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Determining the Content of Silicon Dioxide in Bauxites Using X-Ray Fluorescence Spectrometry

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ABSTRACT: *The X-ray fluorescence spectrometry and the MA.BM.006 reference spectrophotometric methods were used to determine the content of SiO_2 (%) in bauxites from different deposits. The treatment of samples prior to the analysis involved the following steps: annealing, melting using the borax method, and the formation of beads. Certified reference bauxite samples were used for the calibration curve. The calibration curve was produced with the correlation coefficient of r =0.9999 and the standard error of S = 0.0246. The average residual value between the content of SiO_2 determined using the XRF method, and the reference method was 0.045, with a standard deviation of 0.068. The XRF method was statistically verified by the F- and t-tests (using the standard sample and the reference method). The values obtained in the tests show that the XRF method yields accurate results and that there are no standard errors.*

KEYWORDS: *XRF method; Spectrophotometric method; Bauxite; Silicon dioxide*.

INTRODUCTION

Although often classified as a mineral, bauxite is a sedimentary rock, a polymineral aggregate composed of aluminum hydroxide [1]. It is the most important ore in for the production of aluminum, nonmetal products

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and nonmetallurgical applications such as production refractories and abrasive materials [2].

The mineralogy of bauxite depends largely on the geology of the parent rock, as well as some other factors such as climate and biology. The main minerals in bauxite include several forms of hydrated aluminum oxide: gibbsite ($Al_2O_3 \cdot 3H_2O$), boehmite ($Al_2O_3 \cdot H_2O$) and diaspore ($Al_2O_3 \cdot H_2O$). Apart from those, there are over 170 other minerals identified in bauxites. The most important among them are corundum, goethite, hematite, kaolinite, halloysite, anatase and rutile. Gangue and contaminating materials are found in traces, including calcite, dolomite and quartz, along with manganese and phosphate minerals [3]. Silicon occurs in bauxite in a free or a bound form of oxide. Minerals of the free oxide are crystalline forms of SiO_2 (quartz, quartzite, calcedonia) or the amorphous form of SiO_2 (opal). Among the mentioned forms, the most commonly found mineral is quartz, as one of the ploymorphous modifications of silicon oxide, which, in boehmite and diaspore, fills micropores and pores of crystals; it usually represents a constituent element of the binding mass. Among the bound forms of oxides in which it occurs, the most common is kaolinite. It occurs most often in hydrargillite bauxites, and it was found to form thin films on the surface of diaspore and boehmite [1].

The treatment of bauxite ore is generally considered cost-effective on condition that bauxite contains more than 45-50% of Al_2O_3, not more than 20 % of Fe_2O_3 and 3-5% of combined silicon oxide [4].

The quality of bauxite as seen from the aspect of its application in alumina production is determined on the basis of the content of Al_2O_3 and SiO_2. It is defined using the so-called silicon module, which represents the percentage ratio of Al_2O_3 and SiO_2 in bauxite. The bauxites with the silicon module of 10 are considered to be of high quality, those with 8-10 silicon modules are of such quality that can be treated, whereas those below 8 are considered to be of low quality and their treatment is not economically justifiable. This criterion is applied to boehmite and diaspore bauxites. Due to the lower temperature of leaching in hydragillite bauxites (<150 °C), the harmful effect of silicon depends only on the mineralogy forms, which dissolve with NaOH at these temperatures [5].

The chemical composition of bauxite is expressed by the content of Al_2O_3, SiO_2, Fe_2O_3, TiO_2 and CaO, with the loss on ignition at 1075°C. Chemical determination of the content of SiO_2 in bauxites in the alumina factory "Alumina" was conducted using the spectrophotometric method MA.BM.006, which was introduced as compared to the gravimetric method JUS B.G8.518 (1988) and ISO 6607 (1985). The spectrophotometric method for determining the content of SiO_2 in bauxite consists of the following stages:

1. Alkali fusion of bauxite samples with a mixture of Na_2CO_3 and $Na_2B_4O_7$(3:1)
2. Dissolution of the fused mass in HCl (1:1)
3. Formation of a silicomolybdic complex in strictly defined conditions for the pH value, the concentrations of reagents, temperature and time, its reduction using ascorbic acid and spectrophotometric determination at a wavelength of 810 nm.

