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Geochronology of Mesozoic Large Igneous Provinces in NE Brazil

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Tese de Doutorado

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“It is perhaps a little indelicate to ask of our Mother Earth her age, but Science acknowledges no shame and from time to time has boldly attempted to wrest from her a secret which is proverbially well guarded.” (Holmes, 1913)

RESUMO

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A formação de grandes províncias ígneas (LIPs) está diretamente associada a eventos geológicos globais que incluem ruptura de (super)continentes, abertura de oceanos e crises bióticas. Não é à toa, portanto, que o estudo dos LIPs continua sendo um dos temas mais importantes da fronteira científica, cujo perfil multidisciplinar inclui as áreas de Petrologia, Geoquímica, Geocronologia, entre outras. Neste projeto de doutorado, o foco é a caracterização geocronológica de produtos ígneos expostos no NE do Brasil e relacionados à Província Magmática do Atlântico Central (CAMP) e à Província Magmática do Atlântico Equatorial (EQUAMP). A CAMP é datada em ca. 201 Ma e está correlacionada com a abertura do Oceano Atlântico Central. No NE do Brasil, essas rochas ocorrem na forma de diques, derrames basálticos e soleiras com assinatura geoquímica de magmas toleíticos, identificados tanto na Bacia (paleozoica) do Parnaíba quanto na Província (precambriana) Borborema. CAMP é sabidamente representada por lavas máficas na borda oeste da bacia, e corpos intrusivos (diques e soleiras) na borda leste, enquanto EQUAMP se destaca por enxames de diques máficos que somam aproximadamente 2.000 km de extensão, intrusivos no embasamento precambriano, e soleiras máficas restritas à borda leste da bacia o Parnaíba. CAMP e EQUAMP são caracteristicamente constituídas por diabásios toleíticos de baixo-Ti (TiO_2 <2% wt.%) e alto-Ti (TiO_2 >2% wt.%), sendo os magmas CAMP dominados pela assinatura de baixo-Ti, enquanto em EQUAMP predominam magmas de alto-Ti. A caracterização da idade de formação dessas províncias magmáticas exigiu uma abordagem multitécnica, dadas suas dimensões continentais e escassez de dados geocronológicos prévios. As técnicas incluíram a datação K/Ar sem traçador (em rocha total), $^{40}Ar/^{39}Ar$ como suporte às idades K/Ar, e U-Pb por abrasão química e diluição isotópica (em zircão), visando: (1) estabelecer padrões de idades (Jurássico e Cretáceo) dos vários enxames de diques até então não investigados por nenhum método geocronológico; (2) obter idades precisas dos dois eventos – CAMP e EQUAMP, nos vários alvos investigados; (3) discutir sobre duração de cada evento considerando o entendimento moderno sobre LIPs – de curta (<5 Ma) ou longa (>5 Ma) duração, especialmente para EQUAMP; e (4) discutir implicações geodinâmicas e paleoambientais. Além do esforço em obter os dados científicos, este projeto contemplou a implementação de rotinas analíticas, incluindo um novo método de recuperação de zircão a partir de rochas máficas (sub)vulcânicas. Como resultado, essa tese mostra que o magmatismo EQUAMP afeta amplamente o NE do Brasil e inequivocamente se relaciona com o evento Weissert (Valanginiano) de mudanças paleoambientais, enquanto ainda compartilha, em parte, áreas geográficas/geológicas previamente afetadas pelos magmas da CAMP. A relação entre a CAMP e a crise biótica do Triássico é aqui rediscutida sob um ponto de vista diferenciado àquele proposto na literatura.

Palavras-chave: grandes províncias ígneas, método K-Ar, método U-Pb CA-ID TIMS.

ABSTRACT

Oliveira, A.L., 2022, Geochronology of Mesozoic Large Igneous Provinces in NE Brazil [Doctorate thesis], São Paulo, Instituto de Geociências, Universidade de São Paulo, 297 p.

The formation of large igneous provinces (LIPs) is directly associated with global geological events that include rupture of (super)continents, opening of oceans and biotic crises. It is not unexpected, therefore, that the study of LIPs remains one of the most important topics on the scientific frontier, whose multidisciplinary profile includes the areas of Petrology, Geochemistry, Geochronology, among others. In this doctoral project, the focus is the geochronological characterization of igneous products exposed in the NE region of Brazil and related to the Central Atlantic Magmatic Province (CAMP) and the Equatorial Atlantic Magmatic Province (EQUAMP). The CAMP is dated to ca. 201 Ma and is correlated with the opening of the Central Atlantic Ocean. In NE Brazil, these rocks occur in the form of dykes, basaltic flows, and sills with a geochemical signature of tholeiitic magmas, identified both in the (Paleozoic) Parnaíba Basin and in the (Precambrian) Borborema Province. The CAMP is known to be represented by mafic lavas at the western edge of the basin, and intrusive bodies (dykes and sills) at the eastern edge. The EQUAMP is distinguished by mafic dyke swarms that total approximately 2,000 km in length, intrusive in the Precambrian basement, and mafic sills restricted to the eastern edge of the Paraníba basin. CAMP and EQUAMP are characteristically constituted by low-Ti (TiO_2 <2% wt.%) and high-Ti (TiO_2 >2% wt.%) tholeiitic diabases, with CAMP magmas dominated by the low-Ti signature, while in the EQUAMP predominate high-Ti magmas. The characterization of the age of formation of these magmatic provinces required a multi-technical approach, given their continental dimensions and scarcity of previous geochronological data. Techniques included unspiked K/Ar dating (in whole rock) with supporting $^{40}Ar/^{39}Ar$ dates, and U-Pb dating by chemical abrasion and isotopic dilution (in zircon), aiming to: (1) establish age patterns (Jurassic or Cretaceous) of the various dyke swarms until then not investigated by any geochronological method; (2) to obtain precise ages of the two events – CAMP and EQUAMP, in the various investigated targets; (3) discuss the duration of each event considering the modern understanding of LIPs – short (<5 Ma) or long (>5 Ma) duration, especially for the EQUAMP; and (4) discuss geodynamic and paleoenvironmental implications. In addition to the effort to obtain scientific data, this project included the implementation of analytical routines, including a new method for recovering zircon from (sub)volcanic mafic rocks. As a main result, this thesis shows that EQUAMP magmatism largely affects NE Brazilian terranes and is unequivocally related to the Weissert (Valanginian) event of paleoenvironmental changes, while also sharing geographic/geological areas previously affected by CAMP magmas. The relationship between CAMP and the Triassic biotic crisis is discussed on a different point of view to that currently proposed in the literature.

Keywords: large igneous provinces, unspiked K-Ar method, $^{40}Ar/^{39}Ar$ method, U-Pb CA-ID TIMS method.

1. INTRODUCTION

1.1 Presentation, Motivation and Objectives

The study of Large Igneous Provinces (LIPs) is a theme of scientific frontier since they have been commonly associated with important geologic events such as continental breakup, climatic changes, and biotic crisis. Until recently, the Central Atlantic Magmatic Province (CAMP; Marzoli et al., 1999) and the Paraná-Etendeka Magmatic Province (PEMP; Peate et al., 1992) were the only LIPs recognized in South America (Fig. 1) as result of the breakup of the Neoproterozoic Gondwana supercontinent. The igneous products of the CAMP are exposed in the north (Amazonas and Solimões basins and Guyana Shield), northeast (Parnaíba Basin), central (Parecis Basin) and west (Bolivia) regions of South America (Montes-Lauar et al., 1994; Eiras et al., 1994; Deckart et al., 1997, 2005; de Min et al., 2003; Cunha et al., 2007; Merle et al., 2011; Klein et al., 2013; Bertrand et al., 2014; Davies et al., 2017; Heimdal et al., 2018, 2019; Teixeira et al., 2019; Rezende et al., 2021), whereas the PEMP is exposed in south-southeast region of Brazil with marginal occurrences in Paraguay, Argentina and Uruguay (e.g., Hawkesworth et al., 1988, 1992; Piccirilo and Melfi, 1988; Ernesto et al., 1990; Peate et al., 1990, 1992, 1999; Cañón-Tapia, 2018; Foulger, 2018). Both provinces were formed during (or closely prior to) the opening of the Central and South Atlantic Ocean.

