

## Mineralogical evolution of the anorogenic granites of Tirmini (Damagaram Southeast, Niger)

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### Abstract

The present study focuses on the anorogenic granites of Tirmini. The latter are part of the younger granites of the pan-African province of Damagaram-Mounio, which constitutes a connection between the younger granites of the Air in the north and those of the Jos Plateau in the south. The objective of this study is to determine the mineralogical characteristics of the anorogenic granites of Tirmini. The methodological approach used includes X-ray diffraction (XRD) analysis on the one hand, and scanning electron microscope (SEM) and cathodoluminescence munimicroscope observations on the other. Two magmatic lineages form the Tirmini granite complex: one alkaline and the other peralkaline. Qualitative analysis at XRD shows that alkaline rocks include orthose, albite, quartz with little or no arfvedsonite. In addition to these minerals, peralkaline rocks contain aegyrin and arfvedsonite. SEM observation of all the Tirmini rocks shows that ilmenite (FeTiO<sub>3</sub>) is xenomorphic and luminescent in the yellow-orange range. Fluorapatite Ca<sub>5</sub>(PO<sub>4</sub>)F, which is stocky or elongated in shape, is present only in granites. Zircon (ZrSiO<sub>4</sub>) occurs in an elongated prism or quadratic section. Thus, at cathodoluminescence, monazite appears in luminescent zoned crystals in dark gray. Titanite is luminescent and appears in yellow-oranges. Semi-quantitative analysis of alkaline rocks shows the percentage of orthotics 50-65%, albite 6-8%, arfvedsonite quasi-absent 6%. Quartz is present between 30-34% in all rocks compared to 11% in the quartz alkaline syenite of Tirmini. In the peralkaline line, arfvedsonite and aegyrin account for 13% and 7%, respectively. Orthoclase 40-46%, albite 4% and biotite 2%. Quartz is present at 30%. The high orthotic contents show that the Tirmini rocks are persolvated and that the exsolution dome in the albite-orthotic system was well separated from the solidus.

**Keyword:** Anorogenic granites; Persolvus; Damagaram-Mounio; Mineralogy; X-ray diffraction

### 1. Introduction

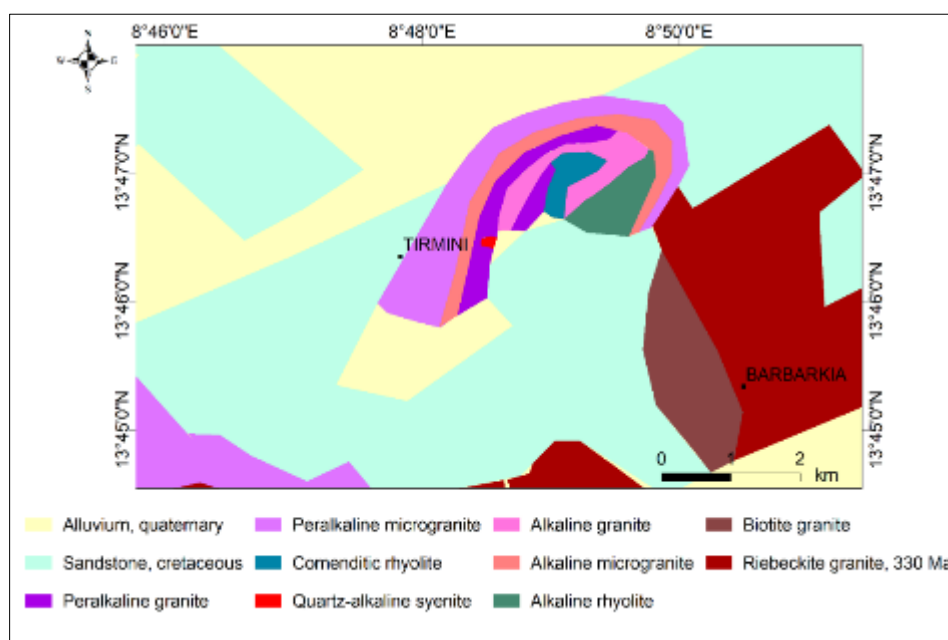
The Paleozoic to Mesozoic anorogenic granites of the Niger-Nigeria province are considered an impressive example of continuous annular complexes over 1300 km in the continental environment, with a gradual decrease in ages from the Air to the Jos plateau in the south [1]–[10]. The Paleozoic magmatic activity of the Damagaram-Mounio province began in the Carboniferous period with the placement of the anorogenic granites of Badaraka, Zinder and Zarniski and ended in the Permian with those of Gouré and Tirmini [11]–[14]. Recent petrological, geochronological and geochemical studies carried out on the anorogenic granites of Tirmini have highlighted an anorogenic sequence typical of the A1 intraplate context [12]. Also, these granites were affected by two stages of deformation: one semi-ductile to brittle and the other purely brittle [13]. However, a detailed mineralogical study applying new analytical techniques is needed to

