# Thermal Denaturing and Study of Bauxite Organics

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## Abstract

The distribution and removal of organic material within pisolitic bauxite, mined in Northern Queensland, Australia, has been studied in this research programme. Although bauxite organics as measured by Total Organic Carbon (TOC) content (< 0.30 %) may appear negligible, it increases oxalate formation disrupting the Bayer process. While previous studies have described the in-situ influence of decaying Bayer organics to increase oxalate production, the study and removal of bauxite organics prior to the Bayer process (pre-Bayer) have remained limited. The current research examined the nature and distribution of organics within pisolitic bauxite. LIBS/ToF SIMS indicate "younger" oxygenated organics evident on the outer perimeter of pisolites while within the pisolite, organics exhibit more of an aromatic character. Furthermore, bauxite organics tend to increase as the particle size distribution decreases and at shallower depths within the deposit. Additionally, the authors have previously investigated several techniques to reduce bauxite TOC (including dissolution, plasma, ultrasonics, abrasion and boric acid esterification). However, low-temperature thermal organics "denaturing" (275–350 °C) proved to have the largest impact on the Bayer process value chain and is reported. Low-temperature thermal organics denaturing reduces bauxite TOC by 42 % yet reduces oxalate formation by up to 86 %. A developing LCMS "fingerprint" programme provided a simple method to show the impact of thermal treatment on the molecular suite of Bayer organics. Bauxite QMID-TGA/DTA results indicate increased thermal stability of organics formed by thermal treatment partially explaining the noted compounding TOC/oxalate relationship. The research highlights the importance of understanding bauxite organics and shows that low-temperature thermal treatment can denature organics effectively, reducing oxalate formation with less energy compared to complete elimination.

**Keywords**: Bauxite organics, Thermal denaturing, Oxalate.

## 1. Introduction

Pisolitic bauxite mined in the Weipa region of north-east Queensland, Australia has been extensively studied by Taylor and Eggelton [1–4]. Bauxite grade (or quality) is typically defined by alumina and silica oxide equivalents from the sum of mineralogical composition [4]. One of the minor bauxite contaminants is organic carbon, which is primarily adsorbed on the outer perimeter of pisolitic bauxite particles through leaching of decaying humic matter, originating in the vegetative overburden. Total Organic Carbon (TOC) numerically defines the organic content of bauxite, which is typically below 0.3 % for most global deposits [5]. Bauxite organics are mostly insoluble in water (at a neutral pH). They comprise micro-organisms, cellulose, lignin, fulvic acid, humic acid and humins [7]. A portion of these organics are soluble within Bayer liquor (pH 12). As humic acids are the most abundant Bayer-soluble organics, their empirical chemical structure may be used as a reference biopolymer [7]. A humic acid molecule comprises several oxygenated molecular functions, primarily hydroxyl and carboxylic acids but additionally (in

lesser concentration) esters, aldehydes and ketones. As such, most of these oxygenated organics are readily extracted into Bayer liquor and susceptible to caustic decay serving as molecular precursors producing stable low molecular weight (LMW) derivatives, including oxalate. Oxalate has limited Bayer solubility, precipitating as sodium oxalate ($Na_2C_2O_4$) at elevated concentration, disrupting the alumina refinery process. Although bauxite organics negatively influence the Bayer process in diverse modes [6–9], the primary focus of this research is the influence on oxalate production.

The impact of bauxite organics within alumina refineries has historically focused on fundamental studies of in-situ decaying organics (referencing model compounds) within Bayer liquor [7, 10, 11]. Subsequently, in-situ methodologies to remediate this impact have focused on Bayer liquor [26] and not pre-Bayer (bauxite pretreated to remove organics) processes. Apart from denoting the numerical TOC value, fundamental studies examining the nature of bauxite organics with the aim of developing pre-Bayer interventions to restrict refinery impact have been limited (as they are minor contaminants in pisolitic bauxite). The geological age of pisolitic bauxite in the Weipa region is estimated to be between 50 and 65 million years [2, 5]. On a molecular level (and over the bauxitization process), occluded humic material within the pisolitic core gradually transforms (decreasing H/C and O/C ratios), acquiring more of an aromatic character [12, 13] which hinders Bayer caustic decay in contrast with their "younger" humic counterparts deposited on the outer perimeter. Thus, the molecular character of organics within a pisolite core and those deposited on the outer perimeter of the cortex differ as would their individual impact within the Bayer process value chain. Increased caustic decay linked with "younger" humic acids is a function of organic oxygenate substitution (especially hydroxyls and carboxylic acids) which facilitate the decay mechanism through hydrogen transfer reactions [10, 14] within the Bayer process.