Besides them, two other magmatic events of Mesozoic age have been long-term reported in the literature but never considered in the context of a LIP (Fig. 1). These are a ‘complex of sills’ formally named Sardinha Formation (Bellieni et al., 1990; Fodor et al., 1990; Baksi and Archibald, 1997) intrusive in the Paleozoic sediments of the Parnaíba Basin, and the ‘Rio Ceará Mirim (RCM) dike swarm’ (Bellieni et al., 1992; De Oliveira, 1993; Hollanda et al., 2006; Ngonge et al., 2016a) intrusive in the Precambrian terranes of the Borborema Province. Together with two hitherto unstudied dike swarms named Canindé and Riacho do Cordeiro, the Sardinha sills and the RCM dike swarm were recently (re)interpreted as part of a single LIP in South America, formed synchronously to the early stage of West Gondwana breakup at the Lower Cretaceous (Hollanda et al., 2019). Hollanda et al (2019) made use of a comparison of lithological/chemical and chronological data published in the literature to collectively refer to the sills and dikes as the Equatorial Atlantic Magmatic Province (EQUAMP). They reinforced the previous agreement that the older Cretaceous volcanic rocks that crop out at the Benue Trough (Matos, 1992, 2000; Maluski et al., 1995; Coulon et al., 1996) would be the African counterpart of the EQUAMP.

This PhD project dedicates to apply geochronology to the mafic sills and dike swarms that occur in NE Brazil in order to provide reliable information to test the validity of the purpose in favor of a single (and major) magmatic province. The hypothesis, if proven correct, would lead to the timing constrain of the EQUAMP, formed by the collective grouping of the Parnaíba basin sills (in its eastern domain), together with the RCM, Canindé and Riacho do Cordeiro dike swarms. The additional implication of such statement is the possible correlation or genetic link between this novel Cretaceous LIP to the other South American (and African) magmatic province further south of the Atlantic Ocean, the PEMP; as well as its relation to environmental crisis that have occurred at the Lower Cretaceous Epoch (i.e., the Weissert Event; Lini et al., 1992; Weissert et al., 1998; Erba et al., 2004). This work was benefited by recent studies dealing with the geochemical characterization of these rocks that were carried out within a MSc. Dissertation (A.R. Dantas, 2021) and a PhD Thesis (A.A. Macêdo Filho, 2021) financed by a major research project – '*The Equatorial Atlantic Magmatic Province: a geochemical and geochronological approach*' (FAPESP 2017/08423-9) that had also financed this study. In the course of the execution of this umbrella project, rocks reported as CAMP representatives were found spatially entangled with the EQUAMP products leading us to also give attention to this older magmatic province and the implications of this event in South America.

1.2 Thesis Structure

The thesis is organized into six chapters. One introductory chapter exposes the main concepts related to LIP studies, particularly geochronology applications, and general information regarding South American LIPs that are invoked in the discussions of the results. The next four chapters encompass the results obtained from the execution of the analytical program. These chapters are presented in the format of scientific articles either published or as manuscript drafts. Two of them deal with the new laboratory routines designed to support geochronological studies in mafic rocks that were developed exclusively during the execution of this PhD project. The other two chapters encompass the resulting high-precision ages discussed in the context of timing and duration of the EQUAMP or CAMP in NE Brazil. Finally, the sixth and last chapter deals with the major conclusion topics of the collective manuscripts and proposes further research topics aroused by these findings. To note, the second chapter of this thesis is presented in English (United Kingdom) because it was accepted at the Geological Society Special Publications, an English journal. All other chapters are presented in English (United States). The strategy adopted to date the EQUAMP rocks was based on a three-fold analytical approach. Taking into account the huge extent of the exposure area of the dike swarms and the variable degree of alteration of the rocks, we firstly applied an exploratory K/Ar method before selecting samples for U-Pb dating. The protocols of the unspiked K-Ar method (Cassignol et al., 1978; Cassignol and Gillot, 1982; Gillot and Cornette, 1986) were setup in a modern noble gas mass spectrometer installed at the Isotope Geology Research Center (or Centro de Pesquisas em Geocronologia e Geoquímica Isotópica – CPGeo, USP), and the results are presented in the article "*Using a 'speedy' unspiked K-Ar methodology to investigate age patterns in giant dyke swarms"* (chapter 2), published in a special volume (n. 518) of the Geological Society of London (Oliveira et al., 2021).

The age intervals indicated by the K-Ar method were then refined with U-Pb zircon geochronology. To make zircon geochronology possible in the execution of this analytical program, we developed a new routine to recover zircon from mafic/ultramafic rocks (chapter 3) during a one-year sandwich doctorate in the Isotope Geology Laboratory (IGL) at Boise State University (BSU). These protocols were organized into the article "*A bulk annealing and dissolution-based zircon concentration method for mafic rocks*" recently published in Chemical Geology (final proof in production). Thus, the precise timing and duration of CAMP (chapter 4) and EQUAMP (chapter 5) rocks in NE Brazil was possible to be established using the CA-ID TIMS (Chemical Abrasion Isotope Dilution Thermal Ionization Mass Spectrometry) U-Pb technique on zircon. These results allowed the connection linkage between these provinces and the global environmental crisis that occurred synchronously to their emplacement.

1.3 Definition of a Large Igneous Province

The term LIP was firstly mentioned by Coffin and Eldholm (1992) to define large continental igneous provinces dominated by mafic rocks (mainly continental flood basalts). Since then, it has been popularized by the geoscientific community and applied to a number of igneous events preserved around the globe (e.g., CAMP, Siberian Traps, Deccan, Karroo-Ferrar; see Lightfoot and Hawkesworth, 1988; Sobolev et al., 2011; Svensen et al., 2012; Ernst, 2014; Latyshev et al., 2018 for examples). More than two decades passed after the introduction of the LIP term and several ‘re-readings’ of it were proposed (Table 1).

Table 1 – Synthesis of definitions published for the “Large Igneous Province” term.

Reference	Definition
Coffin and Eldholm, 1994	"LIPs are massive crustal emplacements of predominantly mafic (Mg and Fe rich) extrusive and intrusive rock which originate via processes other than 'normal' seafloor spreading."
Saunders, 2005	"The key aspect of large igneous provinces (LIPs) is that they represent anomalously high magmatic fluxes. The magma is usually basaltic, but may be rhyolitic. They are large in area, covering many thousands if not millions of square kilometres, and they testify to unusual geological processes, involving large amounts of thermal energy."
Sheth, 2007	"...the term LIP should cover all large volcanic and intrusive igneous provinces, irrespective of emplacement mechanism or compositional affinity [...] a lower limit of 50,000 km^2 [..] I thus suggests that the term LIP be used in its broadest sense, and propose new necessary, more specific terms for the discrete LIP categories."
Bryan and Ernst, 2008	“Large Igneous Provinces are magmatic provinces with areal extents >0.1 Mkm^2, igneous volumes >0.1 Mkm^3 and maximum lifespans of ~ 50 Myrs that have intraplate tectonic settings or geochemical affinities, and are characterized by igneous pulse(s) of short duration (~1–5 Myrs), during which a large proportion (>75%) of the total igneous volume has been emplaced.”

