A New Brazilian Calcined Refractory Grade Bauxite

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Bauxite ores present a wide range of technological applications according to their chemical and mineralogical compositions. In the refractory industry, this raw material has been used in high-alumina refractories, substituting partially or totally other alumina sources in bricks and castables due to its high refractoriness and relatively low cost. Worldwide, there are few refractory-grade bauxite (RGB) mines spread out with remarkable presence of manufacturers in China, Russia, Guiana and India. This paper presents a new Calcined RGB from a Brazilian source with outstanding properties. The characterization of this Calcined RGB reveals high alumina content (~85%) and refractoriness (>1800°C) besides the high density (low porosity) and homogeneity of grains which are mainly constituted by corundum and mullite crystalline phases with iron retained mainly as solid solution. It is important to mention the absence of titanium phases and low amounts of alkaline oxides assuring good performance at high temperatures.

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Introduction

Bauxite is a sedimentary rock also known as aluminum ore being composed mainly by minerals of aluminum hydroxides, containing secondary minerals in variable amounts, based on silica, iron oxides and hydroxides, titania and hydroaluminosilicates. Bauxite mines are normally large opencast operations using surface methods, which involve the removal of topsoil and overburden by bulldozers and scrapers, to expose the underlying bauxite. Selective mining techniques are used to produce various grades of ore, which are sorted according to chemical composition, and then used separately or blended if required. There is a wide variation in bauxite ores, and the ore quality will determine the exact mining and processing route [1].

In the world, around 95% of the bauxite produced is used in alumina refineries (“metallurgical grade-bauxites”), and the remaining 5% is used in numerous non-metallurgical applications, including proppants (20%), Portland cement (20%), brown fused alumina - BFA (17%), refractories (16%) and others [2]. There are relatively few producers of non-metallurgical bauxites and only a handful of these make grades suitable for calcining aiming refractory applications [1].

Refractories demand is largely determined by steel and iron production (73%), followed by cement and lime (13%), and non-ferrous metals (4.5%). The rise in refractory demand is offset slightly by an overall reduction in the specific consumption of refractory per ton of steel produced, as steel production methods and refractory product quality improve. Increased energy cost is a key issue for refractory mineral production. This cost makes it more expensive to produce fused minerals and encourage the use of alternative minerals, such as calcined bauxites with refractory grades [3]. As the energy price increases, the efforts of refractory producers to find cheaper and more cost effective substitutes grow at the same pace.

Guiana has been the pioneer of refractory-grade bauxite since 1940´s and after became available from Suriname, and in the 1970´s from China. Afterwards, other quantities were found in Brazil [4]. Nowadays, the main producers of refractory-grade bauxite are based in China, and the country accounts for over 85% of production [3]. In a column published in the American

Ceramic Society Bulletin, McCracken showed a serious concern about where raw materials really come from and how much they really cost, reporting what he would say to be a major world supply problem on refractory-grade bauxite, warning the buyers who migrated to Chinese market, willing to take advantage of the giveaway low prices. In outburst tone, he remarked issues related to processing, quality control and logistics [5]. Sutton wrote that concerns remain in the industry over the availability and security of calcined RGB supply [6].

At present, a wide range of natural and synthetic raw materials have found use in the production of aluminosilicate refractories. The wear resistance and service efficiency of components and products based on the Al_2O_3-SiO_2 system is mainly determined by the chemical and mineral composition of the starting raw materials. The technological usefulness of natural raw materials increases with their content of Al_2O_3 in ascending order: pyrophyllite → kaolinite → bauxite clay → kyanite, sillimanite, andalusite → refractory-grade bauxite. The corundum (61.4) and mullite (48.0) phases present a high energy density (kJ/cm^3) meaning that a high energy is required for complete destruction of a unit volume of material. Hence, the energy density of alumina-containing materials bears direct relevance to their other properties of practical importance such as enhanced resistance to the corrosive attack by slags, inertness, low thermal expansion, high strength and hardness, thermal shock resistance, thermal stability and refractoriness. There are good reasons to believe that further progress in the field will be achieved by increasing the use of high-alumina refractories at the expense of materials of acid and basic composition. One may assert, with some reservation though, that the unique physicochemical properties (amphoteric) of Al_2O_3 and related compounds provide a wide range of practical applications, including refractories [7]. Refractory-grade bauxites potentially have a high refractoriness (~1840°C) as it can be deduced from the system Al_2O_3-SiO_2 (figure 1) where these materials are represented on the basis of their two main constituents: alumina and silica. However, although in practice their refractoriness is high (>1700°C), under load is low (1450-1550°C) even when compared with other refractory products of lower alumina content. This behavior is normally attributed to the presence of a liquid phase at the last mentioned temperatures due to the impurities present in the bauxites, mainly TiO_2 and Fe_2O_3. Bauxite samples heated at 1500-1550°C show that the liquid content at these temperatures is normally lower than 10%. At first this quantity does not seem to justify such a low refractoriness under load, unless the distribution of liquid between the solid phases, more than its amount, determines this behavior [8].

