---

**Article:**

Escudero-Castejón, L, Taylor, J, Sánchez-Segado, S et al. (1 more author) (2021) A novel reductive alkali roasting of chromite ores for Carcinogen-free Cr6+-ion Extraction of chromium oxide (Cr2O3) – a clean route to chromium product manufacturing! Journal of Hazardous Materials, 403. 123589. ISSN 0304-3894

https://doi.org/10.1016/j.jhazmat.2020.123589

---

# Supplementary Information

## A novel reductive alkali roasting of chromite ores for $Cr^{6+}$-ion free extraction of chromium oxide ($Cr_2O_3$) – *a clean route to chromium manufacturing!*

Authors: L. Escudero-Castejón, James Taylor, S. Sánchez-Segado, A. Jha*

a.jha@leeds.ac.uk

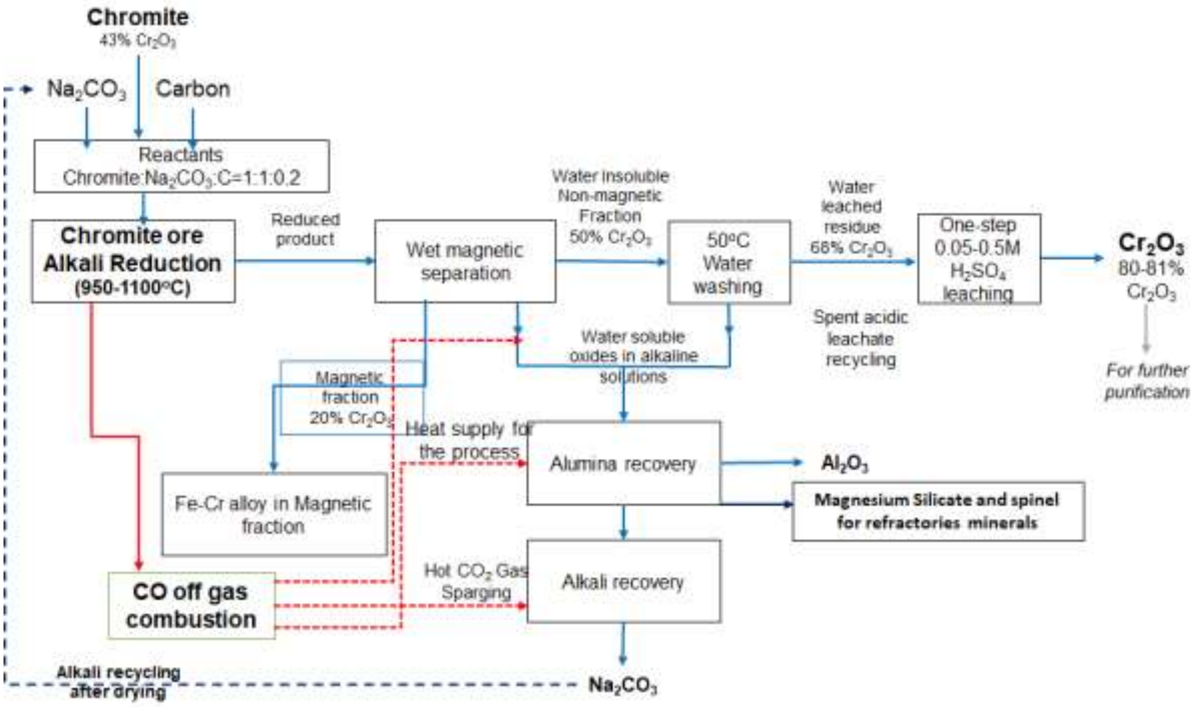


Figure S1: A schematic of Process Materials Flow diagram for alkali reduction of chromite ores with carbon for the extraction of $Cr^{3+}$-value s without generating or discharging $Cr^{6+}$-ionic species.