Bauxite activation examines the influence of thermal treatment (600 °C) on the dehydroxylation of gibbsitic and boehmitic alumina minerals forming amorphous alumina [15]. While this eliminates bauxite TOC (and thus oxalate production potential), it is only a secondary benefit. The primary driver for bauxite activation is to reduce refinery energy by operating a lower temperature Bayer circuit.

The current study aims to increase fundamental knowledge of pre-Bayer bauxite organics by examining their spatial distribution within pisolitic bauxite in conjunction with variance in chemical nature. These fundamental findings were used to explore potential pre-Bayer processes for bauxite TOC reduction. This internal research programme initially explored a diverse array of approaches including solvent dissolution, plasma, ultrasonics, boric acid esterification and abrasion. Low-temperature thermal treatment (275–350 °C) to "denature" bauxite organics was found to be effective and is discussed in this paper, along with its downstream impact within the Bayer process.

## 2. Experimental Methods

### 2.1 Bauxite Organics Characterisation

A Particle Size Distribution (PSD) was conducted using standard screens. PSD fractions that were isolated include > 9.5 mm, 2.0–9.5 mm, 0.6–2.0 mm and < 0.6 mm. The TOC of the fractions was established and is shown in Figure 2.

Two novel methodologies were used to characterize pisolitic bauxite organics:

- Laser-Induced Breakdown Spectroscopy (LIBS): Samples were analysed on a Elemission Coriosity LIBS utilising a pulsed high-energy laser (1064 nm) focused on the bauxite surface producing plasma emitting photons. The photons possess wavelengths specific to

an element (in this case Fe, Al, Ti, Si and C) producing a spectral fingerprint over a 40 × 40 mm domain.
- Time of Flight Simulation (ToF SIMS): This was conducted using an IONTOF M6 (Energy: 30 keV, target current: 0.1 pA and field of view 4 μm/px) to determine organic fragmentation ions.

### 2.2 Low-Temperature Thermal Treatment

Pisolitic bauxite (originating from the Amrun mine within the Weipa region) was sampled from the Yarwun refinery stockpile, at Gladstone, Queensland and oven dried (105 °C) overnight. A control sample was not heat treated (untreated bauxite), while two other samples were thermally treated to 275 and 350 °C, respectively, in a laboratory furnace for one hour. The following bauxite analysis was conducted:

- Bauxite TOC using a Shimadzu SSM-5000A solids analyzer.
- TGA/DTA (QMID) using a Netzsch STA F3 Jupiter simultaneous thermal analysis system with a QMS Aeolos Mass spectrometer with the mass spectrum of the gases leaving the furnace chamber (1 to 300 AMU/charge).
- XRF using a PANalytical Zetium XRF Spectrometer.
- LCMS using a Shimadzu 9030 is a high-performance quadrupole time-of-flight (Q-TOF) mass spectrometer. The LC component is a Shimadzu Nexera HPLC/UHPLC system. The samples were acidified, diluted and filtered, then concentrated and purified using solid phase extraction (SPE). Prepared samples were injected into a Shim-pack Scepter $C_{18\text{-}120}$ column and column guard, with compounds separated using a gradient method.

The bauxite samples were digested using a laboratory Bayer method. The bauxite charge for digestion was calculated using the assay results for the untreated bauxite. Bauxite samples were digested (in triplicate), on the 4-bay bomb unit under Yarwun conditions of high temperature digestion (270 °C). The slurries were quenched to < 95 °C, centrifuged and a sample of liquor filtered and retained for analysis. A blank sample of digest liquor without bauxite was also digested and quenched in triplicate. All liquors were analysed for sodium oxalate (Dionex IC), Total Organic Carbon (Shimadzu TOC-VWS), alumina and caustic (Metrohm Autotitrator).

## 3. Results and Discussion

### 3.1 Influence of Particle Size Distribution (PSD) on TOC

The Amrun bauxite deposit is described as a collection of loose, roughly spherical particles existing within a 10 m layer under 2 m of vegetative overburden [3, 5]. As indicated in previous studies [1, 3] the bauxite PSD comprises:

- Fine bauxite fragments with irregular morphologies (< 0.6 mm)
- Oolites and pisolites (0.6–9.5 mm) including roughly spherical particles comprising an inner core and a series of concentric layers comprising the outer cortex
- Larger nodules and pisolites (> 9.5 mm) exhibiting oblong/spherical morphologies which may include intact pisolites incorporated within core of a nodule (during the bauxitization process)

Photos of the different size fractions are shown in Figure 1.