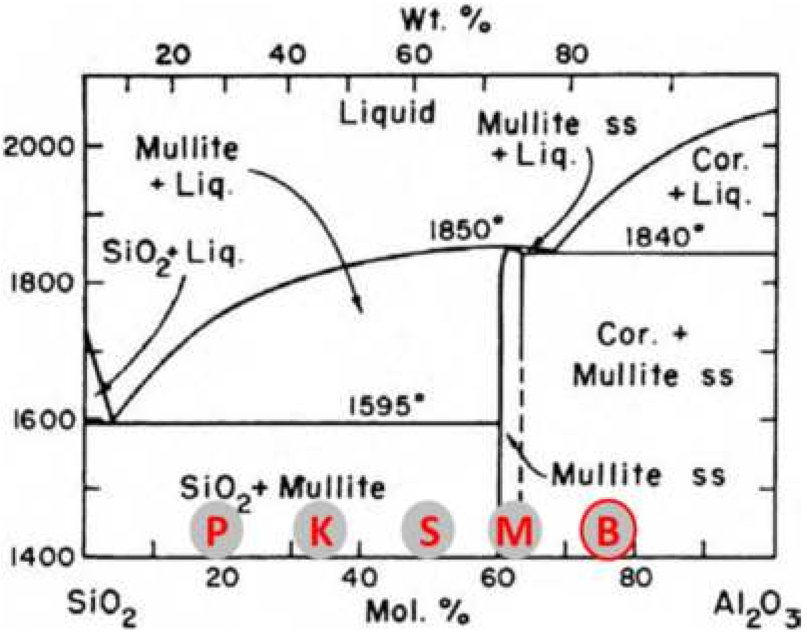

Fig. 1 Stable and metastable phase equilibrium in the Al_2O_3-SiO_2 system, showing the position of typical raw materials used in aluminosilicate refractories production, where P = Pyrophyllite; K = Kaolinite; S = Sillimanite; M = Mullite 3:2; B = Bauxite - RGB *[adapted from 8 and 9]*.

The major requirements for high-quality bauxite aggregates are low porosity, good creep resistance, high-resistance to hot-slag/metal corrosion and erosion, homogeneous phase microstructure and aggregate size variety to allow good packing properties.

The microstructure of calcined bauxites is comprised mainly of corundum (α-Al_2O_3) grains surrounded by a matrix which may contain, among other phases, mullite crystallites, tialite, glassy phase and iron either as oxide compounds or in solid solution [10].

The thermal stability of Calcined RGB depends primarily on the alumina content, since this is the most refractory phase, melting at approximately 2050°C. The presence of mullite in bauxites is favorable because it is associated with high resistance to thermal shock, high refractoriness, good mechanical strength and high-creep resistance. The acicular shape of mullite crystallites yields a three-dimensional network that prevents shearing and deformation at high temperatures [11].

Impurities

Most impurities in bauxites combine to form low melting point (mp) phases, particularly alkali and alkaline earth compounds (Na_2O, K_2O, MgO and CaO). Even though the fluxes work to enhance densification rates on sintering, their presence is detrimental to the physical properties of refractories. Moreover, fluxes promote lower service temperature, lower resistance to corrosion and to slag attack, and increase creeping mechanisms.

Fe_2O_3 and TiO_2 content can vary widely in different bauxites. Iron is considered undesirable because it can contribute to lowering melting point. Whereas, this concern is more valid if iron is in the reduced form (Fe^{2+}). This occurs because of the smaller size of Fe^{3+} in the former which allows it to be easily incorporated into the crystal structure of other compounds as solid solution [10]. Besides hematite phase (Fe_2O_3) also is considered a refractory material (mp = 1595°C) TiO_2 can be found either as tialite (Al_2TiO_5) or rutile (TiO_2). Tialite melts at 1850°C; however, its presence is not desired due to its low mechanical strength and to its thermal instability in the temperature range 750–1350°C which leads to the decomposition [12]. In addition, tialite exhibits anisotropic thermal expansion. On cooling from the firing temperature the expansion mismatch between the grains result in stresses and microcracks formation, impairing thermal shock resistance of refractory [13]. During sintering, densification mechanism and chemical reaction compete against each other, with initial increase of bulk density irrespective of the titania content, until a certain level of temperature. Beyond this temperature, the bulk density decreases due to excessive amount of liquid formation. Gradual addition of TiO_2 up to 4% promotes the densification process (lowering the densification temperature to 1500°C); however it causes much deterioration in high thermal properties [14].

