Evaluation of the particle size of organosolv lignin in the synthesis of resol resins for plywood and their performance on fire spreading

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ABSTRACT: Scientists today are intensively seeking alternatives to petrochemical materials. Among others, lignin is a promising candidate because it is available in large quantities while its chemical structure makes its use possible in a variety of chemical reactions. Lignin, received by numerous methods from various feedstocks, is a promising material for the synthesis of many products like active carbon, thermosetting and thermoplastic polymers, surfactants, phenolic chemicals, etc.

In this paper, the potential of using Biolignin – a trademarked organosolv lignin from straw prepared by Compagnie Industrielle de la Matière Végétale (CIMV; Neuilly-sur-Seine, France) – in the synthesis of phenol-formaldehyde (PF) resins was studied by CHIMAR HELLAS S.A. (Kalamaria, Greece). Before its use, Biolignin was further purified and subjected to mechanical treatment for the reduction of its particle size in order to increase its reactivity. The effectiveness of the treatment was verified by atomic force microscopy (AFM) measurements that were carried out by SYNPO Company (Pardubice, Czech Republic). Resol phenolic resins were prepared with various substitution levels of phenol up to 80%. However, their synthesis process was smooth only up to the substitution level of 50%. The properties of the resins were determined with typical lab analysis. Their thermal behavior was studied with differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) measurements that were conducted by the Aristotle University of Thessaloniki in Greece. Their bonding ability was evaluated by CHIMAR HELLAS via their application in the production of plywood panels of three layers that were prepared following a simulation of the industrial process. The panels were tested for their properties according to the relevant European standards, while their performance relative to fire was studied with cone calorimetry measurements that were performed by the Latvian State Institute of Wood Chemistry (LIWC; Riga, Latvia).

All results were compared with that of a typical PF resin. It was found that the particle size of lignin affects the performance of the resins, while lignin-based PF resins are suitable for the production of plywood panels and have somewhat better performance relative to fire than typical PF resins. This study has been performed within the framework of the European project BIOCORE (biocommodity refinery for biofuels, chemical intermediates, polymers and materials).

Application: This study informs readers that the performance of organosolv lignin in the synthesis of PF resins may be improved with adjustments to its particle size. Lignin-based PF resins render to plywood panels better performance to fire.

Lignin is an amorphous, heterogeneous phenolic polymer found in all vascular plants (trees, plants and agricultural crops), mostly between the cells, but also within the cells, and in the cell walls. It is covalently linked to the polysaccharides and contains several functional chemical groups, such as hydroxyl (phenolic or alcoholic), methoxyl, carbonyl and carboxyl groups, in various amounts, depending on the origin of the material and the separation process applied [1].

Lignin has attracted the interest of scientists and business because it is derived from abundant, renewable sources while it is non-toxic and versatile in performance due to its complex nature and highly functional character. Among the top players in the industry involved in lignin business are Borregaard LignoTech, Sappi, MeadWestvaco, Tembec, Domtar, UPM, Asian Lignin Manufacturing, Northway Lignin Chemical, Green Value, etc. Until today, lignin has been used as raw material for the synthesis of various bio-based products like carbon fibers, cosmetics, foams, dust control systems, batteries, thermosetting and thermoplastic binders, polyolefin-based products, concrete admixtures (as rheology modifier), etc. [2,3].

Especially in the case of phenol–formaldehyde (PF) binders, lignin has been extensively studied as a phenol substitute. However, the phenol substitution levels are usually low. The

reason is that the various lignin types have very large and non-uniform molecular weights (Mw) that affect both the average molecular weight and the polydispersity of the resins, resulting in products with low performance in the various applications [4,5]. Scientists have confronted this by applying various treatments to lignin either before its use or during the formation of the lignin-based products. These methods include fragmentation (pyrolysis, oxidation by chemicals and enzymes), functionalization of its hydroxyl groups (esterification, etherification, phenolation, urethanization), synthesis of new chemical active sites (alkylation/de-alkylation, hydroxyalkylation, amination, nitration) [6] or even employing novel methods to separate lignin, like acid hydrolysis or organosolv process, etc. [3]. However, most of these treatments and processes are quite expensive and thus economically unprofitable for use in industrial scale production.

