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ORIGINAL

Uv-vis spectrophotometry for the determination of iron and silicon in calcined alumina: evaluation of calibration curves

Espectrofotometría uv-visible para la determinación de hierro y silicio en alúmina calcinada: evaluación de curvas de calibración

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ABSTRACT

In laboratories with strict quality requirements, the calibration of equipment is crucial for the reliability of analytical results, especially in the determination of iron and silicon in alumina. This study validated analytical methods by constructing calibration curves for iron (Fe) and silicon (Si) using certified reference materials. The content of iron(III) oxide (Fe_2O_3) and silicon dioxide (SiO_2) in calcined alumina (NIST 699 standard) was evaluated by UV-Vis spectrophotometry. The statistical viability of the calibration models was confirmed with a Student's t-test. The calibration curves for Fe_2O_3 and SiO_2 showed a strong linear relationship (R^2 = 0,99891 and R^2 = 0,9995, respectively). The analysis of the reference standard yielded concentrations of 0,0129 ± 0,0004 % for Fe_2O_3 and 0,0124 ± 0,0005 % for SiO_2, demonstrating the accuracy of the method. Statistical analysis confirmed the consistency of the results with the expected theoretical values. In conclusion, the calibration curves showed robustness, the precision was evidenced by the low standard deviation in the replicates, and UV-Vis spectrophotometry proved to be highly reliable and accurate for the quantitative determination of iron and silicon in calcined alumina certified reference material.

Keywords: Calibration; Spectrophotometry; Iron; Silicon; Student's T-Test; UV-Vis.

RESUMEN

En laboratorios con estrictas exigencias de calidad, la calibración de equipos es crucial para la fiabilidad de resultados analíticos, especialmente en la determinación de hierro y sílice en alúmina. Este estudio validó métodos analíticos mediante la construcción de curvas de calibración para hierro (Fe) y silicio (Si) con patrones de referencia certificados. Se evaluó el contenido de óxido de hierro (III) (Fe_2O_3) y dióxido de silicio (SiO_2) en alúmina calcinada (patrón 699 NIST) por espectrofotometría UV-Visible. La viabilidad estadística de los modelos de calibración se confirmó con una prueba t de Student. Las curvas de calibración para Fe_2O_3 y SiO_2 mostraron una fuerte relación lineal (R^2 = 0,99891 y R^2 = 0,9995, respectivamente). El análisis del patrón de referencia arrojó concentraciones de 0,0129 ± 0,0004 % para Fe_2O_3 y 0,0124 ± 0,0005 % para SiO_2, demostrando la precisión del método. El análisis estadístico confirmó la consistencia de los resultados con los valores esperados. En conclusión, las curvas de calibración mostraron solidez, la precisión fue evidenciada por la baja desviación estándar, y la espectrofotometría UV-Visible demostró ser altamente confiable y precisa para la determinación cuantitativa de hierro y silicio en alúmina calcinada.

Palabras clave: Calibración; Espectrofotometría; Hierro; Silicio; T de Student; UV-Visible.

INTRODUCTION

The primary raw material for aluminum (Al) refining is alumina (aluminum oxide, Al_2O3), a chemical compound of great industrial importance. This white, odorless, and tasteless crystalline solid is abundant in nature as the main component of minerals such as corundum, ruby, and sapphire.(1) Metallic aluminum is produced by a chemical reaction between the anode block, mainly carbon (C) and alumina (Al_2O3). In this reaction, the oxygen in the alumina combines with the carbon (C) in the anode block to produce carbon dioxide (CO_2) in gaseous form and liquid aluminum (Al), which precipitates in the cathode at a temperature of approximately 960°C(2) (equation 1).

Al2O3 (ac) + 3/2C (s) → 2Al (l) + 3/2CO2 (g) (1)

The research stems from the need to guarantee analytical results in the Quality Control Division of CVG Venalum CA, which operates under strict quality standards. Equipment calibration is essential to ensure measurement accuracy, which is particularly critical in determining iron and silicon in alumina. Therefore, calibration curves must be updated periodically to align with internal laboratory policies. In this way, establishing a calibration frequency aims to balance the accuracy needed for the analyses and the available resources, maximizing the reliability of the data obtained. The importance of this research lies in updating the calibration curves for iron (Fe) and silicon (Si) using UV-visible spectrophotometry. A robust and reliable analytical method will be established by optimizing the update frequency to a six-month interval. This will allow for more accurate detection of low concentrations of these impurities, which is essential to ensure the quality of the final product and comply with current industry standards. As a result, the operability and reliability of the Quality Control Division of CVG Venalum C.A. will be ensured.

