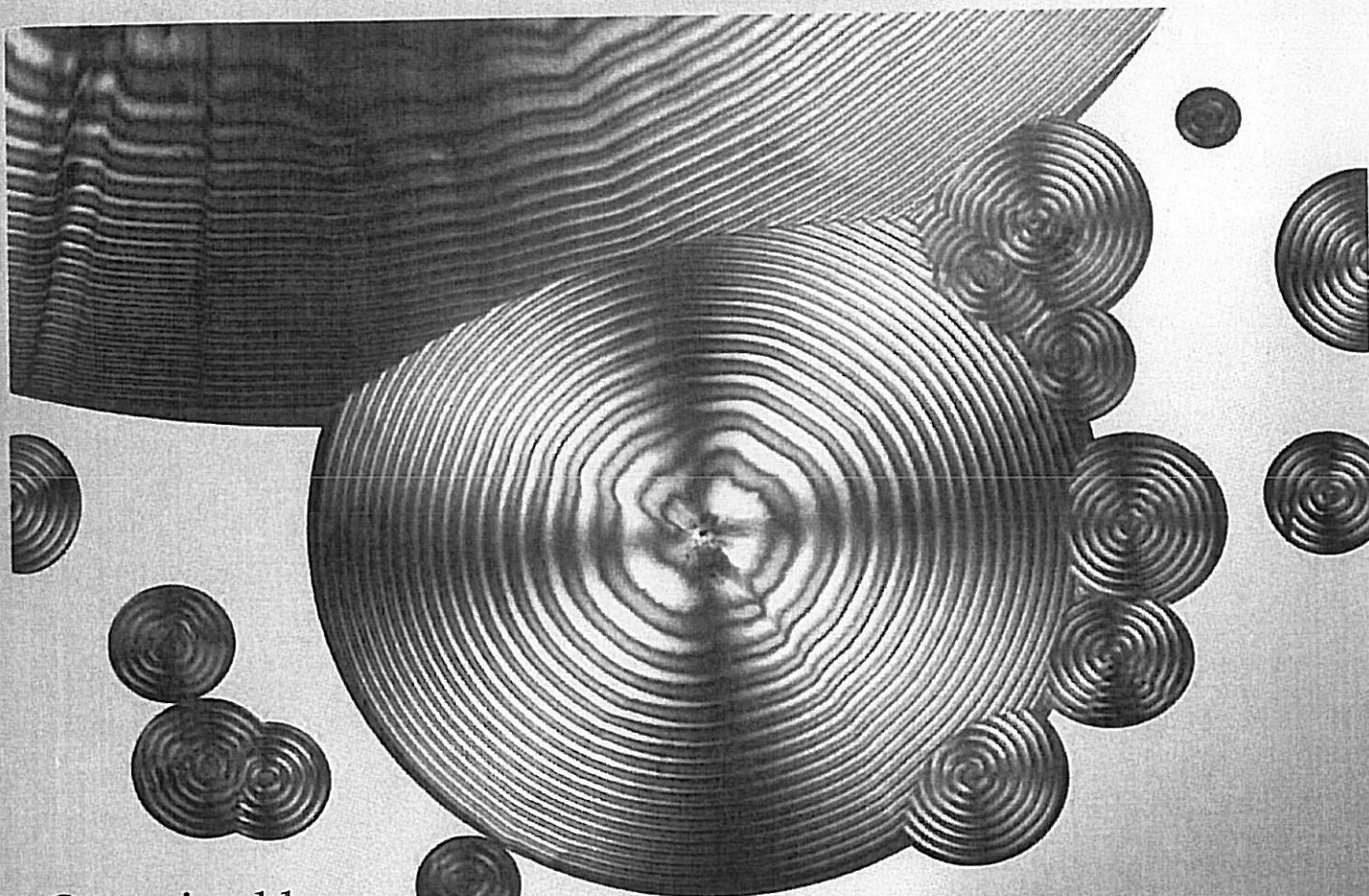
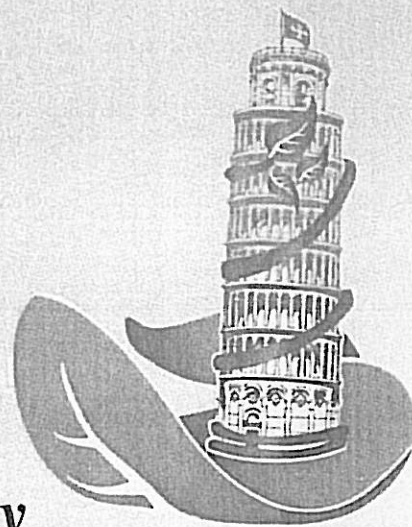