A more economical processing of materials, well known in the industry, that could also be applied in the case of lignin is mechanical treatment by various methods, like ball milling, which facilitates the reduction of particle size. Generally, the mechanical treatment of raw materials offers easy handling, increase in surface area per unit volume, and separation of entrapped components, while the mechano-chemical reaction, which is induced by the input of mechanical energy by grinding in ball mills, allows reactions to occur under simplified and solvent-free conditions at room temperature. The notion of structural change of lignin in the ball milling process can be traced back to the 1920s, but the majority of the related studies are focused on the particle size reduction of milled wood or the synthesis of new products by treating lignin and other chemicals together.

Ikeda et al. [7] studied the structures of milled wood lignin (MWL), cellulolytic enzyme lignin (CEL), and residual lignin (REL) from a loblolly pine, using a modified derivatization followed by reductive cleavage (DFRC) method developed to allow the quantitative determination of three different structural monomeric products originating in lignin: phenolic beta-O-4, alpha-O-4, and etherified beta-O-4 structures. Their study showed that dry vibratory ball milling under a nitrogen atmosphere causes substantial structural changes, including condensation, whereas vibratory ball milling in toluene had little effect on the lignin structure.

Kleine et al. [8] studied a base-assisted ball milling treatment of lignin model compounds and demonstrated that a cleavage process takes place that predominantly involves the breaking of β-O-4 linkages.

Vlček [9], in the patent WO2014/044234A1, discloses the reduction of particle size of lignin powder to nanometers and micrometers in a dispersion device-like ball milling. The resulting lignin is suitable to be dispersed without the use of solvents in a pre-polymer terminated by reactive isocyanate groups upon preparation of single–component polyurethane materials. This method gives a solution to the problem of the insolubility of lignin in polyols that prevents the reaction of OH^- groups of lignin with isocyanate leading to the formation of a polymer network.

Qu et al. [10] applied wet ball milling (WBM) and ionic liquid pretreatment to industrial lignin samples and evaluated their effect on the average molecular weight and polydispersity, surface morphology, and functional groups changes. The results showed that lignin pretreated by WBM with phosphoric acid presented a dramatic decrease of polydipersity (23%) and an increase of phenolic hydroxyl content (9%).

Kandula et al. [11] treated kraft lignin with ball milling and poly-esterified it with ε-caprolactone, in order to create hydroxyl groups that are more easily available to further reactions. The resulted copolymer was still soluble in several organic solvents and suitable for use in various chemical reactions like, for example, in the synthesis of polyurethane.

Guo et al. [12] prepared oleated organosolv lignin (OL) by transesterification between OL and methyl oleate. The oleation reaction was conducted effectively in a ball milling process. The oleated OL displayed improved thermoplasticity, while when blended with PLA, it exhibited improved dispersion, suggesting increased compatibility.

In the current study, organosolv lignin of various particle sizes that resulted from the application of mechanical treatment was evaluated as a substitute for phenol in the synthesis of PF resins for the production of wood-based panels. The effect of the ball milling was verified by observation with atomic force microscopy (AFM), while the resins were studied for their properties with lab analysis, differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA). Their adhesive ability was evaluated via their application in the production of lab scale plywood panels that were tested for their mechanical and wet properties according to the European Union (EU) standards in force. Moreover, the performance of the resins to the spread of fire was evaluated via cone calorimetry tests of the plywood panels prepared with these resins. All results were compared with that of a typical PF resin.