The spectrophotometric method for determining the content of SiO_2 in bauxite was introduced based on the ISO 6607(1985) gravimetric method. This method involves the dissolution of bauxite using three acids (HCl, HNO_3, and H_2SO_4) with the formation of a silicon acid deposit. The deposit obtained is annealed at 1000°C. The resulting solid residue is then treated with hydrofluoric acid and the silicon is isolated as silicon fluoride.

The current researches into the determining of the content of SiO_2 in different composites (samples) were focused on the application of the Graphite Furnace AAS method, the IR-spectroscopy techniques and Inductively Coupled Plasma Optical Emission Spectroscopy [6-9].

The X-ray fluorescence spectrometry is a very powerful analytical technique for determining almost all the potential elements in the sample. XRF radiation occurs when photons with the sufficient level of energy emitted from an X-ray source are focused on the sample. These primary X-rays start various interactions with the atoms of the sample. High levels of energy in photons cause the electrons in the inner orbit to ionize in the photoelectric effect forming voids in the inner orbits (K, L, M...). A rapid transfer of electrons from the outer orbit to these voids causes the emission of characteristic fluorescent radiation. Elements are identified according to their characteristic X-rays. The spectrum of the emitted characteristic fluorescent radiation is measured using the spectrometers with wavelength dispersion (WDXRF) and energy dispersion (EDXRF). In WDXRF analyses,

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the results are given in the form of the the intensity of the spectrum with characteristic lines according to wavelengths [10].

The XRF has a wide application in industrial, pharmaceutical, forensic and scientific researches in terms of determining the presence or absence of elements, or, in some cases, measuring the concentrations of constituent elements or contaminating materials [11].

X-ray fluorescence spectrometry offers a large number of advantages. The spectrums are relatively simple and, spectral interferences are therefore rare. The method can be conducted on samples ranging from those barely visible to massive objects. One of the advantages is that the results can be obtained very quickly (in several minutes). Considering the fact that the intensity of fluorescence is proportional to the mass proportion of all the components present in the sample, modern fluorescence instruments with X-rays can be used for the quantitative analyses of complex materials with the precision which is the same or even exceeds that of the traditional wet chemistry methods or other instrumental methods. Suitable calibration databases, obtained by measuring the intensity of fluorescence X-rays for standard materials, are input in the instrument software. Their purpose is to establish a relationship between the signal read for the analyzed sample and the signal of the standard material. Calibration databases are formed using certified reference materials with familiar, precisely defined qualitative and quantitative chemical compositions [12].

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 SiO_2. as well. Therefore, the methods for determining the contents of these components need to be quick and reliable.

EXPERIMENTAL SECTION

The experimental part involved using bauxites from different deposits (Brazil, Mostar, Bosanska Krupa, Indija, Nikšić and Široki Brijeg), treated at "Alumina" near Zvornik, Bosnia & Herzegovina.

The samples of bauxite were first reduced to the particle size of 200 μm and dried, and then annealed at the temperature of 1075°C. The loss on ignition was also calculated at this temperature.

The next step was to separate 1g of the annealed sample and mix it with 8 g of $Li_2B_4O_7$ in a platinum pot for the purpose of the XRF analysis. After the mixture was gently stirred using a glass rod, the pot was placed on the annealing tool "VULCAN", by Fluxana. After melting for 15 minutes at about 1250°C, the sample was poured into a preheated platinum mould. After cooling for 10 minutes, the obtained bead was recorded by WDXRF "S8 TIGER" (Bruker).

The calibration curve was drawn based on the certified reference samples of bauxite, which were also annealed prior to the procedure, then melted and poured into the moulds, and recorded (Table 1).