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better characterize and quantify the constituent minerals of the anorogenic Tirmini granites. To better identify the different mineralogical phases, samples were analyzed using the DRX method, since these atoms are arranged according to specific crystallized planes.

### 1.1. Petrographic context of Tirmini

Damagaram has a long geological history with a Pan-African basement spread from the southwest to the northeast, dissected by deformation structures and purely anorogenic magmatism [7], [15]–[17]. The anorogenic granites of Tirmini are part of the Paleozoic alkaline province of Damagaram-Mounio, which represents a connection between the younger granites of the Air in the north and those of the Jos Plateau in the south. Tirmini petrography is characterized by two lineages: (i) alkaline and (ii) peralkaline [12]. The first lineage is characterized by a petrographic sequence evolving from alkaline rhyolite, alkaline microgranite, alkaline granite and ends with the establishment of a quartz alkaline syenite. The second peralkaline lineage highlights a magmatic evolution ranging from a comenditic rhyolite, to microgranite and peralkaline granite. These petrographic evolutions are typical of a petrogenetic model by subsidence of [18] and [3]: volcanic phase, hypovolcanic phase and plutonic phase.



**Figure 1** Geological map of the Tirmini complex [1].

## 2. Methodology

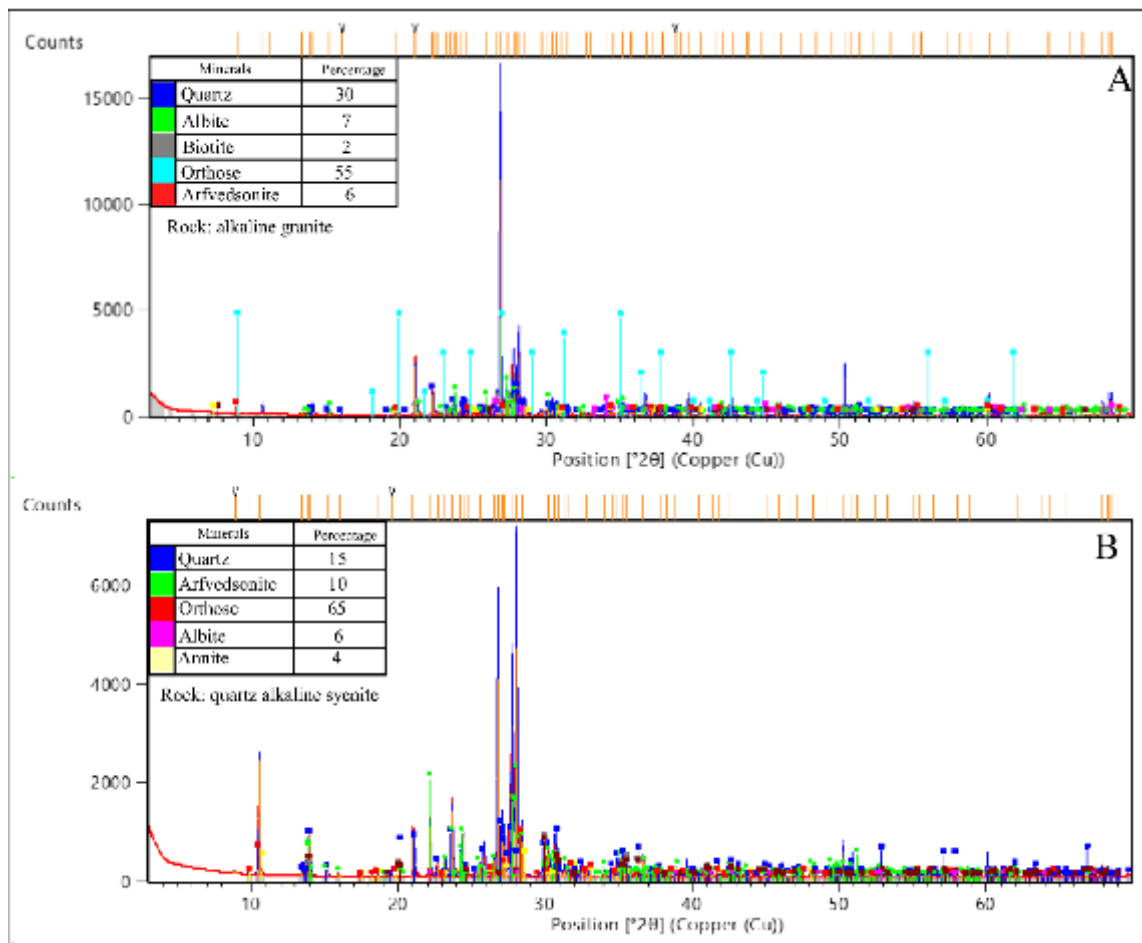
The methodological approach adopted includes: (1) the identification of mineral phases (semi-quantitative) through the position of the peaks of the diffractograms of each mineral, (2) observation with a scanning electron microscope (energy dispersion spectrum) and (3) characterization morphological with a cathodoluminescence microscope. XRD analysis involved twelve (12) samples of crystalline powder from Tirmini rocks. These powders were analyzed at the National Institute of