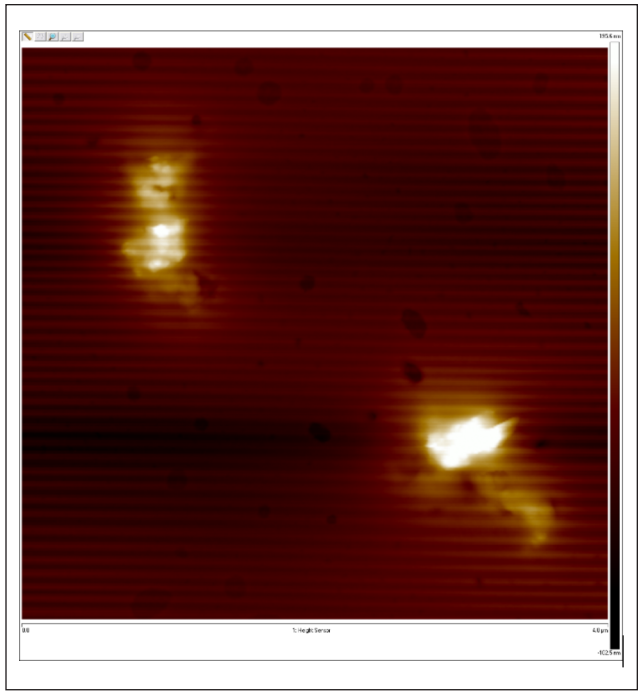
EXPERIMENTAL

Raw materials

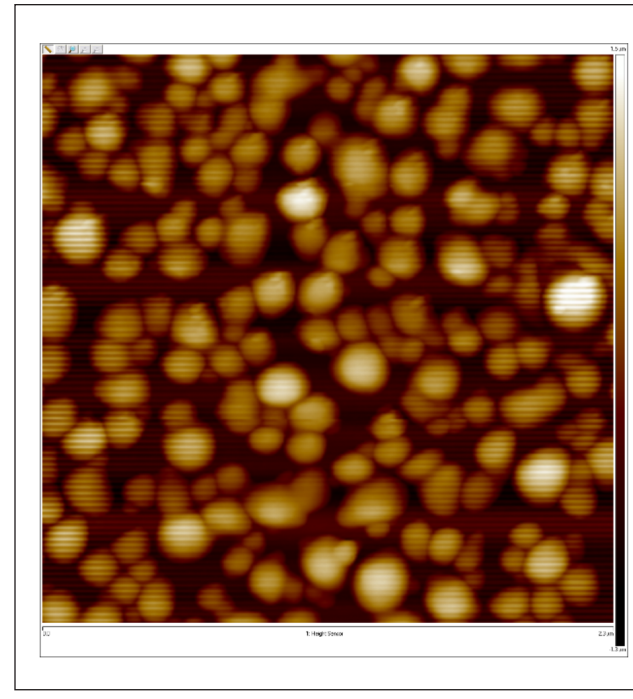
Thermosetting polymers of PF type were prepared using the following raw materials of technical grade in the form of water-based solutions: phenol 90% w/w, formaldehyde 37%w/w, and sodium hydroxide (NaOH) 50% w/w. Experimental resins were prepared by replacing phenol with organosolv lignin from straw provided by Compagnie Industrielle de la Matière Végétale (CIMV; Neuilly-sur-Seine, France) in the form of powder under the trademarked name Biolignin (45%w/w DM).

Lignin treatment

Biolignin, as initially provided by CIMV, was inefficient in the synthesis of resins, and CHIMAR HELLAS S.A. (Kalamaria, Greece) subjected it to treatment in order to increase its reactivity. This treatment consisted of a purification step followed by a particle size reduction step. In this study, this treatment will be named "CH-treatment". The purification step was applied in order to remove any acids and water soluble residues contained in Biolignin that may reduce its performance in the



1. Atomic force microscopy (AFM) image of untreated Biolignin from CIMV. Scan 4 µm and 2.3 µm, map of topology. There are agglomerates with diameter approximately up to 1 µm and height up to about 1 µm.



