

## The Quality of Bauxites from Bosnia & Herzegovina and Montenegro Processed by the Alumina DOO Zvornik between 2014 and 2018

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### Abstract

The majority of bauxite used as raw material in Alumina DOO Zvornik refinery originates from Bosnia and Herzegovina (B&H) and from Montenegro. Over the past 5 years, about 4 million tonnes of bauxite from B&H and Montenegro have been processed. Bauxites mostly come from mines in the areas of Milići, Srebrenica, Jajce, Mrkonjić Grad, Široki Brijeg, Nikšić and Posušje. Mineralogical analyses confirm that bauxite from B&H and Montenegro are mainly boehmitic. Some mixed gibbsitic-boehmitic bauxites are present in the Herzegovina region, and diasporic-boehmitic bauxite is present in the Jajce and Nikšić mines. By chemical composition, the weight % of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, CaO and ZnO are the most important for processing bauxite in Alumina DOO using the Bayer process. Characterisation of bauxite from these sites by the Alumina DOO Laboratory have confirmed their commercial viability, especially for the production of alumina and aluminum hydrate for non-metallurgical purposes, and for the production of various types of zeolites.

**Keywords:** Bauxite, Bayer, Chemical Analysis, Mineralogical Analysis.

### 1. Bauxite

From the beginning of alumina production in Zvornik (in 1978), local mines were the main source of bauxite ore. Initially, the geographically closest mines, Milići (Vlasenica) and Srebrenica provided ~95% of refinery needs. The closure of alumina refineries in ex-Yugoslavia (Podgorica, Obrovac, Kidričevo and Mostar) and in Hungary and Romania, has resulted in Alumina DOO has become the only user in this region. Over the past 5 years (2014-2018) about 4 million tonnes of bauxite from Bosnia and Herzegovina (B&H) and Montenegro (MN) has been processed. Other than bauxites from mines in Milići and Srebrenica, Alumina DOO used bauxite from mines in the areas of Jajce, Mrkonjić Grad, Široki Brijeg, Nikšić (MN) and Posušje. Figure 1 shows the approximate geographical location of these bauxite mines.

#### 1.1. Geology of B&H and Montenegrin Bauxites

Bauxite is quite widespread in B&H and Montenegro. Deposits in B&H are located in western and eastern Herzegovina (reserves of about 80 million tonnes), central Bosnia surrounding Jajce and Mrkonjić Grad, western Bosnia in Bosanska Krupa and Mountain Grmeč (reserves of about 20 million tonnes), and eastern Bosnia, between Srebrenica and Milići (reserves of about 50 million tonnes) [1]. In Herzegovina, the economically most important bauxite deposits are from the Eocene age lying in Karst depressions of upper Cretaceous limestone (floor), and the roof is represented by different lithological elements of the older Eocene [2]. Bauxites of central Bosnia in Ljuša, Liskovica, Poljane, Bešpelj, and Crvena Stijena are from different ages (Triassic, Jurassic, and Cretaceous). Bauxites of western Bosnia are also from different ages (Cretaceous

and Jurassic), while bauxites of eastern Bosnia between Vlasenica and Srebrenica (Braćan, Šumarnica and Kosturi) are from the upper Cretaceous.

According to the stratigraphic nomenclature, bauxites in Montenegro are classified as Triassic, Jurassic, Cretaceous and Eocene. The red karst bauxites of Montenegro are found in carbonate rocks of Mesozoic and Paleocene (reserves of about 130 million tonnes) [1], [3].

