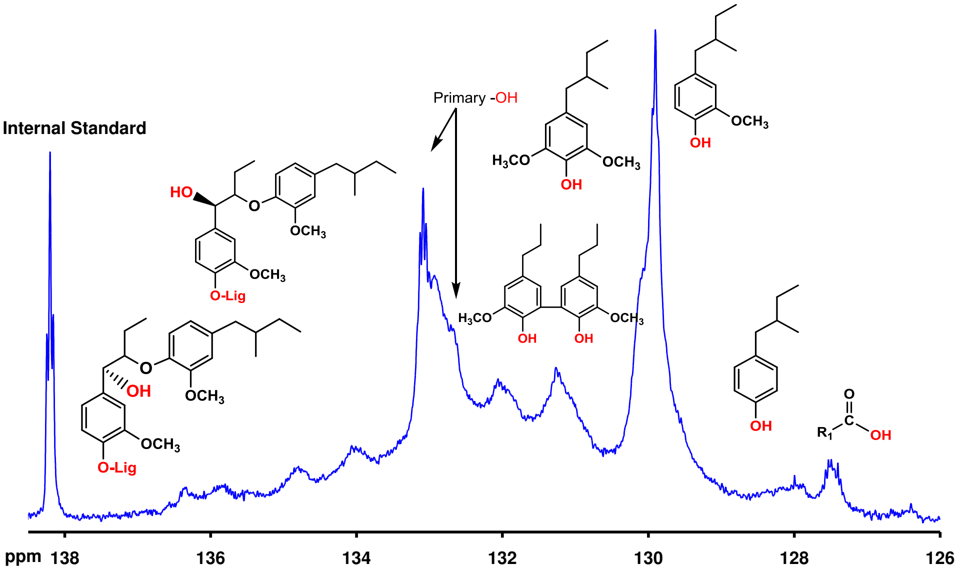


Figure 2. *^{31}P NMR spectrum of Indulin AT lignin with reagent I and signal assignments.*

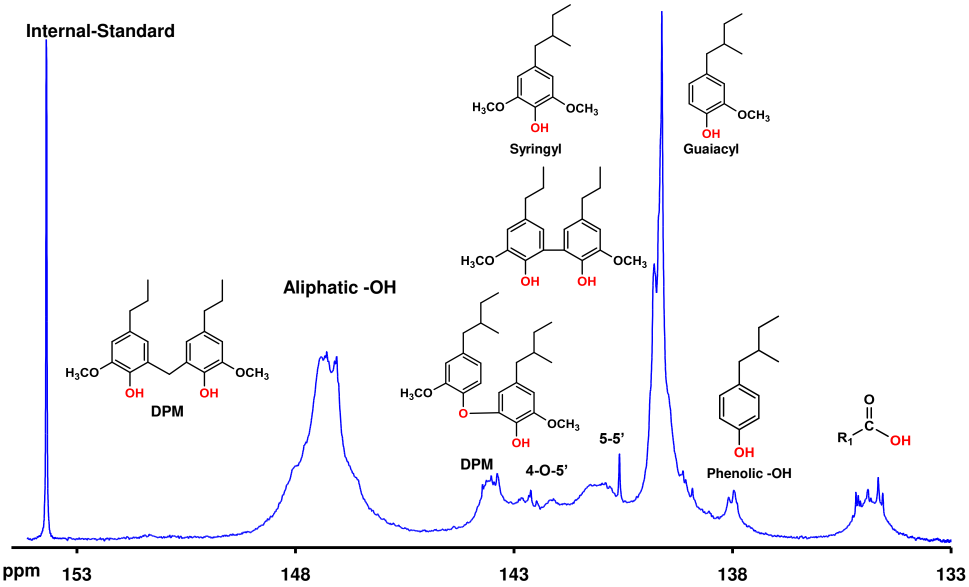


Figure 3. *^{31}P NMR spectrum of Indulin AT lignin with reagent II and signal assignments.*

Table 1. Quantitative ^{31}P NMR determination of carboxylic, phenolic, and aliphatic hydroxyl groups present in Indulin AT lignin.