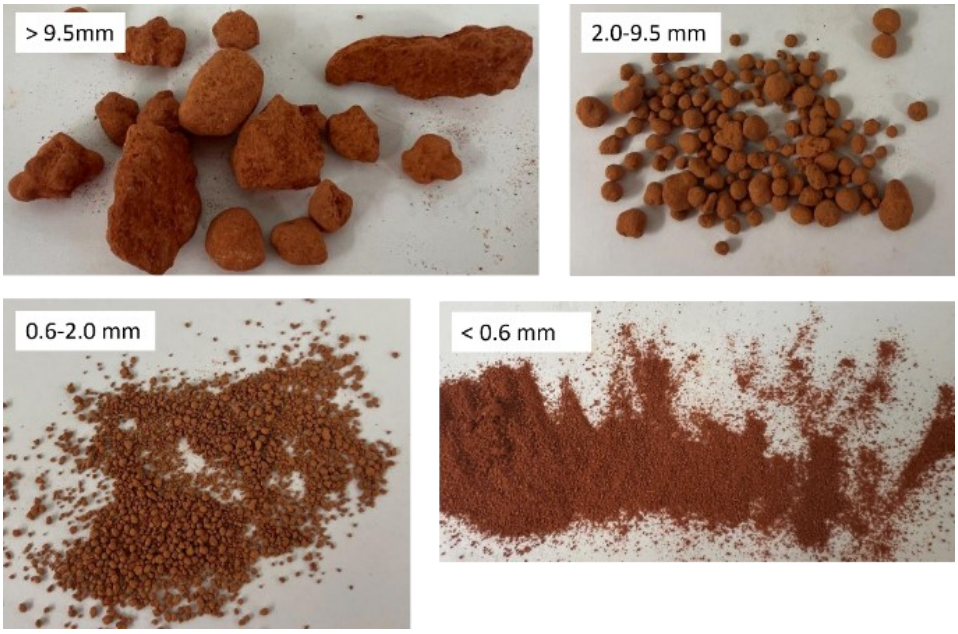


**Figure 1. Photos of Weipa bauxite pisolitic size fractions.**

The influence of bauxite PSD on TOC is shown in Figure 2.

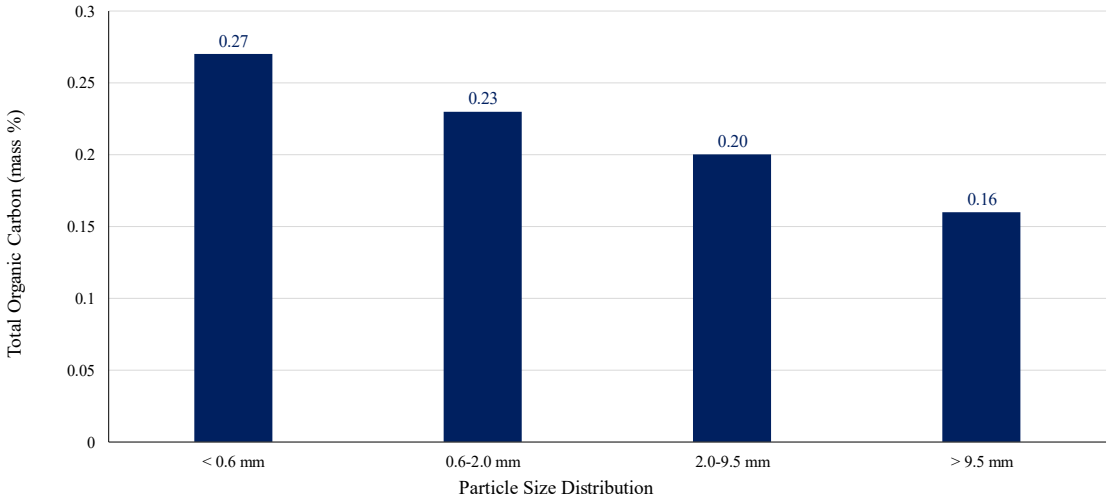


**Figure 2. Influence of bauxite PSD on bauxite TOC.**

As shown in Figure 2, smaller bauxite particles exhibit higher TOC values. This is consistent with the deposition of younger organics (dependent on seasonal rainfall) on the outer perimeter of pisolites and regulated by surface area to weight ratio [16, 17]. These external oxygenated organics represent the bulk of measured TOC (especially those susceptible to Bayer extraction).

While the low TOC (< 0.3 %) of pisolitic bauxite presents analytical challenges, LIBS can identify elemental carbon using a colour coded “heat” map at these low concentrations. A cross-section of four nodule particles exhibiting engulfed pisolites is shown in Figure 3.

