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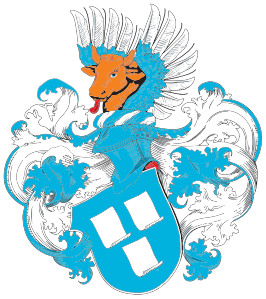

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POLYOLS AND RIGID POLYURETHANE FOAMS DERIVED FROM LIGNIN SIDE-STREAMS OF THE PULP AND PAPER INDUSTRY

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ABSTRACT

Lignin is one of the major components of the side-streams of the pulp and paper industries and of the emerging lignocellulosic biorefineries. Find new strategies for lignin valorisation, apart from e vironmental issues, will contribute to close the lignocellulosic value-chain. In this context, this work will focus on the synthesis of polyols through oxypropylation and their subsequent incorporation into rigid polyurethane (RPU) foam formulations. Two *E. globulus* lignin streams were tested: (i) lignin isolated from industrial kraft liquor and (ii) depolymerized lignin resulting from an oxidation process developed at the LA LSRE-LCM to produce vanillin and syringaldehyde. This work is therefore part of the LA LSRE-LCM global strategy towards the consolidation of the integrated process to valorise lignin from the kraft pulp industry, which accounts with more than 25 years of experience.

Polyols were firstly obtained by an oxypropylation process, using a 450 ml stainless steel PARR autoclave (setpoint temperature of 160 ºC), following a general procedure established in our group. For original lignin, L/PO (lignin/propylene oxide, w/v) ratios of 20/80, 30/70 and 40/60 (catalyst levels of 2, 3 and 5 %, w/w lignin based) were used. For depolymerized lignin, only the 30/70/2 formul tion was tested. The obtained polyols were characterized (homopolymer content (%, w/w), hydroxyl index (IOH) (mg KOH/g) and viscosity (20ºC, Pa.s) and incorporated into RPU foam formulations, alone or combined with 50% of a commercial polyol (Daltfoam TP 32015 polyol) at a NCO/OH ratio of 1.1. For the samples using 100% of the lignin-based polyol the following additives contents were used: 10% of glycerol, 2% of surfactant (SR-321 NIAX silicone), 2% of the catalyst system (DMCHA:NIAX-A1 (50:50)), 2% of water and 20% of n-pentane (%, w/w, based on the polyol amount). The obtained RPU foams were characterised experimentally in what concerns density (kg/m^3), thermal conductivity (W/m.K), heat release rate (HRR, kW/m^2), viscoelastic thermal response and thermo-mechanical properties with increasing temperature. Flame retardancy and thermal stability were also inspected.

The performed work demonstrates the viability of using the lignin side streams of the pulp and paper industry to produce novel materials, namely polyols and RPU foams. The generated polyols were successfully used at a content of 100% to produce RPU foams. The produced foams presented densities ranging from 45-80 kg/m^3, and in some cases thermal conductivity lower than the reference foam (e.g. 38.8 mW/m.K for the 40/60/100 foam (reference foam: 40.2 mW/m.K)). Even lignin is refereed in literature as presenting fire retardancy, leading to the formation of char and volatile compounds from thermal decomposition, its incorporation on RPU foam systems resulted only in slight advantages, nevertheless the HRR decreased for the systems incorporating lignin, especially for the ones using the depolymerized fractions resulting from the vanillin and syringaldehyde process. In fact, and comparatively with the original lignin, these fractions showed interesting characteristics for polymeric materials production. An additional advantage was the elimination of the malodorous typical of kraft lignins, possible resulting from the loss of the side chains of phenylpropane unit (bounded to sulfh dryl moieties, in the case of kraft lignin).

Keywords: Lignin valorisation, integrated process, biopolyols, polyurethane rigid foams