Geophysics and Volcanology in Naples, Italy. Powder X-ray diffraction was performed using a Panalytical Xpert PRO PW 3040/60 diffractometer equipped with a pyrolytic graphite analyzer crystal. Unfiltered  $\text{CuK}\alpha$  radiation was used under the following conditions: 40kV acceleration voltage, 40 mA tube current, range of 3 - 70  $2\theta$ , pitch of 0.02  $2\theta$ , counting time 30 s/step, 0.5 mm divergence slit, 0.1 mm receiving slit, and 0.5 anti-scatter slot according to the procedure described by [19]. All powder samples were laterally loaded to minimize the possible preferential orientation effects. Qualitative identifications were carried out using the High Score Plus software and the ICDD PDF2 database (2012 version, Naples, Italy). A JEOL JSM 5310 scanning electron microscopy (BEM) integrated with an energy dispersive spectrometry (EDS), at the Thin section laboratory of Geoscience Environnement Toulouse (France), was used for the micro-morphological and chemical study of both fragments and polished thin sections. The fragments were fixed on aluminum beads with double-adhesive carbon tape, while thin sections were positioned on a flat aluminum support after carbon coating about 10 nm thick under a vacuum evaporator, observed by SEM and analyzed by EDS. The following conditions were adopted: 15 kV electric current, 50 kV filament current. The analysis of the polished thin sections made it possible to characterize the chemical compositions of the accessory minerals thanks to the energy dispersion spectra. These analyses were performed on a total of five compound polished sections. The morphology and

structure of the accessory minerals was examined using cathodoluminescence imaging of two samples that carry more secondary minerals. CL images of these accessory minerals were also made at the Laboratory of Geoscience and Environment Toulouse using a cathodoluminescence munimicroscope connected to a Gatan minicl system.