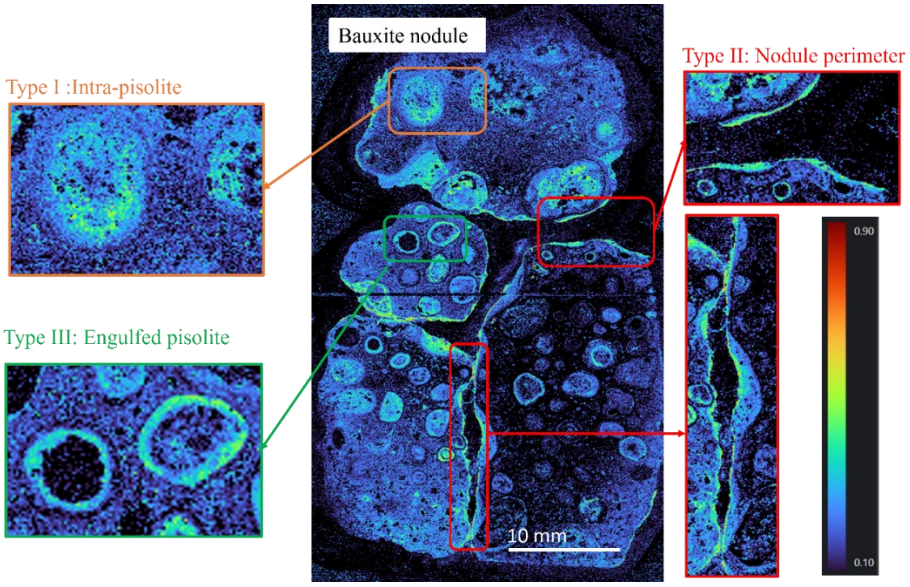


**Figure 3. LIBS of bauxite nodules showing spatial carbon distribution.**

While LIBS examines two-dimensional spatial distribution of elemental carbon, it does not distinguish between organic (oxygenated or aromatic) and inorganic (mineral carbonates) carbon. Approximately 90 % of Weipa pisolitic carbon contaminants are organic and 10 % carbonate [3]. Time of Flight Simulation (ToF SIMS) can be used to examine the chemical make-up of bauxite organics within a specific spatial domain and is shown in Figure 4.

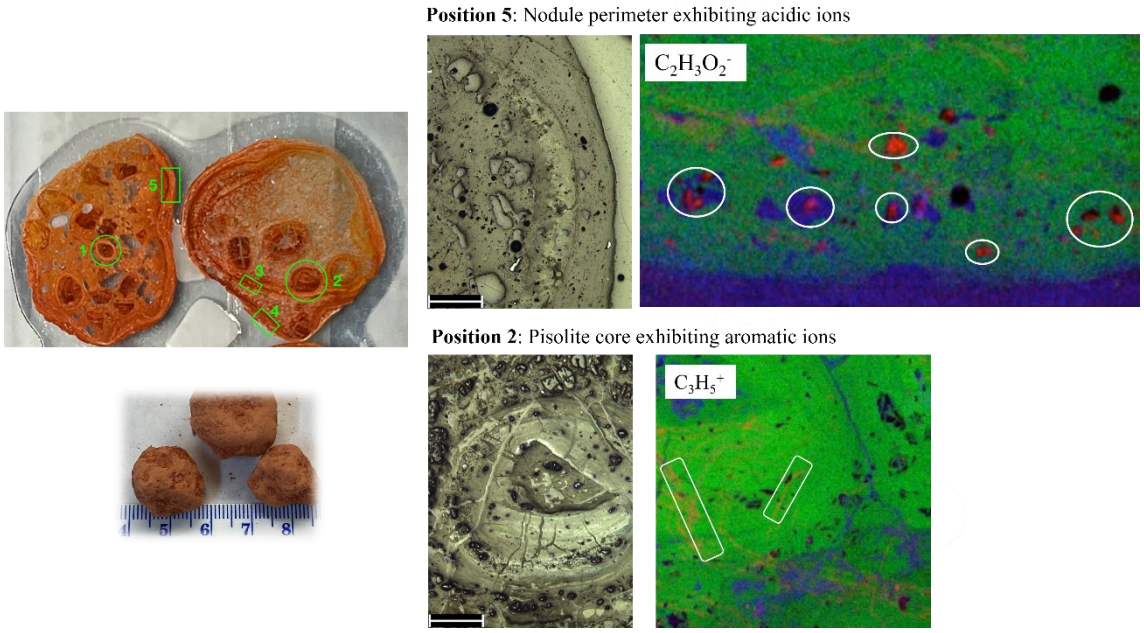


**Figure 4. ToF SIMS of bauxite nodule indicating aromatic (Position 2) and oxygenated (Position 5) organic carbon.**

Using evidence shown in Figures 3 and 4, three organic carbon typologies are distinguished:

- **Type I**: The carbon appears as a finite dispersion within mineral particles located within the core of a pisolite. ToF-SIMS indicates the presence of predominantly "older" aromatic carbon [4, 18], substantiated by evidence in Figure 4, Position 2.
- **Type II**: The carbon appears arranged on the outer perimeter of independent nodules. This represents the majority of younger organic carbon deposited through rainwater

percolation [5, 7, 11] through the overburden and through the bauxite deposit. When correlating this spatial distribution on the outer nodule cortex in Figure 4, Position 5, the organic ion fragments are primarily acidic, indicating the presence of oxygenated organic functions (primarily hydroxyl and carboxylic acids).

