

Determination of Iron Oxide Content in Bauxites Using X-Ray Fluorescence Spectrometry by Pressing: A Comparative Study with Spectrophotometric Method

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Abstract: Bauxite is the primary ore for aluminum extraction. In order to assess the quality of bauxite, it is important to determine not only the content of Al_2O_3 but the content of Fe_2O_3 as well. Determining the composition of bauxite is very important from the aspect of determining the quality of bauxite. Therefore, it is important to use a method that is fast, accurate, and precise. In this paper the results of the comparison of two methods are presented. Bauxites of different deposits were analysed for their content of Fe_2O_3 (mass %), using the X-ray fluorescence spectrometry and reference spectrophotometric method MA. B. M.018. The samples were annealed prior to the process, and beads were prepared by pressing for the purpose of the analysis. Certified reference samples of bauxite were used for producing a calibration curve. The equation for calculating the content of Fe_2O_3 (mass %) in the samples of bauxite was derived from the calibration curve, which was obtained with the coefficient of correlation $r = 0.9989$ and the standard deviation $S = 3.4420$. The XRF method was statistically verified by the F-test and t-test (using the standard sample of the bauxite and the reference method). The values obtained from the mentioned tests showed that the XRF method was imprecise and inaccurate for determination of iron oxide in bauxite, when the samples was prepared by pressing.

Keywords: Bauxite, Iron-oxide, Pressing, Standard Method, XRF Method

1. Introduction

Bauxite is mostly an aluminous rock containing hydrated aluminum oxide as the main component along with iron oxide, silica and titania as the contaminations in varying proportions. The iron oxide in bauxite mineral is present as hematite or goethite [1].

Bauxite is the primary ore for aluminum extraction. It is treated with sodium hydroxide at above 200°C to extract alumina in the Bayer's process [2].

Bauxite residue (red mud) is a waste generated during the Bayer process of alumina production from bauxite. Major elements that are present in the bauxite residue are iron, aluminium, silicon, titanium and calcium. It also contains

some valuable but minor elements such as rare-earth elements (REEs) [3].

Determination of the chemical and mineralogical composition of bauxite of different quality, at the appropriate speed, with acceptable accuracy and precision, is of great importance in the bauxite processing industry.

In addition to determining the content of Al_2O_3 , the determination of the content of Fe_2O_3 is also of great importance in determining the quality of bauxite.

Various methods have been used for determining iron content in different types of samples, such as: diffuse reflectance spectroscopy [4, 5], inductively coupled plasma atomic emission spectroscopy (ICP-OES) [6, 7], voltammetry [8, 9], spectrophotometry [10-12] laser-induced breakdown

spectroscopy (LIBS) [13-16].

In the alumina factory "Alumina", chemical determination of Fe₂O₃ content in bauxite is carried out using the spectrophotometric method MA. B. M.018.

XRF analysis as a technique is widely used in academia, research and development and industry as an analysis tool for the determination of elemental composition of materials [17].

When a sample, used as the target, is irradiated with a source of photons or bombarded with particles of high energy, a X-ray fluorescence is most often observed. The spectrum of this photoluminescence is made up of radiations with wavelengths and intensities that are characteristic to the atoms present in the sample [18].

The quantitative estimation of an element is possible by first measuring the emitted characteristic line intensity and then relating this intensity to the elemental concentration [19].

Some examples in which the XRF technique was used are: determination of the chemical composition of different bauxites [20-23], cement raw meal [24, 25], red mud [26-28], brown fused alumina [29], analysing refractory materials [30, 31].

2. Experimental

2.1. Materials

Bauxites labelled, "Grčka", "Brazil" and "Mađarska" prepared in alumina factory "Alumina", Zvornik, BiH, were used for the experimental part of the research.

2.2. Methods and Instrumentation

2.2.1. Preparation of Pressed Samples

Bauxite samples were first ground to particle size below 200 µm and dried, and then annealed at 1075 °C. The loss on ignition was calculated at that temperature.

