

# Controlled Reduction of Iron Content in Zirconium Ore: A Case Study in Polyphase Mineralization, Poços de Caldas Alkaline Plateau

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**Abstract:** In PCAC (Poos de Caldas Alkaline Complex), in southeastern Brazil, it is observed a polyphase mineralization related to Zr-, U-, Th-, Mo-, and REE-enrichment due to hydromothermal processes which affected alkaline primary rock. Primary Zr-minerals were leached and concentrated as “caldasite”, a rock composed mainly by zircon and baddeleyite in different proportions. Several techniques of mineralogical characterization were applied and results indicated zircon, baddeleyite, magnetite and iron-oxyhydroxides, mainly. Magnetic separation by WHIMS (Wet High-Intensity Magnetic Separation) was performed in order to indicate the efficiency for Fe-concentration removal for potential application in refractory industry.

**Key words:** Zircon, baddeleyite, caldasite, Poos de Caldas alkaline massif, Brazil.

## 1. Introduction

The PCAC (Poos de Caldas Alkaline Complex) is circular with a mean diameter of approximately 33 km, and it is situated about 220 km north of So Paulo in SE-Brazil. PCAC was geologically studied and mapped by Ref. [1], analyzed structurally on the basis of remote sensing by Ref. [2] and geophysically investigated by Refs. [3-5]. Known mineral deposits are of hypogene and/or supergene origin, including bauxite, clays, Zr, Zr-U-Mo, F and Th-REE(-Fe) mineralizations [6-12]. The petrographic associations of Poos de Caldas alkaline rocks are rather complex and as yet unique in Brazil [13]. Potassium-rich phonolites and nepheline syenites are the predominant rock types, with only minor occurrences of eudialyte-bearing nepheline syenites (Fig. 1) [14]. The

PCAC suffered widespread post-magmatic deuteric alteration. At several specific sites there also occurred additional intensive hydrothermal rock/meteoric water alterations, producing low- to medium-grade U-Th-Zr-REE-Mo-F-pyrite mineralizations. Sites typically show the combined effects of argillation and potassic alteration (kaolinite, illite and k-feldspar formation), pyritization and variable U-oxides, jordisite (MoS<sub>2</sub>), sphalerite, zircon-baddeleyite (ZrO<sub>2</sub>), REEs, fluorite, and pyrite mineralizations. Mineralization can be disseminated without any evident structural control, or is associated with either fractured vein/stockwork systems, or local volcanic breccias. The typical wall and host rock is always altered by K-metasomatism, leading to the formation of so-called potassic rock [6], i.e., hydrothermally altered phonolites and nepheline syenites with K<sub>2</sub>O contents normally in excess of 12 wt.%. Hydrothermal mineral occurrences include vein mineralizations of “caldasite” (intergrowth of Zr-minerals such as zircon and

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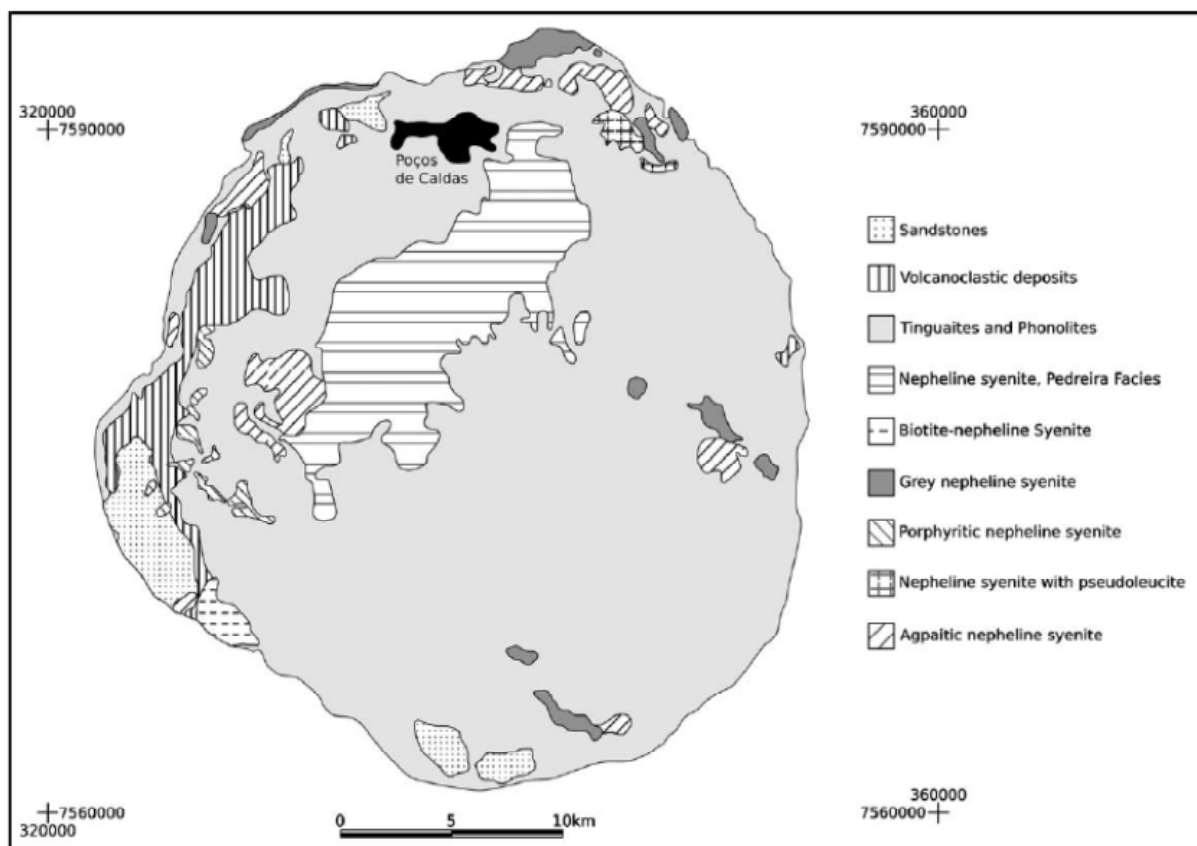
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baddeleyite) [14].

