ALBEMARLE®

Refining Solutions

INTERNSHIP RESEARCH REPORT

Code : [Reportcode (by secr)]
Date : 24-Jun-2016

Department : HPC-PDA
Name : Christian Harmoko

Signature :

Project code : 2342
Project name : Alumina washing

OPTIMIZE SALT REMOVAL IN ALUMINA WASHING

Number of pages: 19

Keywords: filtration, alumina, HPC

Intern student:
Christian Harmoko

Supervisors:
Anatolie Motelica (Albemarle Catalyst)
Johan Krop (De Haagse Hogeschool)

Internship period: 8 Feb – 24 Jun, 2016

FINAL Report

Acknowledgments

Process Development department in Amsterdam of Albemarle Catalyst supported this work. I want to thank Anatolie Motelica, my company's supervisor who continuously supported me in carrying out this research. I also want to thank Johan Krop and Claudia Germano, my university's supervisors, for the encouragement and time taken to evaluate my work.

Summary

Washing of boehmite cake is one of the crucial steps in catalyst production process. Understanding and quantification the influence of washing conditions, on purity of washed cake is of high importance. This allows finding the optimal washing conditions for existing processes and optimal design of new ones. In this report, the focus is on developing a correlation that predicts the amount of adsorbed ions (Na^+ and SO_4^{2-}) on boehmite, under certain washing conditions. The proposed correlation is developed based on theoretical insights and its parameters are determined from several sets of washing experiments. To show the applicability of the correlation, a simple Continuous Stirred Tank Washing model is developed. The calculated trends, for example, influence of slurry pH on the amount and type of adsorbed ion is well captured by the proposed correlation.

Conclusions

- A new model that correlates the amount ions (Na^+ and SO_4^{2-}) adsorbed on alumina boehmite is developed. The main model feature is that it can be employed to calculate the adsorbed amount of ions for various washing conditions (slurry pH, amount of washing water and wash water temperature).
- To fit the model parameters many washing experiments have been done. The reproducibility of the experiments is not always satisfactory. The measured data that we did not trust were eliminated from model parameter fit. The main source of errors is the required multistep handling of the cake and mother liquor to prepare the samples for XRF measurements, as the XRF equipment is not calibrated to measure concentrations in the domain required for our experiment. Aging of the slurry taken from the plant also contributes to difficulties in getting reproducible measurements. Several rules for XRF sample preparation are identified, and employed to mitigate the source of errors.
- The applicability of salt adsorption correlation is shown in modeling a Continuous Stirred Tank Washing process. The predicted trends, and the amount of adsorbed salts on boehmite, upon variation of slurry pH, amount of wash water, temperature and the amount of solids percentage in the cake left on the filter after filtration is as expected.

Recommendations

- Improve the measurement method or experiment strategy for the salts content in the washed cake and mother liquor to get a good reproducibility of the measurements.
- Extend the salts adsorption model for slurries which containing silica.
- It observed that the slurry properties slightly changes after two weeks based on the solid content test. Thus, the experiment must be done directly after get the slurry from the plant.
- Developing a plug flow washing model based on continuous stirred tank washing model and compares the results with the practical experiments.

Table of Content

List of abbreviations

SO_4 - Sulfate ion
Na - Sodium ion
HPC - Hydro Processing Catalyst
Alsul - Aluminum Sulfate
Natal - Sodium Aluminate
WG - Water Glass (Na_2SiO_3)
WW - Wash Water
ML - Mother Liquor
CSTR - Continuous Stirred Tank Reactor
CSTW - Continuous Stirred Tank Washing
PP - Pure Powder
H-water - Deionized water
XRF - X-Ray Fluorescence