For the purpose of preparing pressed beads, 47.5 g of the annealed sample was separated and mixed with 2.5 g of wax and ground in a mill. The sample (10 g) prepared in this way was then transferred to the mould in the press and subjected to a pressure of 150 KN for 10 seconds. The bead obtained in this way was recorded by WDXRF "S8 TIGER" (BRUKER).

The calibration curve was obtained based on the certified reference bauxite samples, which were also annealed prior to the process, then pressed, and recorded afterwards (Table 1).

Table 1. Analysis of standard reference bauxite samples according to the certificate (mass %).

| Components | 69b NBS (Arkansas) | 696 NBS (Surinam) | 697 NBS (Dominican) | 698 NBS (Jamaican) | BXT-09 |
|--------------------------------|--------------------|-------------------|---------------------|--------------------|--------|
| Al ₂ O ₃ | 48.80 | 54.50 | 45.80 | 48.20 | 53.40 |
| BaO | 0.008 | 0.004 | 0.015 | 0.008 | - |
| CaO | 0.13 | 0.018 | 0.71 | 0.62 | 0.010 |
| Co | 0.0001 | 0.00009 | 0.0013 | 0.0045 | - |
| Cr ₂ O ₃ | 0.011 | 0.047 | 0.100 | 0.080 | 0.037 |
| Fe ₂ O ₃ | 7.14 | 8.70 | 20.00 | 19.60 | 14.15 |
| MgO | 0.085 | 0.012 | 0.18 | 0.058 | 0.03 |
| MnO | 0.110 | 0.004 | 0.41 | 0.38 | 0.04 |
| P ₂ O ₅ | 0.118 | 0.050 | 0.97 | 0.37 | 0.07 |
| K ₂ O | 0.068 | 0.009 | 0.062 | 0.010 | - |
| SiO ₂ | 13.43 | 3.79 | 6.81 | 0.69 | 7.57 |
| Na ₂ O | 0.025 | 0.007 | 0.036 | 0.015 | - |
| SO ₃ | 0.63 | 0.21 | 10.13 | 0.22 | - |
| TiO ₂ | 1.90 | 2.64 | 2.52 | 2.38 | 2.98 |
| V ₂ O ₅ | 0.028 | 0.072 | 0.063 | 0.064 | 0.06 |
| Loss on Ignition | 27.20 | 29.90 | 22.10 | 27.30 | 20.80 |

The parameters in the process of recording iron were as follows: Line Fe KA₁, Mask: 34 mm, Mode: Vacuum, 50 kV, 6 mA, Filter: NoneCrystal (nominal): 2d = 4.026 Å, Collimator aperture (nominal) = 0.23 degrees, Detector: scintillation counter LLD = 35, ULD = 300 % of nominal peak. Adjusted peak at 57.486 degrees 2-theta, Wavelength = 1.936 Å.

2.2.2. Preparation of the Samples for Reference Measurement

Spectrophotometric analysis involved preparation of solutions by fusing the sample with the mixture of Na₂CO₃ and Na₂B₄O₇ (3:1) according to a modified method JUS B.G8.

520/92 and ISO 6994/86. The spectrophotometric method MA. B. M.018 is based on the reduction iron (III) by hydroxylamine and the formation of a red-colored complex of ferro-orthophenanthroline under a strictly determined pH. A UV-VIS spectrophotometer Lambda 25 Perkin Elmer was used for measurement.

2.2.3. Mineralogical Characterization of the Samples

Mineralogical characterization of all the samples of bauxite was carried out using X-ray diffraction (XRD analysis) on a powder diffractometer PHILIPS PW 171. Radiation from the copper anticathode with the wavelength of CuKα = 1.54178 Å

and the graphite monochromator were used for the analysis. The operating voltage on the tube was $U = 40$ kV, and the current intensity was $I = 30$ mA. Samples were tested in the range 2θ 5–50° and with time retention of 1s at each step. X'Pert Quantify computer software was used for instrument manipulation, whereas X'PertHighScore was used for data processing.

