Investigation of non-process element chemistry at Elk Falls mill - green liquor clarifier and lime cycle

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Abstract: Extensive sampling and analysis of green liquor clarifier deposits, dregs, grits, lime mud and lime were carried out. Using the quantitative Rietveld XRD method, inorganic compounds were identified in mill process streams that are previously unreported in the pulp & paper literature. Aluminum and silicon were lost from the green liquor clarifier primarily as the silicate diopside, $CaMgSi_2O_6$, and the aluminosilicates pargasite, $NaCa_2Mg_3Fe^{2+}Si_6Al_3O_{22}(OH)_2$, and vermiculite, $Mg_{1.8}Fe^{2+}_{0.9}Al_{4.3}SiO_{10}(OH)_2{\cdot}4(H_2O)$. A new kiln dead load component in lime was identified, $Ca_4Na_2(PO_4)_2(SiO_4)$. Application of these results to mill process improvement is discussed.

The Elk Falls mill was experiencing high dregs concentration in clarified green liquor and low solids content in lime mud from the rotary filter. In addition, lime mud pressure filters required frequent cleaning and the lime mud was dark green in colour. The incremental fuel cost resulting from low solids content of the lime mud to the lime kiln was estimated at more than \$200,000/year, while kiln dead load incremental cost was more than \$150,000/year.

Located near Campbell River on the east coast of Vancouver Island, Elk Falls began operation in 1952 as a single-line newsprint mill. The paper machine was joined with a kraft pulp mill on the same site in 1956, and two other paper machines followed in 1957 and 1982. A kraft paper machine was installed in 1966. Sawdust pulp, a product pioneered at Elk Falls, was first manufactured in 1964, and capacity expanded in 1983. The kraft mill was simplified in 2004, and is now a single-line operation. Elk falls currently produces about 830 t/d of kraft and 1600 t/d of TMP.

A research project was initiated in April 2005 to investigate the contribution of non-process elements (NPEs) to operational problems in the recaust area of the mill. Recent work by Taylor and Bossons suggested that aluminosilicate compounds in lime mud could lead to reduced filtration efficiency [1]. The effects of non-process elements on kraft mill operation have been extensively reviewed [2-12] and include increased scaling, increased lime kiln fuel use, reduced lime mud settling and reduced filtration efficiency. Dregs carryover to lime mud is known to significantly reduce lime mud solids content [13]. Most of these studies have focused on the concentration of individual non-process elements, their source and purge points, with little information on the NPE compounds themselves. Identification of the compounds formed by NPEs would allow a better understanding of their behaviour in the mill process and how to reduce their concentration in critical areas.

For instance, Magnusson et al [6] provide a list of potential dissolved and solid phase forms of NPEs in green liquor and white liquor. This paper has often been cited, but these species were not directly observed. They were proposed based on simple equilibrium chemistry.

Formation of hydrotalcite in lime mud, $Mg_6Al_2CO_3(OH)_{16}$, was first observed by Bennett et al [5] in their studies of wet air-oxidation (WAO) soda recovery. Magnesium from dolomite was intentionally added to make-up limestone to reduce aluminum concentration in the white liquor. Ulmgren [14] suggested that hydrotalcite was formed in the green liquor clarifier. He reported a successful mill trial where aluminum concentration in green liquor was reduced by the addition of magnesium sulfate to the dissolving tank.

The Skoghall mill in Sweden has added aluminum ion to the dissolving tank to reduce excess magnesium ion in green liquor [15]. It was assumed that hydrotalcite was formed, but this was not confirmed by any measurements.

Aluminosilicate scales in black liquor evaporators are well known [8,9,11]. Kaolinite, $Al_2O_3{\cdot}2SiO_2{\cdot}(OH)_4$, and gehlinite, $2CaO{\cdot}Al_2O_3{\cdot}SiO_2$, were reported in Harmac lime by Taylor and Bossons [1].

Experimental equilibrium solubility data for hydrotalcite and several aluminosilicates in black, green and white liquor streams were reported by Wannenmacher et al [8].

Hydroxylapatite, $Ca_5(PO_4)_3(OH)$, has been identified by XRD as the primary phosphorus-containing component of lime mud [16]. Phosphorus is an important dead load component in the lime kiln, since 1 wt% phosphorus in lime mud is equivalent to 5.4 wt% hydroxylapatite.

This paper describes NPE compounds and chemistry occurring in the green liquor clarifier and lime cycle of the Elk Falls mill. This information allowed the design of a successful NPE mass balance mill trial that is reported in a separate paper [17].

METHODS AND MATERIALS

Step-scan X-ray powder-diffraction data (XRD) using the quantitative Rietveld method were collected on a standard Siemens (Bruker) D5000 Bragg-Brentano diffractometer. For SEM/EDS measurements, samples were coated with evaporated carbon and examined on a Philips SL30 SEM equipped with a Princeton Gamma-Tech

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