

**Supplementary Information to**

**Optimization of the neutralization of Red Mud by pyrolysis bio-oil using a Design of Experiments Approach.**

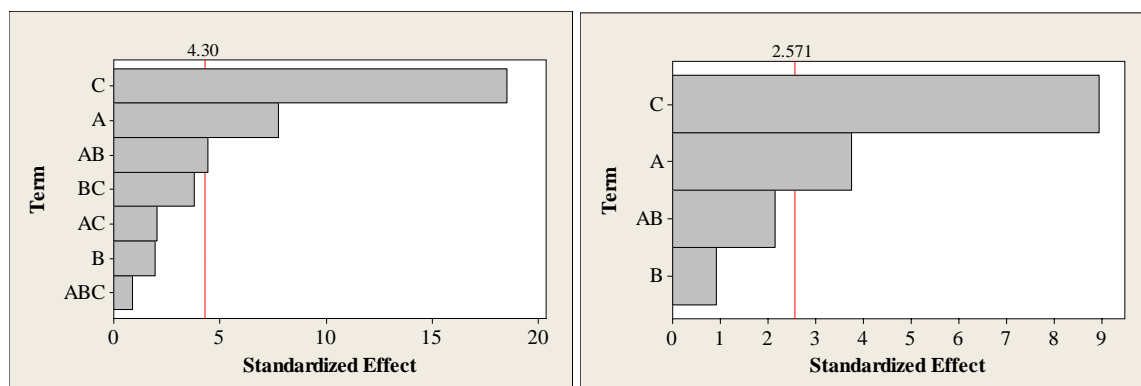
Véronique Jollet, Christopher Gissane and Marcel Schlaf\*

Department of Chemistry  
Guelph-Waterloo Centre for Graduate Work in Chemistry (GWC)<sup>2</sup>  
University of Guelph  
Guelph, Ontario, N1G2W1, Canada  
mschlaf@uoguelph.ca

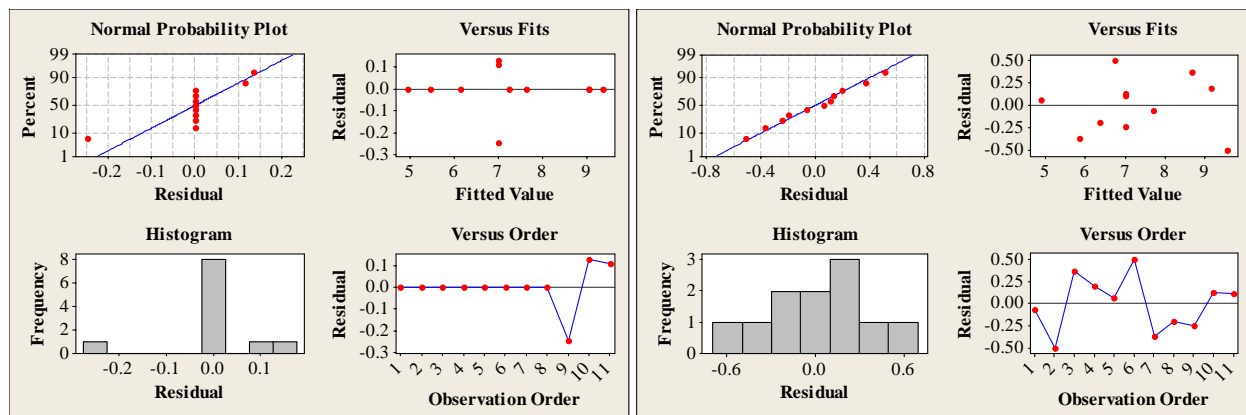
### Experimental data on the residuals.

The standardized effects are determined dividing the value of coefficients  $\beta$  by the square roots of the main diagonal elements of the covariance matrix  $\alpha^2(X'X)^{-1}$ . The line of significance in red is established by the Student's *t*-distribution at the level  $\alpha = 0.05$  and the degree of freedom for the residual error corresponding to  $n-p$ . Any bars extending beyond this line are considered a significant terms.