3. Results and Discussion

According to the XRD analysis, the bauxite samples from different deposits (“Grčka”, “Brazil”, and “Mađarska”) have the following mineralogical composition:

“Grčka” bauxite is diaspore bauxite with a boehmite and hematite contents. In addition to those minerals, the sample also contains calcite and anatase, whereas quartz, kaolinite, gibbsite, goethite, and rutile are present in traces (Figure 1).

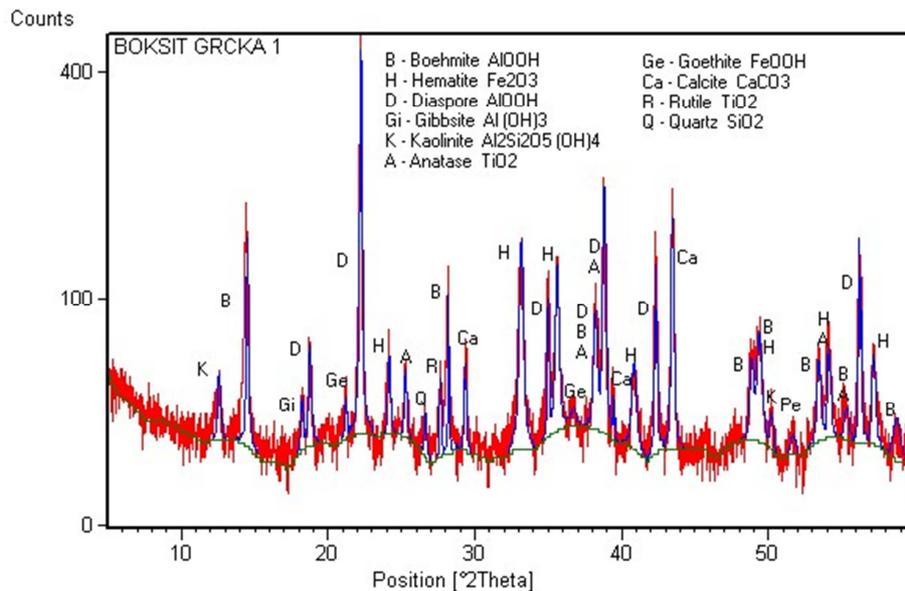


Figure 1. Diffractogram of the bauxite sample “Grčka”.

“Brazil” is a typical hydrargillite type of bauxite. In addition to gibbsite, this sample contains boehmite, hematite, anatase, and kaolinite, all in traces (Figure 2).