In this thesis, the more comprehensive description of Ernst (2014) is favored and includes attributes of volume, areal extent, composition, and chronology, and says:

> A LIP is a mainly mafic (+ultramafic) magmatic province with areal extent > 0.1 Mkm^2 and igneous volume > 0.1 Mkm^3, that has intraplate characteristic, and is emplaced in a short duration pulse or multiple pulses (less than 1-5 Ma) with a maximum duration of < c. 50 Ma. Silicic magmatism (including that of LIP scale, termed Silicic LIPs (SLIPs)) and also carbonatites and kimberlites may be associated.

An important outcome of the study of LIPs concerns the link between large volumes of magma erupted and greenhouse gases expelled in short duration periods, which is considered key to correlate them with important extinction events and environmental changes on Earth (e.g., Saunders, 2005; White and Saunders, 2005; Ernst and Youbi, 2017). Among the "big five" mass extinction events recognized on Earth, four of them are linked to LIPs: Yakutsk-Vilyui (Late Devonian), Siberian Traps (End Permian), CAMP (End Triassic) and Deccan (End Cretaceous), while the fifth and an older one (End Ordovician) is still not totally understood (Bond and Grasby, 2017; Ernst and Youbi, 2017). Similarly sized LIPs erupted in the Early Cretaceous are better related to environmental changes like the Oceanic Anoxic Events (OAE) and Carbon Isotope Excursions (CIE), instead of mass extinction events. The Paraná-Etendeka LIP is a good example in which the timing of lava eruption has been linked with the Valanginian 'Weissert' positive $\delta^{13}C$ excursion (Weissert et al., 1998; Wignall, 2001; Erba et al., 2004; Cavalheiro et al., 2021). Despite the importance as environmental proxies, LIPs have been widely used to reconstruct the paleogeography of ancient landmasses being essential tools for the elaboration of a global barcode throughout the Archean to Mesozoic times with a relatively constant frequency of about 1 per 20 myr since 2600 Ma until 180 Ma, while from 180 Ma to the present day, the frequency becomes 1 per 10 myr (Ernst et al., 2005; Ernst, 2014). Once the timing constrain is implied on the definition of the term, positioning of any LIP on such barcode and testifying an association with supercontinent breakup requires that its igneous products are precisely dated, including as a way to unequivocally correlate coeval events on multiple continents.

1.4 Geochronology Applied to Large Igneous Provinces

The majority of works that deal with the dating of LIP products consider the use of $^{40}Ar/^{39}Ar$ or U-Pb radiometric methods. The former is suitable to K-bearing rocks or minerals since potassium is a major oxide (> 1 wt.%) in many crustal rocks and it is common that these minerals (e.g., sanidine, plagioclase, amphibole or micas) are present as important modal constituents in igneous rocks (e.g., Renne et al., 1996b; Venkatesan et al., 1997; Marzoli et al., 1999; Knight et al., 2003; Jourdan et al., 2004, 2005, 2009; Nomade et al., 2007; Reichow et al., 2009; Ivanov et al., 2009; Merle et al., 2011; Ricci et al., 2013; Baksi, 2014, 2018; Samant et al., 2019; Sprain et al., 2019). Nevertheless, it often pertains as a problematic methodology since mineral alterations and low spatial analytical accuracy can be a major factor that disrupts the measurements and add uncertainties and complexities to the obtained ages.

The determination of K/Ar analysis requires that the sample is completely degassed (usually on a single heating stage), freeing all Ar present in the sample to be measured by the spectrometer, so that it can be calculated relative to the K present on the sample, this is referred as the “total gas” or “total fusion” age (Kelley, 2002b). However, on $^{40}Ar/^{39}Ar$ analyzes, K and Ar contents are measured at the same time (Merrihue and Turner, 1966) and the father-daughter isotopic ratio is determined on each step. Therefore, the most important advancement of the K/Ar radiogenic clock was the development of the $^{40}Ar/^{39}Ar$ step heating method (see McDougall and Harrison, 1999 for a complete review of the $^{40}Ar/^{39}Ar$ method). This method does not have a maximum age of determination (at least not in the geologic timespan) because, technically, measurements became increasingly easier with radiogenic argon accumulation. That is, older samples are easier to date because of the accumulation of ^{40}Ar from the decay of ^{40}K on the sample. Conversely, the less abundance of radiogenic ^{40}Ar ($^{40}Ar^*$) makes it harder to analyze young samples or low-concentration Ar phases, like plagioclase.

The $^{40}Ar/^{39}Ar$ method is capable of analyzing the $^{40}Ar^*$ proportionally to the ^{40}K of the sample on a single aliquot by irradiating samples on a fast neutron flux reactor, which "forces" the conversion of elements in the sample (e.g., K, Cl, Ca) to correspondent Ar isotopes (i.e., ^{40}Ar, ^{39}Ar, ^{38}Ar, ^{37}Ar and ^{36}Ar), subsequently measured by the spectrometer. It also advances the interpretation of the results by correlating the Ar isotope measurements to their related parent element (e.g., a K/Ca ratio derived from ^{39}Ar and ^{37}Ar isotopes that allows the evaluation of the chemical signature of each step). Unfortunately, the irradiation procedure introduces many variables that must be accounted for, like the production of undesirable radioactive elements and Ar isotope isobaric interferences (e.g., ^{37}Ar from ^{37}Ca). The irradiation also introduces the calculation of a calibration factor for the radionuclides produced during the irradiation (integrated neutron flux gradient). For this, a neutron flux (monitor) mineral is used to indirectly determine the integrated gradient that the sample has received, a function of the number of fast-neutron bombardments and the amount of parent nuclides. The monitor mineral age must be known through K/Ar measurements, also named first principle dating, (Lanphere and Dalrymple, 2000; McDougall and Wellman, 2011) or by other geochronological methods such as U-Pb (Min et al., 2000; Villeneuve et al., 2000; Lanphere and Baadsgaard, 2001; Renne et al., 2010) or astronomical dating (Renne et al., 1994; Hilgen et al., 1997; Kuiper et al., 2008).

The branched decay by electron capture (λ_{ε}) or beta particle emissions (λ_{β}) of ^{40}K to ^{40}Ar and ^{40}Ca, is another problematic department regarding the K/Ar system, where the total radioactive decay constant (λ) is a sum of the two decay mechanisms. The ^{40}K λ is a considerable source of error of the method and is usually the subject of constant revision and attempt of advancement by the community (Min et al., 2000; Kuiper et al., 2008; Renne et al., 2010, 2011; Schwarz et al., 2011). The total ^{40}K λ has to take some factors in perspective; primarily, the values used by the geochronology community (Steiger and Jäger, 1977) and physics community (Audi et al., 2003) are not the same. That by itself already represents how controversial and difficult to determine this constant is. The overall values are poorly disclosed with little to no error propagation reports, counting methods, correction values and equations reviews (Min et al., 2000). Furthermore, continuous reviews of values and constants used on the K-Ar system age equations were reported but little to no attention was given to introduce these changes to the calculation of the dates (Min et al., 2000; Renne et al., 2010).