2. AFM image of CH-treated Biolignin. Scan 2.3 µm, map of topology. There are particles with diameter approximately up to 200nm. This sample is nicely dispersed.

polymerization reactions taking place during the synthesis of the PF type resins. The particle size reduction step was applied in order to make its reactive groups more readily accessible during the chemical reactions and thereby increase its activity. For the implementation of the CH-treatment, a quantity of lignin was initially immersed into tap water bath at room temperature (20°C–25°C) for 1 h, under slight stirring. Next, the water was removed by application of filtering using a Büchner funnel (purification step). The water extract was found to be of acidic pH (2–3) that verified the removal of acidic components. The resulting material was oven dried for about 20 h at 70°, and then it was ground in ball milling (particle size reduction step) in order to increase its surface area per unit volume and make more chemical units available for further reactions. In this study, Biolignin resulting from the previously described CH-treatment will be named "CH-treated" Biolignin.

SYNPO Company (Pardubice, Czech Republic), a partner of the BIOCORE project, carried out AFM measurements on treated and untreated Biolignin samples in order to evaluate the result of the ball milling process. The measuring unit was a Fritsch Laser Particle Sizer (Analysette 22, Fritsch GmbH; Idar-Oberstein, Germany). The samples were diluted in EtOH. CH-treated Biolignin was diluted without problem, but this was not the case for the untreated sample. The untreated Biolignin formed agglomerates with diameter approximately up to 1 µm and length of about 1 µm (**Fig. 1**). Such agglomerations were too big to be seen by AFM. On the contrary, the CH-treated Biolignin had particles with diameter approximately up to 200 nm and was nicely dispersed (**Fig. 2**).

Untreated Biolignin, CH-treated Biolignin, as well as Biolignin, were subjected to some mechanical treatment from CIMV in order to receive material of two different average particle sizes (<500 µm and <10 µm), which were studied in the synthesis of PF resins.

Synthesis and analysis of resins

Initially, CHIMAR tried to use untreated Biolignin in the synthesis of a PF type resin. However, the sample performed insufficient dispersion into the resin mixture, which created problems during the manufacturing of plywood panels with the viscosity measurements during the synthesis of the resin and later on with the application of the glue mixture onto the wood veneers. CHIMAR proceeded to use a lignin that consisted of washing and lab-scale ball milling of lignin, as described previously (CH-treatment). This treatment was found to improve its performance, since the resulting material was now successfully incorporated into the resin mixture.

Although PF resins with phenol replacement level up to 80%w/w by CH-treated Biolignin were synthesized, the higher the substitution level the more irregular was the synthesis process. Therefore, only the resin with 50% phenol substitution was selected for a more thorough study because it had the highest phenol replacement level with the smoothest synthesis process. Together, CIMV provided CHIMAR with two Biolignin samples of different particle sizes prepared at pilot scale on CIMV premises. These samples were also used

Properties	PF Resin	Experimental Resins
Water content, %	53–55	53–55
Viscosity, %	250–500	250–500
pH @25°C	10.0–13.0	10.0–13.0
Gel time at 130°C, s	180–350	60–250
Alkalinity, %	7.1–7.7	6.0–8.0
Water tolerance, ml/ml	>1/10	>1/10
Conductivity, mS/cm	~20	~20
Free formaldehyde, %	<0.5	<0.5

I. Properties of the resins (PF = phenol–formaldehyde).

in the synthesis of PF resins with 50%w/w phenol substitution in order to evaluate their performance in comparison with the CH-treated Biolignin. A typical PF resin was also included in the tests.

All resins were subjected to typical lab analysis for the determination of their physicochemical properties, like water content, viscosity, pH, alkalinity, conductivity, water tolerance, gel time and free formaldehyde (**Table I**).

The thermal properties of the resins were determined with DSC and TGA measurements (Figs. **3a**, **3b** and **3c**). The TGA measurements were carried out with a SETSYS 16/18 TGA-DTG device (Setaram; Caluire-et-Cuire, France). The samples (6.0 ± 0.2mg of liquid resin) were placed in aluminum crucibles while an empty alumina crucible was used as reference. The samples were heated from ambient temperature to 1000°C in flow of air (in order to have an environment representative of the real conditions that resins are used), with a heating step 20°C/min.