List of notations

S_{pp} - Solid content in the pure powder (%w)
S_c - Solid content in the cake after 2nd filtration (%w)
S_{liq} - Solid content in the liquid after 2nd filtration (%w)
$S1_c$ - Solid content in cake after 1st filtration (%w)
$S1_{liq}$ - Solid content in liquid after 1st filtration (%w)
$w_{Na2O,pp}$ - Na_2O content in the pure powder (%w)
$w_{Na,pp}$ - Na content in the pure powder (%w)
$w_{SO4,pp}$ - SO_4 content in the pure powder (%w)
$m_{c,pp}$ - Mass of the cakes of pure powder (g)
$m_{s,pp}$ - Mass of the dry solid in pure powder (g)
m_c - Mass of the cakes after 2nd filtration (g)
$m_{s,c}$ - Mass of dry solid in the cake after 2nd filtration (g)
m_{liq} - Mass of the liquid after 2nd filtration (g)
$m_{s,liq}$ - Mass of dry solid in the liquid after 2nd filtration (g)
$M_{w,Na}$ - Molecular weight of Na (g/mole)
$M_{w,Na2O}$ - Molecular weight of Na_2O (g/mole)
m_{slu} - Mass of the slurry used in experiment (g)
$w_{Na2O,c}$ - Na_2O content in the cakes (%w)
$W_{Na,c}$ - Na content in the cakes (%w)
$W_{SO4,c}$ - SO_4 content in the cakes (%w)
$w_{Na2O,c(m)}$ - Na_2O content in the cakes with PP (%w)
$W_{SO4,c(m)}$ - SO_4 content in the cakes with PP (%w)
$m_{Na,c}$ - Mass of Na in the cakes (g)
$m_{SO4,c}$ - Mass of SO_4 in the cakes (g)
w_{ss} - Weight fraction of solids (include salts) in the slurry (%w)
$w_{Na2O,s}$ - Na_2O content in the slurry (%w)
$w_{SO4,s}$ - SO_4 content in the slurry (%w)
$w_{Na,s}$ - Na content in the slurry (%w)
$\rho_{H2O\ 25\ C}$ - Water density at 25°C (g/L)
$V_{liq,c}$ - Volume liquid in the cake (g/L)
$m_{Na,a}$ - Mass of Na adsorbed on boehmite (g)
$m_{SO4,a}$ - Mass of SO_4 adsorbed on boehmite (g)
$m_{Na,liq}$ - Mass of Na adsorbed on liquid phase (g)
$m_{SO4,liq}$ - Mass of SO_4 adsorbed on liquid phase (g)
m_b - Mass of boehmite (g)
m_{HNO3} - Mass HNO_3 added in the liquid (g)
m_T - Total mass of the cakes (g)
$w_{Na2O,l}$ - Na_2O content in the liquid (%w)
$W_{Na,liq}$ - Na content in the liquid (%w)
$W_{SO4,liq}$ - SO_4 content in the liquid (%w)
$w_{Na2O,liq(m)}$ - Na_2O content in the liquid with PP (%w)
$W_{SO4,liq(m)}$ - SO_4 content in the liquid with PP (%w)
c_{Na} - Concentration Na in liquid phase (g/L)
c_{SO4} - Concentration SO_4 in liquid phase (g/L)
LOI_c - Loss on ignition cake at 120°C (%w)
ρ_{slurry} - Density of the slurry (kg/m^3)
$c_{Na,s}$ - Concentration Na in the slurry (g/L)
$c_{Na2O,liq}$ - Concentration Na_2O in liquid phase (g/L)
$c_{SO4,s}$ - Concentration SO_4 in the slurry (g/L)
ρ_{H-H2O} - Density of H-water at 50°C (kg/m^3)
ρ_{HNO3} - Density of HNO_3 at 50°C (kg/m^3)
$HNO_{3\ sol\ content}$ - Solution content of HNO_3 (%w)
pH_{sp} - The pH set point
pH_{mix} - The pH mixture of the slurry and H-water
m_{H-H2O} - Mass of H-water added (g)
$m1_c$ - Mass of cake from 1st filtration (g)
$m1_{liq,w}$ - Mass of liquid from 1st filtration (g)
$m1_{c,w}$ - Mass of cake from 1st filtration (g)
$m1_{s,c}$ - Mass of dry solid in the cake after 1st filtration (g)
$m1_{s,liq}$ - Mass of dry solid in the liquid after 1st filtration (g)
m_{c+} - Mass of the cake used to analyze (g)
m_{pp} - Mass of the pure powder used to analyze (g)
m_{liq+} - Mass of the liquid used to analyze (g)
ρ_{liq} - Density of liquid (kg/m^3)

**The list of abbreviations and notations is used for reading the report and the attachments.*