The values of 5.543 x 10^{-10} a^{-1} with a branching ratio of 0.1171 for λ_{ε}/ λ_{β} reported by Steiger and Jäger (1977) did not consider uncertainties (Min et al., 2000; Renne et al., 2010) and the application of such uncertainties alone could represent small variations on the ages obtained by the method (Min et al., 2000). The review of the constant and the scrutiny of once accepted values became a focal point of discussion on the early 2000's to the 2010's when reported errors of the ^{40}Ar/^{39}Ar method began to fall below 0.2% precision at quoted 1σ level (Min et al., 2000; Kuiper et al., 2008; Renne et al., 2010, 2011; Schwarz et al., 2011). The λ value of radiogenic elements can be determined by (i) disintegrating counting, or, (ii) measuring parent and daughter radionuclides of a material whose age is well known (Min et al., 2000). Notwithstanding, Renne et al. (1998) emphasized that systematic errors and bias on monitor minerals could be drastically improved by inter-calibration of the standards by another independent measurement (like astronomical tuning or the U-Pb method) and that measuring a direct disintegrating value would introduce many random errors, one order of magnitude higher than what could be obtained from the second (ii) approach. However, errors of the total ^{40}K λ still accounted greatly to the precision of the method and a better improvement of the constant would be indispensable for the improvement of the method itself. Similarly, Kuiper et al. (2008) showed that the ^{40}K λ could be used without considering the branch decay (only the total constant value) by applying an inter-calibration factor of the $^{40}Ar/^{39}Ar$ method with the astronomical tuning of cyclic sedimentary sequences. By doing so, the inter-calibration of the $^{40}Ar/^{39}Ar$ system could be fine-tuned to a higher precision method and provide a synchronized (and intercalibrated) age to other geochronometers like the U-Pb method or the astronomical tuning dating technique (Renne et al., 1994; Hilgen et al., 1997; Kuiper et al., 2008). This approach was able to refine the age for the monitor mineral Fish Canyon sanidine (28.201 ± 0.046 Ma) and reduced the uncertainty of the $^{40}Ar/^{39}Ar$ method by one order of magnitude (Kuiper et al., 2008). In the same way, an inter-calibration or the $^{40}Ar/^{39}Ar$ ages to the U-Pb ages of known samples could be used to determine the λ_{ε} and λ_{β} of the λ and intercalibrate the total decay constant from the assumption of a correct age value of a standard (Renne et al., 2010, 2011).

Additionally, the reported uncertainties of the $^{40}Ar/^{39}Ar$ ages often omit the λ error but the comparison to other geochronological methods (e.g., the U-Pb method) must take it under consideration (Renne et al., 1998, 2009). This is where the K/Ar and $^{40}Ar/^{39}Ar$ methods precision fails to achieve absolute precise measurements on pair to the U-Pb technique. However, it can be used to achieve results better than 0.2% precision at the 1σ level, capable of determining relative ages inside the 5 Ma window span of ages of a LIP (Sprain et al., 2019), even though the alteration of plagioclase must be carefully assessed to understand and correct for excess argon and recoil effects (Kelley, 2002a; Jourdan et al., 2007).

The U-Pb method, in turn, is applicable to U-rich (zircon, baddeleyite) and U-bearing (apatite, rutile, titanite) minerals that are preferentially found as trace constituents in evolved, intermediate to felsic rocks. The method has recently gained more visibility on studies carried out to date magmatic events related to LIPs (e.g., Burgess et al., 2015; Davies et al., 2017; Ivanov et al., 2017; Schoene et al., 2019) once it provides higher precision (and reliable) ages in comparison to $^{40}Ar/^{39}Ar$ ages because of: (i) the well-known and precise ^{238}U decay constant (uncertainty of 0.054%; Jaffey et al., 1971), and (ii) the ability of zircon to survive post-magmatic and weathering processes, which are the two main drawbacks of the K-Ar method and its derivative $^{40}Ar/^{39}Ar$ method. Nonetheless, the application of the U-Pb method in LIPs studies requires the proper determination of a mineral age avoiding mixed age zones within the minerals. This stimulated the development of new techniques to surpass or circumvent internal heterogeneous structures to solve erroneous “mixed” ages, noteworthy are the *in-situ* methods (Secondary Ionization Mass Spectrometry – SIMS, and Inductive Coupled Plasma Mass Spectrometry – ICPMS), imaging techniques (backscattered electrons – BSE, and cathodoluminescence – CL), and abrasion techniques like the air abrasion method (Krogh, 1982) and the chemical abrasion method (Mattinson, 2005).

The *in-situ* techniques must use a mineral standard to correlate the sample unknown signals to a previously known measurement (mineral standard ages are defined by the TIMS method), so SIMS and ICPMS can measure the isotopic contents of minerals and compare to a standard value obtained from known crystals. By these methods, zircon minerals can even be measured in their petrographic context or domains, although unfortunately, the ionization of particles from the zircon (or any other desired mineral) often ionizes every other element. Thus, mass fractionation corrections are the most relevant source of analytical errors of *in-situ* measurements. Because of that, *in-situ* techniques never achieved (and probably never will) the precision obtained by the TIMS method (Schoene, 2014).

The development of the chemical abrasion (CA) method (Mattinson, 2005) on crystals that do not have internal heterogeneous structures, is the one that works better to solve the discordance issue caused by Pb loss and produce the best possible age precision currently available. Through the CA method, zircon grains are leached on digestive acids that can remove alpha-particles and fission track totaled zones to obtain a residual, ‘perfectly closed-system’ zircon grains (Mattinson, 2005, 2011). The processes of annealing (heating up to 900 ºC for 60h), and partially dissolving zircon grains in multiple steps of acid treatment (29M HF) at temperatures from 180 to 190 ºC is capable of completely (or partially) removing zones of high-Th-U damage concentration (Mattinson, 2005). By a meticulous multi-step dissolution processes, the CA-ID TIMS method is capable of achieving precision values better than 0.1% on single grain analyzes (subject to variations due to tracer calibration values and decay constants) and is quoted as the most precise dating method currently available (Schmitz, 2012; Schoene, 2014). Furthermore, one of the primordial scientific goals that emerged from the CA treatment was the desire to further improve the precision values obtained on tracer (spike) calibration solutions. As TIMS laboratories are now able to report blank values on the order of a few femtograms (10^{-15} grams), the calibration solutions and clean lab facilities become increasingly important sources of errors (Schmitz, 2012). For this purpose, the EARTHTIME initiative (http://www.earthtimetestsite.com/) was created to better improve tracer solutions purity. As a result, a large aliquot of mixed (^{202}Pb-)^{205}Pb-^{233}U-^{235}U tracer was calibrated to limit spike uncertainties (Schmitz and Schoene, 2007; Schoene, 2014). This is particularly important because the tracer solution is added to the dissolved unknown samples and the uncertainties obtained from those measurements cannot be better than the uncertainty of the tracer calibration itself. Before the EARTHTIME initiative, spike solution uncertainties were around the 0.1% precision mark (often bigger than the CA-ID TIMS precision limit) (Schoene, 2014). Now, the EARTHTIME spike uncertainty is quoted to range between 0.05 to 0.03% (Schmitz, 2012; Schoene, 2014; McLean et al., 2015a) and synthetic solutions are used to account for the reproducibility of analytical parameters (Schaltegger et al., 2021).