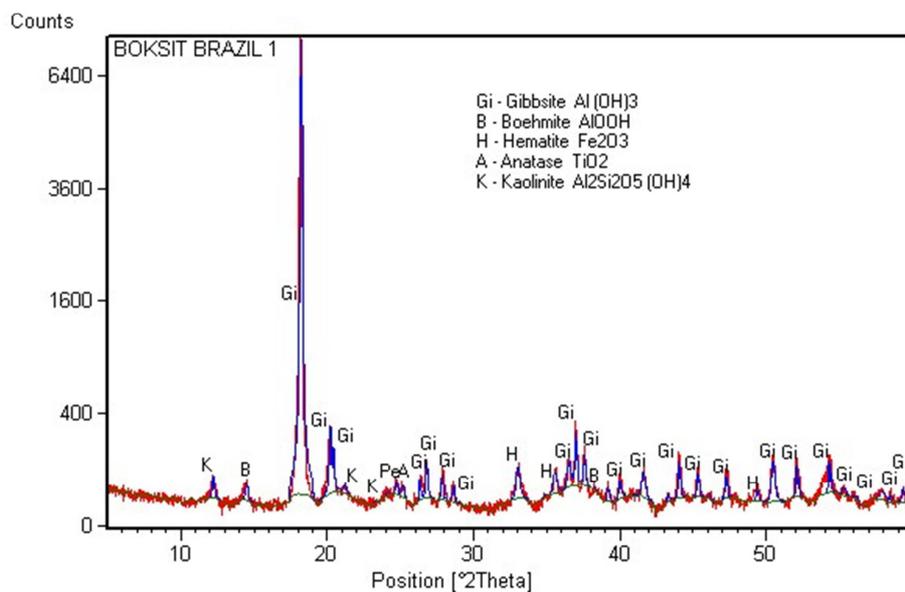


Figure 2. Diffractogram of the bauxite sample “Brazil”.

“Mađarska” is a gibbsite-boehmite combination with hematite and kaolinite contents. In addition to these minerals, the sample also contains goethite and traces of anatase and rutile (Figure 3).

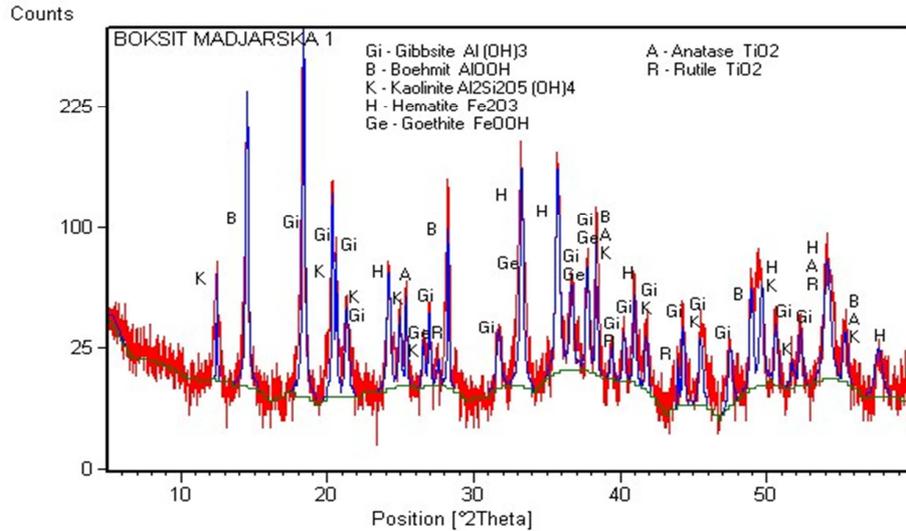


Figure 3. Diffractogram of the bauxite sample "Madarska".

The calibration curve was created based on the data for standard certified reference samples of bauxite and on the obtained values of intensity (Net) for these samples. The content of Fe₂O₃, which was taken as the basis for the development of the calibration curve, was calculated for an absolutely annealed sample (Figure 4).