### 3. Results

#### 3.1. X-ray diffraction on alkaline rocks from Tirmini

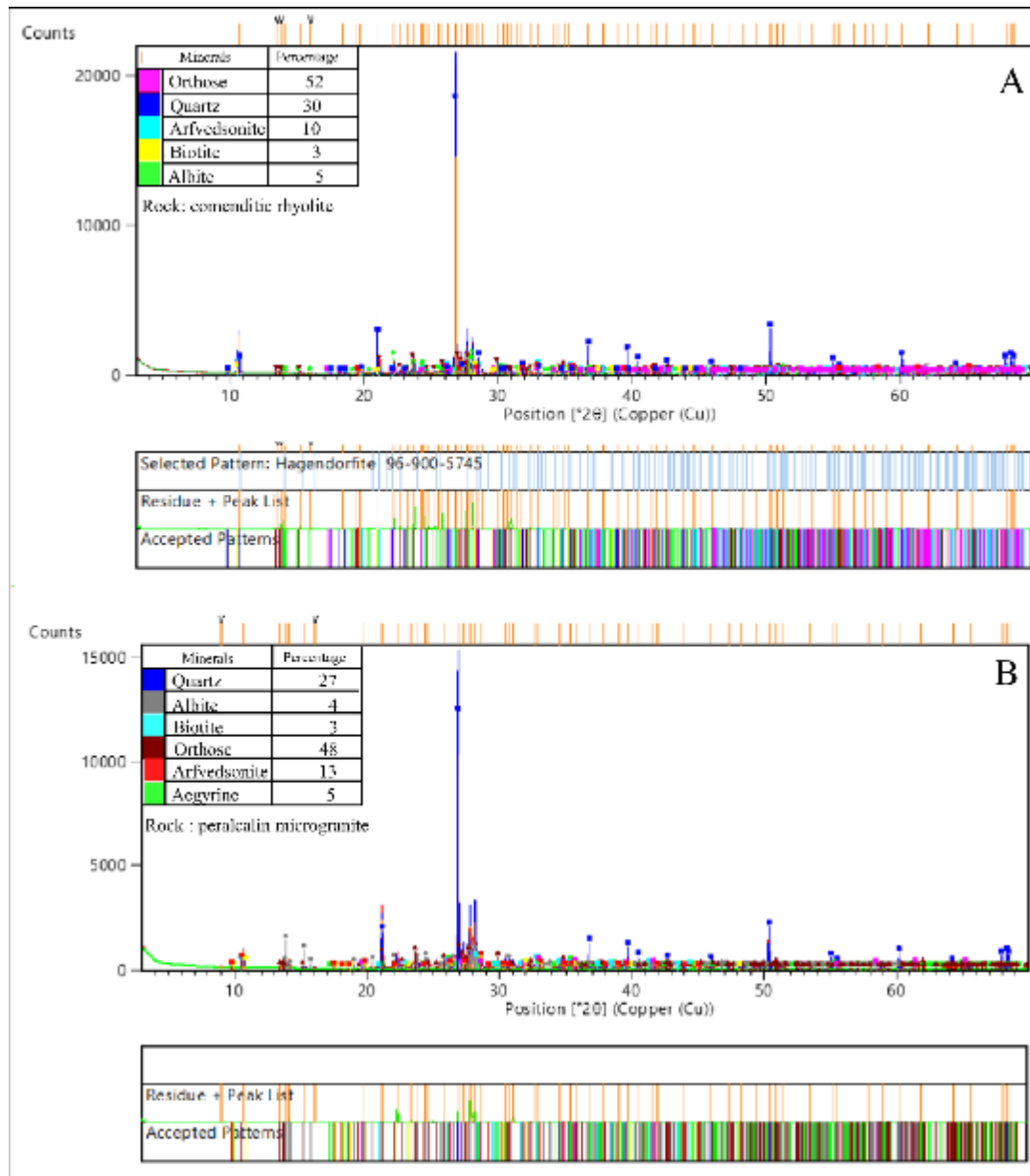
Alkaline feldspar (orthoclase and albite) is the most abundant mineral in all rocks of the Tirmini annular complex. Semi-quantitative XRD analysis shows orthoclase is present between 40-65% and albite 6-8% (Figure 2). The orthoclase records prominent peaks in the range 9 to 65 degrees  $2\theta$  with an intensity of up to 15000 counts (Figure 2A and B). The peaks of albite are minor and close surlignage and are locally recorded along diffractograms (Figure 2). For quartz, the semi-qualitative data in Figure 2 show that the prominent peaks generated are located around 27 degrees  $2\theta$ , the most noticeable of which have an intensity of more than 15000 counts in the granite. Its content varies from 30-34% in the alkaline rocks of Tirmini. On the other hand, in syenite, the quartz content is estimated at about 15%, which explains why it would be a quartz alkaline syenite (Figure 2B). The peaks produced by arfvedsonite in alkaline rocks are detectable around 2000 counts and are recorded along diffractograms between 10 and 65 degrees  $2\theta$  (Figure 2A). In the granite, the peaks of arfvedsonite are minor and of low intensity with an estimated percentage of about 6% (Figure 2A). On the other hand, at the same scale of 2000 counts, the most prominent peaks of syenite are recorded around 22 and 28 degrees  $2\theta$  with a percentage of about 10% (Figure 2B). This show that Tirmini syenite is more alkaline than granite. Annite or ferriferous biotite is only detectable in quartz alkaline syenite.