Since LIPs are rapid igneous events that require high-precision geochronological data, the zircon U-Pb CA-ID TIMS is the most common method applied to that end. The mass fractionation bias of the SIMS and ICPMS analyzes often hinders its use on such context because the precision values that arise are a few orders of magnitude greater than what is obtained from the TIMS method. Still, the LA-ICPMS is commonly used to pre-select (by a non-destructive method) which zircon grains are the best to be treated and analyzed by the much more complex and precise CA-ID TIMS method. Still, U-Pb dating use in mafic rocks has been an analytical challenge once U-bearing minerals (mainly zircon) occur in minor (sometimes negligible) proportion of volume in mafic rocks, making hard or unlikely the separation from the host rock. Even so, U-Pb baddeleyite dating has had historical success for mafic rocks since it can crystallize from the silica-undersaturated mafic melts. The earliest and most common applications of U-Pb baddeleyite geochronology were to Precambrian mafic intrusions (Krogh et al., 1987; LeCheminant and Heaman, 1989; Heaman et al., 1992), in geochronological contexts where a resolution of several millions of years were adequate to address the geological applications. Further application of U-Pb baddeleyite geochronology has been key to understanding ancient continental paleogeography, Large Igneous Provinces (LIPs) magmatism, and tectonic evolution (Heaman and LeCheminant, 2001; Heaman, 2009; Nilsson et al., 2010; Teixeira et al., 2015, 2019). However, baddeleyite is not as reliable as the zircon chronometer because of secondary Pb-loss problems (Rioux et al., 2010). This limitation hinders its use for highly accurate and precise (≤0.1% 2σ error) geochronology (Davis and Davis, 2010; Li et al., 2010; Schaltegger and Davies, 2017; Pohlner et al., 2020). With these recognized limitations of U-Pb baddeleyite geochronology, high-precision/high-accuracy U-Pb dating thus often relies on finding zircon crystals from segregated pods or evolved melts of mafic rocks. This approach applies a very selective filter to rocks and sampling sites, leaving many igneous events undated. Even so, numerous recent works have been published on high-precision zircon geochronology of LIPs to constrain global tectonic reconstructions (Bleeker and Ernst, 2006; Ernst et al., 2013) and to correlate mafic large igneous events to mass extinctions and environmental changes (Davies et al., 2017; Heimdal et al., 2018; Schoene et al., 2019). But it is the rarity and difficulty in concentrating zircon from mafic rocks that hinders its application to a wide range of tectonomagmatic events.

1.5 Brief comments on the CAMP and PEMP events in South America

Manifestation of the CAMP event is reported in several regions of South America including (1) the flood basalts at the western side of the Paleozoic Parnaíba Basin (known as Mosquito Formation; Bellieni et al., 1990; Fodor et al., 1990; de Min et al., 2003; Merle et al., 2011), (2) the sills and/or dikes in the Paleozoic Amazonas and Solimões (the Penatecaua magmatism; de Min et al., 2003), Parecis (Anari and Tapirapuã magmatism; Montes-Lauar et al., 1994) and eastern Parnaíba (Ernesto et al., 2003; Morais Neto et al., 2016; Heilbron et al., 2018; Fernandes et al., 2020) basins, (3) the dikes in the Precambrian Guiana Shield (Deckart et al., 1997, 2005), and finally, (4) the flood basalts and sills in Bolivia (Bertrand et al., 2014). In total, the CAMP covers an area of at least 10×10^6 km^2 with a maximum volume estimated of 3×10^6 km^3 (Marzoli et al., 2018). In all occurrences, CAMP rocks are represented by tholeiitic basalts and basaltic andesites generally grouped by geochemical affinities into: (i) low Ti (TiO_2 <2%) with higher and variable Mg# ((MgO+FeO)/MgO = 0.3 to 0.6) compositions, representing the least evolved rocks, and (ii) high Ti (TiO_2 >2%) and lower Mg# (= 0.1 to 0.2) compositions. As a whole, the CAMP compositions have been attributed to the melting of an asthenosphere enriched by subduction events beneath the Pangea supercontinent, with the variable influence of crustal assimilation and fractional crystallization (see Marzoli et al., 2018 for a review). Merle et al. (2011) had modeled the compositions of the CAMP lavas in the western Parnaíba Basin as a result of contamination of asthenosphere-like sources by ultra-alkaline liquids derived from a metasomatized lithospheric mantle reservoir followed by fractionation of a primitive mineral assemblage consisted mainly of olivine, plagioclase, and augite.

Global-scale warming followed by diffuse melting of the mantle beneath Pangea (McHone, 2000) has been preferentially evoked to explain the elongate boundary (about 8,000 km-long) and chemical signature of the CAMP magmatism from the north to the south hemispheres (Marzoli et al., 2018) instead of the initial supposition of a plume source (White and Mckenzie, 1989; Wilson, 1997). The $^{40}Ar/^{39}Ar$ method was applied on rocks of the South American continent and associated the occurrences to those on the other three continents, which constrained the peak of the magmatic activity around 200 Ma. With that timing interval and age errors, the CAMP magmatism was broadly associated with the Triassic-Jurassic boundary and, consequently, with one of the five major mass extinction events, the end-Triassic extinction event. Using the parameters adopted by Baksi (2003), the CAMP ages were mainly composed of three magmatic peaks on an interval between 205-190 Ma, with the major cluster of ages around 200 Ma (as previously published by Marzoli et al., 1999). However, according to Baksi (2003), an evaluation of ages from north to south suggested that the northern segment of the CAMP (North America and Europe) predated the southern segment (Brazil) by ca. 1.5 myr. He also proposed that the magmatism began at ca. 205 Ma, just before the T-J boundary, and peaked after the age interval of the mass extinction event.

In South America, numerous $^{40}Ar/^{39}Ar$ works have proposed an age range from ca. 181 Ma to 208 Ma (Deckart et al., 1997; de Min et al., 2003; Merle et al., 2011; Bertrand et al., 2014; Heilbron et al., 2018; Fernandes et al., 2020). However, widely younger ages around 181 Ma (de Min et al., 2003; Heilbron et al., 2018) and slightly older ages (> 202 Ma; Fernandes et al., 2020) appear to be derived from alterations or excess argon rather than subordinate magmatic pulses, as in general the bulk data are constrained on an interval between 196-201 Ma, correlative to the global CAMP. Thus, the $^{40}Ar/^{39}Ar$ method applied to the CAMP provided age constraints that attested for a bulk magmatism around 200-199 Ma. Taking this alleged interval as a strategic route, the U-Pb CA-ID TIMS method was used to validate and consolidate this age interval around the global occurrences of the CAMP. The results revealed that the CAMP magmatism was constrained on a much smaller interval of less than 1 myr by one or two magmatic pulses (Schoene et al., 2010; Blackburn et al., 2013; Davies et al., 2017, 2021; Heilbron et al., 2018). Additionally, the interval of ages was older than previously proposed, around 201.3 to 201.6 Ma with error estimations of less than 0.1% at the 95% confidence interval.