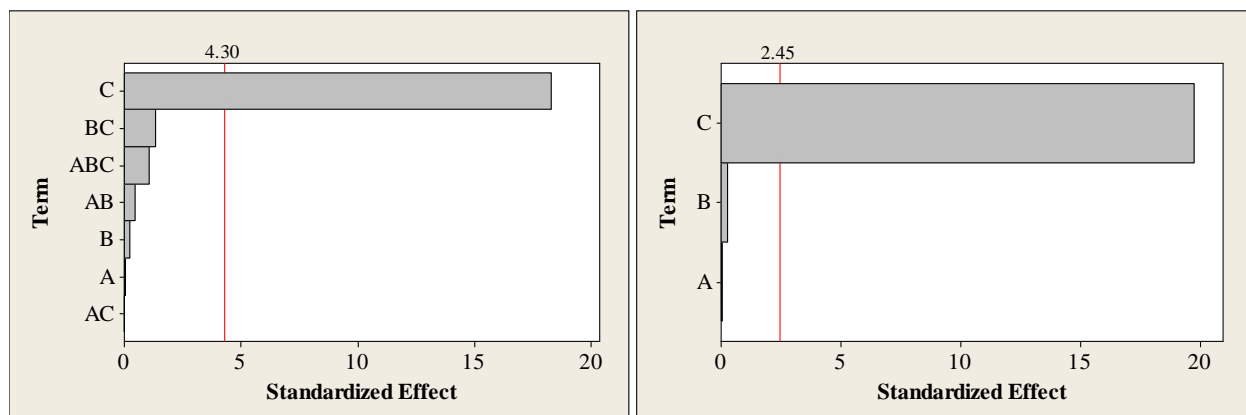
For all graphs A, B and C = X1, X2 and X3.



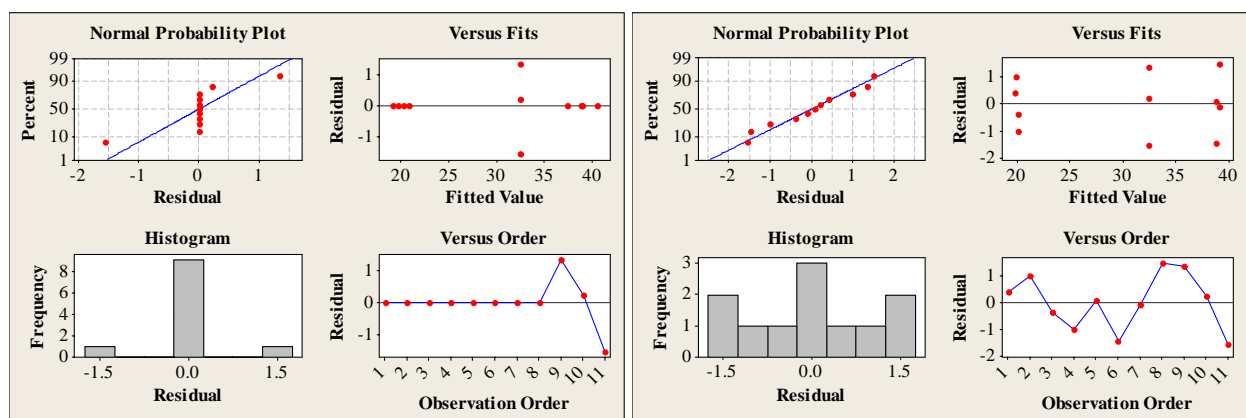
**Figure S1:** Pareto charts of the standardized effects for the pH of the solid (full model in left, reduced model in right).