The mineralization assemblage includes minerals that were formed during the different stages of hydrothermal event and are found as disseminated impregnation in the rocks and/or vein and void infillings, such as pyrite, zircon and Zr-minerals (“caldasite”), fluorite, REE phases, baryte, carbonates, molybdenum minerals and clay minerals [15]. This paper focuses on the mineralogical and technological characterization of “caldasite” due to commercial significance for Zr as refractory material. The so-called “caldasite” was firstly described by Ref. [16] as a uraniferous zirconium ore, a mixture of zircon ( $\text{ZrSiO}_4$ ) and baddeleyite ( $\text{ZrO}_2$ ), with an average zirconium content higher than 60%  $\text{ZrO}_2$  and uranium content of 0.3%  $\text{U}_3\text{O}_8$ . Ref. [16] presented chemical analysis of “caldasite” with contents of  $\text{U}_3\text{O}_8$  (0.3%),  $\text{ZrO}_2$  (61.2%),  $\text{HfO}_2$  (0.9%),  $\text{TiO}_2$  (0.65%),  $\text{Al}_2\text{O}_3$  (3.0%),  $\text{Fe}_2\text{O}_3$  (7.7%),  $\text{MnO}$  (0.30%) and  $\text{SiO}_2$  (22.1%); and

mineralogical analysis resulting of zircon (67.5%) and baddeleyite (15.8%).

Ref. [17] described that “caldasite” was formed due through the destruction by hydrothermal solutions of the primary zirconiferous minerals present in PCAC rocks (eudialite-eucolite, rosenvuschita, lavenite, acmite and mainly zircon). Ref. [8] identified 44 occurrences of “caldasite” in PCAC area, associated or not with radioactive U-Th anomalies. Their economic occurrences are found mainly as alluvial/colluvial deposits or as lenses and veins, associated with rocks intensively altered by hydrothermalism (potassic rocks). Therefore, it was suggested by Ref. [18] that the Zr is subtracted from the crystalline structure of the primary Zr-silicates and re-deposited by the influence of hydrothermalism in the form of leached Zr. With minor economic expression, the “caldasite” can also occur disseminated in the alkaline rocks as small irregular masses of 1 to 4 cm in size.



**Fig. 1** Simplified geological map of Poços de Caldas Alkaline Complex. After Refs. [19, 20]. UTM coordinate system, Zone 23, southern hemisphere.

Ref. [15] described Zr and Zr-minerals as fine-grained (< 1 mm) idiomorphic to hypidiomorphic zircons occur randomly distributed as isolated grains in the rock matrix, and with variable grain-size (0.5-5 mm) in voids, interstices and along fractures. Skeletally developed zircon is found in interstices and along grain boundaries. In the breccia matrix zircon is strongly metamict and merely present as cryptocrystalline mixtures of zircon, baddeleyite and amorphous (hydrous?) Zr-oxides. Single baddeleyite crystals not associated with zircon are presented in several breccia samples. The zircons normally contain trace amounts of  $\text{Yb}_2\text{O}_3$  (0-0.26 wt.%),  $\text{Ce}_2\text{O}_3$  (0-0.37 wt.%) and  $\text{Y}_2\text{O}_3$  (0-0.68 wt.%); Ce and Y are most probably present as minute monazite and xenotime phases, substituting zircon. Ref. [15] also described that  $\text{HfO}_2$  is present in “caldasite” between 0 and 0.8 wt.%, with  $\text{ZrO}_2/\text{HfO}_2$  ratio ranging from 0 to 0.012, values typical from non-metamict zircons from nepheline syenites [21].

It is proposed to discuss here the mineralogy of the “caldasite”, considering it is a Zr-ore of PCAC rocks from previous studies of Refs. [8, 15, 16]. However, it is necessary to understand and describe not only the Zr-minerals but also the mineral assemblage due to the potential variety of applications for Zr-based materials in industry.