The DSC measurements were carried out with a Setaram DSC141 calorimeter. The samples (6.0 mg ± 0.2mg of liquid resin) were placed in stainless steel sealed crucibles while an identical empty crucible was used as reference in each measurement. The samples were heated from ambient temperature (25°C) to 250°C in a 50 ml/min flow of nitrogen gas (N_2), with a heating rate of 10°C/min.

Production of plywood panels

The adhesive ability of the resins was evaluated via their application in the production of plywood panels. The panels were prepared at lab scale following a simulation of the industrial practice. Panels of 3 layers were produced using a veneer of poplar wood for the core and veneers from okume for the two face layers (okume/poplar/okume). Their testing and classification was carried out according to the following European standards:

- EN 314-1:2004 "Plywood – Bonding quality – Part 1: Test methods"
- EN 314-2:1993 "Plywood – Bonding quality – Part 2: Requirements"

Although these standards are valid for industrial production, they also give a good indication of the performance of lab scale panels.

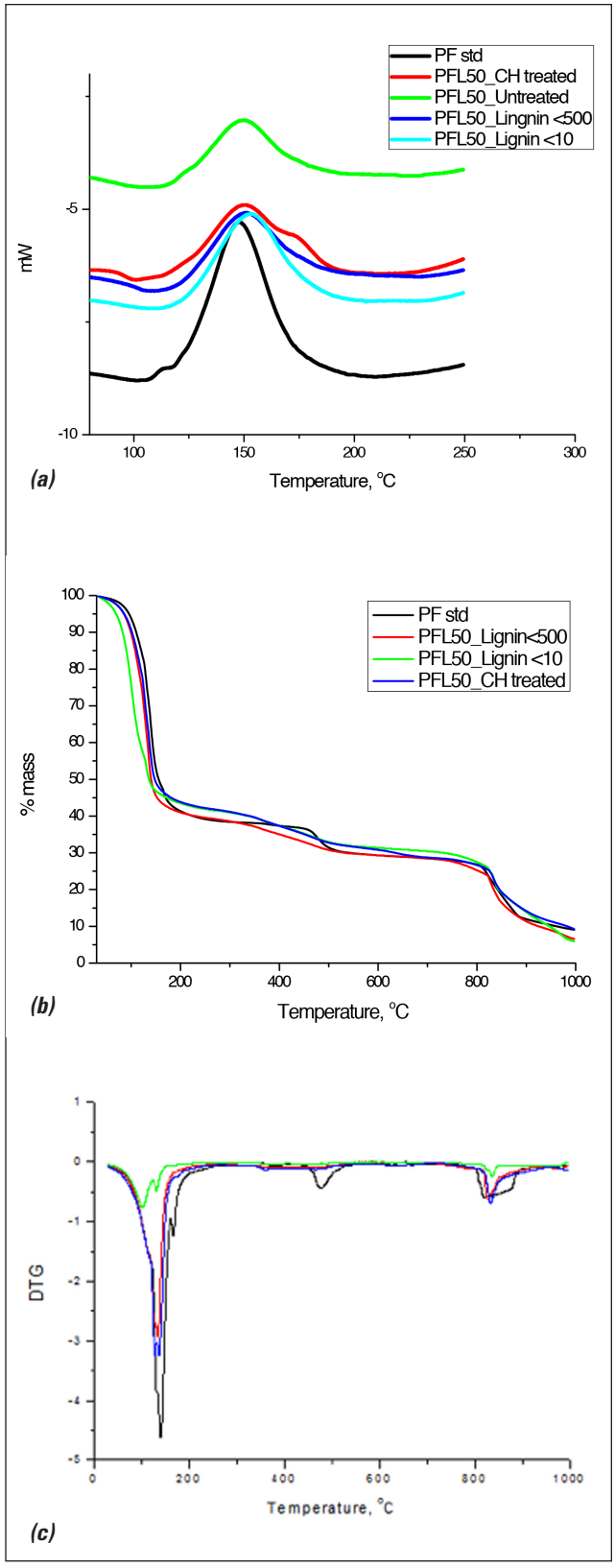
The standard EN 314-1:2004 describes the methods for the testing of the plywood panels, while the standard EN314-2:1993 categorizes the plywood panels and sets threshold values for their mechanical and wood failure performance. In particular, the standard EN314-2:1993 specifies requirements for bonding classes of veneer plywood according to their end use. The designated categories are:

- Class 1 - Dry Conditions: interior applications with no risk of wetting (12% or less moisture content, at 20°C and 65% relative humidity).
- Class 2 - Humid Conditions: protected exterior conditions (20% or less moisture content, at 20°C and 85% relative humidity).
- Class 3 - Exterior Conditions: unprotected exterior conditions (above 20% moisture content).