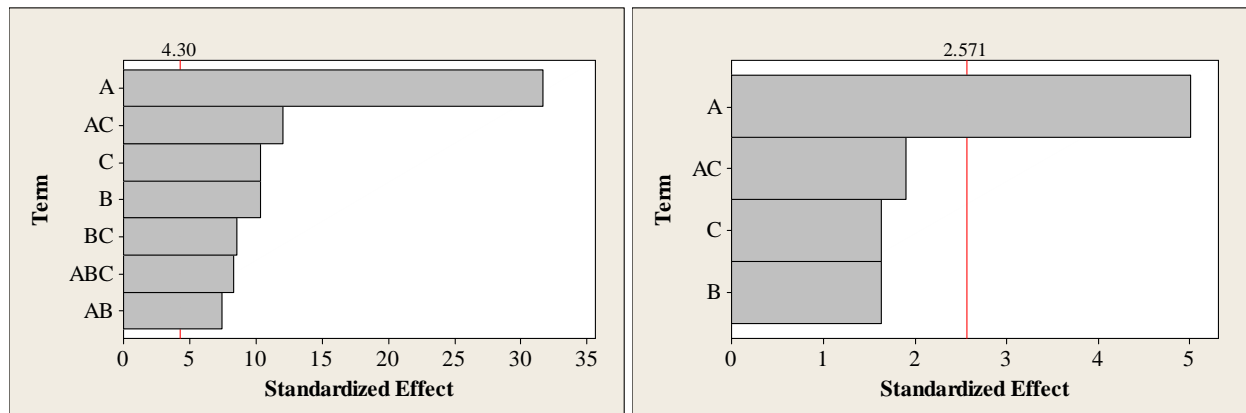
**Figure S2:** Residual plots for the pH of the solid (full model in left, reduced model in right).



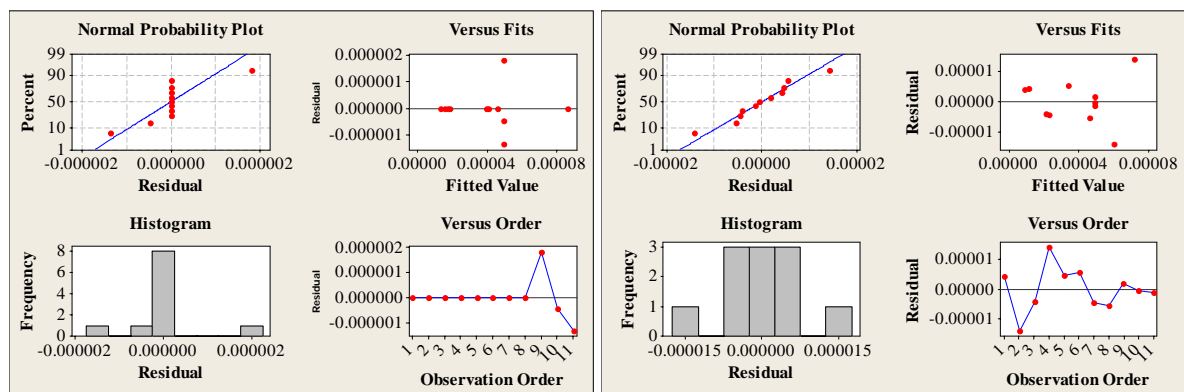
**Figure S3:** Pareto charts of the standardized effects for the percentage of carbon in the solid (full model in left, reduced model in right).



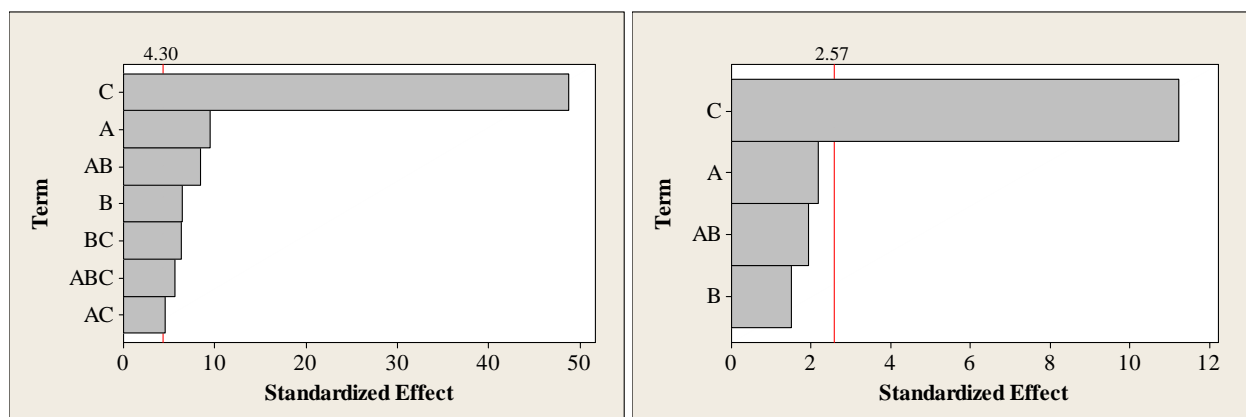
**Figure S4:** Residual plots for the percentage of carbon in the solid (full model in left, reduced model in right).