## 2. Materials and Methods

“Caldasite” samples from PCAC area were crushed using Jaw crusher (Pavitest-Contenco Ind. e Com. Ltda) and milled in a horizontal planetary ball mill (Pavitest-Contenco Ind. e Com. Ltda). The milled samples were then analyzed for chemical composition using X-Ray Fluorescence spectrometer and results tabulated as shown in Table 1. The samples were screened using 13 Sieves (Bertel Ind. e Com. Ltda) with a nominal sieve opening of 12.5, 9.5, 6.35, 4.75, 3.35, 2.36, 1.7, 1.18, 0.6, 0.425, 0.3, 0.212 and 0.1 mm mounted on a sieve shaker (Fig. 2). Samples with 0.1 mm particle size were submitted to XRF (X-Ray

Fluorescence) (Axios PW 4400/40 DY 1686) and XRD (X-ray powder-diffraction) analysis. XRD data were collected over the range  $3-80^\circ 2\theta$  with  $\text{CuK}\alpha$  radiation on a Rigaku-ULTIMA IV diffractometer equipped with a diffracted beam graphite monochromator crystal, step  $0.02^\circ$ , time  $1.5^\circ 2\theta/\text{min}$ . Microscopic analysis of “caldasite” was also done using JEOL JSM-6010 LA SEM-EDS (scanning Electron microscopy with energy dispersion spectroscopy).

After comminution and sieving, particle sizes were analyzed under conventional microscopy techniques and particles size of 0.212 mm presented mineral liberation.

In order to remove iron contents, from iron minerals and iron inclusions, the products of size fractions separation by sieves into a range of +0.212 mm -0.1 mm were subjected to magnetic separation with WHIMS (Wet High-Intensity Magnetic Separation) (model L4 Inbras-Eriez) with field intensity of 2 to 22 A performed in five cycles with increasing intensity.

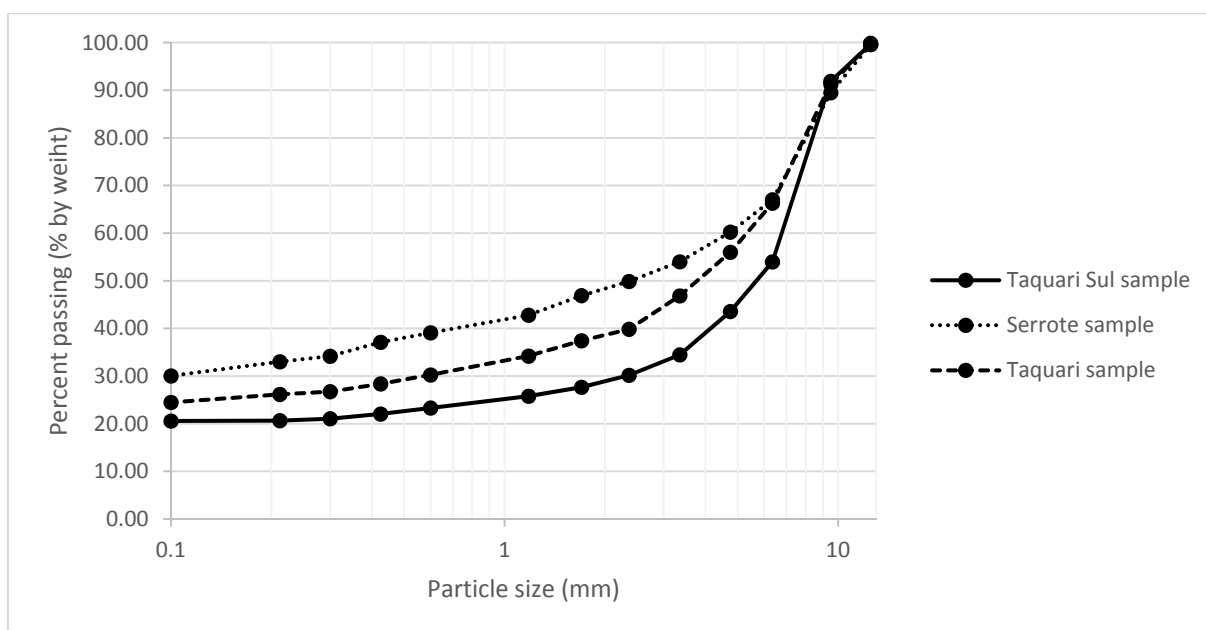
## 3. Results and Discussion

The chemical composition analysis by FRX spectrometry showed that “caldasite” contains  $\text{ZrO}_2$ ,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{U}_3\text{O}_7$  (only in Taquari Sul sample),  $\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  as major elemental concentration. Trace elemental concentrations are: F,  $\text{Nb}_2\text{O}_5$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{ThO}_2$ ,  $\text{HfO}_2$ ,  $\text{CaO}$ ,  $\text{SrO}$  and  $\text{MnO}$ . However,  $\text{V}_2\text{O}_5$ ,  $\text{K}_2\text{O}$ ,  $\text{Ti}_2\text{O}_3$  and  $\text{Cs}_2\text{O}_3$  were not detected in all three samples. FRX results show significant values for  $\text{ZrO}_2$ ,  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$  in all collected samples, which can have potential application for Zr-industry (Table 1). On the other hand, “caldasite” samples presented relatively high concentrations for  $\text{Fe}_2\text{O}_3$  (between 3.6% and 7.15%). It is recommended by refractory industry, in order to obtain chemical stability and high refractoriness, that Zr-materials for refractory applications contains  $\text{Fe}_2\text{O}_3$  as trace elemental concentrations.