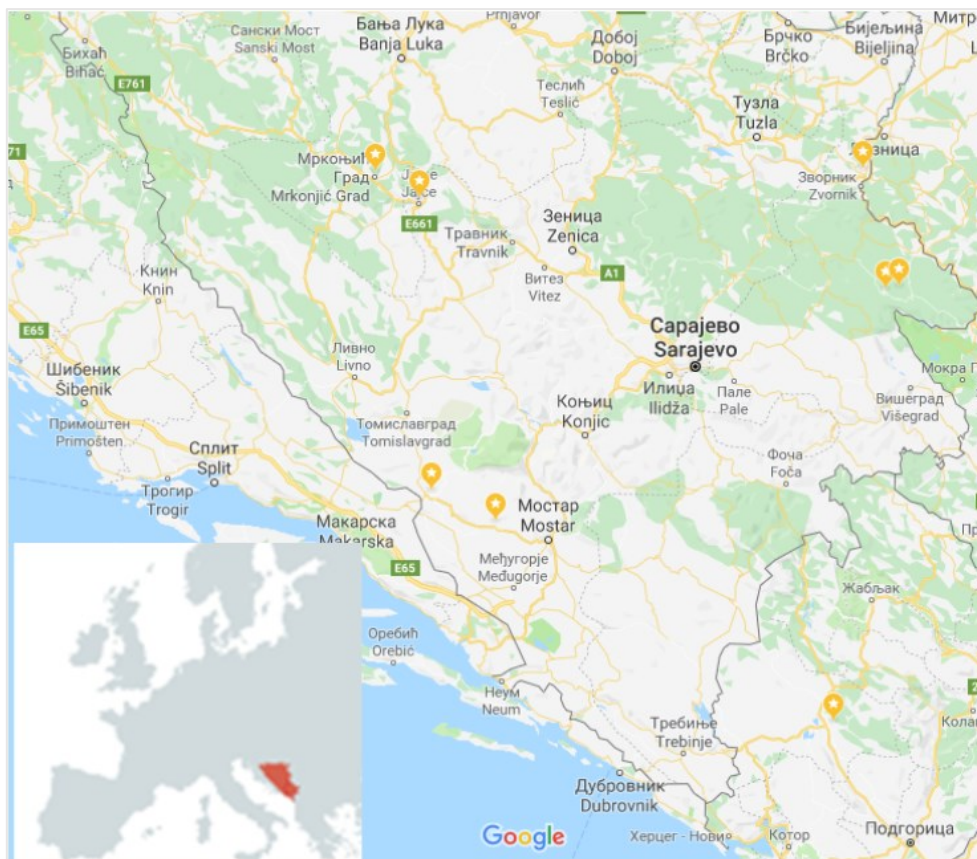
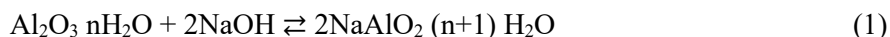


Figure 1. Approximate geographical position of Alumina DOO Zvornik and bauxite mines (Google Maps).

## 1.2. Digestion of Bauxite in Alumina DOO

Of the existing processes for the production of alumina, the most dominant is the hydro-metallurgical Bayer process, which was implemented in Alumina Zvornik in 1978. The process can essentially be represented by the following reaction:



During bauxite digestion, the equilibrium of the above reaction is moved from left to right, and during decomposition (precipitation) of the Bayer liquor, the equilibrium is moved in the opposite direction.

Raw Bauxite is shipped by trucks and railway to the refinery. It is unloaded at the primary bauxite storage area in stockpiles dedicated to a mining location. Since all the bauxites that arrive at the refinery's Mineral Preparation facility differ in quality, in terms of A/S (alumina to silica ratio - wt%  $\text{Al}_2\text{O}_3/\text{SiO}_2$ ), mineralogical composition (boehmite, diaspor, and gibbsite), chemical composition, granulometry and moisture, they have to be homogenised to produce a consistent

feed to the refinery. Homogenisation is done according to the A/S, chemical composition and granulometry, to ensure processing efficiency in the Bayer process. Regardless of the type of transport used for shipping, all material is sampled from the transport units (for each 1000 t about 80 kg of sample) and sent to the Quality Control lab, which determines the type and quality of the bauxite ore. Analyses of the chemical and mineralogical composition of the bauxite arriving at the plant are primarily used for checking the quality and for calculating how bauxite from different mining locations are mixed.

Most often raw bauxite comes to the plant as mined, without any pretreatment. In this case, if needed, the bauxite is sent to primary crushing (using conical crushers) and then to secondary crushing (hammer crushers), with classification of the bauxite particle size to the below the specified top size, and recycling of the oversize to the crushers. After the crushing and classifying of the raw bauxite, it is stored in a dedicated location prior to homogenisation.

Primary homogenisation of the bauxite is done by mixing various qualities of bauxite, with different alumina to silica ratios and contents of ZnO and CaO, from different mining locations, in defined quantities. Determination of the quantities for bauxite mixing is carried out firstly by defining the target of A/S, and concentrations of ZnO and CaO in the homogenised bauxite which will give stable process conditions and ultimately products to meet our customers' needs. Target A/S at the stockpiles is ~12 and target w% ZnO is ~0.015.

Secondary homogenisation is carried out by building stockpiles using "stackers", which are mobile and can create stockpiles of different sizes. Small conical lines are formed for the length of the stockpile. After one line is done, the next is laid down beside the first in the same way. After the entire width of the stockpile is filled, the next row is placed over/between the previous two lower ones until total height is reached. During the building of the stockpile, samples are taken continuously and are composited into one shift sample for analysis.