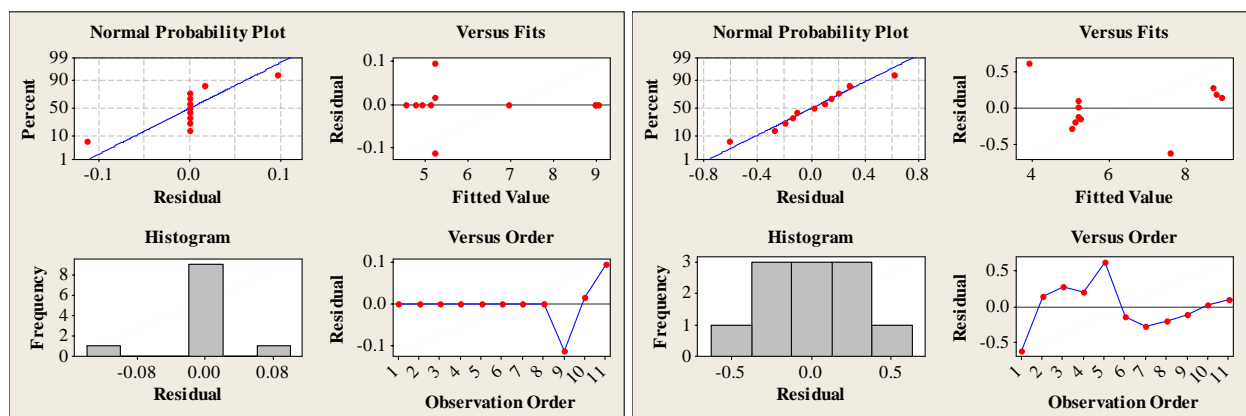
**Figure S5:** Pareto charts of the standardized effects for the mass magnetic susceptibility (full model in left, reduced model in right).



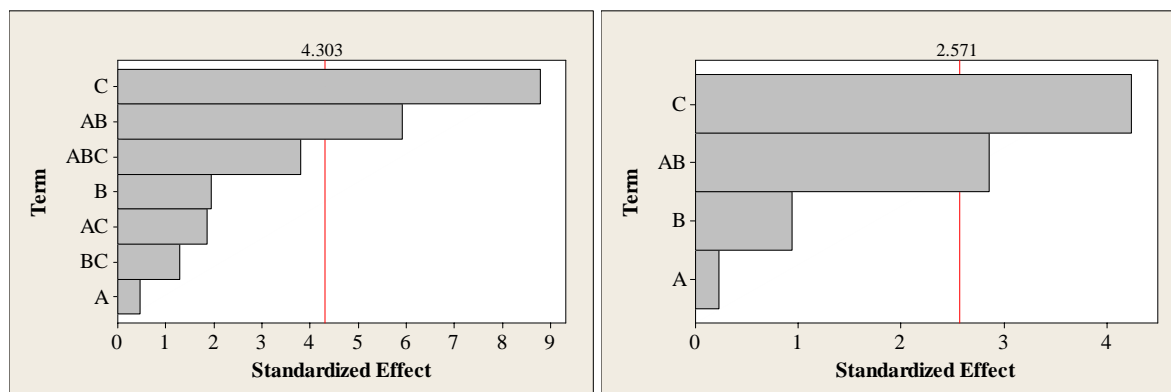
**Figure S6:** Residual plots for the mass magnetic susceptibility (full model in left, reduced model in right).