## EXPERIMENTAL METHODOLOGIES

### *TGA Analysis and 100 g non-isothermal experiments:*

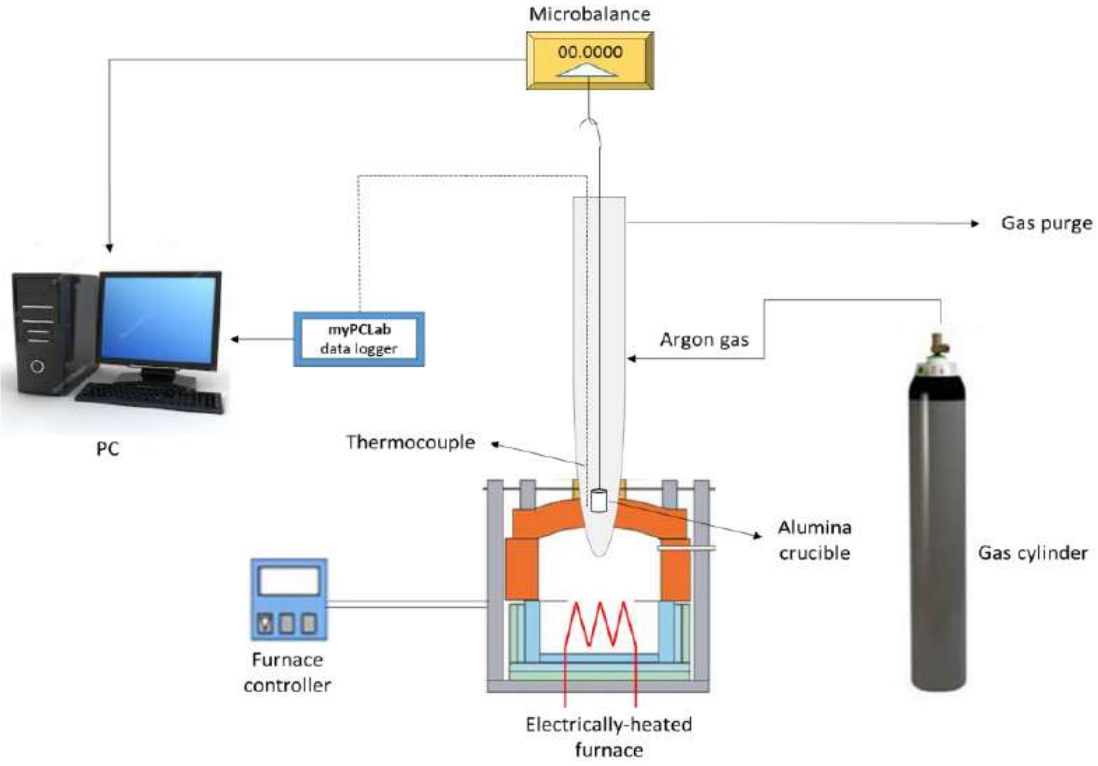
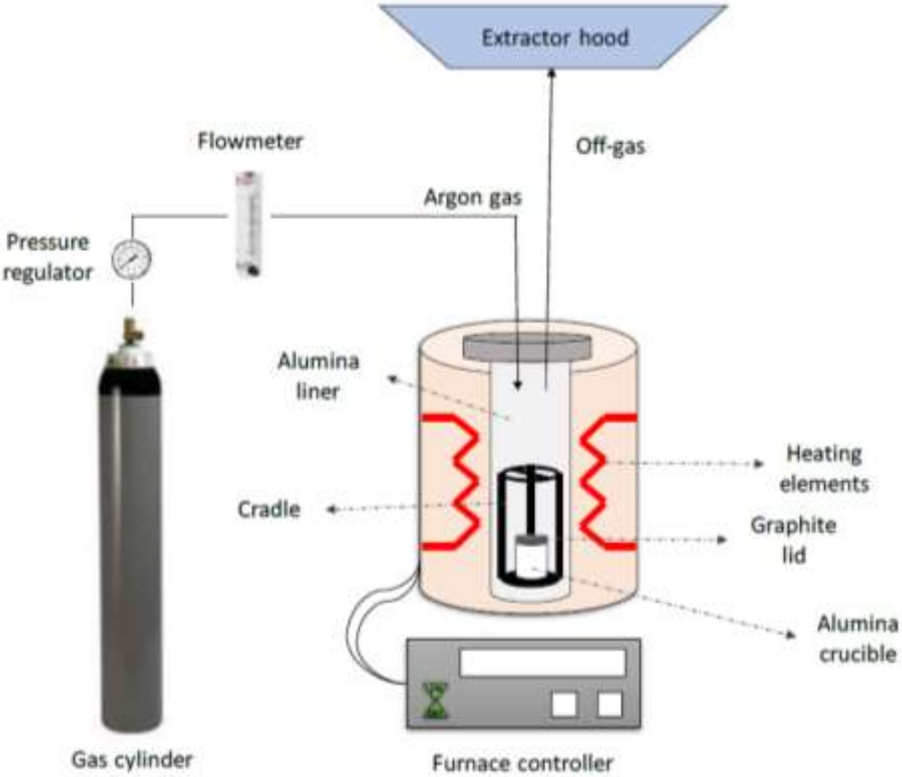