UV-visible spectrophotometry is an indispensable analytical technique for accurately determining the concentrations of iron and silicon in alumina samples. This methodology is based on the ability of metal ions to form colored complexes with specific reagents. When these complexes are irradiated with ultraviolet or visible light, a characteristic absorption occurs that is directly proportional to the concentration of the element in the sample. It is based on the fact that molecules absorb electromagnetic radiation and, in turn, the amount of light absorbed depends linearly on the concentration.(3) Beer's Law is the relationship that describes the absorption or attenuation of monochromatic radiation by matter. The absorbance of the analyte is directly proportional to the optical path through the medium and the concentration of the absorbing species. The absorptivity depends on the units used for the beam path length and the concentration of the absorbing species. When the concentration in Equation 2 is expressed in mol·L-1 and the length in centimeters (cm), the absorptivity is called molar absorptivity or molar extinction coefficient, represented by the symbol "ε" (equation 3). It can be obtained by multiplying the absorptivity by the molecular weight of the absorbing substance.(4,5)

$A = a \times b \times c$ (2)
$A = \varepsilon \times b \times c$ (3)

Where:

Absorbance (A): i is the logarithm of the ratio between the initial energy of a radiation beam (P0) and its energy after passing through an absorbing medium (P).

Absorptivity (a): proportionality constant where absorptivity has as units L·g-1·cm-1.

Molar absorptivity (ε): proportionality constant where absorptivity has the units of L·mol-1·cm-1.

Length (b): length of the radiation beam path (cell width) usually expressed in centimeters (cm).

Concentration (c): concentration of the absorbent chemical species, usually in grams per liter (g·L-1).

The absorbance of a solution is directly proportional to its concentration—the greater the number of molecules, the greater the interaction of light with them—; it also depends on the distance traveled by the light through the solution; at equal concentrations, the greater the distance traveled by the light through the sample, the more molecules it will encounter; and finally, it depends on "ε," a proportionality constant—called the extinction coefficient—that is specific to each chromophore. Since A is dimensionless, the dimensions of "ε" depend on those of 'c' and "l." The second quantity (l) is always expressed in cm, while the first (c) is described in M whenever possible, so the dimensions of ε are M-1·cm-1. This coefficient, expressed in terms of molar concentration units (or an appropriate submultiple), is called the molar extinction coefficient (εM). When, because the molecular weight of the solute is unknown, the concentration of the solution is expressed in units other than M, for example, g·L-1, the dimensions of ε are different, for instance, g-1·L·cm-1, and the coefficient thus expressed is called the specific extinction coefficient (εs).(3)

METHOD

Sample collection

The sample used was a Certified Reference Material (NIST 699) of calcined alumina (figure 1), provided by CVG Venalum C.A. Upon receipt in the Specials Room (Laboratory Division), it was carefully stored in a desiccator at room temperature to preserve its integrity. This material is crucial to ensure the accuracy of the analyses, guaranteeing the quality and reliability of the results in future studies and applications.