The recording conditions for silicon were as follows: Line Si KA_1, Mask: 34 mm, Mode: Vacuum, 30 kV , 20 mA, Filter: None Crystal (nominal): 2d = 8.752 Ao, Collimator aperture (nominal) = 0.23 degrees, Detector: flow counter LLD = 35, ULD = 300% of nominal peak. Adjusted peak at 109.009 degrees 2-theta, Wavelength = 7.1254 Ao.

The spectrophotometric analysis involved using the solutions prepared according to the MA.BM.004–modified method JUS B.G8. 520/92 and ISO 6994/86. UV-Vis spectrophotometer Lambda 25 Perkin Elmer was used.

Mineralogical characterization of all the samples of bauxite was conducted using X-ray diffraction analysis (XRD analysis) on the powder diffractometer type PHILIPS PW 1710. We used the radiation from a copper anticathode with CuKα = 1.54178 Å wavelength and a graphite monochromator. Radiation from the copper anticathode with the wavelength of CuKα = 1.54178 Å and a graphite monochromator were used for the analysis. The operating voltage in the tube was U = 40 kV, the current intensity I = 30 mA. The samples were measured in the range of 2θ 5–50 ° with 0.02 ° rotation movement and the time-lapse of 1 s per every movement. X′Pert Quantify computer software was used for the instrument manipulation, whereas X′Pert HighScore was used for data processing.

RESULTS AND DISCUSSION

According to the XRD analysis and the obtained diffractograms, the standard reference samples of bauxite have the following mineralogical composition:

Standard NBS 69b is a typical hydrargillite bauxite. Apart from gibbsite, this sample also contains kaolinite, hematite, goethite and siderite (Fig. 1).

Standard NBS 696 is a typical hydragillite bauxite. Apart form gibbsite, this sample of bauxite also contains

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Table 1: Analysis of standard reference bauxite samples according to the certificate.

Components	69b NBS (Arkansas)	696 NBS (Surinam)	697 NBS (Dominican)	698 NBS (Jamaican)	BXT-09
Al_2O_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_2O_3	0.011	0.047	0.100	0.080	0.037
Fe_2O_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_2O_5	0.118	0.050	0.97	0.37	0.07
K_2O	0.068	0.009	0.062	0.010	-
SiO_2	13.43	3.79	6.81	0.69	7.57
Na_2O	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_2O_5	0.028	0.072	0.063	0.064	0.06
Loss on Ignition	27.2	29.9	22.1	27.3	20.8

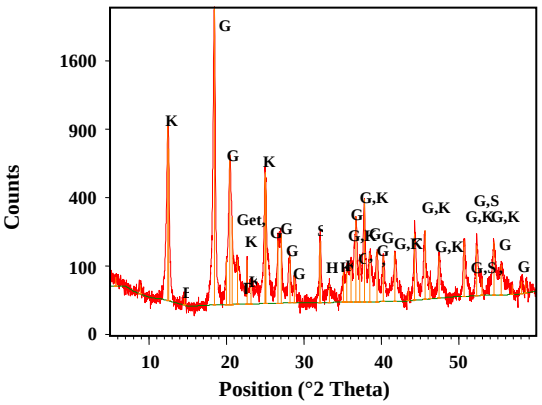

Fig. 1: Diffractogram of the standard bauxite sample NBS 69b (Arkansas).

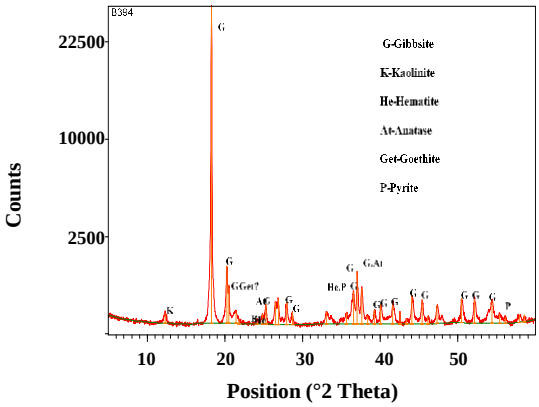

Fig. 2: Diffractogram of the standard bauxite sample NBS 696 (Surinam).

small traces of hematite, anatase, goethite, kaolinite and pyrite (Fig. 2).