Variance in Calcined Refractory Bauxites

In a recent and interesting work, Russian researchers studying molded and unmolded refractories presented different varieties of Chinese Calcined RGB from the same source as basic raw material components having markedly different chemical and mineral compositions. For example, a difference in the main oxides content, Al_2O_3 e SiO_2, reached 4.5 and 4.0% respectively. Such a marked difference is typical for the Chinese Calcined RGB composition, where the difference in corundum content between the lowest and highest was 10%, and for mullite the difference was about threefold. In view of this, original bauxite is blended generating an average value in Calcined RGB. Another typical feature of the Chinese Calcined RGB is the different color of grains: black, brown, grey, beige, white, often a combination of these types. Each grain has its own but variable substance composition, extremely nonuniform microstructure, and sharply differing pore structure. As an example, the porosity for grains of different color varied from 2-4 to 15-17%. Also the mineral composition of differently colored grains varied over wide limits. The variable mineral composition, microstructure and porosity of grains have an unfavorable effect on a number of physicochemical properties of high-alumina refractories. Significant phase and structural heterogeneity of grains is due to the geology of the Chinese bauxite deposits and also raw material treatment technology, in particular lack of necessary blending before firing and the use of shaft furnaces in some plants. As a result of this, refractory objects or concretes based on Chinese bauxite in spite of a high alumina content (86-90%Al_2O_3) have relatively low temperature for deformation under load (<1440°C) [15].

A notorious work about the constitution of Calcined RGB was done by Caballero et al [8]. Four calcined RGB from China (two samples), Suriname and Guyana were studied in chemical, mineralogical and microstructural constitution, being results interpreted considering phase-diagram systems to the understanding about the relationship between constitution and thermomechanical behavior. In this study, all samples had Fe_2O_3 of less than 2.0% and TiO_2 between 2.5 - 6%. For all cases, the theoretically expected phases were corundum, mullite and tialite, and in practice all samples presented rutile meaning that the equilibrium wasn´t achieved due to the non-homogeneity of the raw materials, insufficient calcination temperature and/or soaking and thermal gradients between the outer and the inner part of the bauxite pellets. The data showed that Chinese was the worst calcined bauxite considering the degree of calcination of them. Moreover, the non-homogeneity (microstructural heterogeneity) of these raw materials determines the thermomechanical behavior of bauxite aggregates. Therefore, if a high mechanical stress is applied at these temperatures, all the regions in the aggregate having the glassy phase will deform and likewise the aggregate itself. These situations are worse considering the effect of impurities in lowering the temperature of liquid formation.

The last but not the least, is a sequence of papers published by Brazilian researchers regarding a review of applications in RGB [16-18]. These authors affirm that Chinese raw materials is difficult for processing due to its prime phase diaspore [AlO(OH)] in crude bauxites, contrary to South Americans´ bauxites that contains gibbsite [$Al(OH)_3$] as main source of corundum. The high content of alkalis present in Chinese raw materials compared to South American bauxites implies higher liquid phase content at high temperature being harmful to the mechanical properties of the refractory. The higher content in TiO_2 is also harmful causing cracking and ruptures of pieces due to highly anisotropic thermal expansion which happens in the cooling process of calcined grains. Comparative studies of refractory bauxites from different suppliers from China, Guiana and America showed TiO_2 contents between 2.9 to 4.2% resulting in tialite phase varying from 5.7 to 9.8%. One Brazilian bauxite was also included in the study having 2.2%TiO_2 without identification of tialite. They also observed that higher silica content in set of lower alkalis content produced a higher amount of mullite and lower concentration of glassy phase for the Brazilian sample generated by rotary kiln efficiency which stimulated decreasing in porosity to around 10% against 15% from other countries suppliers [16-18].

In this paper the authors present the properties of a new calcined refractory-grade bauxite which has been produced by Mineração Curimbaba Ltda (MC), using a Brazilian source of RGB as raw material.