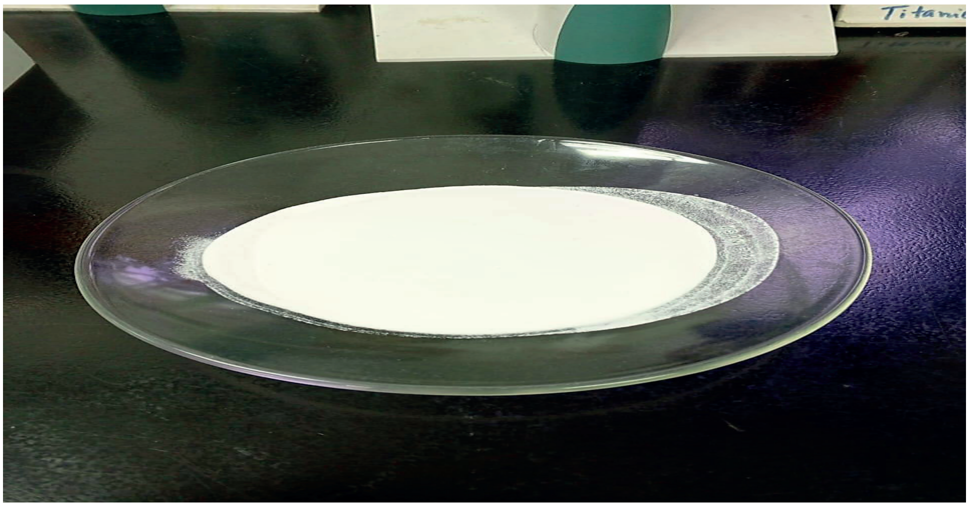

Figure 1. Sample of Certified Reference Material (NIST 699) of calcined alumina

Preparation of alumina solution by alkaline fusion according to ISO 804(6)

1,5 g of the calcined alumina reference standard (699 NIST) was weighed in a platinum capsule. The value was recorded at 0,1 mg, then the analytical balance (Mettler Toledo, AG 245) was tared, and 4,8 g of flux mixture (Na_2CO_3 /H_3BO_3) was weighed, removed and homogenized with the aid of a spatula. The capsule with its contents was placed in a Bunsen burner at a low temperature for 15 minutes; the temperature was increased every 20 minutes, and the temperature was maintained for 60 minutes. It was removed from the burner and placed in a muffle furnace (Thermolyne, Furnatrol, 53600) for 20 minutes. It was removed from the muffle furnace and left to cool to room temperature. Then, the capsule was placed inside a 300 mL beaker, distilled water was added until the capsule was covered, a magnetic stirrer was introduced, and it was placed on a heating plate (Corning) at a gentle temperature until the tablet was completely dissolved. It was removed from the heating plate and cooled to room temperature. The capsule was removed from the beaker with the aid of a rod. Thirty milliliters of concentrated nitric acid (HNO3) was added and heated until the volume was reduced to 100 mL. It was quantitatively transferred to a 200 mL polyethylene volumetric flask and diluted with distilled water.

Calibration for iron (Fe) according to COVENIN 2898(7)

Table 1. Calibration curve parameters for the determination of iron (Fe)

Pattern	Standard solution 10 ppm (mL)	Concentration of Faith (ppm)
White or control	0,0	0,0
1	1,0	0,1
2	2,0	0,2
3	3,0	0,3
4	4,0	0,4
5	5,0	0,5

Starting with a standard iron (Fe) solution of 1000 ppm (Brand). From the previous solution, a standard solution of 100 ppm was prepared by taking 20 mL of the 1000 ppm solution, adding it to a 200 mL volumetric flask, and filling it with distilled water. From the previous solution, a standard solution of 10 ppm was prepared by taking 20 mL of the 100 ppm solution, adding it to a 200 mL volumetric flask, and filling it with distilled water. From the 10 ppm iron (Fe) solution, solutions at 0,1, 0,2, 0,3, 0,4, and 0,5 ppm were prepared, and the aliquots necessary for each concentration, detailed in table 1, were transferred to 100 mL volumetric flasks. To each standard, 5,0 mL of hydroxylamine chloride ($HONH_3Cl$), 25 mL of buffer solution (pH 4,90), followed by 5,0 mL of orthophenanthroline solution ($C_{12}H_8N_2 \cdot H_2O$) were added, stirred and made up with distilled water, then left to stabilize for 60 minutes. Finally, the absorbance was measured in a UV-visible spectrophotometer at 510 nm in a 1,0 cm diameter cell.

Calibration for silica (Si) according to COVENIN 2898(7)

Starting with a standard silicon (Si) solution of 1000 ppm. From the above solution, a standard solution of 100 ppm was prepared by taking 20 mL of the 1000 ppm solution, adding it to a 200 mL volumetric flask, and filling it with distilled water. From the previous solution, a standard solution of 10 ppm was prepared by taking 20 mL of the 100 ppm solution, adding it to a 200 mL volumetric flask, and filling it with distilled water. From the silicon (Si) solution at 10 ppm, solutions at 0,1, 0,2, 0,3, 0,4, and 0,5 ppm were prepared, and the aliquots necessary for each concentration (table 2) were transferred to 100 mL volumetric flasks. The aliquots for each concentration were transferred quantitatively to 50 mL beakers, each standard was diluted to 30 mL with distilled water, then 5,0 mL of ammonium molybdate ($(NH_4)6MO7O24 \cdot 4H_2O$) was added; the pH was adjusted to between 0,85 and 0,90 with nitric acid (HNO_3) solution (1:1) or ammonium hydroxide (NH_4OH) solution (1:10), 5 mL of tartaric acid ($C_4H_6O_6$) and 9 mL of concentrated sulfuric acid (H_2SO_4) were added, it was transferred to a 100 mL polyethylene volumetric flask and allowed to cool to room temperature. Next, 2 mL of ascorbic acid ($C_6H_8O_6$) was added, and the volume was made up with distilled water and then left to stabilize for 20 minutes. Finally, the absorbance was measured in a UV-visible spectrophotometer at 815 nm in a 1,0 cm diameter cell.