XRD analysis resulted in patterns of zircon and baddeleyite, mainly. Reflexions peaks are thin and well

**Table 1** XRF analysis of “caldasite” from (a) Taquari Sul, (b) Taquari and (c) Serrote samples (see text for further explanation).

Oxides	Taquari sul sample (%)	Serrote sample (%)	Taquari sample (%)
ZrO <sub>2</sub>	70.34	68.93	65.15
SiO <sub>2</sub>	15.11	14.94	23.08
Fe <sub>2</sub> O <sub>3</sub>	4.01	7.15	3.6
U <sub>3</sub> O <sub>7</sub>	2.77	-	-
Al <sub>2</sub> O <sub>3</sub>	1.32	3.05	0.78
TiO <sub>2</sub>	1.15	0.84	1.72
F	0.962	-	1.19
Nb <sub>2</sub> O <sub>5</sub>	0.85	0.95	0.31
Y <sub>2</sub> O <sub>3</sub>	0.53	0.36	0.49
Na <sub>2</sub> O	0.43	0.31	0.50
MgO	0.36	-	0.39
ThO <sub>2</sub>	0.18	0.092	0.58
HfO <sub>2</sub>	-	0.84	0.52
V <sub>2</sub> O <sub>5</sub>	0.18	-	-
CaO	0.16	0.21	0.18
SrO	0.16	0.051	0.041
MnO	0.10	0.14	0.23
K <sub>2</sub> O	0.08	-	-
Tl <sub>2</sub> O <sub>3</sub>	0.06	-	-
Cs <sub>2</sub> O	-	-	0.28
LOI	1.19	2.11	0.91

**Fig. 2** Particle size distribution curve of “caldasite” samples.

defined, as excellent crystallinity. However, samples also evidenced peaks of magnetite (Fe<sub>3</sub>O<sub>4</sub>), goethite (FeO(OH)), magnesioferrite (MgFe<sub>3</sub>O<sub>4</sub>), gibbsite (Al(OH)<sub>3</sub>) and hafnium-baddeleyite ((Zr,Hf)O<sub>2</sub>)

(Fig. 3). According to XRD patterns, Taquari is the most variable sample in terms of mineral phases that might be related to the crystallization of caldasite.

Ref. [22] described that zircon consists theoretically

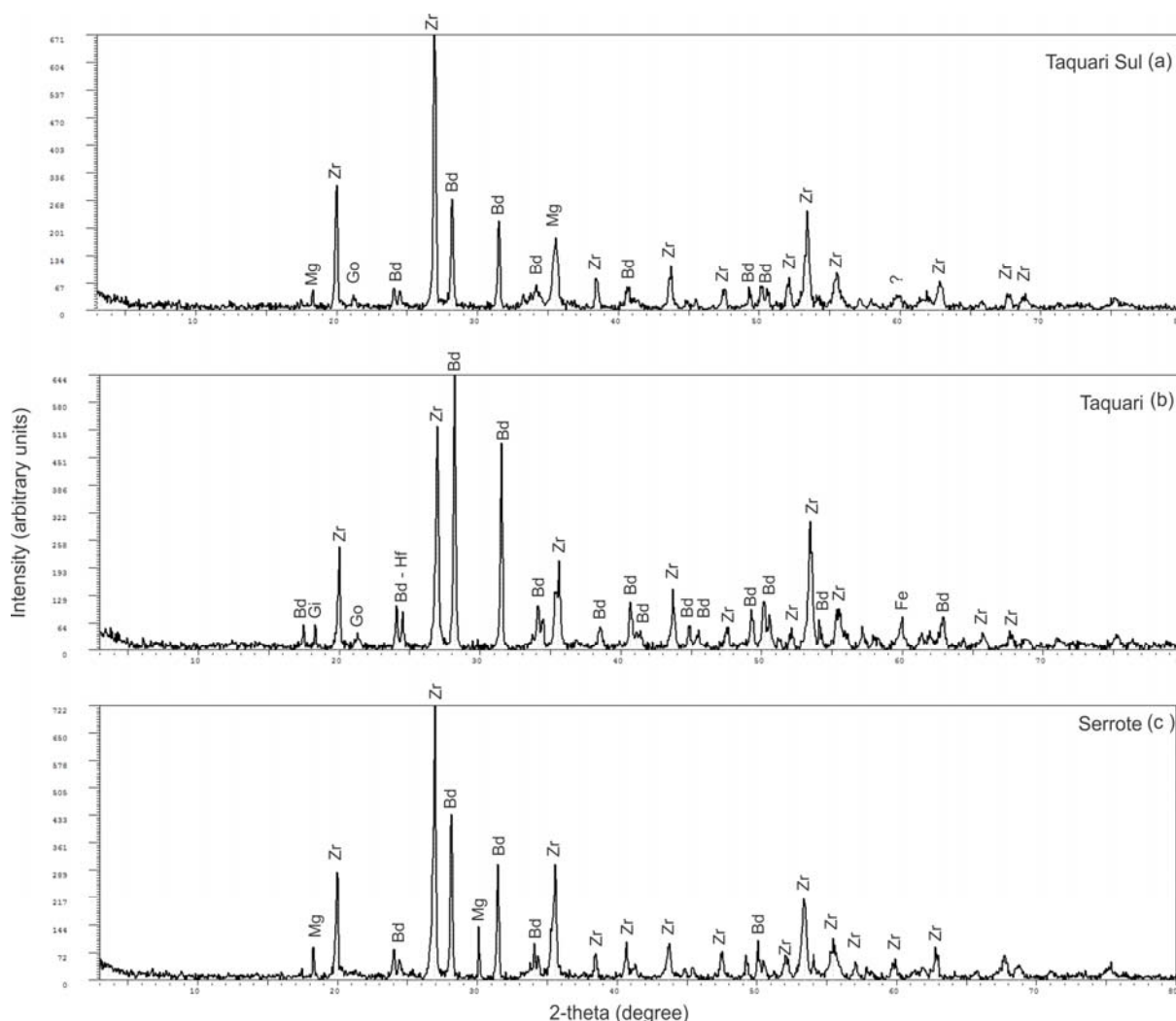
of 67.2% of  $\text{ZrO}_2$  and 32.8% of  $\text{SiO}_2$ , and also contains between 0.4-2.0% of Hf. In all natural occurrences, Zr and Hf always occur together. The similarities of their chemical properties make separation unnecessary for most applications. Inclusions of iron and magnetite can also be present. Also Ref. [23] described baddeleyite as  $\text{ZrO}_2$  with Hf, Ti and  $\text{Fe}^{3+}$ , and Ref. [22] analyzed that it is very common up to 1.7% of Hf in baddeleyite.

