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Local filtration properties of Kraft lignin: The influence of residual xylan

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ABSTRACT

The influence of lignin and xylan interactions on the filtration properties of precipitated LignoBoost lignin was investigated. LignoBoost lignin was (i) suspended in acid water with xylan added and (ii) dissolved together with xylan and then re-precipitated. The resulting lignin-xylan mixtures were more difficult to filter than the original LignoBoost lignin, although the formed filter cake was also found more porous in the case of re-precipitated solids. Furthermore, the pressure dependency of the filtration properties was shown to increase after the addition of xylan. One possible explanation based on the findings presented in this paper is that xylan is sorbed at the surface of the lignin agglomerates: it increases the contact area between solid and liquid, thus making the particle structure more porous.

The influence of ionic strength was also investigated through the addition of sodium sulphate: it was found that increasing the ionic strength of the slurries made the solids easier to separate, possibly due to a decrease in electrostatic repulsive interactions between the solids and the formation of a denser solid structure.

1. Introduction

Lignin, one of the major components of wood (about 20–30%), is the most abundant renewable source of aromatics on Earth. Lignin is also the main organic by-product of the Kraft paper pulp industry: each year, about 50–60 Mt of lignin is dissolved in the cooking liquor and is currently used almost exclusively as biofuel and incinerated in the mills' recovery boiler. In modern Kraft pulp mills, at least 20 to 30% of this dissolved lignin can be extracted without disturbing the operation of the recovery boiler. Thus a minimum of 10–18 Mt of Kraft lignin could be made available annually worldwide for the production of valuable materials and chemicals: adhesives, bio-oils, emulsifiers and carbon fibres are among the large number of potential products that may be made from lignin [12,8,3,4].

Different processes have been developed for extracting lignin from the spent cooking liquor (which is known as black liquor). Acid precipitation followed by filtration and displacement washing is currently the preferred technique since it has been shown to provide a higher yield of lignin at a lower estimated cost compared to other techniques, such as ultrafiltration [16,17]. This technique has actually been used since 1942 in the United States by the Westvaco Company (now MeadWestvaco Corporation) [12]. However, the main challenge of the traditional acid precipitation method is the purification of the precipitated lignin because partial or complete plugging of the filter cake may take place during the displacement washing step. The LignoBoost process, a newer procedure that has recently been introduced on an industrial scale, overcomes this difficulty. Using this process allows lignin with both a high level of purity and a high dry content to be obtained [9,10,11,5]. Moreover, extracting part of the dissolved lignin from black liquor allows the heat capacity of the recovery boiler to be 'debottlenecked', which is generally the parameter that limits production in modern Kraft pulp mills. Therefore, implementing the LignoBoost process in the Kraft process often allows for an increase in pulp production in addition to providing a pure lignin product, making this option highly beneficial to modern Kraft pulp mills.

Cake filtration is one of the major unit operations of the LignoBoost process. The filtration properties of the lignin extracted need to be known for an accurate design of the filtration steps. The compressibility of the lignin filter cake, i.e. its pressure dependency, is also an important parameter that should be considered for the best possible design of the filtration units [14,6]. By measuring the local filtration properties, the pressure dependency can be evaluated during cake formation: this gives a valuable insight of the phenomena taking place during the process.

Depending on the type of wood raw material used in the Kraft process, the filtration of lignin produced during the LignoBoost

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Nomenclature

A	filtration area [m^2]
c	mass of solids per unit filtrate volume [kg/m^3]
$D_{(x)}$	diameter for which x% of the particles by number are smaller than the value stated [μm]
d_γ	average path length of γ-radiation [m]
I	ionic strength [mol/L]
n	Tiller and Leu equations parameter [–]
n_γ	number of counts [–]
$n_{\gamma,0}$	number of counts for an empty filter cell [–]
P_a	Tiller and Leu equations parameter [Pa]
P_{atm}	atmospheric pressure [Pa]
P_c	pressure drop over the filter cake [Pa]
P_l	local liquid pressure [Pa]
P_s	local solid pressure [Pa]
P_{TOT}	total applied pressure [Pa]
ΔP	pressure drop over the filter cell [Pa]
R_m	resistance of filter medium [m^{-1}]
t	time [s]
V	filtrate volume [m^3]
V_l	liquid volume within the final filter cake (included dissolved solids) [m^3]
V_s	non dissolved solids volume within the final filter cake [m^3]
v	superficial liquid velocity in the z-direction [m/s]
z	distance from the filter medium [m]