Figure S2: The Schematic diagram of the TGA instrumentation with a layout of supporting accessories.

100g batches of reactant mixture was used in each batch and heated at a rate of 5ºC/min from room temperature to 1050ºC in an electric muffle furnace (see Figure S3). The



atmosphere was maintained inside the furnace using argon gas maintained at 2 litre/min. Once the desired isotherm was reached, the reactants were held for 0.5 hrs before cooling down to room temperature. After cooling the wet magnetic separation using water at 50°C

### *Chemical analysis of solid samples by XRF, XRD and SEM Analysis Techniques*

XRF: X-ray fluorescence (XRF) was employed for analysing the chemical composition of various ore and separated inorganic phases samples. The sample preparation in XRF required the grinding of the samples until a fine powder was obtained, followed by heating at 900°C in oxidising atmosphere for the determination of the loss of ignition. Each sample was subsequently mixed with a lithium borate-based binder in a weight ratio of 95% binder and 5% sample and total weight of the mixture equal to 7 grams. The mixture was placed in a zirconium crucible ($V = 30$ mL, $h = 32$ mm, $do = 40.5$ mm and straight wall), which was fitted in a Katanax® K1 Prime fusion furnace. A temperature program was set during which the mixture is first heated until fused ($T \approx 1050$ oC), mixed by automatic swinging of the crucible in the furnace, and finally poured on to a mould. The program finishes with the cooling of the mould and sample until approximately 70oC - 80oC. The result was a fused bead which was used for the analysis.

The analysis was carried out in a ZSX Primus II multi-element wavelength dispersive X-ray fluorescence spectrometer with the ZSX Primus II software. A fully-quantitative program was developed using a series of standards, which allowed to obtain calibration curves for the different analytes and to take into account the effects of the sample matrix. The analytes included in the program were Cr, Al, Fe, Mg, Na, Si and Ca. The standards were prepared by mixing pure oxides of the analytes of interest (excepting Na, which was added as Na2CO3) in different weight ratios; and their corresponding fused beads were obtained following an identical procedure than that used for the rest of samples.

### *UV-visible spectroscopic analysis of leachate solutions*

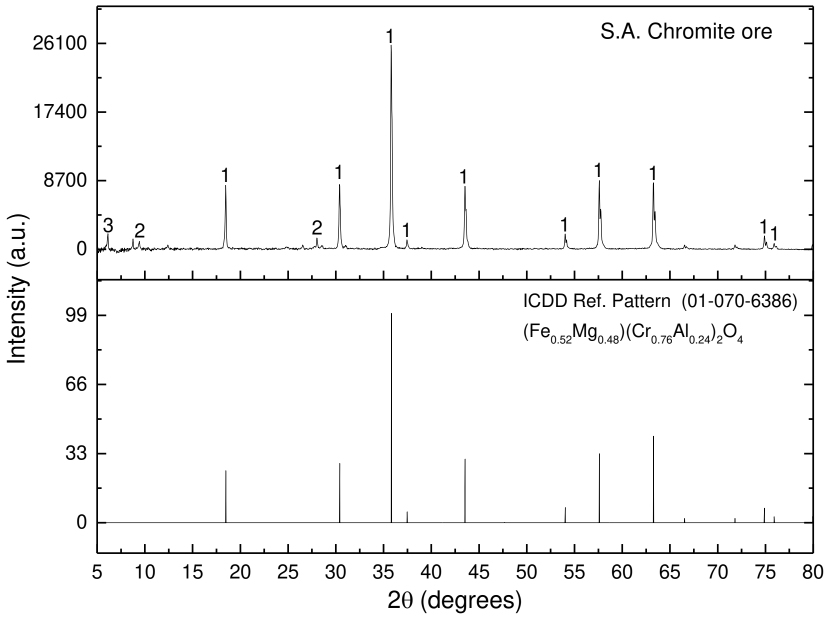


**Figure S2**: A comparison of the X-ray powder diffraction data for the S African Chromite ore with the ICDD data on chromite spinel structures.