Standard NBS 697 is a combined, gibbsite-boehmite type, with a small percentage of hematite. Apart from the mentioned minerals, the sample also contains goethite and kaolinite, whereas anatase and calcite are found in traces (Fig. 3).

Standard NBS 698 is a hydragillite bauxite with a small percentage of hematite. Apart from gibbsite, this sample contains anatase, goethite, and hematite (Fig. 4).

Standard BXT-09 is a gibbsite-boehmite type of bauxite with a small percentage of hematite. Apart from these minerals, the sample also contains kaolinite, whereas anatase and quartz occur in traces (Fig. 5).

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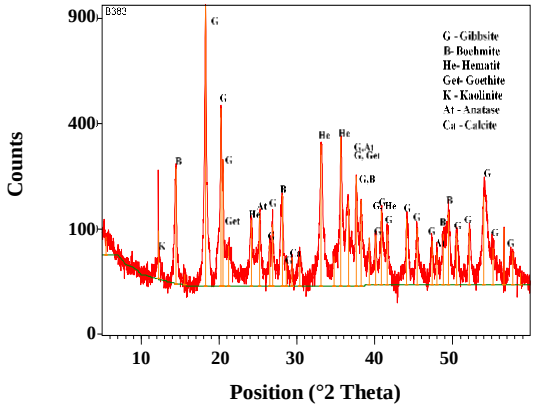

Fig. 3: Diffractogram of the standard bauxite sample NBS 697 (Dominican).

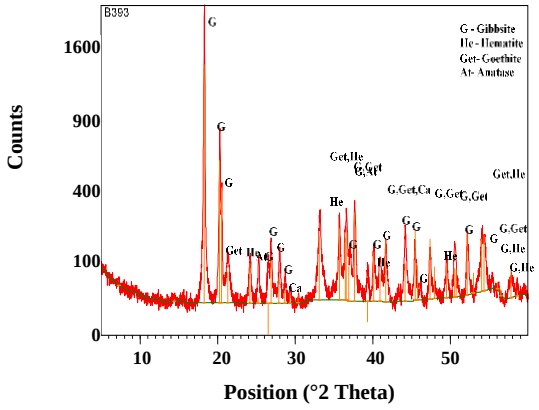

Fig. 4: Diffractogram of the standard bauxite sample NBS 698 (Jamaican).

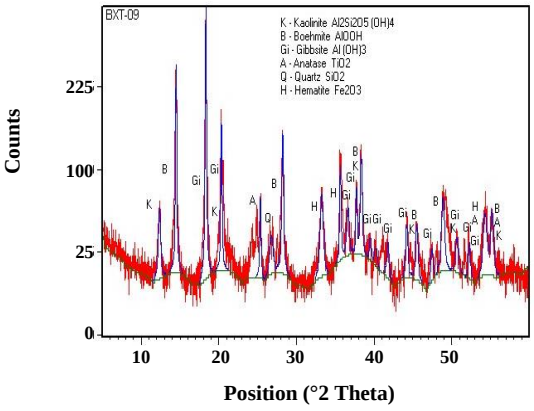

Fig. 5: Diffractogram of the standard bauxite sample BXT-09.

Bauxite samples from different deposits according to the XRD analysis have the following mineralogical composition:

"Brazil" is a typical hydragillite type of bauxite. Apart from gibbsite, this sample contains boehmite, hematite, anatase, and kaolinite, in traces (Fig. 6).

"Mostar" is of boehmite type with traces of gibbsite and a considerable content of hematite. Apart from these, the sample contains anatase and calcite, and kaolinite i s present in traces. The presence of gibbsite is confirmed by a slightly increased loss on ignition compared to the pure boehmite bauxite (Fig. 7).