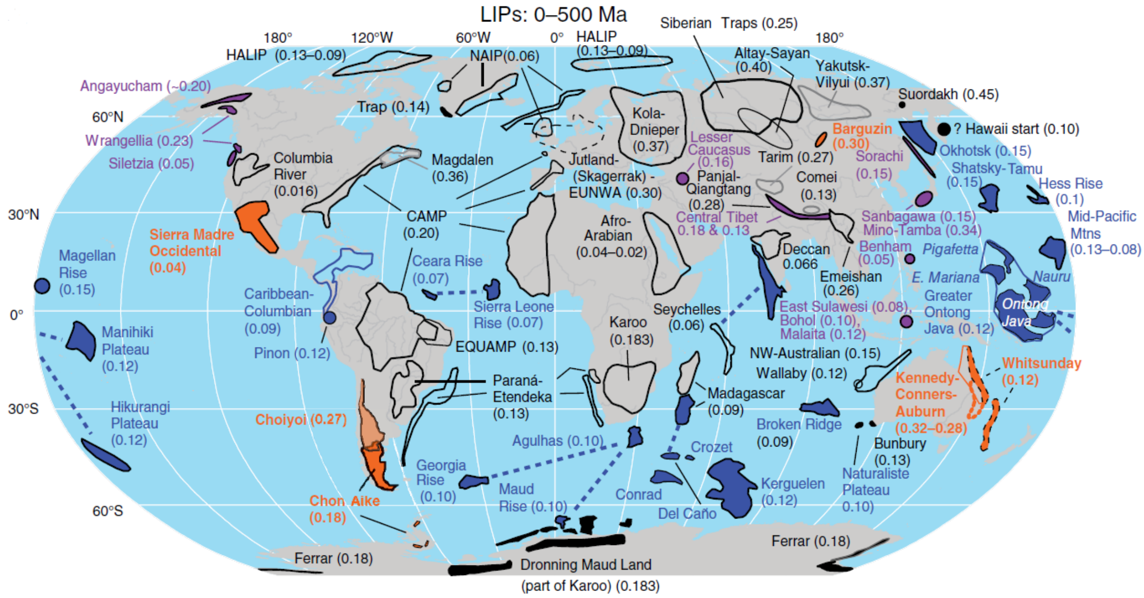

Figure 1 - Global map showing the schematic distribution of LIPs and SLIPs through 0-500 Ma. Abbreviations: CAMP = Central Atlantic Magmatic Province; HALIP = High Arctic Large Igneous Province; NAIP = North Atlantic Igneous Province; EQUAMP = Equatorial Atlantic Magmatic Province; EUNWA = European-Northwest African Magmatic Province. Continental LIPs are highlighted in black, oceanic LIPs in blue, accreted oceanic LIPs in purple, and SLIPs in orange. See review on each event in Ernst (2014). Source: (Ernst et al., 2021).

The PEMP encloses a huge volume of basaltic flows and, at least on the surface, subordinate sills covering large continental areas in Brazil, Argentina, Uruguay and Paraguay (Bellieni et al., 1984; Peate et al., 1990, 1992; Rämö et al., 2016). Dike swarms are currently restricted to coastal areas along the southeast margin of Brazil (Santa Catarina, São Paulo and Rio de Janeiro states) and Namibia, while the Ponta Grossa swarm propagates inland in the Paraná State (Raposo, 1995; Raposo and Ernesto, 1995b, 1995a; Ewart et al., 2004; Gibson et al., 2005). The entire province covers an area of at least 1.2 × 10^6 km^2 with volume estimated of 1 × 10^6 km^3 (Peate et al., 1992), one order of magnitude lower than the CAMP in area, but with roughly equal volume.

Geochemical characterization of the magmas that form the PEMP is a separate subject and requires more than a brief overview presented here and the reader is referred to Peate et al. (1992) as a preliminary, but a comprehensive, reference for it. The PEMP encompasses mainly volcanic rocks of bimodal nature with a remarkable prevalence of tholeiitic basalts and basaltic andesites (<60 wt.% SiO_2) terms relative to those of acid composition (rhyolites and rhyodacites; >64 wt.% SiO_2). These rocks are grouped into low- and high-Ti types according to an arbitrary limit of 2 wt.% of TiO_2 (Bellieni et al., 1984, 1986; Mantovani et al., 1985), which was after equated to a Ti/Y ratio of 310 by Peate et al. (1992). These authors used the Ti-based classification combined with other geochemical parameters to subdivide the PEMP magmas into eight distinct types. Low-Ti compositions are thus arranged into the basaltic magma types Gramado, Esmeralda and Ribeira, and the acid Palmas type, while Paranapanema, Pitanga and Urubici are the high-Ti basalt types and Chapecó is the high-Ti acid type. This classification has been widely adopted in subsequent works dealing with petrological aspects of PEMP. Despite this consensus, there exists a long-term debate whether the origin of these magmas was related to mantle melting by a plume (Schilling et al., 1985; Hawkesworth et al., 1992; Renne et al., 1992; De Min et al., 2018; Pearce et al., 2021) or via alternative mechanisms such as edge-drive convection along craton-orogen discontinuities as proposed by King and Ritsema (2000) or large-scale mantle warming (e.g., Marques et al., 1999; Peate et al., 1999; Coltice et al., 2007; Rocha-Júnior et al., 2012, 2013). The common aspect mentioned in all these works regardless of the preferred model is the fact that the PEMP magmas have typically enriched (subduction-like) signatures requiring the involvement of the metasomatized lithosphere in their genesis.

The geochronology of the province has been comprehensively conceived by the $^{40}Ar/^{39}Ar$ and U-Pb methods (Renne et al., 1992, 1996b; Turner et al., 1994; Thiede and Vasconcelos, 2010; Janasi et al., 2011; Florisbal et al., 2014; Almeida et al., 2018), with recent high precision CA-ID TIMS data (Rocha et al., 2020) and thorough reviews being published (Gomes and Vasconcelos, 2021). The geochemical groups of the PEMP were a first representation of distinct mantellic sources taking place during this igneous event. A stratigraphic evolution from the low- (older) to the high-Ti (younger) lavas was indicated in the southeastern portion of the Paraná Basin (Peate et al., 1990, 1992). However, even though many different magma types were described, the age difference was not considered to be extensive. Reported $^{40}Ar/^{39}Ar$ analyzes of fresh plagioclase samples yielded an interval around 132.9 ± 0.6 to 131.4 ± 1.6 Ma, indistinguishable from a best estimated age of 132.6 ± 1.3 Ma for the entire province (Renne et al., 1992). The interval published by Renne et al. (1992) of around 1.3 Ma for the entire PEMP magmatism was reinforced by the marginally younger Ponta Grossa dikes, supposedly a feeding system for the younger volcanics of the Paraná magmatism, with an age interval from 131.4 ± 0.5 to 129.2 ± 0.5 Ma (Renne et al., 1996a). This interval was also endorsed by the Etendeka volcanic rocks in Africa, which yielded $^{40}Ar/^{39}Ar$ plateau ages ranging from 132.3 ± 0.7 to 131.7 ± 0.7

(Renne et al., 1996b). Thus, the chief volcanic activity in the Paraná Basin, their African counterpart (Etendeka volcanism) and the younger feeding system (i.e., Ponta Grossa dikes) constrained the whole magmatism of the PEMP between 0.6 ± 1.0 myr (Renne et al., 1996b).

Conversely, Turner et al. (1994) suggested, based on $^{40}Ar/^{39}Ar$ analyzes of whole rocks and plagioclase separates of boreholes, that the entire Paraná magmatism was constrained on a larger interval of approximately 10 myr, within 137 to 127 Ma. The inconsistency of reported ages (and intervals) by the two groups were a product of intense debate over the 90's decade. Finally, by re-dating the exact same samples, Thiede and Vasconcelos (2010) revised the ages of Turner et al. (1994) and revealed them to be indistinguishable from a best estimated age of 134.7 ± 1.0 Ma. Subsequently, baddeleyite and zircon U-Pb ages by the ID TIMS method produced high-precision absolute ages of 134.3 ± 0.8 and 133.4 ± 0.2 Ma (Janasi et al., 2011; Almeida et al., 2018). Latter endorsed by zircon U-Pb CA-ID TIMS ages (using EARTHTIME spikes) of Rocha et al. (2020) at the range of 132.72 ± 0.76 and 133.6 ± 0.12 Ma (weighted mean age calculated from 4 units of the low-Ti type) for the acid volcanic types (Chapecó and Palmas, respectively). Although the results by the $^{40}Ar/^{39}Ar$ method disclosed the magmatism of the PEMP to be constrained between approximately 1 myr, according to Janasi et al. (2011) the U-Pb ages combined with all previously reported results, excluding those from Turner et al. (1994), would restrict the PEMP magmatism to a slightly bigger interval of around 3 myr.

Overall, the accepted notion is that the magmatism took place on an interval of roughly 1.6 to 3 myr, from around 135 Ma to 132 Ma (Gomes and Vasconcelos, 2021). In general, both LIPs (CAMP and PEMP) are fast episodic events with over 90% of the total volume emplaced on intervals shorter than 5 myr with magma genesis driven by events that produce a generalized melting of the underlying subcontinental heterogeneous mantle.