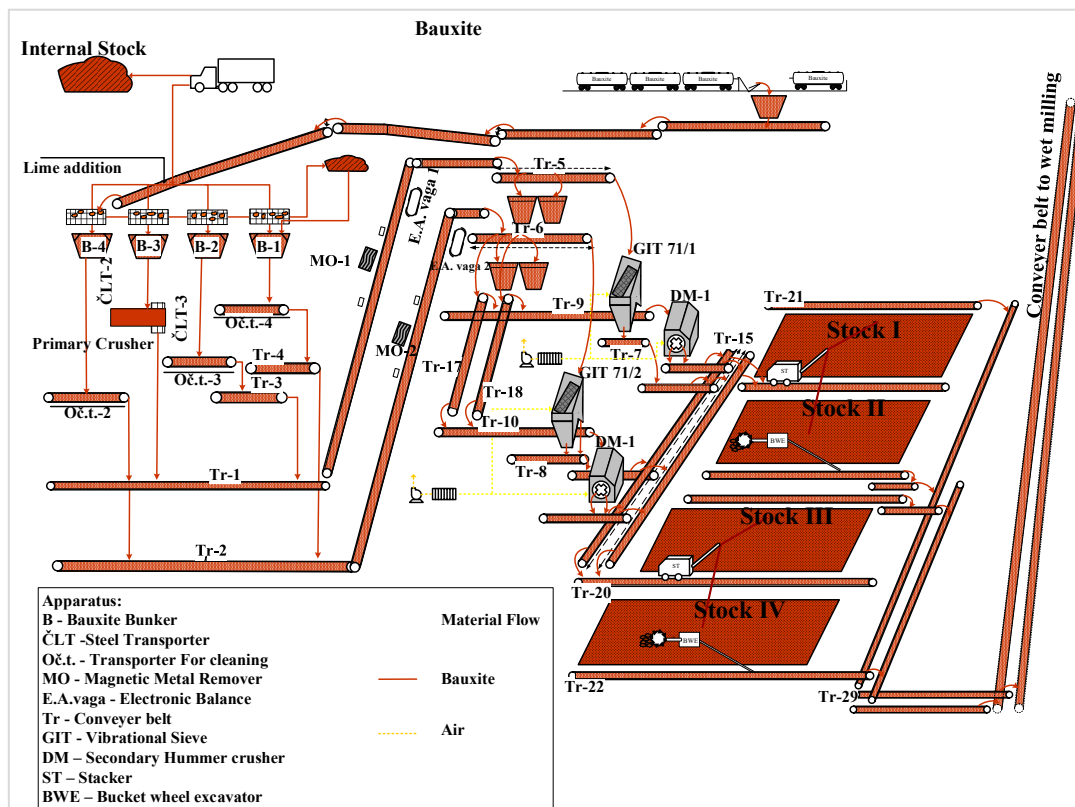


Figure 2. Overview of Bauxite Crushing and Homogenisation in Alumina DOO.

Recovery of the homogenised bauxite from the formed stockpiles is done by Bucket Wheel Excavator (a rotating drum with fixed buckets), and in doing so, a second level of homogenisation is achieved. The more the raw bauxite is mixed, the more homogenous the refinery feed material, but it also increases costs and complexity. For the purposes of stable digestion feed composition, the two steps of homogenisation practiced at Alumina DOO is sufficiently satisfactory. All preparation steps are presented in Figure 2.

## 2. Experimental Methods and Materials

Sampling [4], Preparation [5] and sample analysis were performed by the employees of the Quality Control sector in Alumina DOO.

### 2.1. Chemical Analysis

All chemicals used for the analysis are of Analytical Reagent (AR) grade, and standardised laboratory glassware is provided from the central laboratory of Alumina DOO.

#### 2.1.1. Determination of Al<sub>2</sub>O<sub>3</sub> Content

The Al<sub>2</sub>O<sub>3</sub> method [6] is based on the potentiometric determination of Al<sub>2</sub>O<sub>3</sub> content at pH 5 with EDTA titration by automatic burette Tacussel EBX.

#### 2.1.2. Determination of SiO<sub>2</sub> Content

The SiO<sub>2</sub> method [7] is based on the spectrophotometric determination of the color intensity of silicon-molybdate complex formed under strictly defined ranges of pH, reagent concentration, temperature and time. All measurements were done by Shimadzu UV1800 spectrophotometer.