Mineralogical analyses by conventional microscopy and SEM showed mainly the presence of zircon and baddeleyite and, subordinately, magnetite, iron-oxyhydroxides and gibbsite, very fine-grained.

Regarding the iron minerals, magnetite and

iron-oxyhydroxides were identified. These minerals are mostly isolated crystals and, less frequent, as aggregated to baddeleyite. The magnetite occurs as well-defined euhedral crystals, ranging from 0.2 mm to 0.4 mm in size. Iron-oxyhydroxides are botroidal grains, sized between 0.2 mm and 0.5 mm.

Zircon occurs as isolated grains and aggregates of colorless, or light brown to dark brown color grains. Idiomorphic crystals have grain-size ranging from 50 to 150  $\mu\text{m}$ . Baddeleyite occurs as very fine-grained microcrystalline mass (anhedral) of reddish coloration to reddish-brown (Fig. 4). Very frequently, baddeleyite covers zircon (Fig. 5). In addition, it is very common



**Fig. 3** XRD patterns of “caldasite” from (a) Taquari Sul, (b) Taquari and (c) Serrote samples (see text for further explanation).

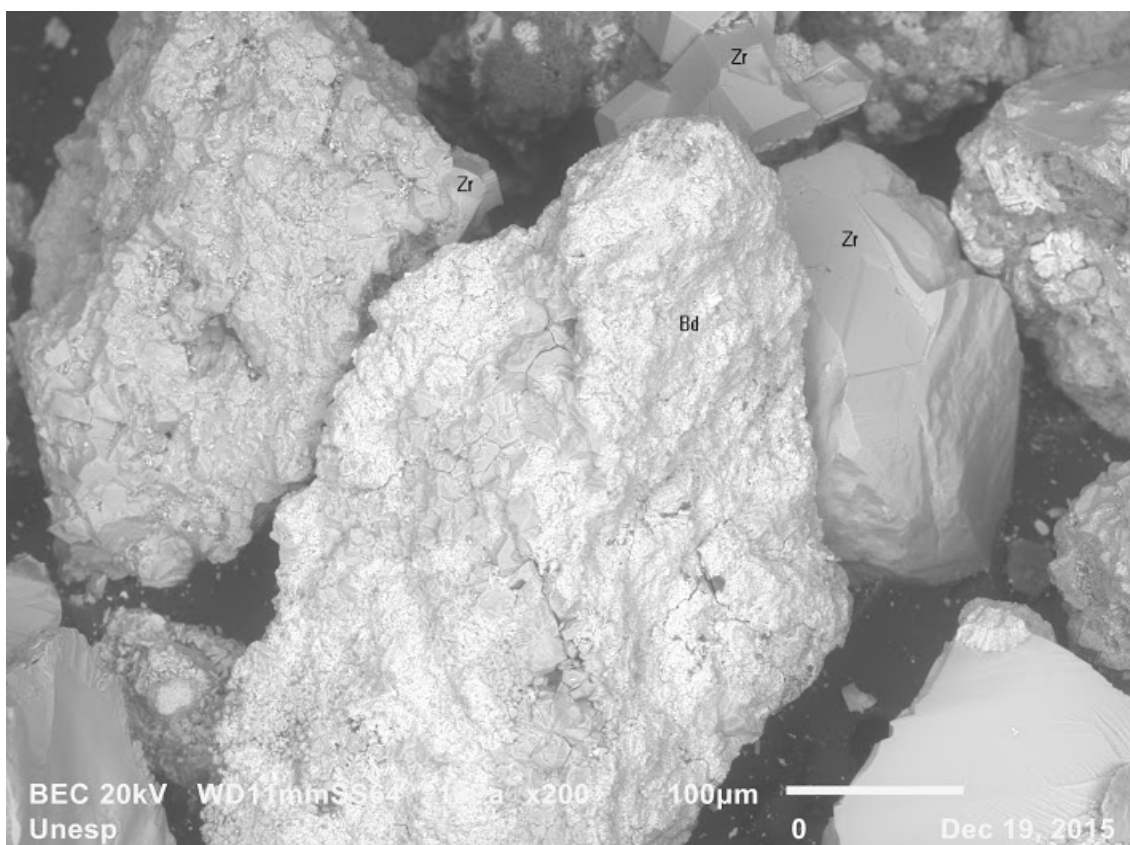
Zr = zircon ( $\text{ZrSiO}_4$ ), Bd = baddeleyite ( $\text{ZrO}_2$ ), Mg = magnetite ( $\text{FeO} \cdot \text{Fe}_2\text{O}_3$ ), Go = goethite ( $\text{FeO} \cdot \text{OH}$ ), Bd-Hf = hafnium-baddeleyite ( $(\text{Zr,Hf})\text{O}_2$ ), Gi = gibbsite ( $\text{Al}(\text{OH})_3$ ), Fe = magnesioferrite ( $\text{Fe}_2\text{MgO}_4$ ).