1.6 The Equatorial Atlantic Magmatic Province - EQUAMP

Giant dike swarms are usually considered to be the main component of LIP plumbing systems representing sub-volcanic feeder channels of the lava flows (Ernst and Buchan, 1997; Ernst, 2014), whereas sill complexes have been interpreted as the result of lateral magma flow within a sedimentary setting usually associated to dikes in deeper crustal levels (e.g., Magee et al., 2016). This scenario has been invoked by Hollanda et al. (2019) to suggest a link between dike swarms and sills presently exposed in NE Brazil, and hitherto treated as products of separate magmatic activities, to form the plumbing system of a new Mesozoic LIP in South America. The EQUAMP (see figure 2) is, therefore, distinguished from the CAMP and PEMP by being exclusively represented by intrusive igneous components. These components are three different dike swarms intrusive in the Borborema Province and one sill province intrusive in the adjacent Parnaíba Basin.

The Borborema Province is a major Neoproterozoic crustal block formed after convergence between West African and Congo-São Francisco cratons together with the Parnaíba block (de Castro et al., 2014). In the pre-drift reconstructions, the Borborema Province shares several geological features with Western Africa (from Gana to Cameroon), enabling a straightforward correlation between them (Arthaud et al., 2008; De Wit et al., 2008; Ganade et al., 2016). For a more detailed overview of the Precambrian geology of the province, see van Schmus et al. (2008) and dos Santos et al. (2010, 2014).

The Parnaíba Basin (Góes and Feijó, 1994; Silva et al., 2003; Vaz et al., 2007) is a ~5 km sandstone-dominated succession including subordinate mudstones/shales and more locally limestones and evaporites, occupying a surface area of approximately 600,000 km^2. These deposits rest unconformably over the West Gondwanan Precambrian basement constituting three successive super-sequences delimited by regional erosive discordances: Silurian, Mesodevonian-Eocarboniferous and Neocarboniferous-Neotriassic (Vaz et al., 2007) representing a long-term (~245 myr) history of subsidence and sedimentation.

On this regional tectonic setting, the EQUAMP was emplaced along several swarms and the main product of the EQUAMP, the RCM dike swarm, totalizes a ca. 1,000 km-long arcuate swarm parallel to the present-day E- and NE-trending Atlantic margins. At least two other sub-sets of dikes of 250-300 km in length occur parallel to the Atlantic coastlines. Sills (also known as the Sardinha Formation), in turn, occur exclusively along the eastern side of the Parnaíba Basin covering an area of ca. 85,400 km^2 (Mocitaiba et al., 2017). Until recently the extent of the RCM dike swarm was estimated to be a ~350 km-long E-trending swarm, but linear structures were identified through high-resolution aeromagnetic surveys (www.cprm.gov.br) constituting a westward continuation of the dikes. At longitude 39ºW, these linear anomalies assume a NE-trend direction to meet the Sardinha sill province in the Parnaíba Basin (Fig. 2). Such lineaments were interpreted as mafic dikes, part of the

RCM on 1:100.000 and 1:250.000 geological maps and technical reports of the Brazilian Geological Survey as well as airborne geophysical mapping (Hollanda et al., 2019; Melo et al., 2021, 2022; Macêdo Filho and Hollanda, 2022). The RCM dike swarm, together with the other two subswarms, would constitute giant dike swarms extending over 1,500 kilometers (or even more) from the present-day eastern Atlantic coast inshore.

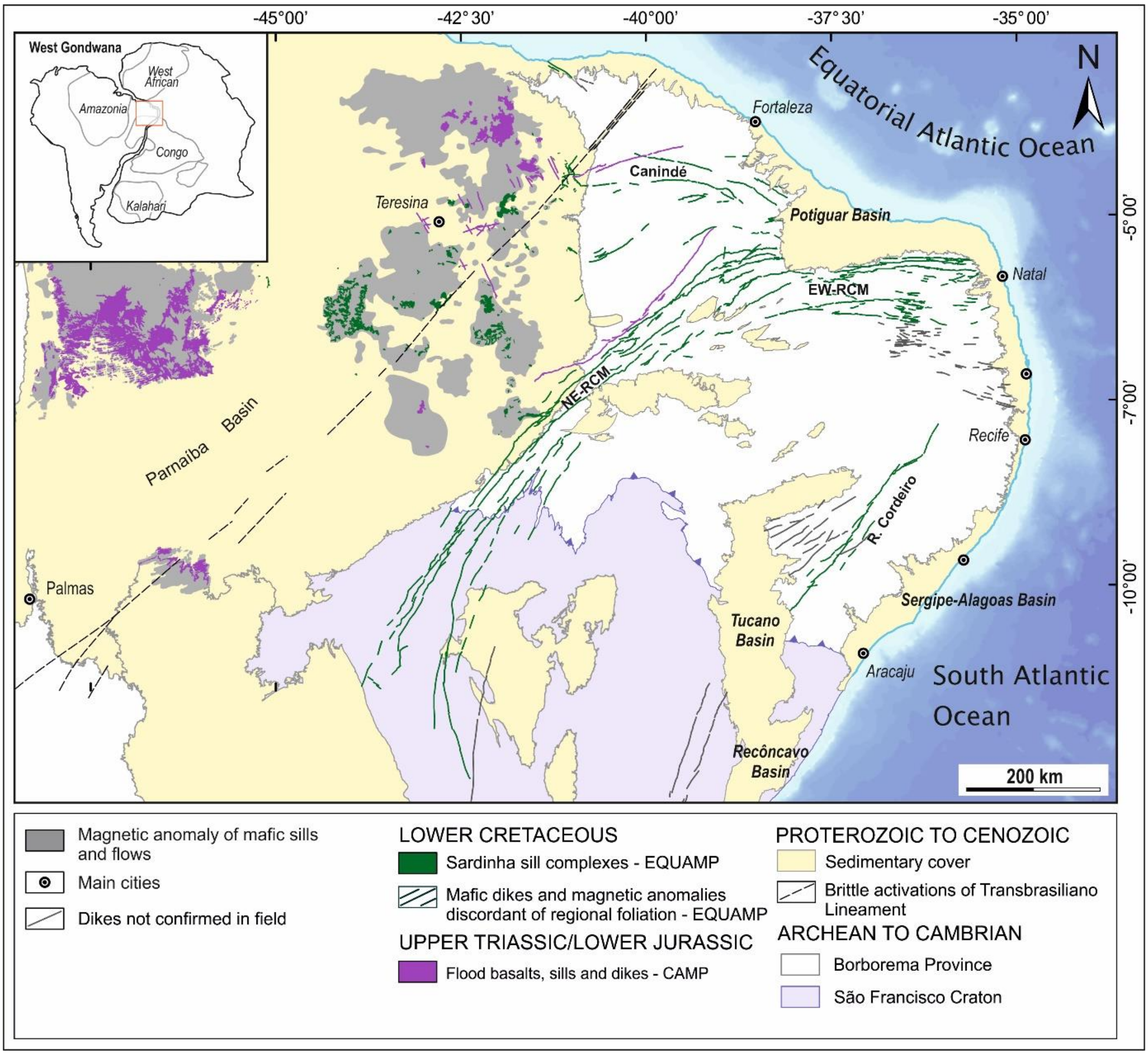

Figure 2 – Map of NE Brazil highlighting Mesozoic (Central Atlantic Magmatic Province – CAMP and Equatorial Atlantic Magmatic Province – EQUAMP) igneous events (modified from Macêdo Filho, 2021).