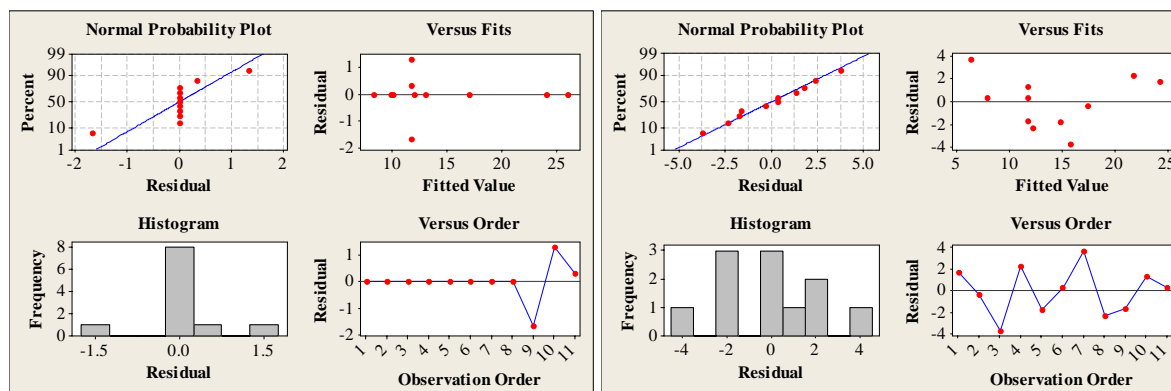
**Figure S7:** Pareto charts of the standardized effects for the pH of the aqueous phase (full model in left, reduced model in right).



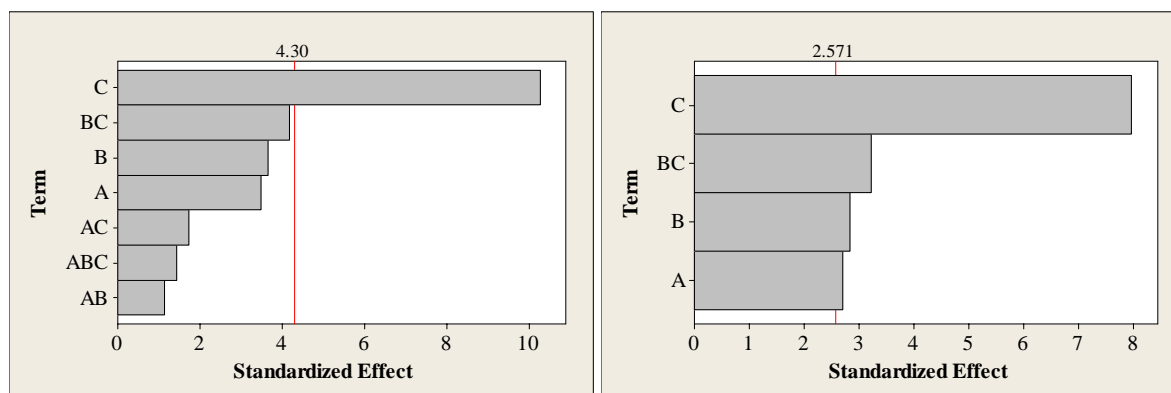
**Figure S8:** Residual plots for the pH of the aqueous phase (full model in left, reduced model in right).



**Figure S9:** Pareto charts of the standardized effects for the sodium content in the aqueous phase (full model in left, reduced model in right).



**Figure S10:** Residual plots for the sodium content in the aqueous phase (full model in left, reduced model in right).



**Figure S11:** Pareto charts of the standardized effects for the sodium content in the aqueous phase (full model in left, reduced model in right).

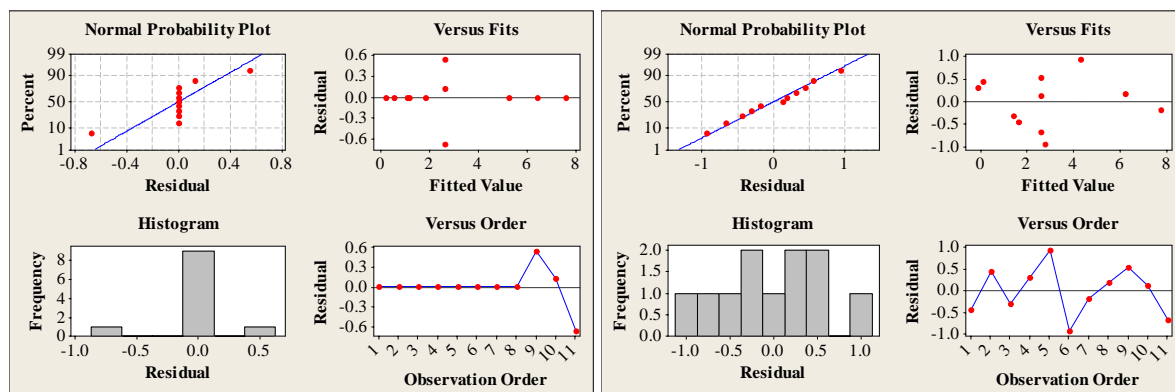


Figure S12: Residual plots for the mass of aqueous phase (full model in left, reduced model in right).