Greek letters

α	local specific filtration resistance [m/kg]
α_{av}	average specific filtration resistance [m/kg]
α_0	Tiller and Leu equations parameter [m/kg]
β	Tiller and Leu equations parameter [–]
$\Delta_{rel}\alpha$	relative experimental uncertainty in local filtration resistance [%]
$\lvert\Delta\phi\rvert$	absolute deviation in local solidosity [–]
$\Delta_{rel}\phi$	relative experimental uncertainty in local solidosity [%]
μ	viscosity of fluid [Pa s]
$\mu_{\gamma,l}$	attenuation coefficient for liquid [m^{-1}]
$\mu_{\gamma,s}$	attenuation coefficient for solid [m^{-1}]
ρ_s	solid density [kg/m^3]
ϕ	local solidosity [–]
ϕ_{av}	average solidosity [–]
ϕ_0	Tiller and Leu equations parameter [–]

Abbreviations

GPC	gel permeation chromatography
HPAEC	high performance anion exchange chromatography
L	lignin
X	xylan

process can vary from being relatively easy and forming weakly compressible filter cakes, to more difficult and forming more compressible filter cakes. If the precipitation conditions are chosen correctly, it has been shown that softwood LignoBoost lignin is a material that is relatively easy-to-filter and forms weakly compressible filter cakes [1]. Such material is therefore relatively easy to handle in the scale-up of the filtration steps. However, other types of Kraft lignin have been found to be much more difficult to filtrate, especially if the precipitation conditions have not been chosen correctly. For example, hardwood lignin has shown to be generally more difficult to filter than softwood lignin. Although the reasons behind this are not fully understood, one is nonetheless related to the content and nature of the residual lignin-hemicelluloses complex present in the extracted lignin [18].

Xylan is known to be the main hemicellulose found in hardwood. The objective of this study is to investigate the influence of the content of xylan on the local and average filtration properties of LignoBoost lignin. Model liquors were prepared through different procedures in which a known amount of hardwood xylan was added to softwood LignoBoost lignin with a very low original content of xylan. The influence of ionic strength during acid precipitation and filtration was also investigated by the addition of sodium sulphate. The materials were characterized and the filtration properties of the solid material in the resulting lignin/xylan suspensions were determined.

2. Experimental

2.1. Materials

The lignin used in this study is a softwood lignin extracted, using the LignoBoost process, from black liquor sourced from a demonstration plant located in Bäckhammar in Sweden. Its residual xylan content is very low: measurements using high-performance anion exchange chromatography (HPAEC) revealed that the concentration of xylan was about 0.28 w%. Such lignin is therefore suitable for studying the influence an addition of xylan has to its filtration properties.

The xylan used in this study is a xylan powder from beech wood. Two batches of beech wood xylan from two different producers were used: a Sigma-Aldrich and a TCI (Tokyo Chemical Industry) batch.

An addition of sodium sulphate (Fisher Chemical, purity > 99.5%) was made in some cases in order to investigate the influence of ionic strength. Finally, the pH of the slurries was adjusted using sodium hydroxide (Sigma Aldrich, purity ≥ 98%) and sulphuric acid (Scharlau, 95–97%).

2.2. Characterization

The solid density of the dried lignin and xylan solid particles was measured using a gas pycnometer (Micromeritics Accupyc 1330).

The sugar compositions of the two batches of xylan used were determined using HPAEC. The set-up consisted of a Dionex ICS-5000 system, equipped with CarboPac™ PA1 columns and an electrochemical detector. The software used was Chromeleon 7, Chromatography Data System, Version 7.1.0.898. NaOH and NaOH + NaAc (0.2 M) were used as eluents.

The amount of monomeric sugar xylose found in both the filtrate and the filter cake was also determined by HPAEC. In the case of the filtrate, the salt content was first eliminated by dialysis tubing (Spectra Por, molecular weight cut-off of 3.5 kDa) before the filtrate was evaporated using a rotary vacuum evaporator (Büchi, R) at a temperature not exceeding 35 °C. Finally, the remaining organic residue was dried at 40 °C for one week before acid hydrolysis was performed. The xylose content that was measured used thereafter to estimate the amount of xylan present in the filter cake.

Furthermore, the amount of lignin dissolved in the filtrate was estimated by subtracting the determined mass of xylan in the