- **Type III**: The carbon appears arranged as thin concentric rings representing original organic deposition on the outer perimeter of a pisolite. However, it is further evident (that during the bauxitization process) this pisolite was incorporated within the core of a larger nodule [4] over a geological time "period" (a geological period is defined between 10 and 100 million years). Although this concentric organic ring was originally Type II, over this period, organics reverted towards a more aromatic chemical make-up [4], akin with Type I organics.

Although they represent a minor fraction of the overall bauxite TOC, older Type I and III organics exhibit an aromatic character. As their original organic oxygen functionalities are depleted, they are less susceptible to Bayer extraction [6] and subsequent in-situ oxalate formation. In contrast, Type II represents the major bauxite fraction TOC and is adsorbed on the cortex perimeter. These organics are oxygenated biopolymers and readily extracted within the Bayer liquor, promoting molecular decay and oxalate formation [7, 10, 14].

### 3.2 Low-Temperature Thermal Treatment of Pisolitic Bauxite

Low-temperature thermal treatment of pisolitic bauxite (275 and 350 °C) was conducted to establish the potential to "denature" Type II organics (at lower comparative temperatures to bauxite activation) without the requirement to "eliminate" them (600 °C) as previously described [15]. It was further examined if this impact could be translated to the Bayer process value chain.

Thermo-gravimetric analysis (TGA)/Quasi multi-ion detection (QMID) was conducted to examine the influence of thermal treatment on the evolution of specific molecular weight ions namely water ($H_2O$; molecular mass 18) as shown in Figure 5 and carbon dioxide ($CO_2$; molecular mass 44) in Figure 6.

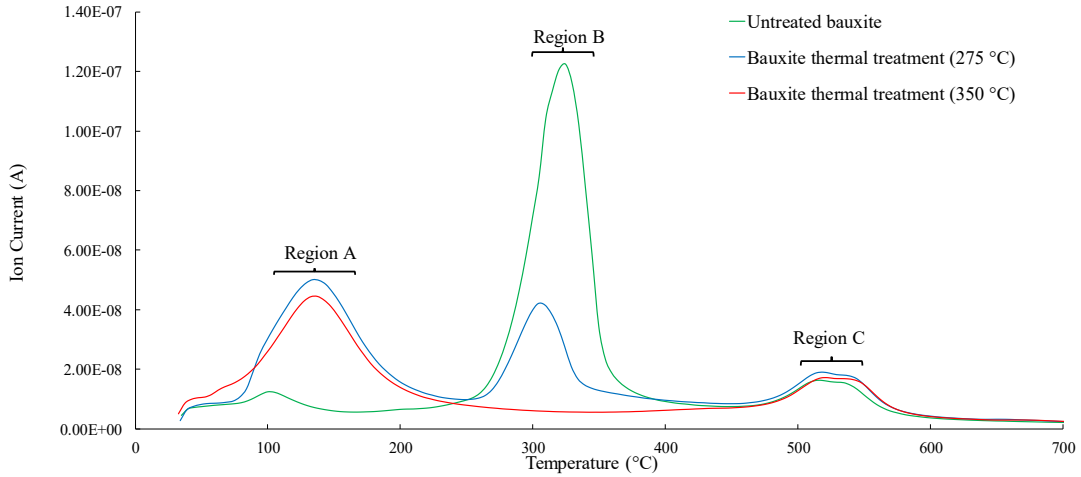


**Figure 5. TGA/QMID examining the influence of thermal treatment on $H_20$ (m18) evolution.**

Bauxite dehydration occurs over three temperature regions:

- **Region A** (100–140 °C): Evaporation of physically bound water
- **Region B** (280–340 °C): Gibbsite [$Al(OH)_3$] dehydroxylation (evolution of three water of hydration or chemically bound water molecules)

- **Region C** (500–550 °C): Boehmite [AlO(OH)] and Kaolinite [$Al_2Si_2O_5(OH)_4$] dehydroxylation (evolution of one water of hydration or chemically bound water molecule)

Both thermal treated bauxite samples indicate physical (free) water (produced during gibbsite dehydroxylation reactions during thermal treatment) in Region A. Within Region B, gibbsitic dehydroxylation during thermal treatment at 350 °C appears to be complete whereas at the lower temperature of 275 °C it is only partial. Some of the gibbsite may revert to boehmite under these conditions so the water balance may not be complete. Region C comprises the evolution of chemically bound moisture (boehmitic and kaolinitic), thus as none of the samples were exposed to high enough temperatures, these peaks appear intact over the three samples.