**Figure 2** Diffractograms of Tirmini's alkaline sequences. A: diffractogram of alkaline granite; B: quartz alkaline syenite diffractogram. The semi-quantitative proportions of the minerals are the result of Rietveld refinement of the quantitative data of the intensities of the peaks.

### 3.2. X-ray diffraction on Tirmini peralkaline rocks

The peaks generated by orthoclase in peralkaline rocks are minor and of low intensity (Figure 3), compared to those produced in alkaline rocks. They were recorded in the range 3 at 70 degrees  $2\theta$  with an intensity barely reaching 50 counts. Although these peaks are low intensities, the semi-quantitative evaluation estimated the percentage of orthoclase 46-52% and albite 3-5% (Figure 3).



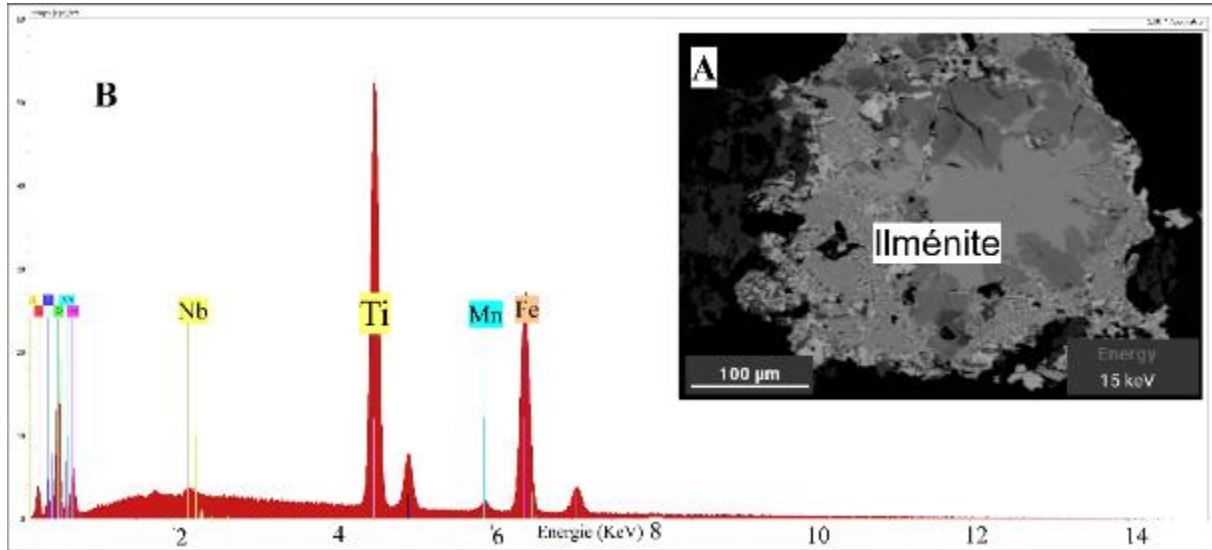
**Figure 3** Diffractograms of Tirmini's hyperalkaline sequences. A: diffractogram of comenditic rhyolite; B: peralkaline microgranite diffractogram. The semi-quantitative proportions of the minerals are the result of Rietveld refinement of the quantitative data of the intensities of the peaks.

The diffractogram shows that quartz generated prominent peaks around 27 degrees  $2\theta$  with an intensity reaching 15000 counts in all the peralkaline rocks of Tirmini. Slightly minor peaks are recorded locally between 11 and 70 degrees  $2\theta$  can estimate the percentage at about 30% (Figure 3). Thus, the peaks generated by arfvedsonite in peralkaline rocks are only detectable from 3000 counts. Taking into account the scale, these peaks are of low intensity and are recorded between 10 and 70 degrees  $2\theta$ . Its percentage can be estimated at about 10% in comenditic rhyolite (Figure 3A). In peralkaline granites and microgranites, the high concentrations of peaks are in the range of 23 and 26 degrees  $2\theta$  (Figure 3B). In microgranites, the percentage varies between 10-13%, which explains why the arfvedsonite

content increases from comenditic rhyolite to microgranite and hyperalkaline granite. Aegyrin is present at about 5% (Figure 3B).