Experimental Procedure

The production of the calcined refractory-grade bauxite (RGB) utilized a blend of Brazilian gibbsitic bauxites with high alumina content and low amount of impurities. The raw material was dried, homogenized, milled and then mixed in a wet route before conformation. After conformation the material was dried and sintered in a rotary kiln reaching temperatures over 1650°C. After cooling the product was crushed and finally sieved in vibratory screens to produce different split sizes. The splits were submitted to several physicochemical analyses aiming the characterization as aggregate for the refractory industry. Figure 2 summarizes the production process of the Calcined RGB produced at the Mineração Curimbaba facility in Brazil.

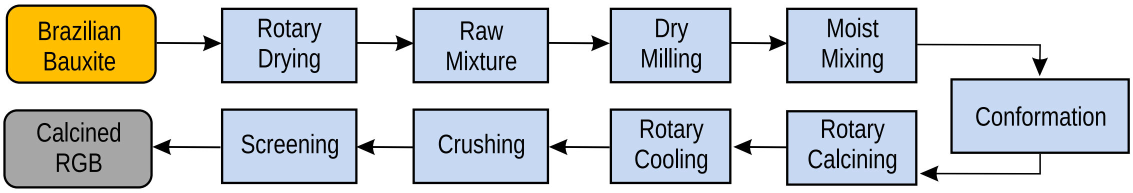

Fig. 2 Flowchart for the Calcined RGB production at Mineração Curimbaba

Split sizes obtained during the process were sampled following internal standard procedures of quality assurance. The RGB grains were characterized chemically and mineralogically. Density, porosity and refractoriness were also measured. A high alumina LCC (Low Cement Castable) refractory formulation was prepared to produce a refractory brick for evaluation of refractoriness under load (RUL).

All tests using the Brazilian Calcined RGB were compared with a Chinese Calcined RGB, following the same methods for data collection and analyses as long as refractory formulations.

The chemical composition was determined by X-ray fluorescence spectrometry (XRF-1800, Shimadzu) with sample preparation by the glass bead method in an automatic fusion machine of high frequency induction (HA-HF 16/2, Herzog). The mineralogical composition was analyzed by X-ray powder diffraction (XRD-6000, Shimadzu) with CuKα radiation scanning 2θ region from 14° to 80°, using the Rietveld method (RM) of crystalline structures refinement for quantitative phase analysis (QPA) and microstructural evaluation of the Calcined RGB through the GSAS software and EXPGUI user interface [19, 20]. The bulk density and apparent porosity were determined by the hydrostatic balance according to ISO 8592 [21]. Finally, the refractoriness was determined by the pyrometric cone equivalent (PCE – ASTM C16) [22] and a traditional brick was prepared utilizing the Calcined RGB as aggregate (0.044mm to 9.5mm) bonded in matrix of CAC, microsilica (MS) and tabular alumina for the RUL test (ASTM C16) [22].

The firebricks were conformed in a LCC formulation with firing at 1000°C during 5 hours.

Results and Discussions

The Calcined RGB produced presented an outstanding homogeneous aspect being composed by grains with same color tones due to reinforcements of milling and homogenization during all process long since the beginning with crude Brazilian bauxite selection and in the stream of process using rotary thermal techniques. The Chinese RGB presented particles with inhomogeneous colors.

This homogeneity in color is related with microstructural and chemical uniformity of the Brazilian RGB that means homogeneity in the different sizes of the aggregate granting a good refractory behavior for firebricks and refractory castables.

According to the typical chemical composition achieved (table I), comparing the Brazilian Calcined RGB and Chinese Calcined RGB, they can be classified as a high-alumina bauxites with 80-90wt% alumina content. It is noted the low impurities concentration, especially the alkali ones (~0.15%). For the Brazilian RGB, it was also expected that the percentage of silica would promote an intermediate concentration of mullite, being capable to keep titanium and iron in solid solution, preventing mainly the tialite formation which impairs the mechanical resistance of the refractory.

The mineralogical composition (figure 3) confirms the main phases expected for the Brazilian Calcined RGB, having corundum as the major phase (78.8%) and mullite as a secondary phase (19.4%). As a minority phase, the accurate mineralogical evaluation shows hematite (Fe_2O_3) obtained in low concentration of 1.80%. In addition to this, the X-ray diffractogram indicates good crystallinity of the Calcined RGB denoting the low content in glassy phase, in agreement with the silica content required to form mullite. By the stoichiometry of the phases, there is only a small quantity (<2%) of silica available to form glassy phase.