#### 2.1.3. Determination of CaO and ZnO Content

The method [8] is based on atomic adsorption spectrophotometric (AAS) determination. Calibration samples are prepared in a matrix very similar to the bauxite by addition of known amounts of Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>. Samples are prepared by diffusion in 3:1 Na<sub>2</sub>CO<sub>3</sub>:Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> mix and analysed by Shimadzu AA7000 Spectrophotometer.

### 2.2. Mineralogical Analysis

The powder XRD data were collected by Bruker Endeavor D8 Diffractometer with these characteristics:

**Table 1. Setup and characteristics of the XRD system.**

|                  |   |
|------------------|---|
| Goniometer       | D8 ENDEAVOR Theta/Theta                                 |
| Radius           | 401mm   |
| Lamp             | 1.8 kW Co long fine focus K $\alpha$ =1.78897Å          |
|                  | 35 kV / 40 mA   |
| Primary optics   | Motorized divergence slits 0.3°<br>4° Soller slits      |
| Sample holder    | Standard rotation stage,<br>Motorized Air-Scatter Knife |
| Secondary optics | 4° Soller slits   |
| Detector         | LYNXEYE-XET, 4.1° opening                               |

The samples were hand pelleted and scanned in the range 5-90 2 $\theta$  (d=1.26-10). Software used for the integration of mineralogical phases is DIFFRAC.EVA with the help of COD (Crystallography Open Database). Table 2 shows the most intense d values and COD codes for the identified phases in the examined samples [9]:

**Table 2. Characteristic d values of known mineralogical forms.**

|                 |                     |                     |                     |                           |                     |
|-----------------|---------------------|---------------------|---------------------|---------------------------|---------------------|
| <b>Mineral</b>  | Gibbsite            | Boehmite            | Diaspore            | Hematite                  | Calcite             |
| <b>COD code</b> | 9008237             | 9012274             | 9005763             | 9015964                   | 9000095             |
| <b>d values</b> | 4.85; 4.36;<br>4.32 | 6.11; 3.16;<br>2.35 | 3.99; 2.31;<br>2.13 | 2.69; 1.69;<br>3.68; 2.51 | 3.03; 1.87;<br>3.85 |
| <b>Mineral</b>  | Kaolinite           | Quartz              | Anatase             | Rutile                    | -                   |
| <b>COD code</b> | 9009230             | 1011097             | 5000223             | 9007432                   | -                   |
| <b>d values</b> | 7.16; 3.56;<br>4.41 | 3.34; 4.26;<br>1.82 | 3.52; 1.89;<br>2.38 | 3.25; 1.69;<br>2.49       | -                   |

### 3. Results

The average annual w% concentrations of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, CaO and ZnO for the samples of bauxite, for the five-year period 2014 - 2018 are presented below. During this total period about 3 900 samples were prepared and analysed. Additionally, mineralogical analysis of typical samples of bauxite from the mines in Nikšić, Jajce, Mrkonjić Grad, Posušje, Široki Brijeg, Srebrenica and Milići were carried out.

#### 3.1. Chemical Results

Average annual w% concentrations of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, CaO and ZnO, along with production volumes per year from all mines are shown in the Table 3.