the occurrence of iron-oxyhydroxides and gibbsite with baddeleyite (Fig. 5).

The analytical and mineralogical characterization techniques employed in the samples defined a mineralogical profile of zircon and baddeleyite in “caldasite”. The results indicated that zircon and baddeleyite present mineralogical characteristics very specific: the zircon presents colorless, or light brown to dark brown color, pleochroic, and may occur in the form of isolated or aggregates crystals, euhedral to subhedral, and variable in size. Baddeleyite is variable in color and it ranges from colorless to dark brown/reddish brown color. It frequently occurs as very-fined grained aggregates (microcrystalline mass), and a superficial oxidation and inclusions of rutile ( $\text{TiO}_2$ ) is also frequent.

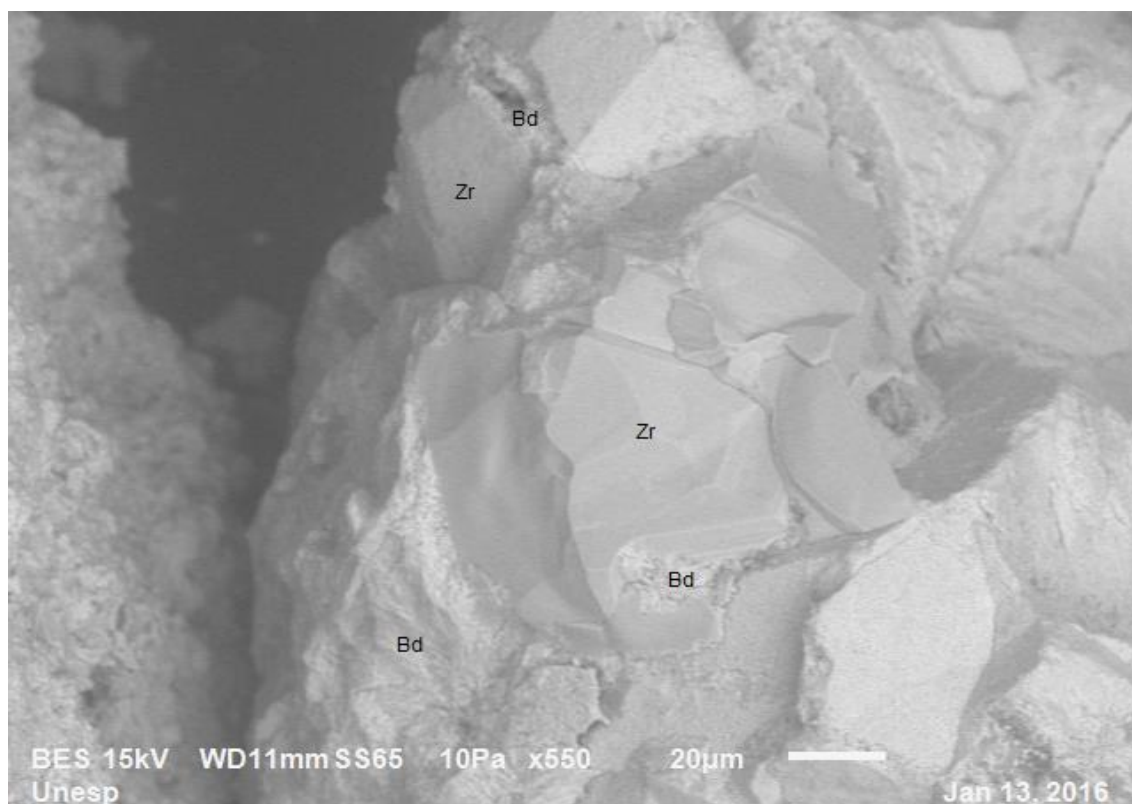
XRD results of indicated, mainly, zircon, baddeleyite, iron-oxyhydroxides and magnesioferrite. Gibbsite was identified in XRD and SEM analysis but it represents a very low concentration on the mineral assemblage (approximately less than 5%). According to the chemical composition of major elements, caldasite presented between 65% and 70% of  $\text{ZrO}_2$ , 15% to 23% of  $\text{SiO}_2$  and 3.6% to 7% of  $\text{Fe}_2\text{O}_3$ .  $\text{U}_3\text{O}_7$  was presented only in one sample (2.77%, Taquari Sul).