For the Chinese RGB, the quantitative analysis presented corundum (79.7%), mullite (15.4%), tialite (4.39%) and rutile (0.49%). These mineralogical results are in compliance with table I, since they show the formation of tialite and rutile for Chinese sample (sum = 4.9%) against the hematite (1.8%) formation for the Brazilian sample emphasizing the solid solution formation with the impurities (Fe_2O_3 + TiO_2) in the mullite for the Brazilian RGB.

Tab. I. Chemical composition of Calcined RGBs (%).

	Al_2O_3	SiO_2	Fe_2O_3	TiO_2	RO*	R_2O**	Others
Brazil	84.8	8.06	3.80	1.63	0.39	0.14	1.18
China	86.5	7.01	1.60	3.59	0.45	0.15	0.7

*RO = Cao + MgO; **R_2O = K_2O + Na_2O

Patent Pending. Released in September, 2017.

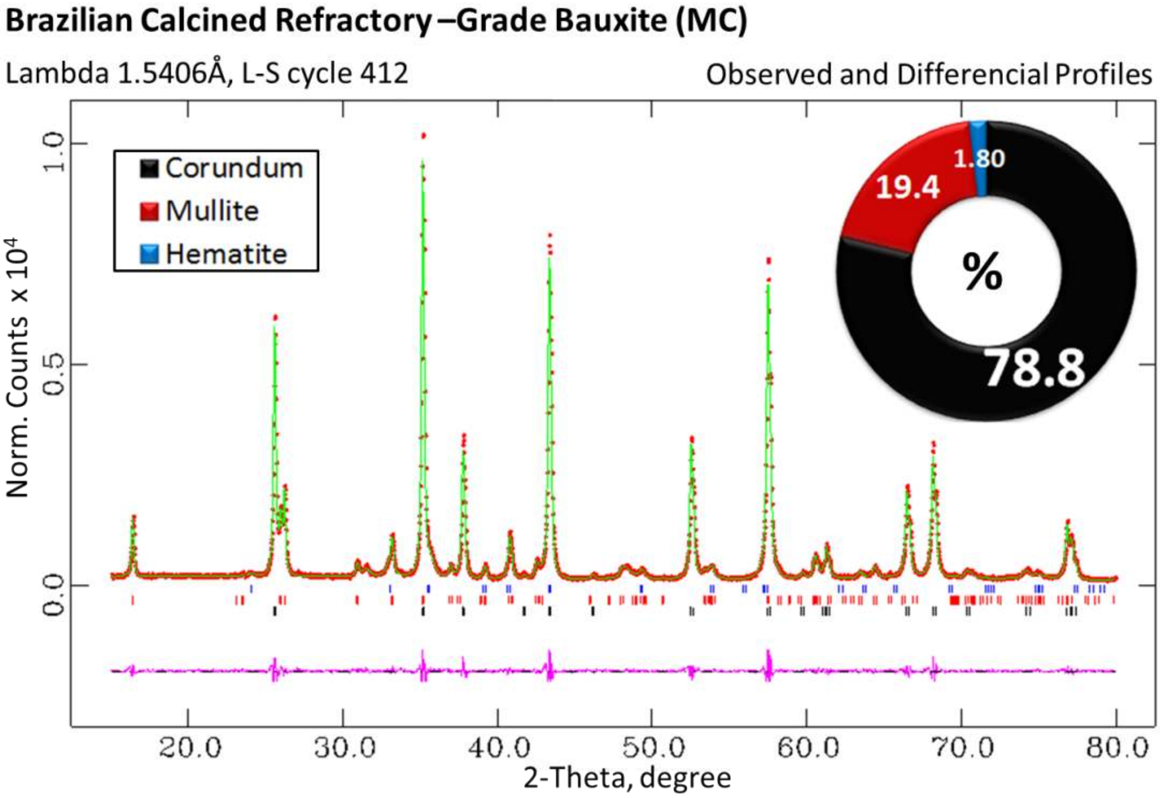

Fig. 3 Rietveld graphic and mineralogical composition of the Brazilian Calcined RGB after XRD characterization