### Factorial plots

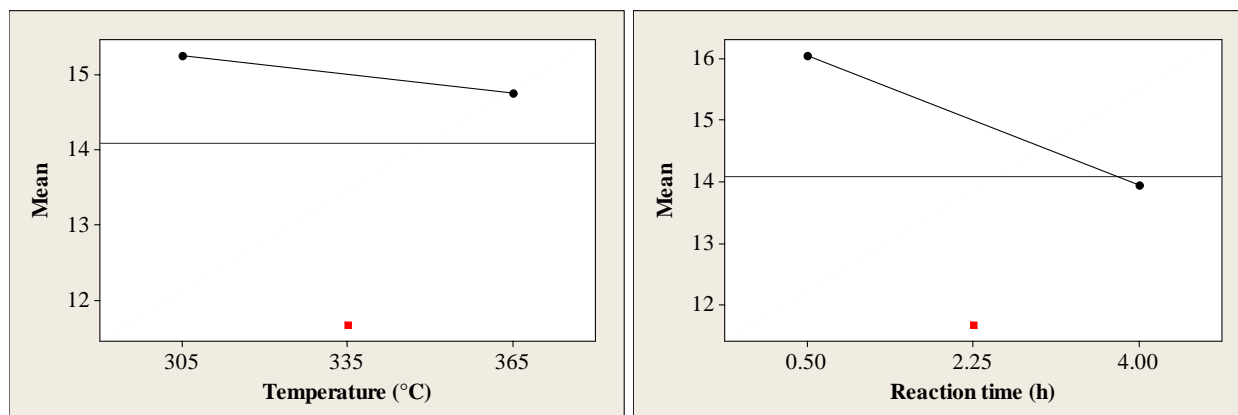
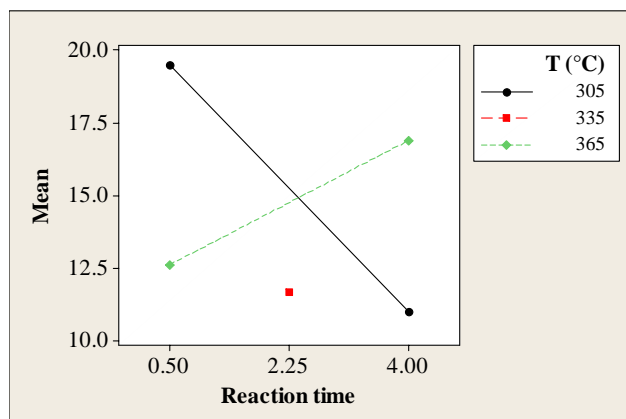


Figure S12: Mean effects plot for the sodium content (according to the temperature in left, and the reaction time in right). The reference lines correspond to the total average of responses. The points represent the average of responses at the border and the center of the domain.



**Figure S14:** Interaction plot for the sodium content between the reaction time and the temperature. The points represent the average of responses at the border and the center of the domain.

### Magnetic separation experimtns:

**Procedure:** The solid phases from the replication and the scale up experiments were separated in 6 samples (<20 grams each) according to the mass of solid obtained for wet magnetic separation testing. Each sample was to be individually processed using the Wet High Intensity Magnetic Separator [WHIMS]. Each sample was individually processed; each was slurried with approximately 300 mL of tap water and poured into the canister of the separator, which contained medium expanded metal matrix (MEX) with a background magnetic field of 1000 Gauss. The canister was then rinsed with 350 - 400 mL of tap water to flush out entrapped non-magnetic material, which was collected in a clean pail. The magnetic field was turned off and the magnetic material was rinsed from the matrix with tap water into a clean pail. The magnetic fraction was filtered, dried, weighed, and bagged. The non-magnetic fraction was reprocessed using the same matrix and a background magnetic field of 5000 Gauss. The magnetic and final non-magnetic fractions were filtered, dried, weighed, and bagged.