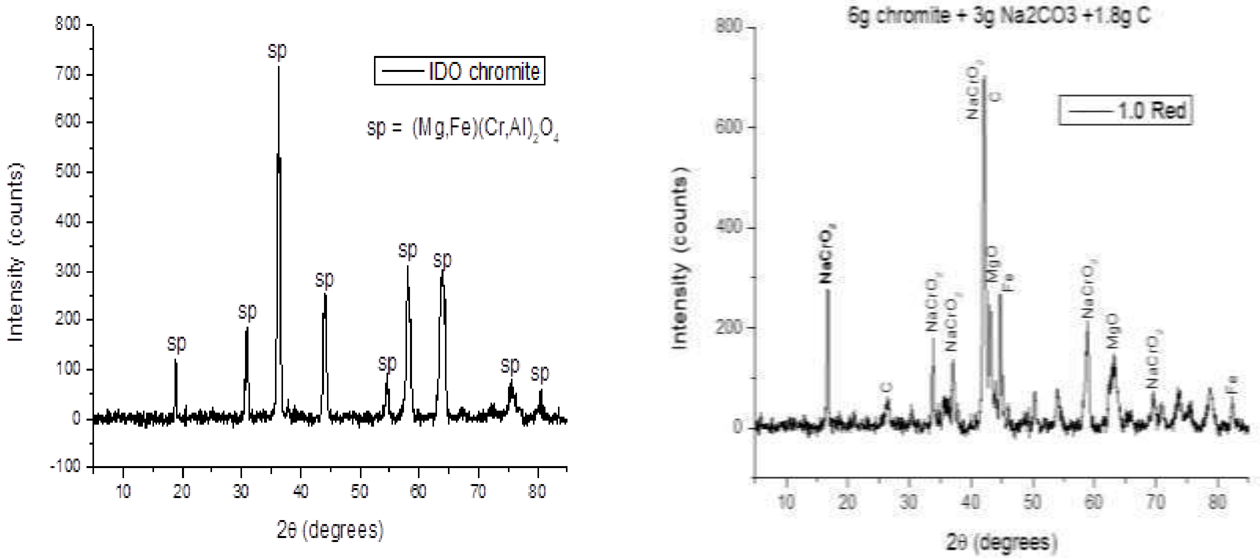
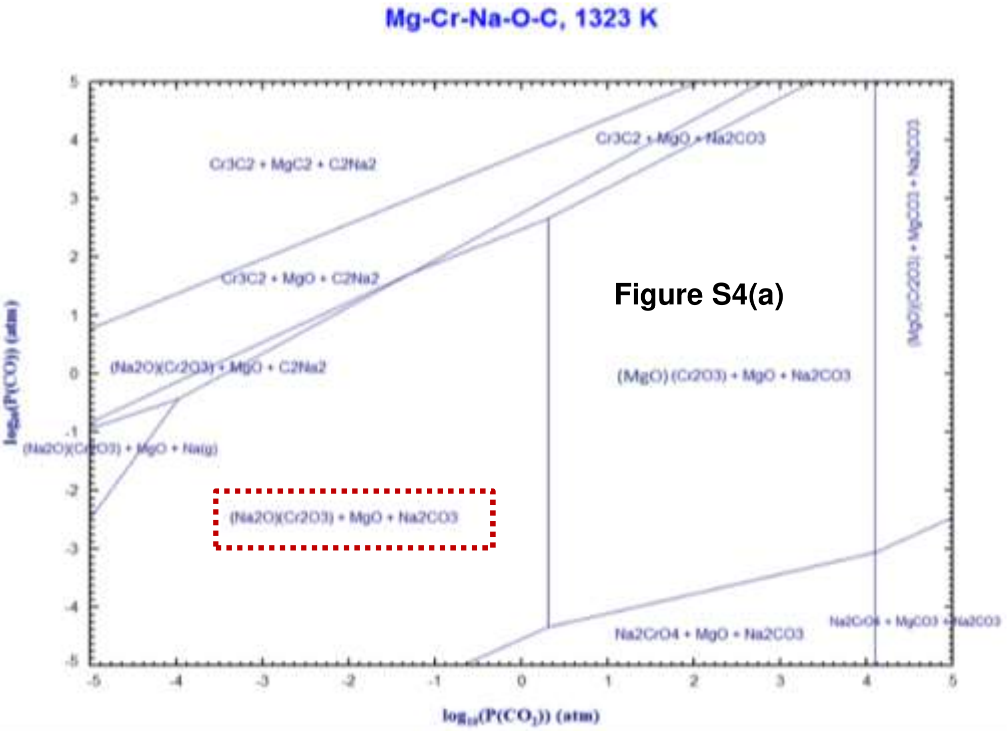