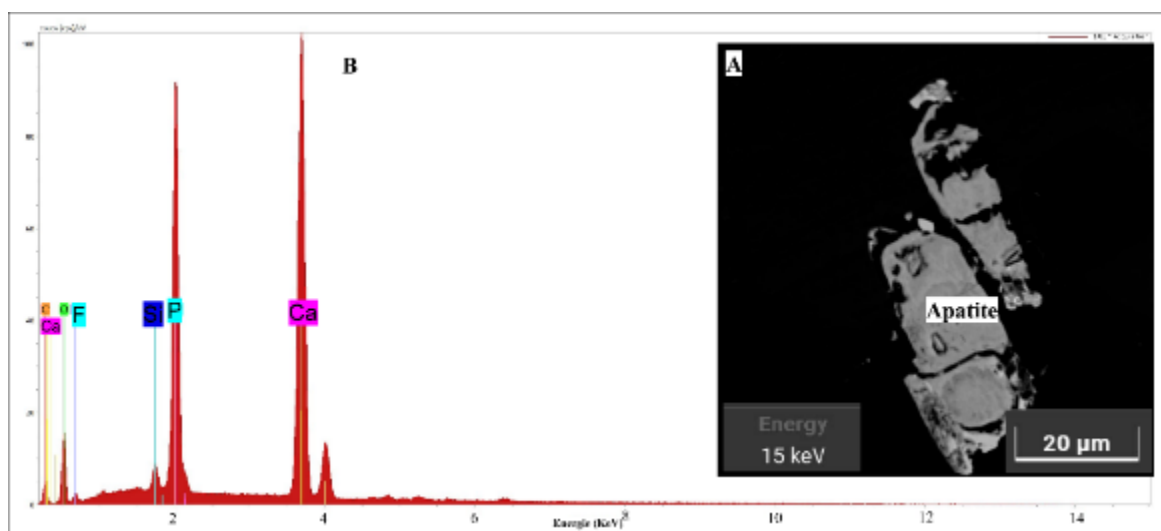
### 3.3. Scanning electron microscope (SEM) observation

Ilmenite is the only secondary mineral detected by SEM and is present in all Tirmini rocks. Apatite and zircon complete the mineralogical composition. Ilmenite ( $\text{FeTiO}_3$ ) occurs in large xenomorphic crystals (Figure 4A) of several tens of micrometers up to 100  $\mu\text{m}$ . It appears as an alteration product of annite to arfvedsonite. The high concentrations obtained belong to titanium, iron and oxygen. The intensities of manganese and niobium are low (Figure 4B).



**Figure 4** Ilmenite of the Tirmini rocks. (A) microphotograph of a xenomorphic ilmenite crystal located in the Tirmini rocks; (B) Energy dispersion spectrum showing the chemical composition of ilmenite. Nb: niobium; Ti: titanium; O: oxygen; Fe: iron; Mn: manganese.