"Bosanska Krupa" is a boehmite-diaspore bauxite type with a small percentage of hematite. The sample also contains anatase and traces of calcite and kaolinite (Fig. 8).

"Indija" is a typical hydargillite type of bauxite. Apart from gibbsite, the sample also contains boehmite, hematite, anatase, calcite, and kaolinite (Fig. 9).

"Nikšić" is boehmite bauxite with a considerable content of hematite. The sample also contains calcite and anatase, and ghoetite is present in traces (Fig. 10)

"Široki Brijeg" is boehmite bauxite with a considerable content of hematite and calcite. The considerable quantity of calcite is confirmed by an increased loss on ignition compared to the boehmite bauxite. The increase in loss on ignition is in accordance with the presence of calcite. The sample also contains goethite and anatase, and rutile is present in traces (Fig. 11).

The calibration curve was created based on the data for the standard certified reference samples of bauxite and on the obtained values of intensity (Net) for these samples. The content of SiO_2, taken as the basis for the calibration curve, was calculated to an absolutely annealed sample (Fig. 12).

The equation for calculating the percentage (%) of silicon dioxide in the annealed bauxite was derived based on the obtained calibration curve, Eq. (1):

$$SiO_2(\text{annealed})/\% = 4.227569562 \times \text{Net} - 0.017968435 \quad (1)$$

The real content of silicon dioxide in the bauxite samples was calculated according to the following pattern, Eq. (2):

$$SiO_2/\% = SiO_2(\text{annealed})/\% \times \frac{100 - LOI_{1075^\circ C}}{100} \quad (2)$$

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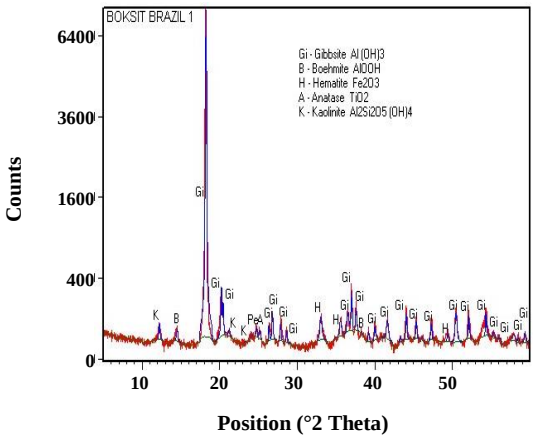

Fig. 6: Diffractogram of the bauxite sample "Brazil".

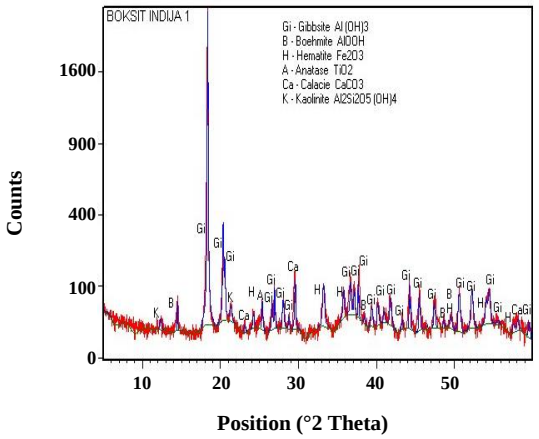

Fig. 9: Diffractogram of the bauxite sample "Indija".

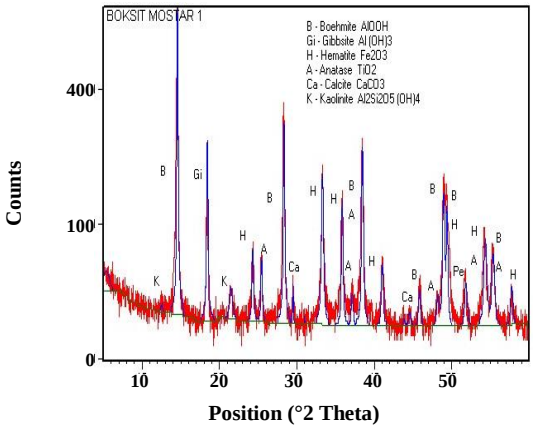

Fig. 7: Diffractogram of the bauxite sample "Mostar".

