High-Throughput Bauxite Characterization and Process Monitoring via Automated QXRD Integrated with Cluster Analysis and PLSR Modelling

Sheida Makvandi[1], Matteo Pernechele[2], Uwe Koenig[3], Lin Wang[4] and Grace Zhang[5]
1, 2. Application Specialist
3. Global Mining Segment Manager
Malvern Panalytical, Almelo, Netherlands
4, 5. Application Specialist
Malvern Panalytical Division - Spectris Instrumentation & Systems Shanghai, Shanghai, China
Corresponding author: *sheida.makvandi@malvernpanalytical.com*
https://doi.org/10.71659/icsoba2025-bx010

Abstract

DOWNLOAD FULL PAPER

The aluminium industry faces growing challenges due to declining bauxite grades, compositional variability, and the demand for sustainable refining. This study demonstrates a fully automated, high-throughput XRD workflow using Malvern Panalytical's Aeris (Minerals Edition) compact diffractometer for mineralogical and process-oriented characterization of bauxite and red mud, integrating Rietveld refinement, cluster analysis, and Partial Least Squares Regression (PLSR). Besides enabling mineralogical discrimination between lateritic bauxite, karstic-diasporic types, and red mud- critical for Bayer feed quality assessment, residue valorisation, and environmental risk mitigation- the method also identified five distinct subtypes within the lateritic group. These were defined by diagnostic mineralogical profiles (e.g., gibbsite-rich, kaolinite-dominant, high-iron, boehmitic), each linked to specific digestion strategies. Amorphous content was quantified using a corrected external standard approach, and kaolinite crystallinity (via FWHM of the 001 reflection) was evaluated as an indicator of silica reactivity.

PLSR models trained on full-pattern XRD data demonstrated excellent predictive power for Total Extractable Alumina, Total Al_2O_3, Reactive Silica, and LOI, with R^2 values ranging from 0.989 to 0.999, minimal bias, and strong agreement with reference data across all parameters. This integrated approach enables simultaneous extraction of mineralogical and compositional parameters within minutes, providing a robust decision-support tool for ore classification, blending, and Bayer process optimization – especially for complex and low-grade bauxites.

Keywords: Quantitative XRD, Bayer process parameters, kaolinite crystallinity, amorphous content, HighScore Plus.

1. Introduction

Bauxite, the principal ore of aluminium, is geologically diverse and mineralogically complex. It primarily contains aluminium hydroxide minerals – gibbsite [$Al(OH)_3$], boehmite [γ-AlO(OH)], and diaspore [α-AlO(OH)] – alongside iron oxides (goethite, hematite), silica (mainly as kaolinite and quartz), and titanium-bearing phases such as anatase and rutile [1]. This variability critically influences grade classification and processing performance, directly affecting digestion efficiency, caustic soda consumption, desilication product (DSP) formation, and red mud generation during the Bayer process [2].

As global aluminium demand grows – driven by its lightweight, corrosion-resistant properties and broad industrial use – bauxite mining and alumina refining face increasing pressure to enhance efficiency, reduce waste, and meet environmental regulations. These challenges are compounded by declining ore grades, greater feed variability, and the need for improved residue management.

Conventional grade control methods like X-ray fluorescence (XRF) provide elemental data but limited insight into mineralogical phases that govern reactivity. The form of alumina phases and the presence of reactive silicates such as kaolinite strongly influence the choice of digestion temperature (which depends on the dominant aluminium-bearing mineral in the bauxite), caustic loss, and red mud characteristics. Bauxites with high reactive silica (> 8.0 wt%) are often uneconomical due to excessive soda loss and DSP formation [3].

To overcome these limitations, X-ray diffraction (XRD), especially with Rietveld refinement and multivariate analysis, has become vital for bauxite quality assessment. XRD enables the identification and quantification of both crystalline phases (via the Rietveld method) and amorphous content (via calibration), allowing accurate estimation of process-critical parameters such as the Total Extractable Alumina (TEA) and reactive silica. The mineralogical composition also guides digestion strategy; for example, gibbsite-rich bauxites are typically digested at 150 °C, while those containing significant boehmite require higher temperatures, generally 225 °C.

Recent advances in detector technology, low-power instruments, and high-throughput handling have improved the speed, precision, and sustainability of XRD, making it suitable for both mine-site screening and in-plant control. Comprehensive mineralogical profiling also aids in identifying by-products such as iron, titanium, and gallium. This article discusses bauxite's mineralogical complexity, its impact on the Bayer process, and the increasing role of XRD in enabling efficient, sustainable alumina production.