For all bonding classes, each glue line must satisfy two criteria: the values of mean shear strength and mean apparent cohesive wood failure must have the correlation described in **Table II**. The previously mentioned threshold values stand for any class of plywood panels, but according to their intended application, the panels are subjected to different pretreatments before their testing (EN314-1:2004).

The formaldehyde emissions were determined with the desiccator method (ISO 12460-4 "Wood-based panels – Determination of formaldehyde release – Part 4: Desiccator method"). Moreover, samples of all plywood panels were subjected to cone calorimetric flammability testing according to the ISO/TR 5660-3:2003 Standard "Reaction-to-fire tests – Heat release, smoke production and mass loss rate – Part 3: Guidance on measurement," which was performed by another partner of the BIOCORE project, the Latvian State Institute of Wood Chemistry (LIWC).

The parameters of the measurement are cited in **Table III**. Samples of the panels were measured for their heat release rate (HRR) and smoke production rate (SPR). The results are presented in **Figs. 4** and **5**.



3. a) Differential scanning calorimetry (DSC) graph of resins, b) thermogravimetric analysis (TGA) graph of resins, and c) differential thermal analysis (DTA) graph of resins (PF = phenol–formaldehyde).

Mean Shear Strength fv, N/mm²	Mean Apparent Cohesive Wood Failure w, %
0.2 ≤ fv < 0.4	≥ 80
0.4 ≤ fv < 0.6	≥ 60
0.6 ≤ fv < 1.0	≥ 40
1.0 ≤ fv	No requirement

II. Standard bonding performance requirements for European Standard EN 314-2:1993.

Heat flux	35 kW/m²
Test time	2 min
Ignition source	Electrical spark
Sample dimensions	10 cm x 10 cm x ~ 0.5-0.6 cm
Orientation	Horizontal

III. Parameters of the cone calorimetry measurements.

RESULTS AND DISCUSSION

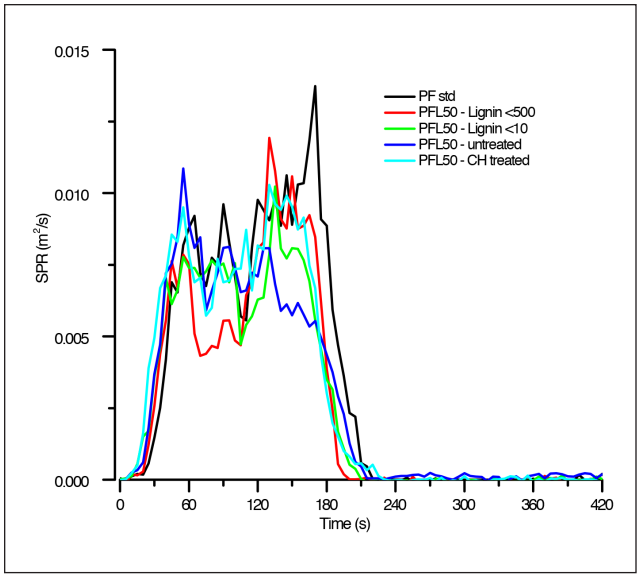
Characterization of resins

The physicochemical properties of the resins are cited in **Table I**.