It is extremely important to note that the excess of mullite for Brazilian RGB is not only related to the silica and alumina contents, but to the unique mineralogical characteristic of the Brazilian gibbsitic bauxite. In this case, the formation of mullite occurs in three stages, having the kaolinite as origin in raw mixture. In the first stage (1000 to 1500°C), metakaolinite [$Al_2O_3.2SiO_2$], which is an amorphous phase, is formed below 1000°C due to the dehydration of kaolinite [$Al_2Si_2O_5(OH)_4$]. In the second stage, metakaolinite produces the mullite phase with 3:2 stoichiometry ($3Al_2O_3.2SiO_2$) and a liquid phase due to the excess of silica released from the kaolinite. Simultaneously, there is the stabilization of alpha-alumina (α-Al_2O_3) in the corundum phase after a series of transformations that brings the gibbsite to the most stable alumina phase (corundum). Thus, the main advantage of having these series of transformations lies in the preventing the growth of corundum grains in this second stage, since densification in the process of firing will compete with the chemical reaction between alumina (corundum) and silica (glass). As the grains are still small, there will be reactivity of the corundum phase with the excess of silica concentrated in the liquid phase, consequently generating a solid solution (S.S) rich in alumina in the third stage, the as called 2:1 mullite ($2Al_2O_3.SiO_2$).

Although there is a considerable concentration of impurities (Fe_2O_3 + TiO_2) in the Brazilian bauxite, only a small amount (1.80%) is in the hematite phase (Fe_2O_3). The balance, although, is dispersed in a solid solution with the corundum phase and mainly in the mullite as it will be shown.

A simplified scheme of the main reactions that occur during the calcination of the Brazilian grade refractory bauxite is presented in figure 4.

For the Chinese bauxites, the same mechanism does not occur since the transformation from the initial phase (diaspore) to corundum involves less steps, causing the well-developed corundum phase in the second stage and preventing the reactivity of the alpha-alumina with the glassy phase. Additionally, even with a concentration of impurities lower than the Brazilian RGB, during the production process occurs the formation of the tialite phase ($Al_2O_3.TiO_2$) in considerable content (4.39%) impairing the high temperature properties of the refractory even though with a high chemical concentration of alumina (> 85%) in the RGB.

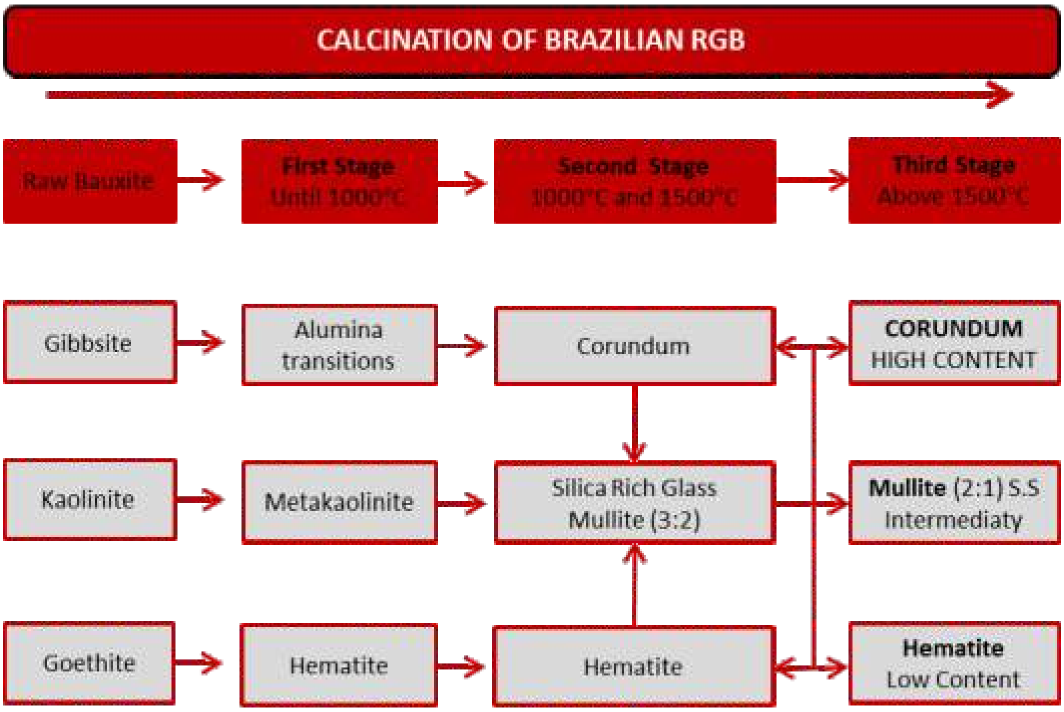

Fig. 4 Firing: Reaction Sequence of Solid State in Brazilian Refractory-Grade Bauxite.