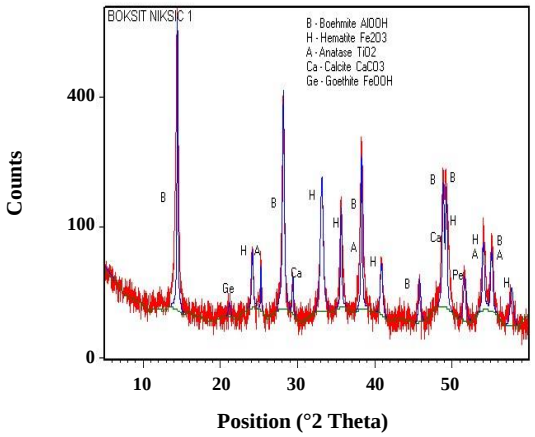

Fig. 10: Diffractogram of the bauxite sample "Nikšić".

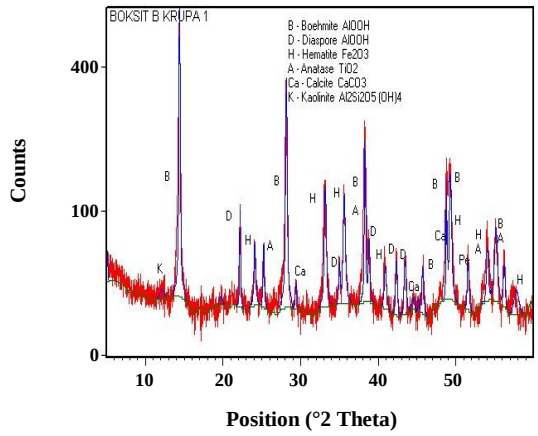

Fig. 8: Diffractogram of the bauxite sample "Bosanska Krupa".

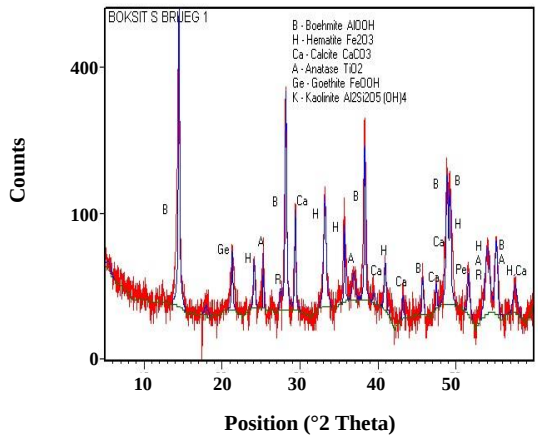

Fig. 11: Diffractogram of the bauxite sample "Široki Brijeg".

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Table 2: The loss on ignition for bauxites from different deposits.

Bauxite	LOI (1075 °C)/ %
BRAZIL	27.84
MOSTAR	12.75
BOSANSKA KRUPA	12.47
INDIJA	26.53
NIKŠIĆ	12.97
ŠIROKI BRIJEG	14.80

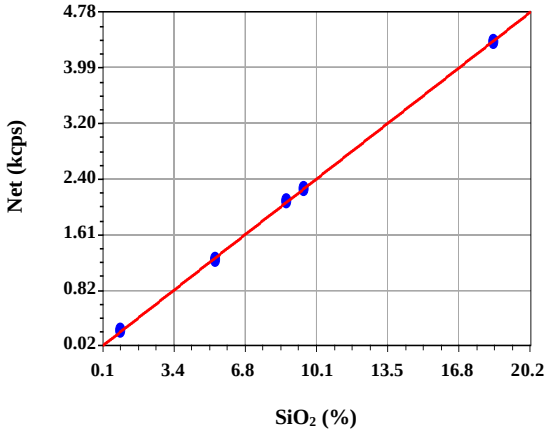

Fig. 12: Calibration curve.