Considering that iron content is a potential contaminant for application of Zr-materials in industry, size fractions of +0.212 mm -0.1 mm were performed into WHIMS magnetic separation in 2A, 10A, 18A, 22A and 0A (for equipment cleaning) field intensity in order to estimate the efficiency of Fe removal from “caldasite” samples.



**Fig. 4** Scanning electron microscopy images from Taquari Sul sample.

Zr—zircon, Bd—baddeleyite. The analytical conditions for BSE images were: 15 kV accelerating voltage, 20 nA electron beam current, WD (working distance) of 11 mm and SS (spot size) of 65.



**Fig. 5** Scanning electron microscopy images from Taquari Sul sample showing Zr-aggregates.

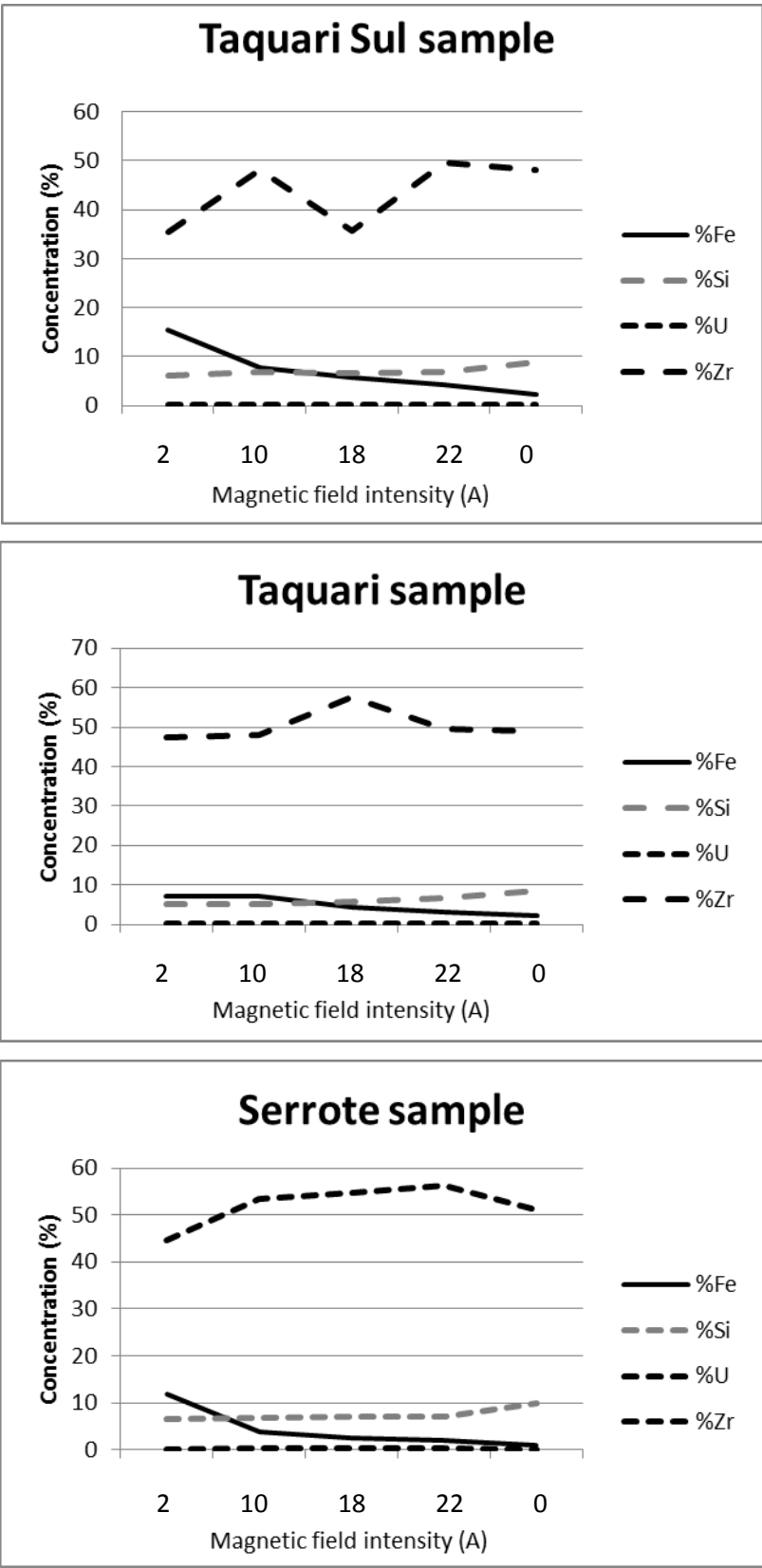
Zr—zircon, Bd—baddeleyite. The analytical conditions for BSE images were: 15 kV accelerating voltage, 20 nA electron beam current, WD (working distance) of 11 mm and SS (spot size) of 65.