**Figure S3**: X-Ray powder diffraction (XRPD) data for an Indonesian ore before (left) and after reduction (right) at 1100°C. The peaks not labelled in XRPD of this ore after reduction is for complex and multicomponent (Al, NA, Mg, Ca) silicates which are apparent in the scanning electron microscopic analysis and may be partly amorphous.



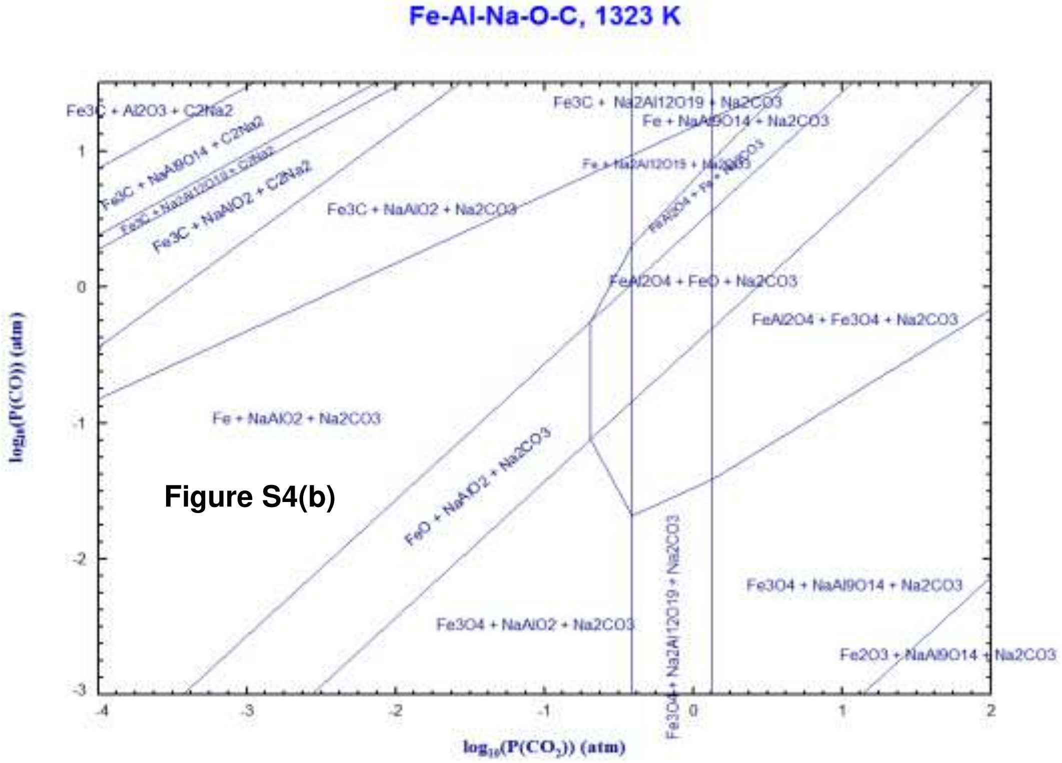


**Figure S4**: Computed multicomponent phase equilibrium assemblages at 1323K (1050°C) for the, a) Mg-Cr-Na-C-O and b) Fe-Al-Na-C-O systems. These assemblages were computed using the Fact Sage program [23]