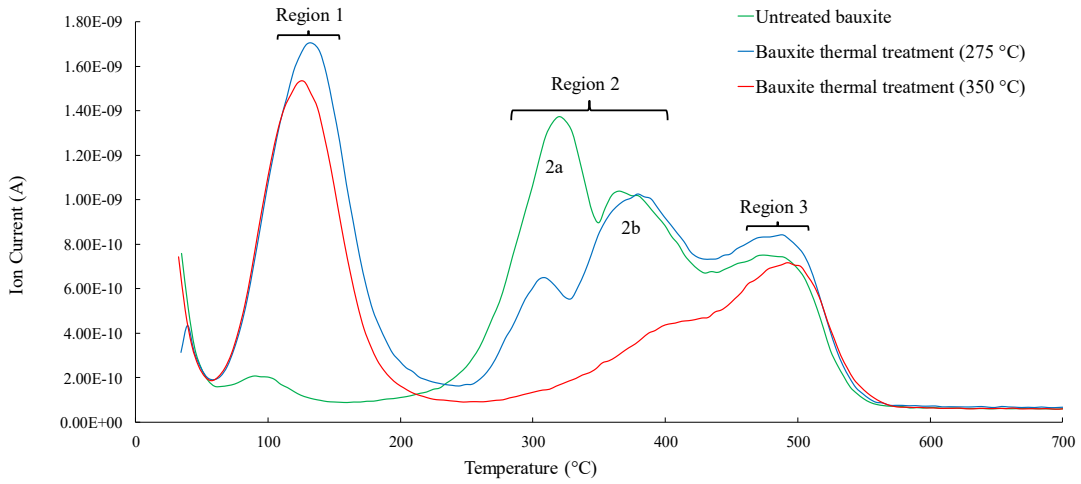


**Figure 6. TGA/QMID examining the influence of thermal treatment on $CO_2$ (m44) evolution.**

As mentioned, a challenge associated with the study of bauxite organics is the comparatively low TOC concentration (< 0.3 %). However, using TGA/QMID it is possible to study $CO_2$ evolution from bauxite at low organics concentrations as a function of chemical nature (measured by thermal stability in an oxidative atmosphere). In Figure 5, the bauxite comprises around 20 % water by weight (thus the y-axis is indicated as multiples of $10^{-8}$). By comparison, $CO_2$ evolution (representing the organic carbon mass) is measured by two orders of magnitude less (than water evolution), on a smaller scale (Figure 6) of $10^{-10}$.

The chemical makeup of thermal organics denaturing may be measured as a function of oxidative stability (organic combustion) and presents in three regions, shown in Figure 6:

- **Region 1** (110–140 °C): Although $CO_2$ evolution within this region represents a false peak, examining it is important and the rationale underpinning the examination of moisture in Figure 5. Bauxite thermal treatment produces $CO_2$ which dissolves in free water (from gibbsite dehydroxylation) trapped within closed pores, as there is excess water [19]. However, as noted in Figure 5, when the TGA temperature exceeds 100 °C, free water is evaporated followed by gaseous $CO_2$ evolution.
- **Region 2** (280–380 °C): This broad region comprises an overlapping doublet of $CO_2$ evolution peaks (indicated in Figure 6 as 2a and 2b). Compared to the untreated bauxite, thermally treated bauxite (275 °C) shows a reversal of peak intensities between 2a and 2b, indicating a slight increase in thermal stability. Thermal treatment at 350 °C eliminates peak 2a and reduces peak 2b, migrating toward Region 3. The evolution of this peak morphology due to organics is consistent with previous work [20, 21].

- **Region 3** (480–520 °C): This region is associated with the oxidation of thermally stable poly-aromatic hydrocarbons [18, 22, 23] requiring higher activation energies, for which thermal treatment (350 °C) is the major peak.

Incremental thermal treatment (untreated → 275 °C → 350 °C) migrates $CO_2$ evolution peaks to higher TGA temperatures indicating increased thermal stability to oxidation. This corresponds with the gradual depletion of molecular hydrogen and oxygen (characteristic of organic oxygenates) and development of poly-aromatic carbon [22, 24, 25] with a higher thermal stability.

The influence of bauxite thermal treatment on elemental oxide composition is given in Table 1.

