Detailed characterization of poor settling green liquor dregs

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ABSTRACT: At a northern bleached softwood kraft (NBSK) mill in western Canada, poor settling green liquor dregs caused high non-process element levels in lime mud and white liquor pressure filter plugging. Dregs samples were collected during poor settling and normal settling conditions. Samples were examined by qualitative analysis, elemental analysis, quantitative X-ray diffraction (XRD) analysis, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscope/energy dispersion X-ray (SEM/EDX) spectroscopy. Poor settling dregs were caused by an inorganic gelatinous material. The inorganic gel was determined to be an amorphous magnesium silicate compound of approximate composition $Mg_2(Si_{1-x}Al_x)O_4$, with a molar ratio of silicon to aluminum of approximately 5:1. The density of the inorganic gel was only slightly higher than the green liquor, causing it to settle very slowly. When calcite particles were trapped by the gel, the average density increased, which increased the settling rate. The inorganic gel was present during normal settling, but contained more aluminum (silicon to aluminum ratio of approximately 2:1). During normal settling, the gel was more dense and contained more trapped particles of calcite.

Application: This work shows how the chemical composition of dregs can result in poor settling at an NBSK mill, outlines options to improve settling, and helps process engineers to understand dregs behavior and improve the settling efficiency of green liquor clarifiers.

At a northern bleached softwood kraft (NBSK) mill in western Canada, high levels of suspended solids occurred in clarified green liquor on a regular basis (once or twice a week). This resulted in higher non-process element (NPE) levels in lime mud, and increased pressure drop across the white liquor pressure filter. The mill uses oxygen delignification with magnesium sulfate addition. Flocculating anionic polymer was used in the green liquor clarifier, but varying the type and concentration did not correct the settling problem. Lime mud addition to the dissolving tank was used to improve dregs settling.

Mills with oxygen delignification and high magnesium levels in black liquor often suffer from poor dregs settling properties and high clarifier rake torque [1,2]. An excess of magnesium ion has been reported to lead to the formation of brucite, $Mg(OH)_2$, and poor dregs settling [1]; however, there is little evidence to support this mechanism.

Ulmgren [3] investigated magnesium precipitation chemistry in green liquor. He proposed that magnesium ion co-precipitates with aluminum ion in green liquor to form hydrotalcite-like compounds, $Mg_{1-x}Al_x(OH)_2(CO_3)_{x/2} \cdot nH_2O$, where the Mg:Al ratio can vary from 2:1 to 5:1 in different samples. Brucite and hydrotalcite have closely related crystal structures [4]. Brucite contains octahedral layers of $Mg(OH)_2$. In hydrotalcite, aluminum ions substitute for some of the magnesium ions in brucite. Hydrotalcite formation has been proposed as a mechanism for aluminum ion removal from green liquor [3].

The literature contains very little detailed information on dregs composition. Published dregs data contain primarily the overall elemental composition (Richardson et al. [5]), not the distribution of compounds that are present. Some work has identified crystalline dregs components by XRD [6], but not amorphous components.

The goals of this project were to fully characterize and identify the suspended solids that occur in green liquor during periods of poor and normal settling. This allowed a good understanding of the factors leading to high suspended solids content in clarified green liquor at the mill.

MATERIALS AND METHODS

Step-scan, X-ray powder-diffraction (XRD) data using the quantitative Rietveld method were collected on a standard Siemens (Bruker) D5000 Bragg-Brentano diffractometer at the University of British Columbia (UBC) Department of Earth and Ocean Sciences, Vancouver, BC, Canada, in the laboratory of Dr. Mati Raudsepp.

For scanning electron microscope/energy dispersion X-ray (SEM/EDX) spectroscopy analysis, the green liquor dregs sample was prepared by first washing the sample with deionized water to remove soluble sodium compounds. The sample was filtered and dried at 100°C to give a fine powder. The powder sample was prepared in epoxy, and the hardened mixture was polished in stages down to 6 μm diamond polish and mounted as a thick section slide by Vancouver Petrographics. This mounted sample was examined at UBC in variable-pressure mode in a Hitachi S-3000N SEM (Hitachi; Tokyo, Japan), producing back-scattered electron images. The EDX measurements were made with a light-element-capable Quartz X-One

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