Lignin sample	*-OH (mmol/g)*							
	COOH	*Phenolic*			*Condensed phenolic*	*Primary aliphatic OH*	*Secondary aliphatic OH*	*Total phenolic*
		G[*]	*S*[‡]	*H*[§]				
Indulin AT	*0.21*	*1.80*	*0.0*	*0.12*	*1.28*	*3.27*	*1.09*	*3.2*

[*] G: guaiacyl phenolic OH; [‡] S: Syringyl phenolic OH; [§] H: p-hydroxylphenyl phenolic OH.

Table 2. Elemental analyses and empirical formula of phenylpropane structural unit of Indulin AT lignin.

Lignin sample	*Elemental unit (%)* C	*H*	*N*	*O*	*S*	*OCH_3*	*C9 Structural unit*	*Mw g/mol*
Indulin AT	*60.63*	*5.75*	*0.87*	*29.59*	*1.69*	*12.92*	$C_9H_{8.67}O_{2.79}N_{0.121}S_{0.102}(OCH_3)_{0.809}$	*191.55*

Table 3. Molecular weight distribution determination of Indulin AT lignin by GPC.

Lignin Sample	*Concentration in THF (mg/mL)*	*Solubility*	M_w	M_n	M_z	M_w/M_n
Indulin AT	*1.77*	*Fully Soluble*	*6050*	*1138*	*68409*	*5.32*