Table 2. Calibration curve parameters for the determination of silicon (Si)

Pattern	Standard solution 10 ppm mL)	Si concentration (ppm)
White or control	0,0	0,0
1	1,0	0,1
2	2,0	0,2
3	3,0	0,3
4	4,0	0,4
5	5,0	0,5

Determination of iron (III) oxide (Fe2O3) according to COVENIN 2898(7)

An aliquot of 20 mL of alumina solution was taken and transferred to a 100 mL volumetric flask. Add 5,0 mL of hydroxylamine chloride solution ($HONH_3Cl$), 25 mL of buffer solution adjusted to pH 4,90, and 5 mL of orthophenanthroline solution ($C_{12}H_8N2 \cdot H_2O$), shake, and make up to volume with distilled water. Allow to stabilize for 60 minutes. Finally, the absorbance was measured in a UV-Visible spectrophotometer at 510 nm in a 1,0 cm diameter cell.

Calculations

$$Fe2O3=(Abs/m\times Fd \times V(L))/(Pm\ (mg))\times 100\times Fc \qquad (4)$$

Where:
Abs: Measured absorbance.
m: Pending the straight.
V: Final volume of the solution, expressed in liters (L).
Pm: Sample weight, expressed in milligrams (mg).
Fc: Conversion factor.
Fd: Dilution factor.

Determination of silica dioxide (SiO2)(7)

A 10 mL aliquot of alumina solution was taken and transferred to a 100 mL polyethylene beaker. It was diluted with distilled water to approximately 30 mL, then 5 mL of ammonium molybdate ($(NH_4)6MO7O24$) was added.$4H_2O$), and the pH was adjusted to between 0,85 and 0,90 with nitric acid (HNO_3) solution (1:1) or ammonium hydroxide (NH_4OH) solution (1:10). 5 mL of tartaric acid ($C_4H_6O_6$) and 9 mL of concentrated sulfuric acid ($H2SO_4$) were added. It was transferred to a 100 mL volumetric flask and allowed to cool to room temperature. Then, 2,0 mL of ascorbic acid ($C_6H_8O_6$) was added, stirred, and made up to volume with distilled water. It was left to stabilize for 20 minutes. Finally, the absorbance was measured in a UV-Visible spectrophotometer at 815 nm in a 1,0 cm diameter cell.

Calculations

$\%_{SiO2}$ =(Abs/m×Fd×V(L))/(Pm (mg))×100×Fc (5)

Where:
Abs: Measured absorbance.
m: Pending the straight.
V: Final volume of the solution, expressed in liters (L).
Pm: Sample weight, expressed in milligrams (mg).
Fc: Conversion factor.
Fd: Dilution factor.

Student's t-test

Statistical tests are used to decide whether an experimental value is equal to a theoretical or known value or whether two or more experimental values are identical within a given confidence level; it uses "s" and "x" when reasonable estimates of 'σ' and "μ" are not available.(8) The test is used to determine whether the mean of a sample is statistically different from a known or hypothetical population mean. This test is used when the population does not follow a normal distribution, or the sample size is small (less than 30). The test is based on the calculation of the "t" statistic, which is obtained by dividing the difference between the sample mean and the hypothetical or known mean by the standard deviation of the sample divided by the square root of the sample size.(9) Steps for performing a Student's t-test:

Define the null hypothesis (H0) and alternative hypothesis (Ha) before gathering data. The null hypothesis states that there is no significant difference between the two means, while the alternative hypothesis states that there is a significant difference. Select the appropriate t-test type: this will depend on whether the samples are independent or related. Calculate the mean, standard deviation, and sample size for each group. Calculate the t-statistic using the appropriate formula, which will take into account the difference between the means, the variability of the data, and the sample size. Determine the critical value of t using a Student's t-distribution table and the desired significance level, usually α = 0,05 and degrees of freedom (n - 1).(9,10)

Calculations

$t=(\bar{x}-\mu)/(s/\sqrt{N})=((\bar{x}-\mu)\sqrt{N})/s$ (6)

Where:
t: Student's t-distribution.
$\bar{x}$: Sample average.
μ: Theoretical Value.
s: Sample standard deviation.
N: Sample size.