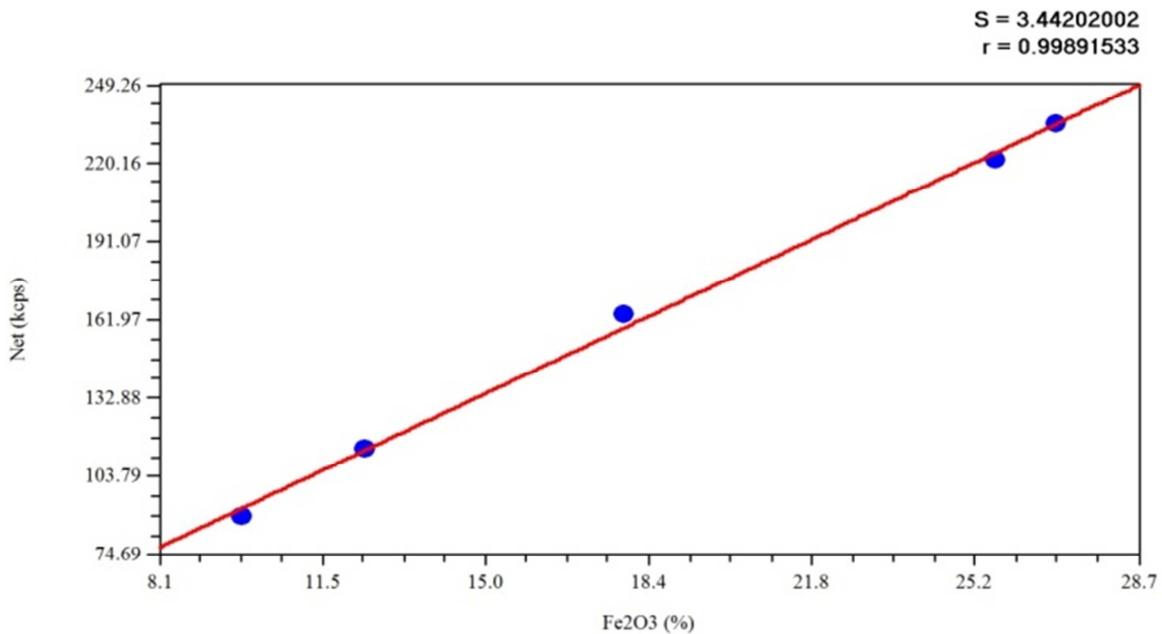


Figure 4. Calibration curve.

The equation for calculating the percentage (mass %) of iron-oxide in annealed bauxite was derived based on the calibration curve obtained for the beads resulting from the process of pressing, (1):

$$\% \text{Fe}_2\text{O}_3 \text{ (annealed)} = 0,120108218 \cdot \text{Net} - 1,233740095 \quad (1)$$

The actual content of iron-oxide in the samples of bauxite was calculated according to the following equation, (2):

$$\% \text{Fe}_2\text{O}_3 = \% \text{Fe}_2\text{O}_3 \text{ (annealed)} \cdot \frac{100 - \text{LOI}_{1075^\circ\text{C}}}{100} \quad (2)$$

This calculation is based on the values of the loss on ignition (LOI) at 1075 °C (Table 2).

Table 2. Loss on ignition for bauxites from different deposits.

| Bauxite | LOI (1075°C)/% |
|----------|----------------|
| Grčka | 12.12 |
| Brazil | 27.84 |
| Mađarska | 19.05 |

On the basis of the calibration curve, the samples of different deposits prepared by pressing were recorded and the content of Fe₂O₃ was calculated according to the above equations (Table 3).

Table 3. The content of Fe₂O₃ in bauxites from different deposits, calculated using spectrophotometric and XRF method.

| Bauxite | Fe ₂ O ₃ (mass %) | |
|----------|---|---------|
| | Spectrophotometry | XRF |
| Grčka | 21.462 | 20.9512 |
| Brazil | 10.731 | 11.4940 |
| Mađarska | 30.274 | 28.5913 |

The data presented here were obtained as a result of recording ten different beads from every sample, as well as recording a bead from each sample ten times. Moreover, ten samples for every bauxite were prepared for spectrophotometric analysis of the content of Fe₂O₃. The results for “Grčka” bauxite are shown in the tables below (Table 4, Table 5, and Table 6).

Table 4. The content of Fe₂O₃ in ten different beads of the “Grčka” bauxite sample calculated using the XRF method.