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

On the basis of the calibration curve, the samples of different deposits were recorded and the content of SiO_2 was calculated according to the mentioned patterns (Table 3):

The data shown were obtained by recording ten different beads for every sample, and one bead of every sample was recorded ten times as well. All the measurements were conducted in terms of repeatability. They were performed by a single person, with the same equipment in the same laboratory and within a short time frame. Moreover, ten samples for each bauxite were prepared for spectrophotometric calculation of SiO_2 content. The results for the "Brazil" sample are given in the following tables (Table 4, Table 5, Table 6):

The accuracy of the XRF method was tested by an F-test, where the zero hypotheses hypothesis according to which the variances in the spectrophotometric method for calculating the content of SiO_2 in bauxites-MA.BM.006 and a non-standard XRF method were equal. The following value of the F-test was obtained:

$$F=\frac{S_1^2}{S_2^2}=\frac{0.0622^2}{0.0594^2}=1.096$$

Table value at α = 0.05 is $F_{9.9}$ = 3.18. Therefore, with a risk of 5%, the zero hypotheses hypothesis on the equality of variances can be confirmed. The existing differences are caused by random errors.

The test of accuracy for the XRF method used in determining SiO_2 was performed on the standard bauxite BXT-09 as well as compared to the reference method. Ten beads of this standard were prepared and recorded. The results obtained are given in Table 7.

These served as the 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.

$$|t|=\left|\frac{\mu-\bar{x}}{s}\right|\cdot\sqrt{n}=\left|\frac{7.57-7.563}{0.0739}\right|\cdot\sqrt{10}=0.299$$

Table value at α = 0.05 is $|t|$ = 2.262. As $|t|<|t|_{tab}$, with the risk of 5%, the zero hypotheses can be accepted and we can state that the XRF method does not have systematic errors.

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

$$S_p=\sqrt{\frac{(n_1-1)\cdot S_1^2+(n_2-1)\cdot S_2^2}{n_1+n_2-2}}\cdot\sqrt{\left(\frac{1}{n_1}+\frac{1}{n_2}\right)}=$$

$$\sqrt{\frac{(10-1)\cdot 0.0594^2+(10-1)\cdot 0.0622^2}{10+10-2}}\cdot\sqrt{\left(\frac{1}{10}+\frac{1}{10}\right)}=0.0272$$

$$t=\frac{\bar{x}_1-\bar{x}_2}{S_p}=\frac{5.2034-5.194}{0.0272}=0.346$$

Table value at α = 0.05 is $|t|$ = 2.101. Due to the fact that $|t|<|t|_{tab}$, the hypotheses hypothesis of the equality of arithmetic means for the two methods can be accepted and we one can conclude with the risk of 5% that the XRF method does not have systematic errors.

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Table 3: The content of SiO_2 in bauxites from different deposits calculated using spectrophotometric and XRF method.

Bauxite	SiO_2 (%)		Residual value
	Spectrophotometry	XRF method	
Brazil	5.19	5.20	0.01
Mostar	0.94	0.96	0.02
Bosanska Krupa	2.98	2.80	0.18
Indija	3.98	3.99	0.01
NIKŠIĆ	1.63	1.66	0.03
Široki Brijeg	1.59	1.61	0.02
$\bar{x}$	-	-	0.045
S	-	-	0.06758245

Table 4: The content of SiO_2 in ten different beads of the "Brazil" bauxite determined using the XRF method.