Table 3. Comparison criteria for the value of the calculated $t_{calculated}$ with the critical $t_{ocritic}$ (9,10)

Comparison	Interception
$t_{calculated} < t_{ocritic}$	If the value of $t_{calculated}$ is less than the value of $t_{critical}$, the null hypothesis cannot be rejected.
$t_{calculated} > t_{ocritic}$	If the value of $t_{calculated}$ is greater than the value of $t_{critical}$, the null hypothesis that the two means are equal is rejected and it is concluded that there is sufficient evidence to state that the sample mean is significantly different from the hypothetical or known mean. The alternative hypothesis is therefore accepted.

Interpret the results appropriately and conclude whether or not there is a significant difference between the two means. In summary, the Student's t-test for one sample is a useful tool for analyzing whether a sample of data is representative of a larger population and for determining whether the difference between the sample mean and the population mean is statistically significant.(9)

RESULTS AND DISCUSSION

Calibration curves for iron (Fe) and silicon (Si) using the external standard technique

For instrument calibration, a technique was used that allowed a correlation to be obtained between the concentrations of iron (Fe) standards and the absorbances. From this correlation, an R2 factor of 0,99891 was derived (figure 2). This value indicates a strong correlation between the iron concentration (ppm) and the measured absorbance, demonstrating the excellent linearity of the calibration curve obtained.

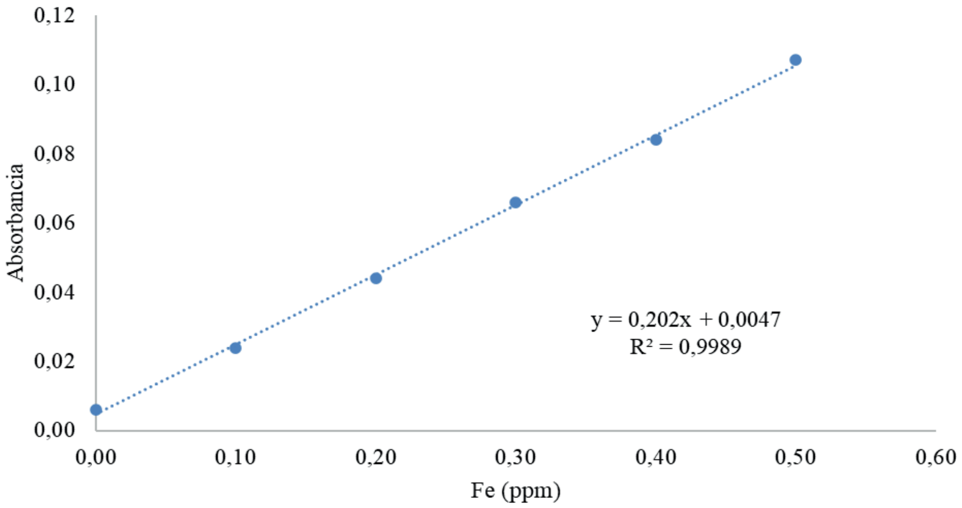

Figure 2. Calibration curve for the determination of iron (Fe)

Regarding the technique used to calibrate the instrument, a correlation was obtained between the concentrations of the silicon (Si) standards and the absorbance values obtained. From this correlation, a correlation factor $R^2 = 0{,}9995$ was derived (figure 3), indicating a strong relationship between the silicon concentration (ppm) and the measured absorbance. Therefore, the calibration curve obtained shows excellent linearity.