2. Materials and Methods

This study refines a methodology originally established in 2012 [4], which demonstrated the use of rapid X-ray diffraction (XRD) and cluster analysis for fast mineralogical grade control of bauxite based on Rietveld quantification [5] and calculated process parameters including the TEA and reactive silica. Building on this foundation, we incorporated advancements in quantitative XRD (QXRD), Rietveld refinement strategies, and multivariate statistical tools to enhance scalability and accuracy. The updated workflow was applied to a dataset of 40 industrial samples and 10 certified reference materials from NIST and ALCAN. As industrial bauxites are commonly blended ores tailored to meet specific grade specifications, the use of certified standards with well-defined mineralogy is critical – they act as anchors for model calibration and validation, ensuring reliable quantification across diverse compositions. All samples were prepared using automated milling and pressing to ensure consistency and reproducibility. Notably, gibbsite quantification may be affected by preferred orientation due to its platy morphology, potentially leading to overestimation. While careful sample preparation helps minimize this effect, minor bias may persist.

In addition, the abovementioned samples, a larger dataset of 110 bauxite mining samples, with complementary chemical reference data, was used to explore the potential of Partial Least Squares Regression (PLSR) calibration in HighScore Plus for predicting key compositional parameters.

2.1 System Features and XRD Measurements

XRD measurements were performed using the Aeris Minerals Edition compact diffractometer (Malvern Panalytical), equipped with a Co-anode tube (40 kV, 15 mA), 145 mm goniometer, and Bragg–Brentano geometry. A high-capacity sample changer (HCSC, 67 positions) enabled high-throughput analysis. Detection was carried out using the 1Der energy-resolving detector, which provides automatic control of the energy window, allowing fluorescence-free, high-resolution data acquisition without the need for Kβ filters. This is particularly advantageous for compositionally complex samples like bauxite that commonly contain amorphous and/or disordered phases. While Co radiation was used in this study, the system also supports the use of

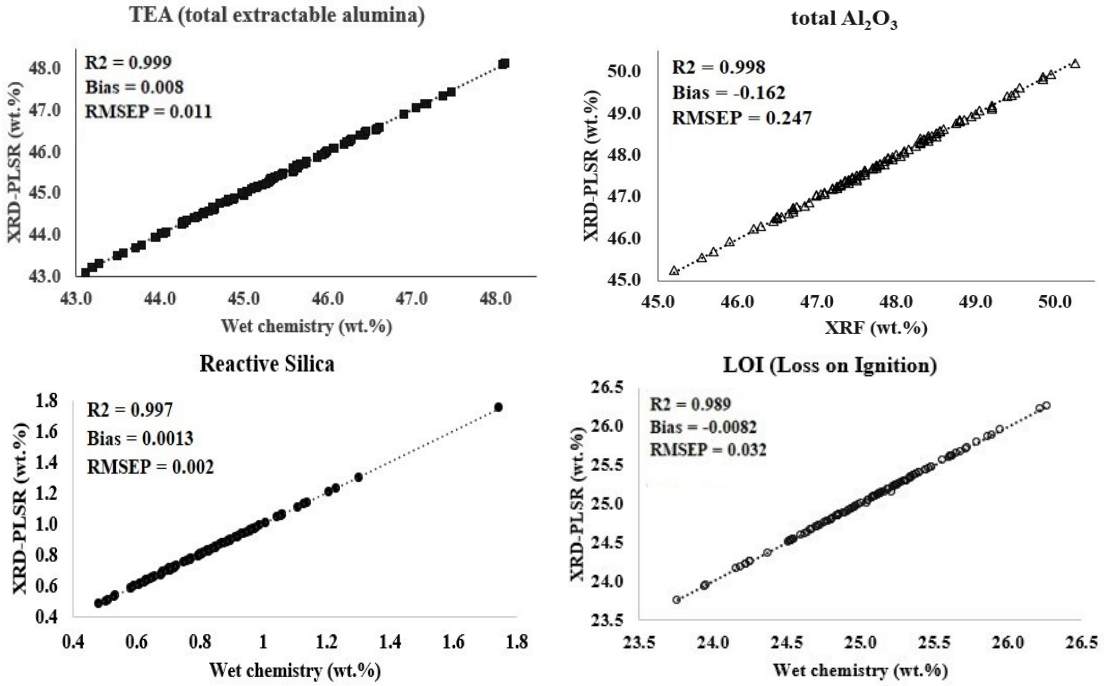

Figure 7. Correlation plots between XRD-PLSR predictions and reference values for TEA, total Al_2O_3, reactive silica, and LOI, showing excellent linearity (R^2), low root mean square error of prediction (RMSEP) and minimal bias across all Bayer-relevant parameters.

