

Scale up of electrochemical lignin depolymerization

C. Ecrabey, C. Roux, J. Bennetsen, R. Tschentscher, SINTEF, Process Technology, Oslo, Norway

Introduction

As the Kraft pulp mill industry is developing solutions to diversify

A liquid sampling system enables to draw samples during the operation. After a test the system can easily be cleaned by several cycles of hot water.

their product portfolio beyond pulp and green energy, the abundant lignin present in the kraft black liquor is a rich source of valuable aromatic compounds. Previous works on lab scale lignin depolymerization have produced industrial results combining electrolytical oxidation and reduction in an undivided cell. In this work we present the design and commissioning of a pilot reactor (TRL4) for electrochemical depolymerization of lignin.

Pilot reactor design

This rig, shown in Figure 1, enables to convert weak and intermediate black liquor as obtained from the industrial Kraft liquor cycle in volumes of up to 3 liters. The conditions with respect to temperature and black liquor composition are closely matched with the condensation train of a Kraft process, which enables simple integration into the Kraft liquor cycle.





Results and discussion

Running a (2:1:1) black liquor/white liquor/water solution and nickel foams as both cathode and anode, chrono-amperometric tests have shown stable currents for cell voltages up to 2.25 V. At voltages above 2.25 V the current drops indicating the formation of lignin-based deposits at the electrodes. Electrolyte foaming is prevented by adjusting the liquid flow rate at 32,2g/s.



Figure 3: Chronoamperometry results showing the impact of the circulation flow on the current stability



Figure 1: Pilot test rig for lignin depolymerization (left) and its scheme (right)

The rig consists of a double walled glass reservoir for black liquor. A thermostatically controlled oil pump enables to heat the reactor homogeneously up to 100°C and control the temperature inside the reactor with a connected thermometer. The black liquor is pumped from this reservoir through the cell and then either collected in a product reservoir (single-path operation) or circulated back into the reservoir (multi- path operation).

The cell used is a through flow design with opencell metal foam electrodes (Figure 2) with a geometric surface area of 78 cm2, having a significantly increased specific surface area of more than 1000 m2 per m3 of electrode material. Gas products are analyzed by online GC-TCD/PDD utilizing a nitrogen sweep gas (10ml/min) flowing



To verify current stability over time, constant voltage runs at 1.25 V for 4 hours resulted in stable current densities of 20-50 mA/cm2.



Figure 4: Chronoamperometry results for a 1,25V run (left)

Product analysis

Liquid analysis is currently performed by HPLC and GPC to identify Gas products are analyzed by online GC-TCD/PDD and estimate the produced monomers the extent of depolymerization and repolymerization. Initial data show the utilizing a nitrogen sweep gas (10ml/min) flowing Figure 2: Cell assembly production of both phenolic compounds and aromatic hydrocarbons, through the reservoir. A condenser is installed to but longer runs will be operated to convert most lignin in monomers for the pilot test rig prevent water vapor entering the GC. and be able to quantify them.