Fig. 6 shows the pattern of Fe, Si, U and Si-contents for all samples along the five cycles of the magnetic separation. The last cycle (cycle 5) refers to equipment cleaning (0 A field intensity) in which diamagnetic minerals were collected because they were not separated in the previous steps. All “caldasite” samples show the same pattern concerning iron removal: successive increasing of iron concentration as field intensity is increased. It is also observed that WHIMS magnetic separation has not influenced Zr- and U-concentrations in the analyzed samples because there is no clear evidence of a change in the Zr- and U-pattern through field intensity alteration. Only Si-concentration presented variable concentration as field intensity is increased.

The successive increasing of iron concentration as field intensity increases is an evidence for iron removal efficiency from WHIMS in “caldasite” samples (Table 2), considering that Zr-concentration presents

diamagnetic properties. According to Table 2, Taquari Sul sample presented 42.64% of iron removal and 83.15% of mass recovering. Serrote sample had 75.0% of iron removal and 68.81% of mass recovering. Taquari sample had 70.6% of iron removal and 79.5% of mass recovering. During the magnetic separation processing, Taquari sample did not present iron removal between cycles 1 and 2 (Fig. 6) as observed in the other samples, which can be explained by a difference on the mineralogical composition in this sample. It was observed in Taquari Sul and Serrote samples that as long as field intensity is increased, iron removal is higher (Fig. 6).

In comparison of iron removal efficiency, mineralogical composition from XRD (Fig. 3) and chemical analysis from XRF (Table 1), it is observed that Serrote sample has zircon, baddeleyite and magnetite as mineral composition, and  $\text{Fe}_2\text{O}_3$ , as iron concentration for magnetic separation, is 7.15% (higher



**Fig. 6** Magnetic field intensity (A) vs. Fe-, Si-, U- and Zr-concentrations.  
Cycle 1: 2A; Cycle 2: 10A; Cycle 3: 18A; Cycle 4: 22A; Cycle 5: 0A.



**Table 2** Iron removal efficiency and massic recovering obtained from WHIMS magnetic separation of Taquari Sul, Taquari and Serrote samples.

Samples	Iron removal efficiency (%)	Massic recovering (%)
Taquari Sul	42.64	83.15
Taquari	70.62	79.52
Serrote	75.0	68.81

value concentration considering the analysed samples). On the other hand, Taquari Sul and Taquari samples presented  $\text{Fe}_2\text{O}_3$  of 4.01% and 3.6%, and mineralogical composition is also very similar and a little more complex than Serrote sample with gibbsite, goethite and magnesioferrite in addition with zircon, baddeleyite and magnetite. This observation can explain that Serrote sample had presented considerable iron removal during the first cycles of magnetic separation (cycles 1 and 2, from 11.9% to 3.6% Fe-removal) although it presents a less complex mineralogical composition and the higher Fe-concentration in comparison of all samples. There could be two reasonable reasons for this: (a) magnetite is presented as magnetite-free although it could not be observed in SEM analysis, or (b) magnetite is presented as inclusions in zircons (as baddeleyite is a botrioidal mass) and could be removed in this size fraction (+0.212 mm -0.1 mm).

#### 4. Conclusions

Based on the collected samples and the results obtained, it is possible to conclude that “caldasite” is a very complex rock, mainly in terms of identification and mineralogy quantification.

The integration of FRX and DRX results indicates a rock composed of zircon, baddeleyite and magnetite and iron-oxyhydroxides, mainly. Only one sample registered  $\text{U}_3\text{O}_7$  concentration in a relative low concentration. The hypothesis, formulated by Garda (1990), that caldasite was formed by Zr-leaching of primary alkaline rocks during hydrothermal processes and deposited as a mixture of zircon, baddeleyite and magnetite is coherent with the FRX, DRX and SEM results presented here. Ref. [15] admitted that

iron-oxyhydroxides, kaolinite, goethite, illite and smectite were formed because of supergene weathering. It is not very clear how U- and Zr-concentration are related. “Caldasite” does not present very high U-concentration but presents Th-concentration (even though in low proportions).

The mineralogical characterization performed by DRX and conventional microscopy analysis was very important to understand how Fe-minerals could be removed. Magnetic separation by WHIMS presented a considerable reduction of 46% to 71% for Fe-concentrations removal. This is also a clear evidence that comminution processes for 0.212 mm particle size were appropriate, and the magnetic separation by WHIMS was efficient. A very positive result is the maintenance for high Zr-concentrations even after magnetic separation. All results obtained here indicate “caldasite” has a potential application in refractory industry due to high Zr-value concentrations even though Fe-concentration removal is necessary.

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