The solubility of titanium and iron oxides from South American refractory bauxites was studied by Schneider and Wohlleben [23]. This study provides a linear relation between the amount of impurities present in the mullite and the lattice parameters of the phase allowing the estimation of Fe_2O_3 and TiO_2 content in solid solution. The various bauxite samples taken in this study were separated by color where the mullite phase presented in light-colored fragments (low impurities) showed lattice dimensions close to the 3:2 mullite configuration. By increasing the impurity content in darker color samples (and consequently decreasing the SiO_2 content), the lattice parameters expand and the configuration approximates the 2:1 stoichiometric ratio. The researchers used bauxites which sum of impurities (Fe_2O_3 + TiO_2) in the mullite phase ranged from 0.43 to 6.9%. Thus it is interesting to compare the refinement data of crystalline structure by RM with those found by these authors. For this comparison, initially it was plotted the results encountered by Schneider and Wohlleben (blue points) being obtained a linear correlation (figure 5). Hence, it was obtained the linear equation and added in the graphic the cell parameter b of mullite (red point) obtained in RM calculations with XRD data for the Brazilian calcined RGB.

Figure 5 shows that most of the impurities (Fe_2O_3 + TiO_2) were incorporated in the orthorhombic structure of the mullite, since the value achieved through the Schneider and Wohlleben correlation was higher than 7%, surpassing the sum of Fe_2O_3 and TiO_2 found in the typical chemical analysis for Brazilian Calcined RGB (table I). This super estimated value based in the linear correlation can be related to the different firing conditions used in Curimbaba process which stimulate the growing in comparison to calcined samples used in the study from those authors [23]. Whereas it was clear the solid solution formation in mullite through the increase in cell parameter caused by the presence of Fe^{3+} and Ti^{4+} in the crystalline structure. This solid solution is very important to not forming tialite and not releasing these oxides to form glassy phase.

An important comparison between the unit cell volume for the two main phases was done showing the greatest solubilization of the impurities in the Brazilian sample. The higher volume of corundum (255.491$Å^3$) and mullite (168.941$Å^3$) unit cells for Brazilian RGB was observed in comparison with Chinese sample (255.208$Å^3$ and 168.475$Å^3$, respectively), indicating a higher solubility of the impurities in the corundum and mullite structures in the Brazilian RGB.

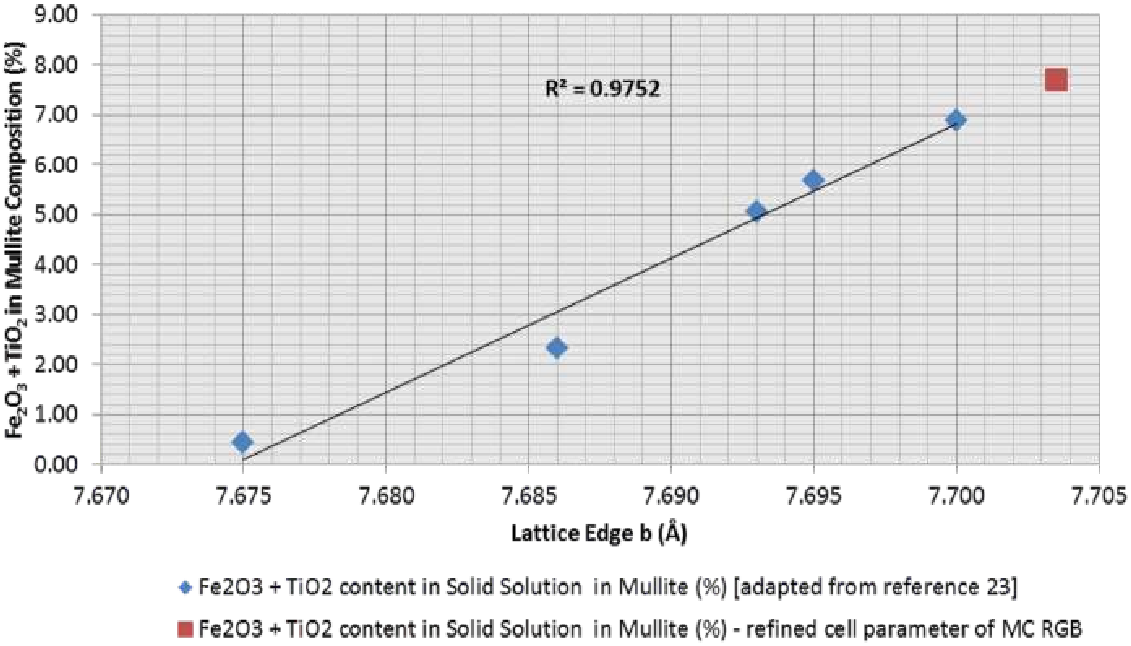

Fig. 5 Correlation between the lattice edge b and the Fe_2O_3 + TiO_2 content in solid solution in mullite [adapted from 23].