**Table 1. Influence of thermal treatment on bauxite mineral composition.**

| Analysis | Unit | Untreated bauxite | Bauxite thermal treatment (275 °C) | Bauxite thermal treatment (350 °C) |
|---|---|---|---|---|
| $Al_2O_3$ | % | 54.79 | 60.28 | 63.17 |
| $SiO_2$ | % | 8.36 | 9.23 | 9.60 |
| $Fe_2O_3$ | % | 11.09 | 12.63 | 14.10 |
| $TiO_2$ | % | 2.77 | 3.03 | 3.19 |
| Loss on Ignition (LOI) | % | 22.7 | 14.5 | 9.6 |
| Total Available Alumina (TAA) | % | 50.4 | 56.7 | 60.1 |

The major influence of thermal treatment is chemical moisture loss, increasing elemental oxide composition by a similar multiple. Thermally treated bauxite samples were digested in caustic liquor in a laboratory Bayer process. Photos of Bayer liquor from untreated and thermally treated (350 °C) bauxite are shown in Figure 7.

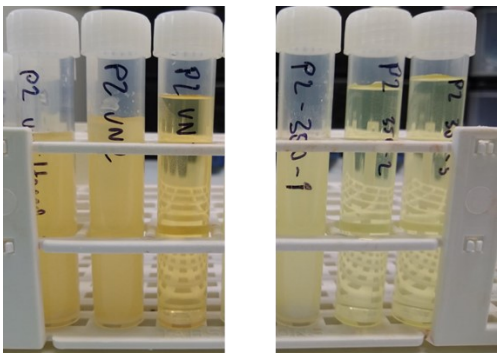

**Figure 7. Photos of Bayer liquor charged with untreated (left) and thermally treated (350 °C) bauxite (right).**

Bayer soluble organics usually impart a yellow brown colour to Bayer liquor [7], which deepens with increasing TOC concentration. This colour is largely associated with the presence of Bayer extractable humates. As shown in Figure 7, the untreated bauxite (on the left) imparts a deeper yellow colour to the Bayer liquor compared to bauxite thermally treated at 350 °C. This visual comparison indicates less extractable organics and serves as a preamble to describe the impact of denatured bauxite organics within the Bayer process. This impact on Bayer TOC solubility and oxalate production is given in Table 2.

**Table 2. Influence of thermal treatment on bauxite TOC, Bayer liquor TOC and sodium oxalate.**

| Analysis | Unit | Untreated bauxite | Bauxite thermal treatment (275 °C) | Bauxite thermal treatment (350 °C) |
|---|---|---|---|---|
| Bauxite TOC | % | 0.19 | 0.17 | 0.11 |
| Liquor TOC | g/L | 0.31 | 0.18 | 0.12 |
| Sodium oxalate | g/L | 0.42 | 0.37 | 0.06 |

Bauxite thermal treatment (275 °C) exhibits minimal impact on bauxite TOC, yet a slightly greater impact on liquor TOC. The comparative influence on sodium oxalate formation is minor. This agrees with previous work [9] indicating transformation of organic hydroxyl functions to carboxylic acids (below 300 °C) but overall retaining an oxygenated character.

Thermal treatment (350 °C) partially eliminates bauxite organics retaining denatured organics exhibiting a condensed poly-aromatic character. Subsequently, when comparing thermally treated (350 °C) and untreated fractions, the bauxite TOC reduces by 42 %, liquor TOC by 62 % and sodium oxalate by 86 %. Caustic decay of bauxite organics within the Bayer process is governed by hydrogen transfer reactions [10, 14] facilitated by the presence of oxygenated organic molecules. However, reduced molecular oxygen functionalities inhibit Bayer deportment, retarding molecular decay and subsequent oxalate production. This (thermal treatment at 350 °C) impact of denatured organics is not linear through the Bayer process value chain (bauxite TOC → liquor TOC → oxalate) but rather compounded. The impact of denatured organics through the Bayer process value chain is compounded by decreasing the bauxite TOC by 42 %, decreasing the Bayer liquor TOC by 62 % and sodium oxalate production by 86 %, due to changes in deportment pathways compared to untreated bauxite organics.

A minor fraction of oxalate (within Bayer liquor) has been reported to stem from the bauxite itself [7]. This reported natural oxalate fraction (within bauxite) may stem from vegetative decay as discussed. However, geological carbon dioxide reduction (via a carbonite intermediate [27]) may additionally contribute to the production of mineral oxalates during the bauxitization process.

### 3.3 Liquid Chromatography Mass Spectroscopy (LCMS) of Bayer Liquors

A review of the complex suite of Bayer organic intermediates using advanced analytical techniques within Bayer liquors was performed by Greg Power and coworkers in 2011 [7]. Advances in data analytics have since progressed the scientific value thereof. A pioneering multi-dimensional technique, namely LCMS-MS, was first used by Picard and co-workers in 2002 [28]. Given the complexity associated with Bayer organics, a simplified variant thereof using LCMS (in the negative mode) was used to compare “fingerprint” traces of Bayer organic anions. This was conducted to establish a potential link between denatured bauxite organics and the suite of organic intermediates presenting in the Bayer liquor. The rationale underpinning this approach was the sequential caustic decay of larger organic molecules producing smaller molecules within Bayer liquor. Should this decay value chain be impeded (reducing the production of stable LMW molecules, including oxalate) it could be linked to bauxite thermal treatment. An overlay of LCMS “fingerprint” traces between untreated and thermally treated (350 °C) bauxite in Bayer liquor are shown in Figure 8. Although Bayer molecules generally elute according to molecular size, their inherent anionic polarity may influence elution patterns and thus only level of accuracy akin to “fingerprint” traces are currently considered.