**Table 3. Chemical analysis of bauxite samples.**

| Bauxite source | Parameter                          | Year    |         |         |         |         | Total     |
|----------------|------------------------------------|---------|---------|---------|---------|---------|-----------|
|                |                                    | 2014    | 2015    | 2016    | 2017    | 2018    |           |
| Milići         | <i>Amount (t)</i>                  | 340 190 | 408 320 | 361 229 | 419 042 | 425 553 | 1 954 333 |
|                | Al <sub>2</sub> O <sub>3</sub> (%) | 53.60   | 54.21   | 53.80   | 53.98   | 53.44   | 53.81     |
|                | SiO <sub>2</sub> (%)               | 5.13    | 5.07    | 4.94    | 4.89    | 5.55    | 5.12      |
|                | CaO (%)                            | 0.32    | 0.43    | 0.54    | 0.82    | 0.85    | 0.61      |
|                | ZnO (%)                            | 0.015   | 0.009   | 0.012   | 0.012   | 0.013   | 0.012     |
|                | A/S                                | 10.45   | 10.69   | 10.89   | 11.04   | 9.63    | 10.51     |
| Srebrenica     | <i>Amount (t)</i>                  | 115 879 | 143 691 | 124 726 | 43 474  | 125 458 | 553 228   |
|                | Al <sub>2</sub> O <sub>3</sub> (%) | 54.37   | 55.23   | 54.20   | 53.85   | 53.11   | 54.23     |
|                | SiO <sub>2</sub> (%)               | 5.39    | 5.77    | 6.71    | 7.14    | 7.84    | 6.48      |
|                | CaO (%)                            | 0.19    | 0.21    | 0.15    | 0.28    | 0.19    | 0.19      |
|                | ZnO (%)                            | 0.010   | 0.009   | 0.012   | 0.007   | 0.009   | 0.010     |
|                | A/S                                | 10.09   | 9.57    | 8.07    | 7.55    | 6.77    | 8.37      |
| Posušje        | <i>Amount (t)</i>                  | 38 937  | 86 504  | 67 836  | 86 355  | 92 757  | 372 389   |
|                | Al <sub>2</sub> O <sub>3</sub> (%) | 54.06   | 54.08   | 54.38   | 55.83   | 54.43   | 54.63     |
|                | SiO <sub>2</sub> (%)               | 1.34    | 1.23    | 1.38    | 1.35    | 1.34    | 1.32      |
|                | CaO (%)                            | 0.78    | 1.24    | 1.18    | 0.93    | 0.97    | 1.04      |
|                | ZnO (%)                            | 0.033   | 0.025   | 0.041   | 0.030   | 0.039   | 0.033     |
|                | A/S                                | 40.34   | 43.97   | 39.41   | 41.24   | 40.62   | 41.23     |

| Bauxite source          | Parameter                          | Year           |                |                |                |                | Total            |
|-------------------------|------------------------------------|----------------|----------------|----------------|----------------|----------------|------------------|
|                         |                                    | 2014           | 2015           | 2016           | 2017           | 2018           |                  |
| Mrkonjić Grad           | Amount (t)                         | 9 432          | 12 569         | 7 751          | 11 784         | 3 026          | 44 562           |
|                         | Al <sub>2</sub> O <sub>3</sub> (%) | 57.86          | 57.63          | 58.27          | 57.47          | 58.05          | 57.78            |
|                         | SiO <sub>2</sub> (%)               | 1.99           | 1.84           | 1.78           | 1.65           | 2.06           | 1.83             |
|                         | CaO (%)                            | 0.92           | 1.48           | 1.18           | 1.27           | 0.89           | 1.21             |
|                         | ZnO (%)                            | 0.000          | 0.000          | 0.000          | 0.000          | 0.002          | 0.000            |
|                         | A/S                                | 29.08          | 31.32          | 32.72          | 34.84          | 28.18          | 31.64            |
| Jajce                   | Amount (t)                         | 114 041        | 118 930        | 110 206        | 105 784        | 107 385        | 556 345          |
|                         | Al <sub>2</sub> O <sub>3</sub> (%) | 55.25          | 58.86          | 58.89          | 58.15          | 58.81          | 57.98            |
|                         | SiO <sub>2</sub> (%)               | 2.23           | 2.13           | 1.73           | 1.61           | 1.88           | 1.92             |
|                         | CaO (%)                            | 1.29           | 1.10           | 0.95           | 1.03           | 1.09           | 1.09             |
|                         | ZnO (%)                            | 0.001          | 0.000          | 0.000          | 0.000          | 0.000          | 0.000            |
|                         | A/S                                | 24.78          | 27.63          | 34.06          | 36.21          | 31.28          | 30.15            |
| Široki Brijeg           | Amount (t)                         | 24 566         | 33 349         | 39 589         | 38 599         | 39 324         | 175 427          |
|                         | Al <sub>2</sub> O <sub>3</sub> (%) | 54.43          | 56.22          | 57.07          | 55.70          | 56.14          | 56.03            |
|                         | SiO <sub>2</sub> (%)               | 1.79           | 1.80           | 1.57           | 1.78           | 1.73           | 1.73             |
|                         | CaO (%)                            | 2.75           | 1.59           | 1.01           | 1.65           | 1.56           | 1.63             |
|                         | ZnO (%)                            | 0.024          | 0.018          | 0.021          | 0.024          | 0.024          | 0.022            |
|                         | A/S                                | 30.41          | 31.23          | 36.27          | 31.24          | 32.45          | 32.42            |
| Nikšić                  | Amount (t)                         | 5 118          | 51 565         | -              | 34 144         | 155 923        | 246 750          |
|                         | Al <sub>2</sub> O <sub>3</sub> (%) | 61.90          | 60.14          | -              | 59.51          | 59.26          | 59.53            |
|                         | SiO <sub>2</sub> (%)               | 1.75           | 2.03           | -              | 2.59           | 2.99           | 2.71             |
|                         | CaO (%)                            | 0.21           | 0.81           | -              | 1.20           | 1.05           | 1.00             |
|                         | ZnO (%)                            | 0.021          | 0.022          | -              | 0.031          | 0.036          | 0.032            |
|                         | A/S                                | 35.37          | 29.63          | -              | 22.98          | 19.82          | 21.98            |
| <b>Total Amount (t)</b> |                                    | <b>648 162</b> | <b>854 927</b> | <b>711 337</b> | <b>739 183</b> | <b>949 424</b> | <b>3 903 034</b> |

