

Determination of Phenolic Hydroxyl Groups in Technical Lignins

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Lignin is one of the most abundant biomaterial available and used in many applications such as fuel in kraft process and raw material source for chemicals *e.g.* DMSO and vanillin. Yet it's characteristics are widely unknown since lignin acts as a by-product of kraft industry and only minor amount is used in commercial applications (roughly 2%). Lignin still provides huge potential for future applications *e.g.* in petrochemical industry as a renewable resource or source material for carbon fibre. [1, 2]

Lignin is a complex and highly cross-linked copolymer, consisting three units namely p-coumaryl, coniferyl, and sinapyl alcohols [3]. The amount of phenolic hydroxyl groups is the main parameter to indicate the reactivity of ligning. Thus, reliable methods to determine hydroxyl content are needed.

Two technical lignins, Indulin AT and Milox, were investigated. Indulin AT is a well-studied and commercially available lignin which was used as a reference material. Milox is yet uncharacterised. In this study we investigated the reliability of Ionization Difference Ultraviolet Spectrophotometry (IDUS) method in characterization hydroxyl content of lignin and used ¹³C-NMR as reference method [4]. ¹³C-NMR has proven to be a precise analytical method to determine hydroxyl content in lignin [5]. By NMR methods one is able to distinguish different types of hydroxyl groups separately and quantitatively (see Fig. 1). Obtained hydroxyl content for Indulin AT was in good agreement with previous studies. Also obtained values with IDUS method compare well with NMR results when limitations of IDUS method are taken into account. Therefore we can predict reliable results for Milox.

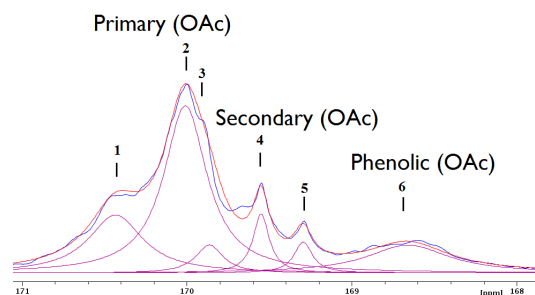


Figure 1: Quantitative ¹³C-NMR spectrum of Milox. Blue line corresponds experimental spectra, purple fitted peaks and red sum of the fit.

[1] *NNFCC factsheet*: Renewable chemicals factsheet [online 2.2.2016].

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[3] N. Lewis & E. Yamamoto *Annu. Rev. Plant. Physiol. Plant. Mol. Biol.*, **41** 455-496 (1990).

[4] W. M. Goldmann, J. Ahola, **O. Mankinen**, A. Kantola, S. Komulainen, V.-V. Telkki & J. Tanskanen, manuscript in preparation.

[5] J. W. Choi & O. Faix, *J. Ind. Eng. Chem.*, **17**, 25-28 (2011).