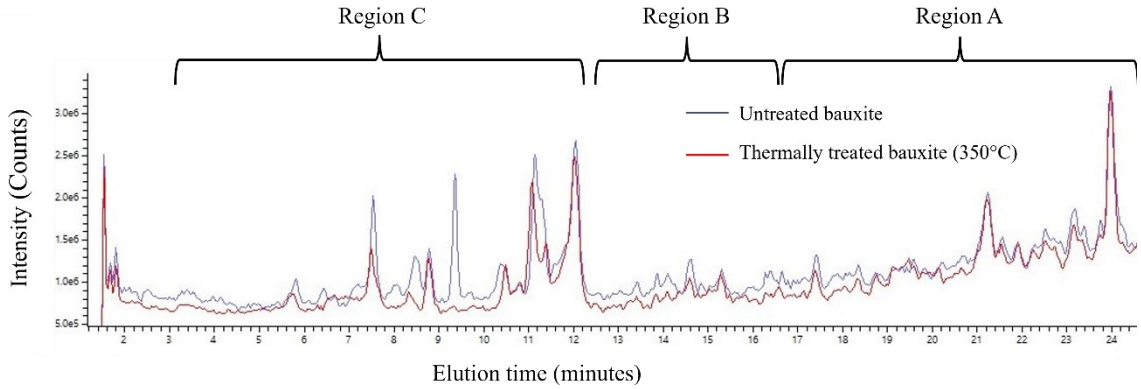


**Figure 8. LCMS "fingerprint" trace of Bayer LMW Bayer organics.**

Only untreated and thermally treated bauxite (350 °C) Bayer liquor traces are presented in Figure 8 to increase the clarity of comparison:

- Elution of larger molecules within Region A (19–25 minutes) are of a slightly lower intensity for thermally treated bauxite compared to those stemming from untreated bauxite, as may be expected based on the results in Table 2
- Within Region B (13–19 minutes) as molecular decay progresses, the difference in peak intensities increases slightly
- Within Region C (2–12 minutes) some comparative peaks are largely or completely eliminated. As thermal treatment (350 °C) depletes LMW molecules of suitable precursors, the formation of stable carboxylates (oxalate and formate) eluting before 2 minutes is retarded

Denatured organics retard the caustic decay of larger molecules as simply demonstrated in Figure 8. Advanced LCMS comparisons are currently limited as the methodology is still in development.

### 3.4 Conclusions and Further Work

The rationale underpinning this research was to present novel evidence regarding the spatial distribution and nature of bauxite organics. This was used to design a simple experiment and demonstrate the impact of low-temperature thermal treatment (275 and 350 °C) on bauxite organics and translate this to impacts within the Bayer process value chain.

The following conclusions are drawn:

- Older occluded organics within the core of a bauxite (occluded over a geological period) exhibit an aromatic character and would be less susceptible to caustic Bayer extraction/oxalate production. However, these carbons may include a small amount of mineral carbonates
- Younger organics deposited on the outer perimeter of individual pisolites/nodules are oxygenated readily promoting Bayer deportment and oxalate production
- Pisolitic bauxite TOC increases with diminishing particle size based on a higher surface area to weight ratio
- TGA/QMID offers a method to study the oxidative thermal stability as it relates to changing chemical composition and the impact of the Bayer process
- Low-temperature thermal treatment (350 °C) does not completely eliminate but rather denatures organics with a compounding impact through the remaining Bayer process value chain. Although similar work has previously been conducted to show the numerical influence of heat treatment at similar temperatures [29], this is the first

research to provide a comprehensive scientific explanation of alternation to the nature of the organic content

- An overlay of rudimentary LCMS "fingerprint" traces provides a simple method to compare larger scale changes associated bauxite organics denaturing within the suite of Bayer organic intermediates

This research presents a practical approach to study and demonstrate the impact of denaturing bauxite organics through the Bayer value chain (from bauxite TOC to oxalate). As this research is relatively novel, it naturally invites further detailed investigation. Further work may include enhanced analytical methods to characterize bauxite TOC. This may be supplemented by further developing practical Bayer in-situ analysis techniques (for example FTIR and LCMS) to better understand denatured organics and the impact on caustic decay mechanisms. Future research should ultimately promote practical solutions to mitigate the impacts of organics within an alumina refinery.

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