| Bead | Intensity (kcps) | Fe ₂ O ₃ (mass %) | |
|-----------|------------------|--|--------------|
| | | annealed | not annealed |
| 1 | 209.2056 | 23.2056 | 20.3922 |
| 2 | 209.3058 | 23.9056 | 21.0073 |
| 3 | 209.6672 | 23.9490 | 21.0454 |
| 4 | 209.4718 | 23.9255 | 21.0248 |
| 5 | 209.1433 | 23.8861 | 20.9901 |
| 6 | 209.2188 | 23.8952 | 20.9981 |
| 7 | 209.4572 | 23.9238 | 21.0233 |
| 8 | 209.3171 | 23.9070 | 21.0085 |
| 9 | 209.2176 | 23.8950 | 20.9979 |
| 10 | 209.4669 | 23.9250 | 21.0243 |
| Min | 209.1433 | 23.2056 | 20.3922 |
| Max | 209.6672 | 23.9490 | 21.0454 |
| \bar{x} | 209.3471 | 23.84178 | 20.9512 |
| S | 0.163938 | 0.224316 | 0.197101 |

Table 5. The content of Fe₂O₃ in one bead of the bauxite sample “Grčka” calculated using the XRF method.

| Number of measurements | Intensity (kcps) | Fe ₂ O ₃ (mass %) | |
|------------------------|------------------|---|--------------|
| | | annealed | not annealed |
| 1 | 209.1875 | 23.8914 | 20.9948 |
| 2 | 209.2240 | 23.8958 | 20.9987 |
| 3 | 209.0736 | 23.8777 | 20.9828 |
| 4 | 209.2877 | 23.9034 | 21.0054 |
| 5 | 209.8495 | 23.9709 | 21.0647 |
| 6 | 209.3462 | 23.9105 | 21.0116 |
| 7 | 209.3204 | 23.9074 | 21.0089 |
| 8 | 209.5856 | 23.9392 | 21.0368 |
| 9 | 209.6316 | 23.9447 | 21.0416 |
| 10 | 209.8477 | 23.9707 | 21.0645 |
| Min | 209.0736 | 23.8777 | 20.9828 |
| Max | 209.8495 | 23.709 | 21.0647 |
| \bar{x} | 209.4354 | 23.92117 | 21.0209 |
| S | 0.275392 | 0.033072 | 0.029054 |

Table 6. The content of Fe₂O₃ in ten different samples of “Grčka” bauxite calculated using spectrophotometric method MA. B. M.018.

| Number of analysis | Fe ₂ O ₃ (mass %) |
|--------------------|---|
| 1 | 21.40 |
| 2 | 21.48 |
| 3 | 21.42 |
| 4 | 21.37 |
| 5 | 21.28 |
| 6 | 21.71 |
| 7 | 21.48 |
| 8 | 21.51 |
| 9 | 21.57 |
| 10 | 21.40 |
| Min | 21.28 |
| Max | 21.71 |
| \bar{x} | 21.462 |
| S | 0.11887 |

In order to check the precision of the XRF method for the beads prepared by pressing, a F-test was conducted, where in the zero hypothesis tested was that the variances in the spectrophotometric method for calculating the content of Fe₂O₃ in bauxites-MA. B. M.018 and a non-standard XRF method were equal. The following value of the F-test was obtained:

$$F = \frac{S_1^2}{S_2^2}, F = 3,56, \nu_1 = n_1 - 1, \nu_2 = n_2 - 1 \quad (3)$$

Critical value at $\alpha = 0.05, \nu_1 = 9, \nu_2 = 9$ is $F_{9,9} = 3.18$. The calculated result is higher than the critical value, which means that the difference between the variances of the two methods is significant, and with a risk of 5 % the zero hypothesis on the equality of variances can be rejected. Existing differences are caused by a systematic error.

The test of accuracy for the XRF method used in determining Fe₂O₃ was performed on the standard bauxite B-010, as well as compared to the same reference method. Ten beads of this standard were prepared by pressing and then recorded. The results obtained are given in Table 7.