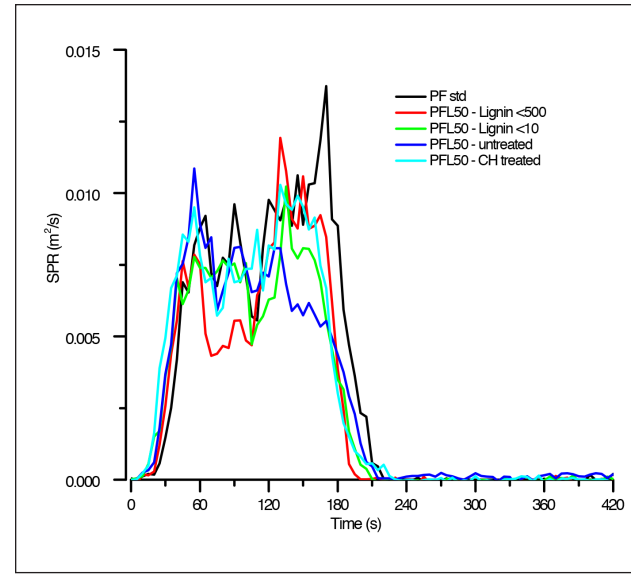
The lab analysis results show that the experimental resins have properties that fall within the limits of a typical PF resin and thus may be used in the production of wood-based panels. Furthermore, it is worth noting that the lignin-based resins have shorter gel time and thus develop their crosslinking network and set faster than typical PF resins.

The TGA measurements of the resins reveal their performance during thermal degradation. As the temperature increases, the resins lose mass at various steps. The first step from room temperature up to about 200°C is due to the loss of water and small molecules like free formaldehyde as well as further crosslinking and loss of byproducts [13]. After 200°C, the thermal degradation of the polymer starts. Until about 450°C, the control resin seems to be the most stable one, while the lignin based resins lose mass at a faster rate. At this region of temperatures, the pyrolytic degradation of polymers takes place while inter-unit linkages break down and release small chemical moieties, like monomeric phenol, for example, into the vapor phase. After about 450°C, the decomposition of aromatic rings takes place [14]. The resin with the CH-treated lignin leaves the highest percentage of ash at the end of the heating, up to 1000°C. According to literature references, the high ash content prevents the spread of fire.

The DSC thermographs show that all resins perform an exothermic phenomenon at around 150°C and thus it can be said that they have similar curing performance. However, in the case of lignin-based resins, the exothermic peak is shifted somehow to higher temperatures, which is in accordance



4. Smoke production rate (SPR).



5. Heat release rate (HRR).

with literature references [15]. The peak temperature of the resin prepared with CH-treated Biolignin is the closest to the control resin (**Table IV**).

The results of the bonding quality tests of the plywood panels are presented in **Table V**. All lignin-based resins have performance slightly lower than the control. Among the experimental resins, the one with lignin of particle size <500 µm as well as the CH-treated sample are the best performed after the severest pre-treatment (5.1.3) and meet the requirements of the standard EN314-2:1993 (Table I) for use in exterior applications. Between these two resins, the one prepared with the CH-treated Biolignin gave panels with significantly lower formaldehyde emissions.

The results of the cone calorimetry measurements of the plywood panels (**Figs. 4** and **5**) show that the control PF resin gives somewhat more smoke than the lignin-based resins, no matter the particle size of the lignin. Furthermore, the typical PF resin is burned more vigorously and emits more heat during the burning process. The three peaks of each plywood sample on the HRR test (Fig. 5) are attributed to their delamination during the burning process. When the layer of plywood is delaminated, a larger surface of the sample is exposed to flame, and hence more heat is released. The peak heat release rate was 268.6 kW/m² for the typical PF resin and 233.8–228.5 kW/m² for the lignin-based resins. All samples were burned for about 420 s until the material was fully charred. The cone calorimeter shows the kinetics of a material when it is burned under constant heat flux, and in the case of the typical PF resin, they were comparably faster. When materials burn at a slower rate, there is more time to put out a fire or evacuate people from a burning building. Among the experimental resins, there are no big differences, although it seems that the lower the particle size, the better the performance of the resin (lower HRR and SPR values). These findings are in accordance with other literature references where it is noted that lignin contributes to a material's resistance to the spread of fire due to a high charring ability and a low heat release when burning [16].