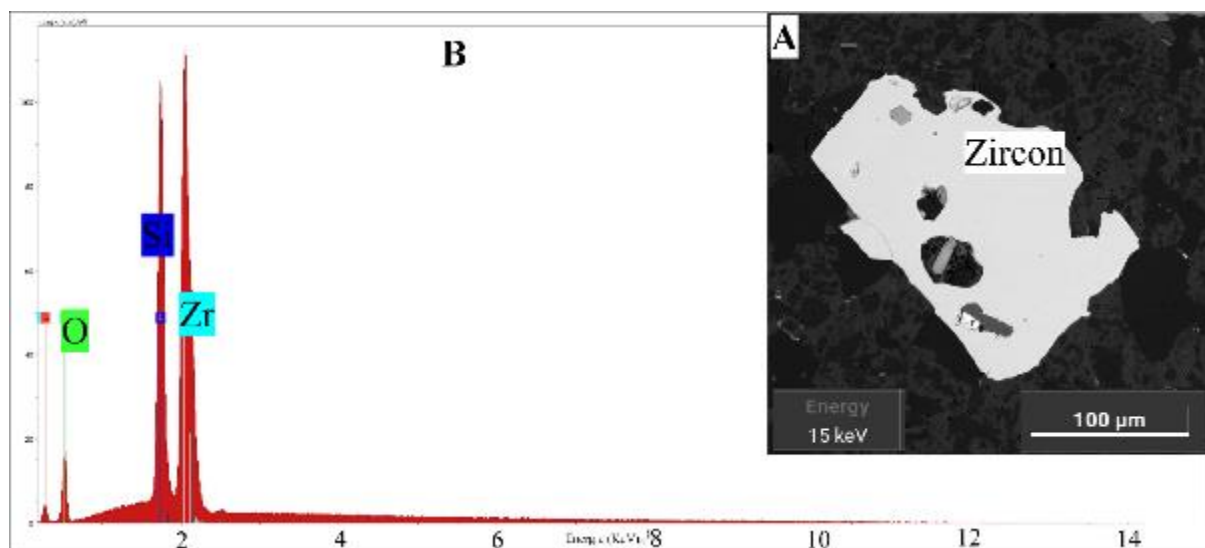
Apatite crystallized early in the form of stocky or elongated prisms, clear with high relief (Figure 5A). It has only been observed in granites. Small crystals are scattered throughout the matrix while large crystals can be as large as 50  $\mu\text{m}$  (Figure 5A). The composition of apatite is dominated by calcium and phosphorus (Figure 5B). Low concentrations are recorded for silicon and fluorine. The fluoride content (Figure 5B) favours a fluorapatite  $\text{Ca}_5(\text{PO}_4)_3\text{F}$ . The presence of Si would be compensated by rare earths, but the intensity of the peaks is barely detectable.



**Figure 5** Apatite from the rocks of Tirmini. (A) microphotography of subautomorphic hydroxylapatite grains; (B) Energy dispersion spectrum showing the chemical composition of apatite. Ca: calcium; P: phosphorus; Si: silicon ; F: fluorine; C: carbon and O: oxygen.



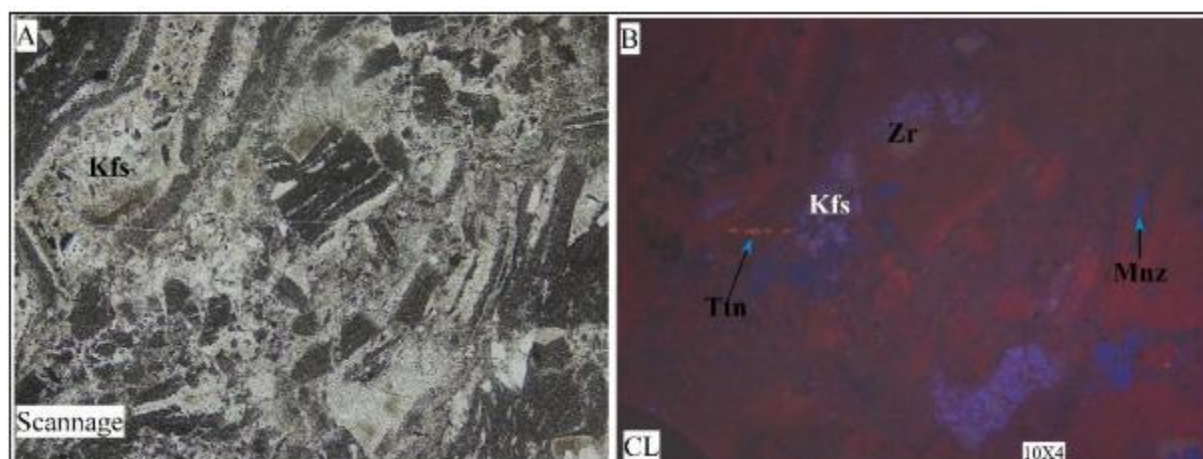
Zircon is an early mineral in the crystallization sequence compared to other accessory minerals. It is included in amphibole, orthosis and quartz. With the simple formula  $ZrSiO_4$ , zircon is present in the Tirmini rocks in the form of quadratic cross-sectional crystals or in elongated prisms (Figure 6A). Figure 6B shows the chemical composition of zircon where only the peaks of oxygen, silica, and zirconium are detected by the software.