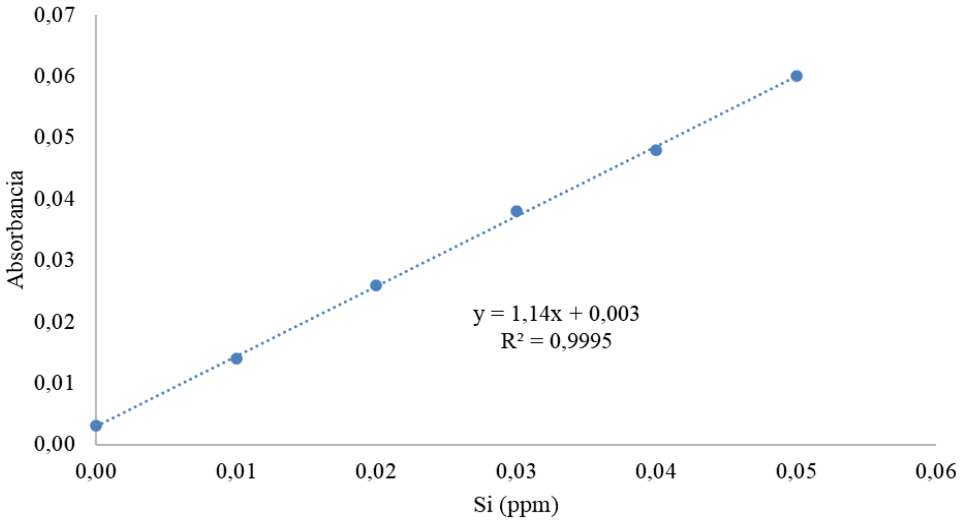

Figure 3. Calibration curve for the determination of silica (Si)

Iron oxide (III) (Fe2O3) and silicon dioxide (SiO2) content in a Reference Material (699 NIST) using UV-Visible spectrophotometry

The analyses were performed on the Reference Material (699 NIST). The iron oxide (III) content ($\%_{Fe2O3}$) reveals remarkable consistency among the eight replicates. The $\%_{Fe2O3}$ values are predominantly clustered around 0,0132 %, observed in replicates 1, 3, 4, and 8, suggesting a well-defined central trend. Although slight variations are observed, with three replicates (5, 6, and 7) recording 0,0127 % and replicate 2 presenting the lowest value of 0,0122 %, the total range of dispersion is extremely narrow. This minimal oscillation between the maximum and minimum values (0,0132 % - 0,0122 % = 0,0010 %) strongly indicates the high precision and reproducibility of the analytical method used to quantify iron oxide. The uniformity of the results minimizes the influence of random errors, providing confidence in the reliability of the iron (III) oxide measurements. Similarly, the %SiO_2 values analysis demonstrates excellent precision and consistency. Most replicates (1, 2, 4, 6, and 7) show an identical value of 0,012 %, establishing this as the most frequent value. Only replicates 3, 5, and 8 deviate slightly, recording a value of 0,013. This minimal variation between the maximum and minimum values (0,013 % - 0,012 % = 0,001 %) underscores the robustness of the analytical procedure used to determine silicon dioxide (figure 4). The high agreement between the measurements in the different replicates minimizes the uncertainty associated with the results, reinforcing the validity of the data obtained and highlighting the efficiency of the %SiO_2 measurement process.

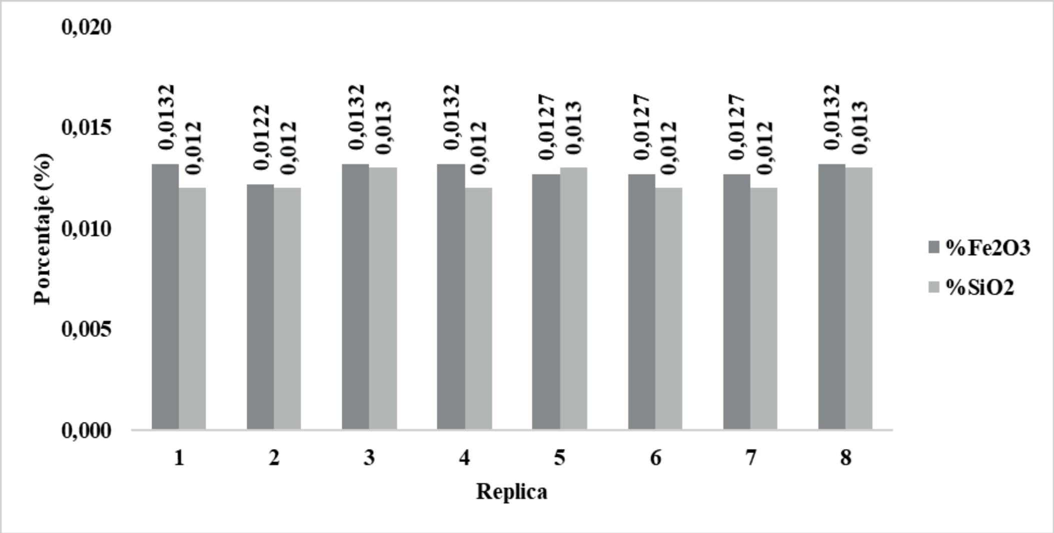

Figure 4. Experimental results in the determination of iron (III) (Fe_2O_3) and silicon dioxide (SiO_2)