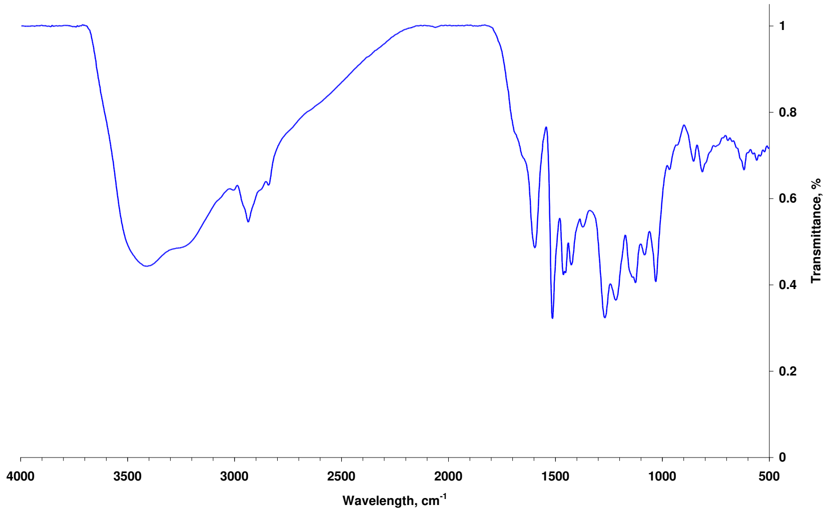


Figure 4. *FTIR spectrum of the commercially available Indulin AT.*

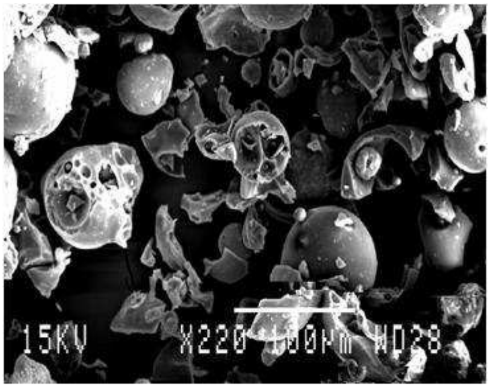
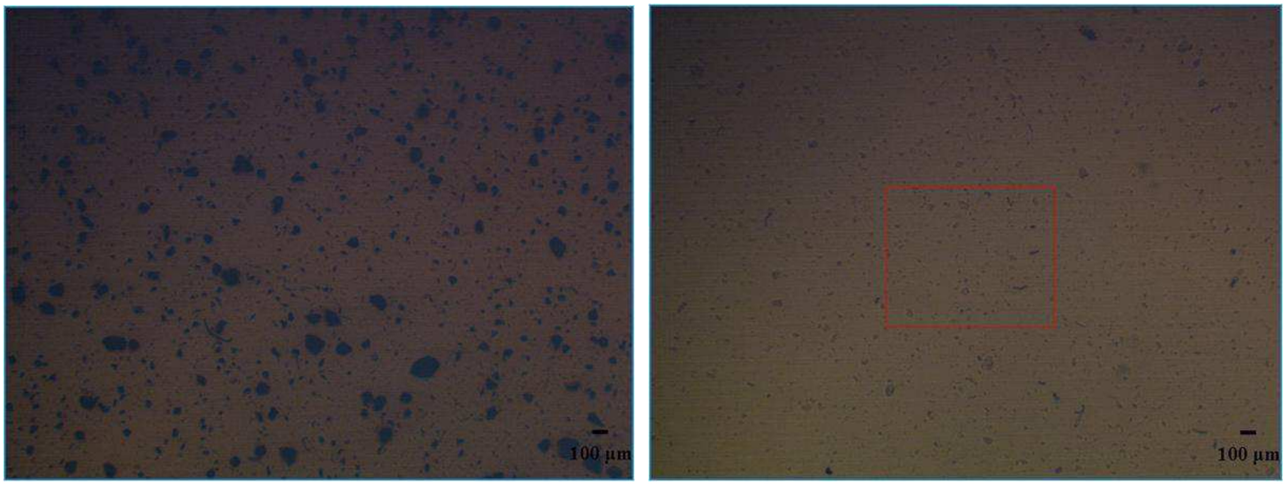


Figure 5. *SEM image of the lignin particles.*

Lignin Dispersion in Polyol

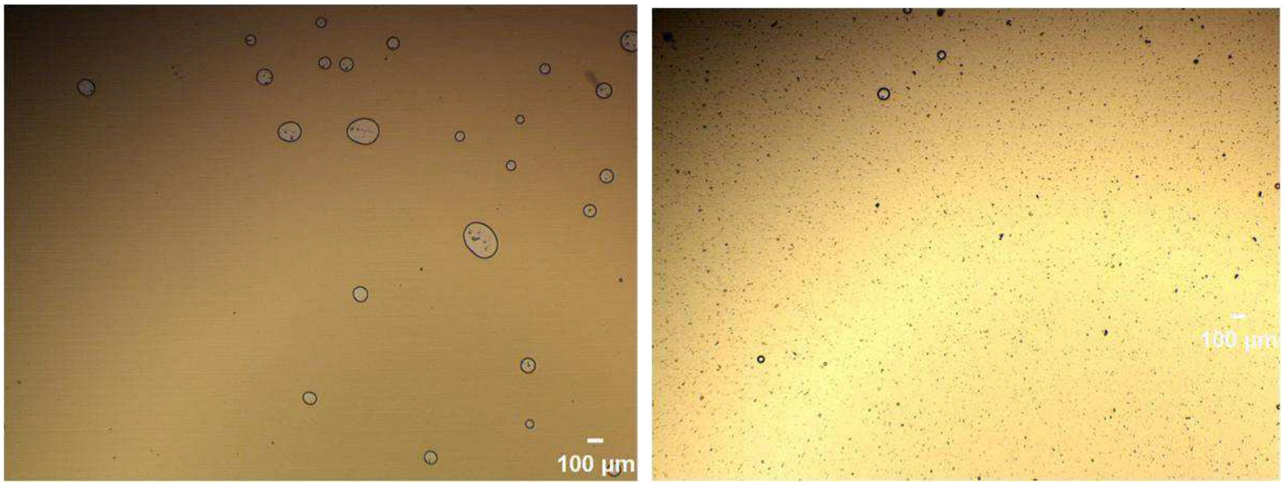
Mixing method and temperature have tremendous effect on the quality of dispersion of lignin in the given polyol. Figure 6a shows the increase of the lignin particle sizes after mixing with the PEG at room temperature (RM) indicating that the particles were swollen up (as compared to Figure 5) by the diffusion of PEG molecules into the porous lignin particles. B increasing the mixing temperature to 90 ^{o}C the large lignin particles almost disappeared (Figure 6b) indicating that they have either been broken in to smaller particles or dissolved into the PEG. Although the density of fine particles does not increase significantly it cannot be concluded if these large lignin particles really dissolved in the PEG since the magnification of OM is very limited (one cannot observe the finer particles). With high temperature and high shear efficiency of the TS mixing method the dispersion of lignin greatly improved as lignin particles cannot be observed anymore under the OM (Figure 7a). However, it remains unclear if they really dissolved in the PEG or the particle sizes are too fine to be detected by the OM, as discussed earlier. In general, high mixing temperature above the Tg of lignin Indulin AT (around 65 ^{o}C) increases the flexibility of lignin structure that facilitates the diffusion of polyol into the lignin particles resulting in swollen and softer lignin particles. With the aid of high shear force those particles could be easily broken into smaller particles and even can dissolve into the polyol if the thermodynamics of the system is favorable.