All the previous findings show that CH-treated Biolignin has advanced performance compared to the other lignin sam-

Resins	Onset Temperature, °C	Peak Temperature, °C	Endset Temperature, °C
PF std	105.753	146.457	202.253
PFL50-Lignin<500	110.671	150.109	190.004
PFL50-Lignin<10	113.138	152.607	205.019
PFL50-untreated	114.369	150.880	197.205
PFL50-CH treated	102.474	150.172	199.766

IV. Differential scanning calorimetry (DSC) characteristic temperatures for resins.

Resin	PF std	PFL50	PFL50	PFL50	PFL50
Biolignin particle size	-	<500µm	<10µm	untreated	CH treated
EN 314-1:2004 Tests					
Pre-treatment 5.1.1:	*Immersion in water at 20°C for 24 h*				
Shear strength, N/mm²	1.28	1.17	0.75	0.84	0.98
Wood failure, %	85	40	25	25	70
Pre-treatment 5.1.3:	*4 h boiling – 16 h drying at 60°C – 4 h in boiling water – 1 h in cool water*				
Shear strength, N/mm²	1.13	0.81	0.57	0.60	0.78
Wood failure, %	75	45	35	30	60
Formaldehyde emissions/ Desiccator values, mg/L	0.083	0.114	0.061	0.041	0.044

V. Bonding quality test results of plywood panels produced with Biolignin from CIMV.

ples tested in this study and verify the consideration that reduction of lignin particle size by the ball milling process (CH-treated Biolignin) can increase its reactivity. The CH-treated Biolignin not only made possible synthesis of the resin, contrary to the untreated sample, but the resin prepared from it also had the closest curing temperature to the control resin (DSC results) and gave acceptable mechanical properties and the lowest free formaldehyde emissions when applied to the production of plywood panels of all samples tested. It can be justified by the fact that the ball milling treatment enables more reactive sites on lignin to become available for reaction with formaldehyde, resulting in the creation of more cross-links between lignin and formaldehyde and thus faster curing and lower free formaldehyde emissions. Based on cone calorimeter results, plywood panels with lignin-based resins burn at a slower rate than that prepared with a typical PF resin. Also, according to the TGA results, the resin prepared with the CH-treated Biolignin gave the highest percentage of ash, signifying that this resin has the highest ability to prevent spreading of fire compared to all other lignin samples studied.

CONCLUSIONS

The results show that organosolv Biolignin from the CIMV process may be used in the synthesis of PF resins as phenol substitute. The treatment applied by CHIMAR (CH-treatment) improved its performance and resulted in resins with a clear advantage over the other experimental resins of this study. Such resins are suitable for the manufacturing of plywood panels and offer slightly improved performance to the spread of fire compared with a typical PF resin. However, in order to establish the CH-treatment as a general method for lignin improvement, more thorough studies are needed for determination of the optimum limits of the particle size of each type of lignin and its application. **TJ**

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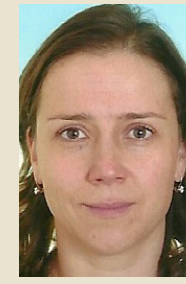

Švíglerová

Benjelloun-Mlayah

We chose this research topic because it was the subject of a European Union (EU) project where co-author Papadopoulou was the principal investigator on behalf of the company CHIMAR HELLAS S.A. In our study, a special treatment of lignin was developed that enabled this material's use in the synthesis of resins for wood-based panels. It was a novel approach in the field that promoted the state-of-the-art in lignin-based thermosetting resins.

The most difficult aspect of the research was to find a way to use the available lignin in the synthesis of resins. It was addressed by the development of a lignin treatment that enabled its successful utilization. A major discovery in this research was that the mechanical treatment of lignin leads to the reduction of its particle size and improves its performance.

Our most interesting finding was that an easy and low-cost treatment of lignin can change its performance and that lignin-based resins are more resistant to fire than typical PF resins.

Mills can apply this simple treatment method to lignin and make it usable when it otherwise cannot be used. Our next step is a scale up of the process.

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