# 4<sup>th</sup> Workshop

## Green Chemistry and Nanotechnologies in Polymer Chemistry

4-6 September 2013 – Pisa, Italy



Organised by

*Department of Civil and Industrial Engineering,  
University of Pisa*

*Institute for Composite and Biomedical Materials, CNR*

*National Interuniversity Consortium of Materials Science and Technology, INSTM*



**4TH WORKSHOP GREEN CHEMISTRY  
AND NANOTECHNOLOGIES IN POLYMER CHEMISTRY**  
4-6 September 2013 – Pisa, Italy



**POSTER**

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**POSTER**

COMPREHENSIVE INSIGHTS INTO LIGNIN OXYPROPYLATION REACTION

Cateto c.<sup>a,b,c</sup>, Barreiro M. F.<sup>a,\*</sup>, Rodrigues A.<sup>b</sup> and Belgacem N.<sup>c</sup>

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Concept and objectives

Polyols resulting from oxypropylation are a mixture of oxypropylated lignin and some low molecular weight products (polypropylene oxide oligomers). Normally these oligomers are left in the final mixture since they constitute useful co-monomers, lowering the Tg and the viscosity of the resulting liquefied lignin. In this work a series of polyols based on Alcell lignin have been produced using L/PO ratios (Lignin/Propylene oxide, w/v) of 10/90, 20/80, 30/70 and 40/60 and catalyst contents (KOH) between 2-5% (C/(C+L), w/w). The produced polyols and the corresponding extracted fractions (homopolymer and oxypropylated lignin) have been analyzed by size exclusion chromatography (SEC). The obtained results, together with the determined homopolymer content, were analyzed with respect to the used formulations (L/PO ratio and catalyst content) and the polyol properties (IOH, viscosity and Tg).

Results and Discussion

Figure 1 shows the chromatographic profile evolution of the two polyol fractions (homopolymer and oxypropylated lignin) as a function of the L/PO ratio for a fixed catalyst content (3%). As the L/PO ratio increases, the homopolymer content decreases and the chromatographic profile of both homopolymer and

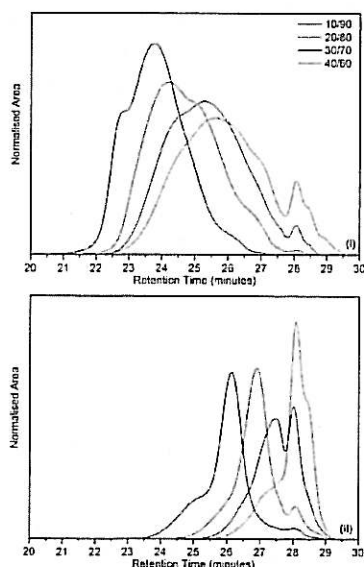


Figure 1. Chromatographic profile as a function of L/PO ratio (3% catalyst) for (i) oxypropylated lignin and (ii) homopolymer fractions.

oxypropylated lignin fractions shift towards lower apparent molecular weights (lower hydrodynamic volumes). This behavior is accompanied by an increase of the viscosity, the hydroxyl index and the Tg of the final polyol. In conclusion, for a fixed catalyst content, an increase of the L/PO ratio seems to favor hydroxyl activation ( $I_{OH}$  increases) together with the formation of short grafts, which corroborates the observed decrease of hydrodynamic volume of the oxypropylated lignin fraction and the increase in polyol viscosity. The same trend is observed for a fixed L/PO ratio, when the catalyst content increases. Tg and the associated transition temperature range increases with increasing L/PO ratio, whereas the catalyst content influence can be neglected. This tendency is related with the homopolymer content but also with the length of the grafted chains. Summarizing, as the L/PO ratio increases, the homopolymer content decreases and the grafted chains become shorter contributing for the achievement of higher Tg values. For the series with low lignin content (10/90 and 20/80), the presence of higher homopolymer content together with long graft chains, contribute to achieve lower Tg values closer to that of the homopolymer fraction.

Acknowledgments

FCT (grant SFRH/BD/18415/2004), French-Portuguese Scientific Cooperation (actions F-13/06 and F-32/08) and LSRE (strategic project PEst-C/EQB/LA0020/2011).