### 3.2. Mineralogical Results

Mineralogical results are from the analysis of 10 representative samples from each mine in 2018. COD codes of target minerals are given in Table 2. Average, minimum and maximum mineralogical content is given in figures 4 - 10.

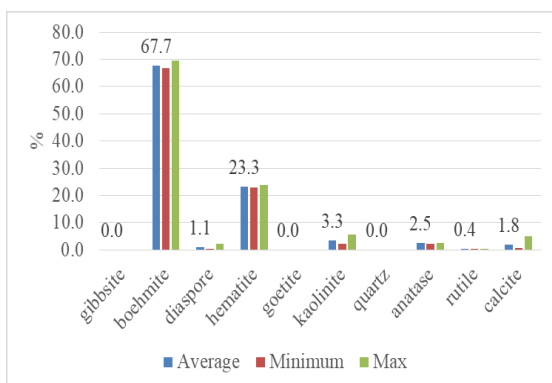


Figure 4. XRD data from Jajce mine.

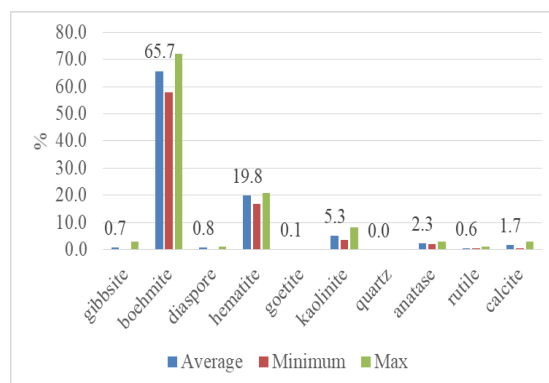


Figure 5. XRD data from Nikšić mine.

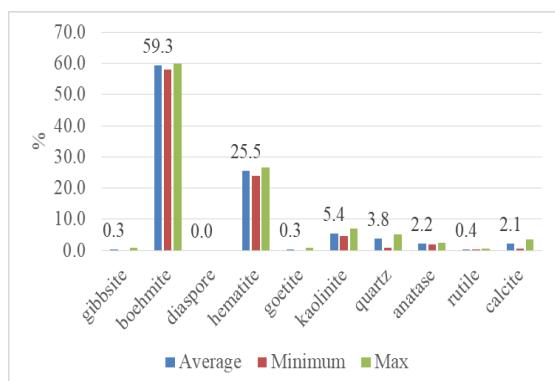


Figure 6. XRD data from Milići mine.

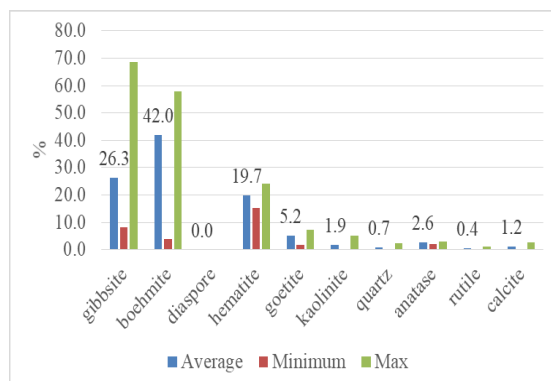


Figure 7. XRD data from Posušje mine.

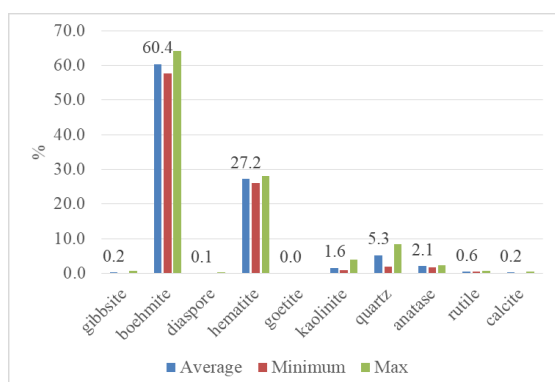


Figure 8. XRD data from Srebrenica mine.