Regarding to the physical characteristics, both samples presented apparent porosity <8.00%, but the Brazilian Calcined RGB presented a notorious higher bulk density (3.21g/cm^3) conferring superior physical characteristic to the product developed (Table II).

Tab. II. Apparent Porosity (AP) and bulk density (d_{bulk}) for Calcined RGBs.

RGB/Property	AP	(d_{bulk})
Brazil	7.88%	3.21g/cm³
China	6.38%	3.01g/cm³

The refractoriness was determined in the sample ground at minus 70 mesh screen by Orton pyrometric cone equivalent (PCE), with plastic deformation not observed until 1804°C (Orton cone equivalent 36 as reference) for both samples. The refractoriness under load (RUL) test was performed by Orton Ceramics Materials Testing and Research Center - USA in two ordinary bricks sent for each RGB (Brazilian x Chinese).

In the case of the Brazilian RGB, both specimens supported the load of approximately 340lb in a furnace at 1600°C with soaking time of 1.5h, without cracking or rupture after completed test. The Chinese material could not support the load and test conditions and were completely destroyed during the execution of the test run.

Figure 6 presents two bricks produced with each RGB (Brazilian and Chinese) after the RUL test as previously described.

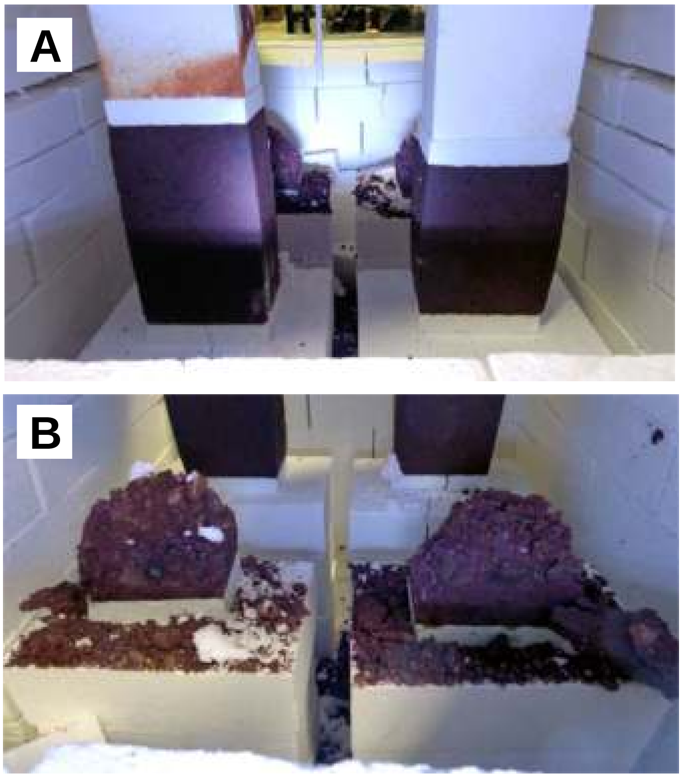

Fig. 6 Hot load test – ASTM C16. A) Specimens Brazilian RGB-1 and Brazilian RGB-2 inside the furnace after 1.5 hours at 1600°C; B) Specimens Chinese RGB-1 and Chinese RGB-2 inside the furnace after 1.5 hours at 1600°C.

These results demonstrated the importance of tialite absence and mullite presence in an intermediate content as a toughening agent around the homogeneous matrix of corundum. After manufacturing a shaped brick with LCC, the Chinese sample still with higher alumina content and lower porosity of the aggregates did not support the RUL test.

Conclusions

The new Calcined RGB from Mineração Curimbaba has been presented as an excellent alternative to the current RGB present in the market. The Brazilian RGB is more homogeneous and most of its impurities are forming solid solutions with mullite and corundum phases not impairing its thermomechanical properties. The low alkali content, typical of the Brazilian bauxites, also promotes chemical stability and high performance of the product.

The high alumina content (80-90%), the absence of the tialite and a suitable concentration of mullite (19.4%) in a homogeneous matrix combined with high density and low porosity makes the Mineração Curimbaba RGB one of the best products available in the market for the refractory industry.

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