Table 7. The content of Fe₂O₃ in the reference bauxite sample B-010 calculated using the XRF method.

| Bead | Intensity (kcps) | Fe ₂ O ₃ (mass %) | |
|-----------|------------------|---|--------------|
| | | annealed | not annealed |
| 1 | 252.6107 | 29.1069 | 25.8586 |
| 2 | 252.7972 | 29.7972 | 26.4718 |
| 3 | 252.4369 | 29.0860 | 25.8400 |
| 4 | 252.5775 | 29.1029 | 25.8550 |
| 5 | 253.2693 | 29.1860 | 25.9288 |
| 6 | 252.2215 | 29.0601 | 25.8169 |
| 7 | 253.5394 | 29.2184 | 25.9576 |
| 8 | 253.4861 | 29.2120 | 25.9519 |
| 9 | 252.9920 | 29.1527 | 25.8993 |
| 10 | 253.2800 | 29.1873 | 25.9299 |
| Min | 252.2215 | 29.0601 | 25.8169 |
| Max | 253.5394 | 29.7972 | 26.4718 |
| \bar{x} | 252.9211 | 29.21095 | 25.9510 |
| S | 0.460706 | 0.213227 | 0.189428 |

These results served as a starting point for the t-test, where the hypothesis on the equality of the reference value and the average value of the results was tested. The following value of the t-test was obtained:

$$|t| = \left| \frac{\mu - \bar{x}}{s} \right| \times \sqrt{n}, |t| = 38,552, \nu = n - 1, \nu = 9 \quad (4)$$

Critical value at $\alpha = 0.05$, $\nu = 9$ is $|t| = 2.262$. Since $|t| > |t|_{\text{critical}}$, with the risk of 5 % the zero hypothesis can be rejected. It can be concluded that the XRF method in this case shows a systematic error.

Testing the accuracy of the XRF method using the same reference method involved testing the hypothesis of the equality of arithmetic means of the two methods. The following values were calculated for this purpose:

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{\sqrt{\frac{s_1^2}{N_1} + \frac{s_2^2}{N_2}}}, t = 6,36 \quad (5)$$

$$\nu = \frac{\left(\frac{s_1^2}{N_1} + \frac{s_2^2}{N_2} \right)^2}{\left(\frac{s_1^4}{N_1^2(N_1-1)} + \frac{s_2^4}{N_2^2(N_2-1)} \right)}, \nu = 13,69 \quad (6)$$

Critical value at $\alpha = 0.05$, $\nu = 14$ is $|t| = 2.14$. Since in this case $|t| > |t|_{\text{critical}}$, then, with the risk of 5 %, the hypothesis of the equality of arithmetic means of the two methods must be rejected. It can be concluded that the XRF method for the beads prepared by pressing does not yield the same average value as the reference method, which means that it shows a systematic error.

When materials like rocks, mineral ores, etc. are analyzed with the pressed powder pellet method, analysis errors due to the heterogeneity effect are generally present. This effects can be classified into mineralogical effect, segregation and grain size effect. It is important for the powder method to minimize the heterogeneity effect and to make standard samples and unknown samples under the same condition (having the same heterogeneity effect). To eliminate the heterogeneity effect, there is a sample preparation method called the fusion bead method [14].

4. Conclusion

Based on recording the intensities of the beads made from certified reference bauxite samples, prepared by pressing, the calibration curve was obtained with the correlation coefficient of $r = 0,9989$ and the standard deviation of $S = 3,4420$. The calibration curve was the basis for the equation used for calculating the content of Fe_2O_3 (%) in the bauxite samples from different deposits. The XRF method was then tested for

precision and accuracy. The F-test results show, with the risk of 5 %, that the zero hypothesis on the equality of variances can be rejected, which means that the XRF method is not precise. A t-test was conducted to test the accuracy (using the reference method and the standard bauxite sample B-010). With the risk of 5 %, it can be concluded that the reference values and average values of the results investigated differed, as well as the arithmetic meanings of the two methods and that the method showed a systematic error.

Based on the results obtained, it can be concluded that the XRF, as a method for calculating the content of Fe_2O_3 in bauxite, proved imprecise and inaccurate, when beads are prepared by pressing. This method shows a systematic error, which is a consequence of insufficient homogeneity of the sample.

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