List of separation data for each sample.

Sample Id <sup>a</sup>	Field (Gauss)	Fraction Id <sup>a</sup>	Fraction (w%)
20 <sup>b</sup>	1000	magnetic	96.60
		non magnetic	3.40
	5000	magnetic	3.20
		non magnetic	0.19
20 <sup>b</sup>	1000	magnetic	97.24
		non magnetic	2.76
	5000	magnetic	2.62
		non magnetic	0.13
20 <sup>b</sup>	1000	magnetic	97.01
		non magnetic	2.99
	5000	magnetic	2.19
		non magnetic	0.58
18	1000	magnetic	93.63
		non magnetic	6.37
	5000	magnetic	5.75
		non magnetic	0.55
19	1000	magnetic	92.19
		non magnetic	7.81
	5000	magnetic	7.22
		non magnetic	0.55
19	1000	magnetic	90.76
		non magnetic	9.24
	5000	magnetic	8.21
		non magnetic	0.91

<sup>a</sup> Identification <sup>b</sup> Samples from experiments 14, 15, 16, 17 were mixed in one fraction called 20.





LABORATORY SERVICES  
Agriculture and Food Laboratory

FINAL Report  
Submission# 12-107712  
Reported: 2013-Jan-11

Submitted By:  
UNIVERSITY OF GUELPH  
VERONIQUE JOLLET  
DEPT OF CHEMISTRY  
U OF G  
GUELPH, ON N1G 2W1

Owner:  
MARCEL SCHALF

Phone: 519 824-4120  
Sampling Date: Not given Received Date: 2012-Dec-14

---

**Other**

Date Authorized: 2013-Jan-11 10:26

Sample ID	Client Sample ID	Specimen	Sampling date / time	Test	Result	Note
0001	RED MUD	Other		Other	See below	

**Comments:**

SN-Other reported as ug/g as received:

Vanadium 727  
Chromium 811  
Manganese 76.7  
Cobalt 11.4  
Nickel 24.2  
Copper 21.7  
Arsenic 22.4

Using methods SNL-013 and SNL-014

Test method(s): Non-routine

Supervisor: Nicolaas Schrier MSc 519 823 1268 ext. 57215 nschrier@uoguelph.ca

This report may not be reproduced except in full without written approval by Laboratory Services.  
These test results pertain only to the specimens tested.

2002-07-26

**Subject : Typical Composition of the Process By-product of the Extraction of Alumina from Bauxite, often called Red Mud or Bayer Process Residue.**

It is worth to mention that this composition varies with the source and the composition of the bauxite itself.

1. Elementary composition expressed as the oxide by XRF :

	<u>%</u>
Al <sub>2</sub> O <sub>3</sub>	15-25
Fe <sub>2</sub> O <sub>3</sub>	30-40
SiO <sub>2</sub>	10-20
TiO <sub>2</sub>	3-8
CaO	1-6
Na <sub>2</sub> O	5-10
H <sub>2</sub> O (chemical)	10

2. Phases identified :

Hematite	Fe <sub>2</sub> O <sub>3</sub>
Goethite	FeO(OH)
Bayer Sodalite	sodium aluminium silicate
Anatase	TiO <sub>2</sub>
Rutile	TiO <sub>2</sub>
Gibbsite	Al <sub>2</sub> O <sub>3</sub> .3H <sub>2</sub> O
Boehmite	Al <sub>2</sub> O <sub>3</sub> .H <sub>2</sub> O
Katoite	calcium aluminium silicate
Quartz	SiO <sub>2</sub>

3. Particle size distribution :

	<u>%</u>
+ 250 microns(sand)	10-15
10-250 microns	10-30
-10 microns	70
median	about 5 microns with sand
median	about 1 micron without sand

4. Other physical properties (when completely washed) :

Dried solid, inert, non toxic, slightly alkaline  
Specific density : 3.0-3.5 g/cc  
Specific surface area : 10-40 m<sup>2</sup>/g