Further, the lignin dispersion depends also on the polyol chemistry. It is interesting to observe in Figure 7 that the polyether polyol, PEG, provides better dispersion than the polyester one, CAPA. The two studied polyols have similar molecular weights, however, PEG has greater polarity than CAPA because of the shorter chain length of its monomers while CAPA generates greater steric effect than the PEG because of its carbonyl groups in the ester linkages. The lignin Indulin AT appears to be more compatible with the more hydrophilic PEG.



a b

Figure 6. *OM photographs of lignin particles dispersed in PEG using a) RM and b) TM mixing.*



a b

Figure 7. *OM photographs of lignin particles dispersed in a) PEG and b) CAPA using TS mixing.*

Characteristics of Ligno-PU

The dispersion of lignin in the PUs is illustrated in Figure 8. As compared with Figure 7, the dispersion of lignin in the PEG-PU one is worse than that in the PEG system while the dispersion of lignin in the CAPA is more or less the same as that in the CAPA-PU. However, the dispersion of lignin in the two PU systems is quite comparable. The poor dispersion of lignin in PEG-PU can be explained by the fact that although PEG has better compatibility with the Indulin AT lignin than the CAPA as shown earlier but during the polymerization PEG was lost due to the reaction with isocyanate. If lignin cannot react well with isocyanate they could be left (partially or fully) to be free in the system and eventually form aggregates as governed by thermodynamics rules. It is important to stress here that the quality of dispersion of lignin in the final PU products can be controlled by both processes: i) the dispersion in the polyol and ii) the polymerization of the PU. The ideal case would be that lignin is well dispersed or even dissolved in the polyol and reacts well with the isocyanate.