The statistical data for iron oxide (III) (Fe_2O_3) and silicon dioxide (SiO_2) (table 4) show a low standard deviation (s) of 0,0005 % for both compounds. This suggests that the method is highly reproducible and that random errors are minimal. Therefore, the results obtained allow us to interpret that there is excellent precision in the determination of Fe_2O_3 and SiO_2 contents. In summary, the results demonstrate the feasibility of the method for the quantitative determination of Fe_2O_3 and SiO_2 in certified reference material of calcined alumina. These data, being accurate and reproducible, support the reliability of the results.

Table 4. Summary of the percentage composition of iron oxide (III) (Fe_2O3) and silicon dioxide (SiO2) in the certified reference material of calcined alumina

Substance	N	Media (%)	Standard Deviation (%)
Iron oxide (III) (Fe_2O_3)	8	0,0129	0,0005
Silicon dioxide (SiO_2)	8	0,0124	0,0005

Evaluate the statistical feasibility of calibration models for iron (Fe) and silicon (Si) by applying a Student's t-test

Table 5. Results of the Student's t-test for the concentration of iron oxide (III) (Fe_2O3) and silicon dioxide (SiO_2) in the certified reference material of calcined alumina

Substance	N	Media (%)	Standard deviation (%)	Theoretical value (%)	$t_{critical}$	$t_{calculated}$
Fe_2O_3	8	0,013	0,00046291	0,013	2,3646	-1,5275
SiO_2	8	0,012	0,00051755	0,012	2,3646	2,0494

The results of the t-test used to compare the mean of a sample (table 5). For iron oxide (III) (Fe_2O3) and silicon dioxide (SiO_2), a critical t-value of 2,3646 was established. This value corresponds to a confidence level of 95 % and 7 degrees of freedom. Specifically, for iron oxide (III) (Fe_2O3), a $t_{calculated}$ value of -1,5275 was obtained, while for silicon dioxide (SiO_2), a $t_{calculated}$ value of 2,0494 was obtained. In both cases, the $t_{calculated}$

value is less than the tcrit value. Since the value of $t_{calculated}$ is lower than the value of $t_{critical}$, the null hypothesis is not rejected. This implies that there is no statistically significant evidence to claim that the mean of the values obtained differs from the theoretical value. Therefore, the results obtained are consistent with the expected theoretical value. Consequently, the method used to determine the concentration of iron (III) oxide (Fe_2O3) and silicon dioxide (SiO_2) works adequately and reliably.

CONCLUSIONS

The calibration curves for iron (Fe) and silicon (Si) showed excellent linearity, with R2 values of 0,99891 and 0,9995, respectively. This indicates a strong correlation between concentration and absorbance and confirms the accuracy and reliability of the instrument for quantifying both elements. The analytical method demonstrates excellent precision and reproducibility for the quantification of iron (III) oxide (Fe_2O3) and silicon dioxide (SiO_2) in calcined alumina, with low standard deviations that minimize random errors and support the reliability of the results, suggesting that the method is highly reproducible and accurate. The Student's t-test in the analyses of iron oxide (III) (Fe_2O3) and silicon dioxide (SiO_2) demonstrate that the method is reliable, as the experimental values do not differ significantly from the theoretical values. The update of the calibration curves for iron and silicon in calcined alumina using UV-visible spectrophotometry was successfully achieved and proved to be a highly accurate and reliable method. The excellent linearity of the curves (R2 of 0,99891 for iron and 0,9995 for silicon) confirms the instrument's reliability. Furthermore, the low standard deviation in the quantification of Fe_2O3 and SiO_2 in calcined alumina, together with the Student's t-test results, validate the method's accuracy and reproducibility. In conclusion, the updated calibration curves are suitable and reliable for the routine analysis of these elements in calcined alumina.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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