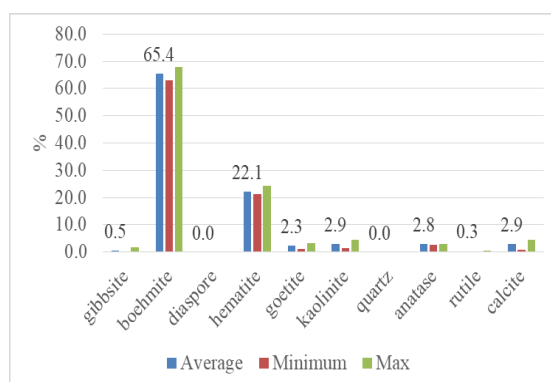


Figure 9. XRD data from Široki Brijeg mine.

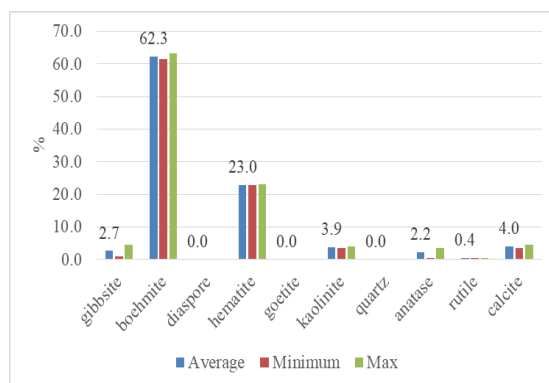


Figure 10. XRD data from Mrkonjić Grad mine.

#### 4. Discussion

The results of the chemical analyses show that target bauxites are of generally good quality. Nevertheless, it is possible to range the bauxites by quality in three groups according to:

##### 1. A/S, ie. w% $\text{Al}_2\text{O}_3/\text{SiO}_2$ :

- Average A/S > 15: bauxites of Herzegovina, Široki Brijeg, Posušje, bauxites of central Bosnia, Jajce and Mrkonjić Grad and bauxite of Montenegro, Nikšić.
- Average A/S < 12: bauxites of east Bosnia, Milići and Srebrenica.

## 2. w% concentration of CaO:

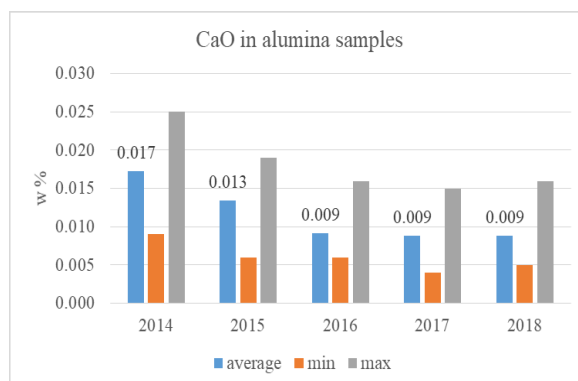
- Higher average concentration of CaO > 1 %: bauxites of Herzegovina, bauxites of west Bosnia, bauxite of Montenegro, Nikšić.
- Lower average concentration of CaO < 1 %: bauxites of east Bosnia.

## 3. w% concentration of ZnO:

- Higher average concentration of ZnO: 200 – 600 ppm, bauxites of Herzegovina, bauxites of west Bosnia, bauxite of Montenegro, Nikšić.
- lower average concentration of ZnO: < 200 ppm - bauxites of east Bosnia,
- Without the presence of ZnO: bauxites of central Bosnia.

Although the Alumina DOO refinery is designed to work most efficiently with the quality of bauxite from Milici and Srebrenica (ie. A/S 10 – 14), in the last five years, a large quantity of better-quality bauxite has been processed with the higher A/S from Nikšić, Jajce, Posušje, Široki Brijeg and Mrkonjić Grad. Consumption of NaOH per ton of bauxite significantly decreases when treating bauxite with lower % SiO<sub>2</sub> and % CaO. Although they have a lower quality in terms of A/S, bauxites from Milići and Srebrenica are better regarding w% CaO and w% ZnO, than bauxite from Nikšić, Posušje and Široki Brijeg. While the best bauxite in terms of the w% ZnO parameter are from Jajce and Mrkonjić Grad.