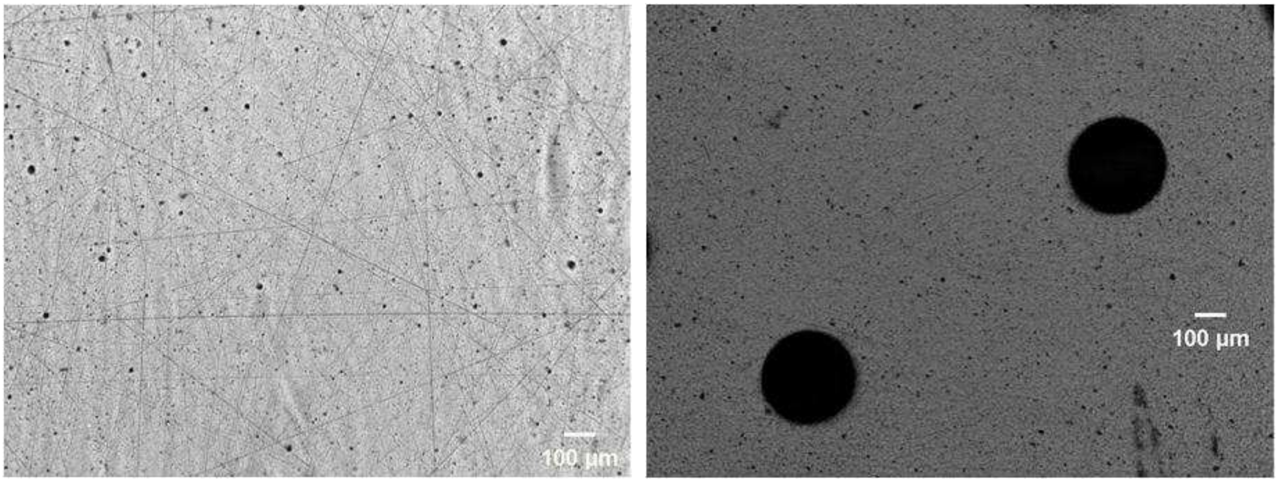
As seen in Table 4, the gel time of the PEG system was much shorter than that of the CAPA one regardless whether lignin was absent or present. The longer gel time for the CAPA system is mainly due to the steric effect of the carbonyl group in its structure that slows down the reaction with isocyanate. It is also noted that in the PEG systems the presence of lignin increases the gel time which could be related to some impurities in lignin.

Lignin is considered to be a polyol, which has greater functionality than the studied diols, PEG and CAPA. If lignin can react with isocyanate its presence should restrict greatly the PU chains hence increasing significantly the Tg of the PU systems. Table 4 shows that the presence of lignin reduces slightly the Tg of the PEG system while it increases significantly the Tg of the CAPA system. This indicates that the Indulin AT lignin was capable of participating in the CAPA based PU network structure but not in the PEG based one. It can be speculated that the slower reaction rate between CAPA and isocyanate (longer gel time) allows lignin to have more chance and more time to react with isocyanate in the CAPA system.

The presence of lignin in the PEG system reduced significantly the PU strength and modulus which could be a strong indication that lignin has affected negatively the formation of the PU network structure. In contrast, the presence of lignin in the CAPA system increased significantly both the strength and modulus of the ligno-PU which should prove for the participation of lignin in the PU network. These results are quite coherent with the Tg results.

Table 4. Characteristics of the PUs

Sample	*Gel time (min)*	*Tg (^oC)*	*Young's Modulus (MPa)*	*Tensile strength (MPa)*
PEG system				
Reference	*2*	*44*	*2280*	*41*
10% Lignin	*10*	*41*	*1220*	*22*
CAPA system				
Reference	*15*	*47*	*1180*	*24*
10% Lignin	*15*	*54*	*1740*	*29*



a *b*

Figure 8. *OM photographs of ligno-PUs based on a) PEG and b) CAPA.*

CONCLUSIONS

The results have demonstrated that the applied shear force, mixing temperature, and chemistry of polyol have also great influence on the dispersion and interaction of lignin with polyols thus on the PU performance. It is important that the lignin must be well dispersed and preferably soluble in the polyol prior to mixing with isocyanate. The mixing temperature and shear forces are among the most important parameters to control the dispersion of lignin. In addition, competing reactions among isocyanate – polyol and isocyanate – lignin can also affect the participation of lignin in the PU network structure which is a determined key for the improvement of the PU performance. A good dispersion of lignin in the polyol should facilitate the interaction between lignin and isocyanate during the PU formation.

It has been found that in this development the kraft lignin was well dispersed in both the studied polyols and the final PUs using the high shear mixer at high temperature, however, the lignin can only participate on the polyester polyol based PU net work structure hence increases significantly the mechanical performance. It demonstrates that if the ligno-polyol is well prepared good improvement in the performance could be obtained.

ACKNOWLEDGEMENT

The authors acknowledged the funding from the joint NRC- Agriculture and Agri-Food Canada- Natural Resources Canada National Bioproducts Program, Project #2. The authors also would like to thank Dr. Michel Drouin pour the thoughtful discussions.

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BIOGRAPHY

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