**Figure 6** Zircon from the rocks of Tirmini. (A) Microphotography of an automorphic crystal of zircon at SEM; (B) Zircon spectrum showing the chemical composition of zircon. Si : silicon ; Zr: zirconium; and O: oxygen.

### 3.4. Observation under a cathodoluminescence microscope

Cathodoluminescence analyses revealed the presence of monazite and titanite in trace amounts in the mesostasis (Figure 7). Titanite is luminescent in yellow-orange with some extinct zonations. As for monazite, it comes in zoned crystals and luminescent in dark gray. The zircon that appears as an inclusion in the orthosis crystallized late.



**Figure 7** Observation under a cathodoluminescence microscope. (A) Scanned photo and B: cathodoluminescence imaging of Tirmini comenditic rhyolite; Kfs: potassium feldspar; Ilm: ilmenite; Ap : apatite ; Mnz: monazite; Ttn: titanite; Zrn: zircon and CL: cathodoluminescence.

## 4. Discussion

The minerals in common for the two anorogenic Tirmini suites are orthose, quartz, albite, zircon, apatite, monazite and ilmenite. The magmatic evolution from the alkaline lineage to the hyperalkaline lineage of Tirmini, explained by [1], is marked by an increase in the relative proportion of sodium coloured minerals (arfvedsonite and aegyrine), characteristic of felsic magmatism [4], [8], [13], [20], [21]. This magmatic evolution is explained by the petrogenetic model [18] and [3] next phase: volcanic phase, subvolcanic phase and plutonic phase. Quantitative analysis at XRD

showed that orthoclase present in all Tirmini rocks is estimated at around 40-65%; 6-8% for albite. This shows that all the rocks of Tirmini are granites and hypersolvus syenites emplaced at shallow depths. In such situations, the exsolution dome in the albite-orthoclase system was well separated from the solidus and the orthoclase (contained in the alkaline feldspar) that underwent the exsolution of the albite could have a composition of 95% plus or minus 2% [22], [23]. This is consistent with the orthoclase contents in all the rocks of Tirmini. In subsolvated and hypersolvated rocks, the content of 95% orthoclase contained in alkaline feldspar is at least 400°C [24], Temperature at which the orthoclase enters the microcline's stability field [25]. This shows that the orthoclase-microcline transition did not occur. The absence of the microcline in all the Tirmini rocks would be linked to the absence of the orthoclase-microcline transition. Thus, the assembly of alkaline feldspars (orthoclase-albite) in these rocks is in equilibrium from the point of view of chemical composition. This association could be explained by: (1) the evolution of a magma with crystallization of a single homogeneous feldspar at a high temperature (400°C), (2) the exsolution of the homogeneous feldspar into two alkaline feldspars, one sodium and the other potassium [25]. Because of the asymmetry of the dome, the albite lacks K and the orthoclase contains some Na (about 95 Or) once the rock is cooled.

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## 5. Conclusion

Qualitative analysis at XRD showed that the alkaline lineage is characterized by orthoclase, albite, quartz with little or no arfvedsonite. Aegyrin and arfvedsonite characterize the hyperalkaline lineage. At SEM, ilmenite (FeTiO<sub>3</sub>) is xenomorphic and luminescent in yellow-orange shape in all Tirmini rocks. Fluorapatite Ca<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>F with a stocky or elongated shape is present only in granites. Zircon (ZrSiO<sub>4</sub>) is present in rocks following quadratic or elongated prism sections. At cathodoluminescence, monazite occurs in zoned crystals and is luminescent in dark gray. Titanite is luminescent in yellow-oranges. Semi-quantitative analysis showed that the percentage of orthoclase 40-65%, albite 6-8%, arfvedsonite 6%, quartz is present between 30-34% in all alkaline rocks compared to 11% in the quartz alkaline syenite of Tirmini. In the hyperalkaline line, sodium colored minerals are estimated to be between 10-13% for arfvedsonite and 4-5% for aegyrin. The contents of orthoclase and albite make the Tirmini rocks hypersolvus rocks rich in arfvedsonite and little aegyrin.

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## Compliance with ethical standards

### *Disclosure of conflict of interest*

No conflict of interest to be disclosed.

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