Analysis results of alumina samples, that had been produced out of these bauxites, for the past five years, for % SiO<sub>2</sub> (0.006 ± 0.001) and % CaO (Figure 11), doesn't show that different concentration of these analytes caused changes in the final product.

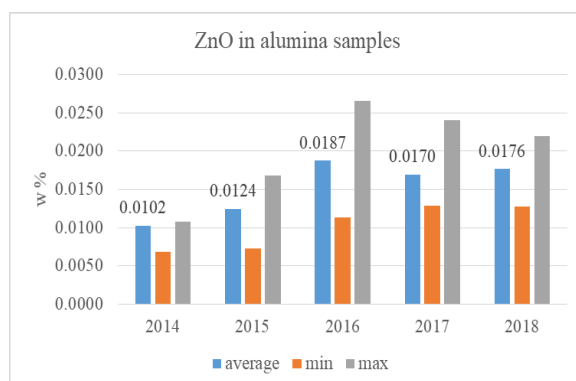


**Figure 11. Annual average, min and max values of w% CaO in alumina samples 2014 – 2018.**

Due to the strict w% ZnO specification for metallurgical alumina buyers, and the tendency to require lower ZnO as impurity in Aluminium, it is almost impossible to incorporate Herzegovinian and Montenegrin bauxites into the production of metallurgical alumina. Figure 12 shows the annual w% ZnO in alumina samples that had been produced by Alumina DOO in the period 2014 - 2018.

However, the production orientation of Alumina DOO has turned toward refractory alumina and hydrate where the requirements regarding the concentration of ZnO are significantly less. This has resulted in a significant increase in the Herzegovinian and Montenegrin bauxite contribution to production at Alumina DOO. The concentration of ZnO has accordingly increased in our alumina products. The production of the other very important group of products in Alumina DOO, zeolites (LTA, FAU, ZSM, and MOR), were not as affected as alumina products. The same aluminate solution is used for production of hydrates and various type of zeolites. ZnO

concentrations in aluminate solutions change as a consequence of ZnO in bauxite introduced. Our target for ZnO in bauxites mixes at stockpiles of < 0.015 % is sufficient to have a good quality of zeolites.



**Figure 12. Annual average, min and max values of w% ZnO in alumina samples 2014 – 2018.**

The XRD results have confirmed previous characterisations of B&H bauxites. Almost all known crystalline phases of aluminum were detected: monohydrate boehmite and diaspore and trihydrate gibbsite. The analysed bauxites are mostly of boehmitic type. Small amounts of diaspore were detected in bauxites from Jajce and Nikšić, and gibbsite in bauxites from Posušje, and in samples from Nikšić, Široki Brijeg and Mrkonjić Grad.

Silica is present in the form of kaolinite in all bauxites, additionally as quartz in samples from Milići and Srebrenica. It is important to note that a certain quantity of aluminum is present in kaolinite, but unavailable due to the precipitation of desilicatisation products (DSPs) in the Bayer Process.

Iron is present mainly as hematite, and goethite is detected in samples from Herzegovinian bauxites. Calcium is present as calcite. Refinery Alumina DOO is designed to process boehmitic bauxites where  $\text{Al}_2\text{O}_3$  is extracted at temperatures of maximum 245 °C, and a pressure range of 31 - 34 bar [10]. With existing autoclaves it is virtually impossible to extract all  $\text{Al}_2\text{O}_3$  from diaspore, and for this reason the presence of diaspore in bauxites from Jajce and Nikšić make these bauxites the least attractive in this category.

## 5. Conclusion

Bauxites of B&H and Montenegro are mostly low silica bauxites, which makes them very suitable for digestion by Bayer process in the Alumina DOO Zvornik refinery. According to the trend of quality and quantity and possible further reserves, at this point, long-term exploitation from the surveyed sites can be expected. According to the chemical properties of bauxite in B&H and MN, they can be grouped according to the A/S, w% CaO and w% ZnO, and by mineralogy into boehmite, mixed boehmite-gibbsite and mixed boehmite-diaspore ores.

The most preferred bauxites would be those with high A/S, low content of CaO and ZnO, and with no diaspore. The established mixing procedure in Alumina DOO allows suitable stockpiles feeding the refinery to be formed from available bauxites for the production of alumina and aluminum hydrate for non-metallurgical purposes and for production of various types of zeolite. With the prices of Aluminium fluctuating over the past years the strategy to produce higher value hydrate, alumina and zeolite products using optimum local bauxite blends has been central to maintaining Alumina DOO's economic future.

## 6. Reference

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