Bead	Intensity (kcps)	SiO_2(%)	
		annealed	not annealed
1	1.7121	7.22005	5.2098
2	1.7122	7.22048	5.2102
3	1.7192	7.25007	5.2315
4	1.6880	7.11817	5.1363
5	1.6886	7.12071	5.1382
6	1.7186	7.24753	5.2297
7	1.7068	7.19765	5.1937
8	1.7325	7.30630	5.2721
9	1.7022	7.17820	5.1796
10	1.7196	7.25176	5.2327
Min	1.6880	7.11817	5.1363
Max	1.7325	7.30630	5.2721
$\bar{x}$	1.70998	7.211092	5.2034
S	0.014061	0.0594417	0.042896

CONCLUSIONS

Recording the intensities of the beads made of certified reference bauxite samples, we produced the calibration curve with the correlation coefficient of r = 0.9999 and the standard deviation of S = 0.0246. The calibration curve was the basis for the equation used for calculating the content of SiO_2 (%) in the samples of bauxite from different deposits. The average residual value between the contents of SiO_2 determined by the XRF method and the reference method, MA.BM.006 was 0.045 with a standard deviation of 0.068. The XRF method for determining the content of SiO_2 in the samples of bauxite was tested for accuracy and precision. The obtained value of the F-test shows that, with the risk of 5%, the variances in both methods are equal, which means that the XRF method is precise. The accuracy test was performed by means of the t-test (the reference method and the standard sample of bauxite BXT-09).

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Table 5: The content of SiO_2 in one bead of "Brazil" bauxite sample determined by XRF method.

Number of measurements	Intensity (kcps)	SiO_2(%)	
		annealed	not annealed
1	1.6778	7.07505	5.1052
2	1.7034	7.18327	5.1833
3	1.7047	7.18877	5.1873
4	1.7015	7.17524	5.1775
5	1.7049	7.18961	5.1879
6	1.7169	7.24035	5.2245
7	1.7000	7.16890	5.1729
8	1.6875	7.11606	5.1348
9	1.6733	7.05602	5.0915
10	1.7043	7.18708	5.1861
Min	1.6733	7.05602	5.0915
Max	1.7169	7.24035	5.2245
$\bar{x}$	1.69743	7.15804	5.1651
S	0.013581	0.057414	0.041434

Table 6: The content of SiO_2 in ten different samples of the "Brazil" bauxite determined by spectrophotometry MA.BM.006.

Number of analysis	SiO_2(%)
1	5.27
2	5.10
3	5.12
4	5.12
5	5.17
6	5.2
7	5.24
8	5.25
9	5.23
10	5.24
Min	5.10
Max	5.27
$\bar{x}$	5.194
S	0.062218

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Table 7: The content of SiO_2 in the certified bauxite sample BXT-09 determined by XRF method.

Bead	Intensity (kcps)	SiO_2(%)	
		annealed	not annealed
1	2.2491	9.49026	7.5163
2	2.3047	9.72531	7.7024
3	2.2827	9.63230	7.6288
4	2.2335	9.42431	7.4641
5	2.2777	9.61117	7.6120
6	2.2617	9.54353	7.5585
7	2.2481	9.48603	7.5129
8	2.2390	9.44756	7.4825
9	2.2758	9.60313	7.6057
10	2.2583	9.52915	7.5471
Min	2.2335	9.42431	7.4641
Max	2.3047	9.72531	7.7024
$\bar{x}$	2.26306	9.54928	7.5630
S	0.022091	0.09339	0.073946

With the risk of 5%, it can be concluded that the reference values and average values of the results tested are equal, that the arithmetic means of the two methods do not differ, and that the method does not have any systematic errors.

It can be observed that the standard deviation obtained by recording ten different beads and the standard deviation obtained by recording a single bead for ten times do not vary significantly.

The results obtained show that the X-ray fluorescence spectrometry is an accurate method for calculating the content of SiO_2 in bauxite. Apart from that, this method is more rapid compared to the traditional method, as the measurement takes only a couple of minutes. The preparation of the sample is also faster and requires fewer chemicals, which makes this method economically justifiable as well.

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