4. Conclusions

This study demonstrates the effectiveness of X-ray diffraction (XRD) for comprehensive characterization of bauxite and red mud, supporting both process optimization and sustainable resource utilization. The combined use of quantitative phase analysis (QXRD), cluster analysis, and Partial Least Squares Regression (PLSR) enabled the classification of bauxite types, evaluation of ore grade, prediction of Bayer process behaviour, and assessment of residue composition. Key methodological advances included the quantification of amorphous content using an optimized external standard approach and the estimation of kaolinite crystallinity to better predict its reactivity during digestion. PLSR models developed from industrial XRD datasets showed excellent correlation with reference values obtained via wet chemistry or XRF for process parameters TEA, Total Al_2O_3, Reactive Silica, and LOI. By integrating stoichiometric phase quantification via Rietveld refinement with pattern-based statistical modelling, the approach enables simultaneous extraction of mineralogical and compositional information. All outputs can be generated through a fully automated workflow within minutes of data acquisition, positioning XRD as a practical and efficient tool for routine quality control and process decision-making in alumina refining.

5. References

1. S.H. Patterson, H.F. Kurtz, J.C. Olson, C.L. Neeley, World Bauxite Resources- Geology and resources of aluminium, U.S. Geological Survey Professional Paper 1076-B, U.S. Government Printing Office, 1986.
2. M. Authier-Martin, G. Forte, S. Ostap, J. See, The mineralogy of bauxite for producing smelter-grade alumina, the journal of the *Minerals, Metals & Materials Society*, 53,2001, 36–40.
3. P. Smith, The processing of high silica bauxites — Review of existing and potential processes, Hydrometallurgy, 98, Issues 1–2, August 2009, Pages 162-176.

4. U. König, R.S. Angélica, N. Norberg, L. Gobbo, Rapid X-ray diffraction (XRD) for grade control of bauxites. Proceedings of *19th International Conference and Exhibition of ICSOBA*, 2012. Belém, Brazil.
5. H.M. Rietveld, A profile refinement method for nuclear and magnetic structures, Journal of Applied Crystallography 2 (2), 1969, 65-71.
6. Thomas Degen, Mustapha Sadki, Egbert Bron, Uwe König and Gwilherm Nénert, The high score suite, *Powder Diffraction* 29, 2014, S13-S18.
7. St. Gates-Rector, T. Blanton, The powder diffraction file: a quality materials characterization database, *Powder Diffraction* 34, 2019, 352-360.
8. H. Wold, Estimation of principal components and related models by iterative least squares, In P.R. Krishnaiaah (Ed.), *Multivariate Analysis*, 1966, 391-420.
9. U. König, R.S. N. Norberg, Partial Least Square Regression (PLSR) – A new alternative XRD method for process control in aluminium industries, *The 32nd International Conference and Exhibition of ICSOBA, 2014*, Zhengzhou, China.
10. J.M. Amigó, J. Bastida, M.J. García Agramut, M. Sanz, J. Galván, Crystallinity of Lower Cretaceous kaolinites of Teruel. In: Galán E, Pérez-Rodríguez JL, Cornejo J, editors. EUROCLAY Conf.; Sevilla' 87; Sevilla, Spain, 1987, 74–75.
11. P. Aparicio, E. Galán, Mineralogical Interference on Kaolinite Crystallinity Index Measurements, *Clays and Clay Minerals*, 1999, 47, 12–27.
12. M. Kumar Kar, M.A.R. Önal, C.R. Borra, Alumina recovery from bauxite residue: A concise review, *Resources, Conservation and Recycling*, 2023, v. 198, 107158.
13. I. Vangelatos, G.N. Angelopoulos, D. Boufounos, Utilization of ferroalumina as raw material in the production of ordinary Portland cement, *Journal of Hazardous Materials*, 168, 2009, 473–8.