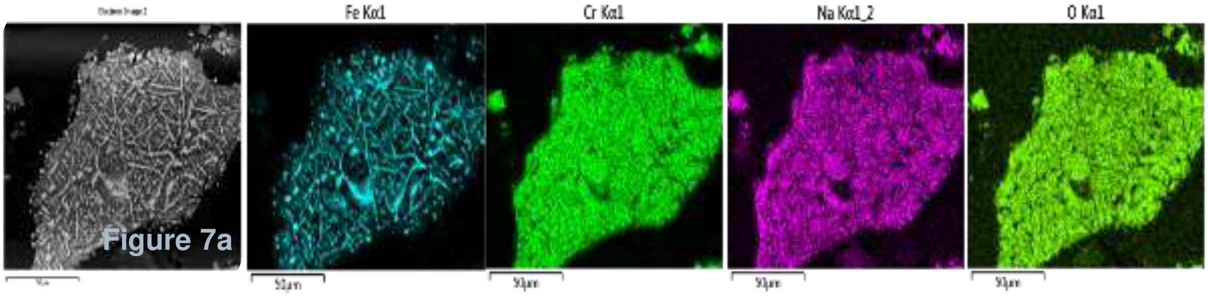


**Figure S5**: Backscattered scanning electron microscopic image and EDX elemental map of Indonesian chromite ore reduced with alkali and charcoal at 1100°C (1373K) for 1.5 hrs. The chromite:$Na_2CO_3$:C wt. ratio used was 1:1:0.2.

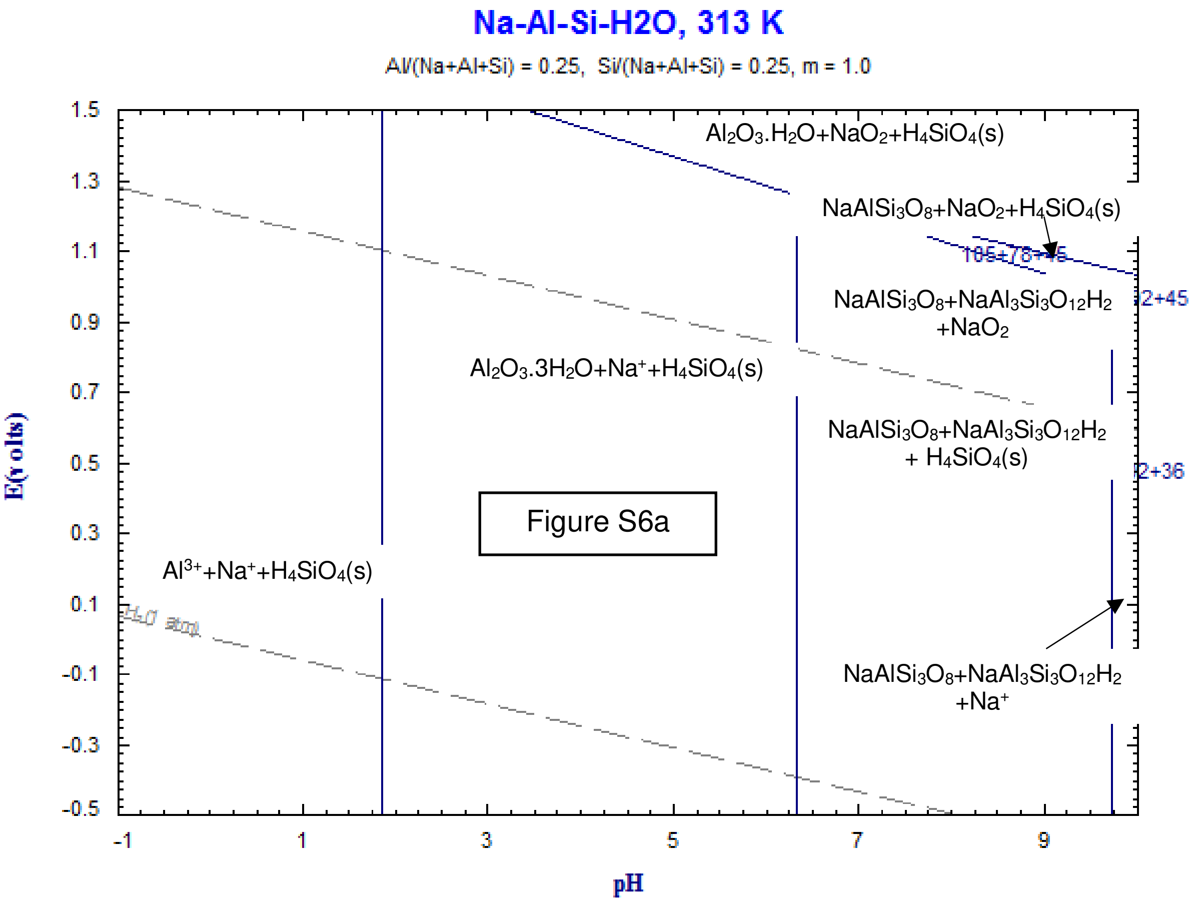
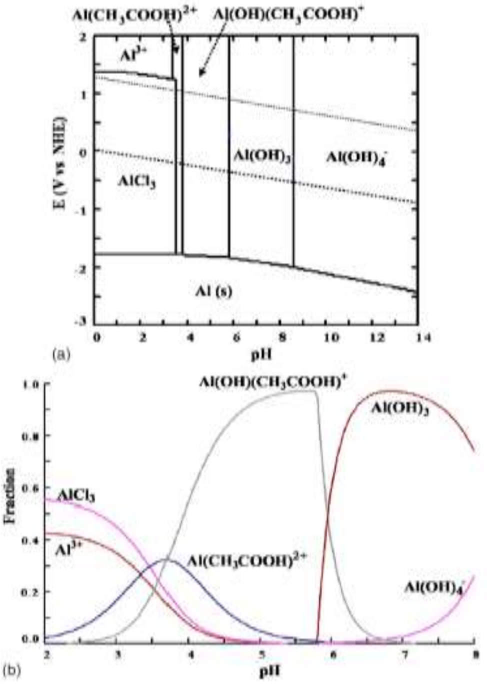


Figure S6b



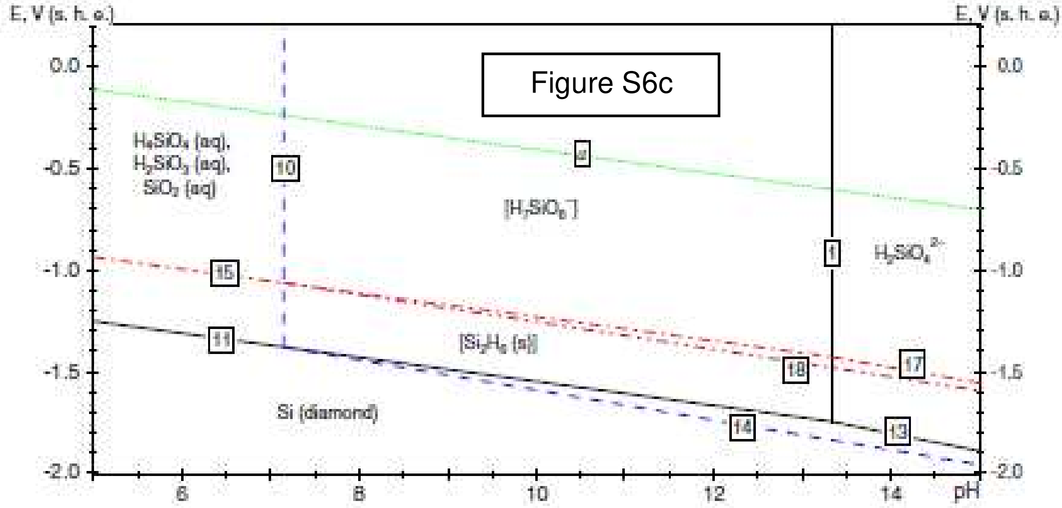


Figure S6: The computed EH-pH diagrams for the **a**) Na-Al-Si-$H_2O$ multicomponent system, relevant for chemical separation and purification of $Cr_2O_3$ and $Al(OH)_3$ from silicate complexes, present in water. Figures S5b and S5c for alumina and silica precipitation from aqueous media [30,31].