The mineral assemblage of the EQUAMP tholeiites consists of plagioclase, clinopyroxene (± olivine) and Fe-Ti oxides (Ngonge et al., 2016a; Dantas, 2021; Macêdo Filho and Hollanda, 2022). Elemental and isotope geochemical signatures were discussed in Bellieni et al. (1992), Hollanda et al. (2006), Ngonge et al. (2016a), Oliveira et al. (2018), Dantas (2021), Macêdo Filho (2021) and Macêdo Filho and

Hollanda (2022). Mostly, these rocks were divided into three groups: high-Ti olivine tholeiites, evolved high-Ti tholeiites (TiO_2≥2.0 wt.%; Ti/Y~360), and low-Ti tholeiites (TiO_2≤2.0 wt.%; Ti/Y≤360), all exhibiting distinct degrees of enrichment in incompatible elements relative to the Primitive Mantle. Negative to neutral Pb anomalies are common in high-Ti groups, while positive Pb are found in the low-Ti tholeiites (Macêdo Filho and Hollanda, 2022). Ngonge et al. (2016a) found that the initial isotopic compositions of the olivine tholeiites reveal a likely contribution of the FOZO (FOcal ZOne) component in their genesis ($^{87}Sr/^{86}Sr$ = 0.70339–0.70373, $^{143}Nd/^{144}Nd$ = 0.512518 to 0.512699 and $^{206}Pb/^{204}Pb$ >19.1). The other tholeiitic groups however showed variable Sr-Nd ratios with relatively consistent $^{206}Pb/^{204}Pb$ ratios clustering towards an enriched mantle (EM1) component on isotope diagrams. Such an enriched signature points to the involvement of a subduction-modified lithospheric mantle or a mantle plume as the source of the low- and high-Ti tholeiites (Macêdo Filho and Hollanda, 2022). Paleomagnetic data show that magnetization was mostly acquired in the Cretaceous, in accordance with other Early Cretaceous poles of South America and Africa (Ernesto et al., 2003).

No precise dating was performed on these dikes and the available K-Ar ages spread over a large interval of 35 myr (145 - 110 Ma; Bellieni et al., 1992; Mizusaki et al., 2002), with most data spanning throughout an interval between 145 and 125 Ma (Mizusaki et al., 2002). An imprecise $^{40}Ar/^{39}Ar$ age of 126.9 ± 4 Ma for an evolved high-Ti tholeiite confirms that the dike emplacement must have occurred in the Lower Cretaceous Epoch (Ngonge et al., 2016a). Even considering misinterpretations related to the K–Ar method, which is unable to recognize Ar loss or excess components, the ages available for the E-trending dikes show good correlations with $^{40}Ar/^{39}Ar$ ages between 147 to 123 Ma obtained for the tholeiitic basalt flows of the Northern Benue Trough in West Africa (Maluski et al., 1995). These basaltic magmas have been interpreted as originated from interaction of the St Helena plume with the sub-lithospheric mantle during the opening of the Equatorial Atlantic (Coulon et al., 1996) and could represent African counterparts of the EQUAMP. The timing of emplacement of the Parnaíba sills was investigated by the K-Ar method, yielding ages between 133 to 126 Ma (Mizusaki et al., 2002). A few $^{40}Ar/^{39}Ar$ ages published by Baksi and Archibald (1997) indicate, however, a more restricted interval of 129 to 124 Ma. Additionally, other K-Ar and $^{40}Ar/^{39}Ar$ ages have been presented in national scientific meetings as a result of collaborations between Brazilian universities and Petrobras

(Morais Neto et al., 2016), all of them pointing out to the same time interval at the Lower Cretaceous Epoch. From the available data, it is apparent that the main igneous components present in NE Brazil (i.e., the RCM dike swarm and the eastern Parnaíba Basin sills) are synchronous. Nonetheless, it remains clear that thorough and comprehensive dating constrain still needs to take place, especially considering that these rocks have not yet been dated considering a single event approach, that is, as a single LIP in NE Brazil as suggested by Hollanda et al. (2019).

6. CONCLUSIONS

The geochemical characterization of the EQUAMP igneous rocks in NE Brazil contributed from the recent work of Macêdo Filho and Hollanda (2022) and the post-graduate theses of Macêdo Filho (2021) and Dantas (2021; unpublished). These works successfully discriminated the EQUAMP products into the RCM, Canindé and Riacho do Cordeiro dike swarms, and the sills in the eastern Parnaíba Basin, altogether assembling both high- and low-Ti mafic magmas as in other Gondwanan LIPs in South America. CAMP, in turn, is a large-scale and well-known LIP (e.g., Marzoli et al., 2018), but until now only restrictedly recognized in NE Brazil, at the western Parnaíba Basin. Here, we proposed an age and timing for the emplacement of EQUAMP magmas, to support the hypothesis of a LIP as originally formulated by Hollanda et al. (2019). In addition, the precise time definition (and overall recognition) of the CAMP event in the NE region of Brazil and how it correlated to other CAMP occurrences in Brazilian sedimentary basins was understood. The success of the geochronological approach herein presented was much due the previous systematic petrologic studies performed on these rocks, as well as methodological developments achieved during the execution of the project. Coordinates for all samples dated in this work can be found in Macêdo Filho (2021) or Macêdo Filho and Hollanda (2022)

It is demonstrated that the geochemical magmas characterized as EQUAMP components were all emplaced at the Early Cretaceous and collectively pertain to a single igneous event nowadays cropping out over ca. 0.8 Mkm^2 in area, in a period of time not more than 800 kyr, which fulfill the prerequisites of area and duration described in the modern LIP concept (Ernst, 2014). Even more, CAMP rocks share the same geographic areas (and structural trends) of the EQUAMP rocks, illustrating a complex history of recurrence of magmatism along the equatorial segment of the Atlantic rift.

The first stage of the analytical program consisted of performing a fast and low-cost survey to recognize age patterns in the dike swarms. We chose the unspiked K-Ar technique of Cassignol and Gillot (1982; also, Gillot and Cornette, 1986) because of the restriction of the ^{38}Ar spike in the ARGUS VI noble gas mass spectrometer hitherto dedicated to $^{40}Ar/^{39}Ar$ measurements. By adapting the instrumental settings, we were able to date over one hundred samples (in replicates) in only a couple of months. The variability observed in the individual (replicate) ages were strongly influenced by the grain size texture of the diabases. Even though, the statistical processing provided two 'estimate' isochron ages that supported the previous $^{40}Ar/^{39}Ar$ and K/Ar data available in literature for Mesozoic magmatic events in NE Brazil (Bellieni et al., 1992; Baksi and Archibald, 1997; Mizusaki et al., 2002; Merle et al., 2011; Ngonge et al., 2016a), and they are comparable with those supposedly related to EQUAMP (ca. 133 Ma) and CAMP (ca. 201 Ma) dates. In addition, ten samples from the eastern Parnaíba sills and RCM and Riacho do Cordeiro dikes were selected to be dated by the $^{40}Ar/^{39}Ar$ method. The $^{40}Ar/^{39}Ar$ dates more precisely reproduce the same intervals found by the K/Ar technique and previous published EW-RCM and Sardinha ages. From these results, we selected representative samples of each geographic (e.g., different dike swarms) and geochemical group to proceed with CA ID-TIMS U-Pb dating. The analytical settings for running unspiked K-Ar analysis are now in routine at the noble gas lab of the CPGeo-USP, but we recommend its use only as a primary tool for investigating large set of (volcanic) samples, as usually found in LIP studies.

From the insights provided by the K-Ar work, the second approach was realized during an overseas stage in the Boise State University on the laboratory of Dr. Mark Schmitz. Three rock samples were selected as candidates to represent the CAMP event, collected from dikes that constitute the Canindé dike swarm and NE-trending branch of the RCM swarm (i.e., the NE-RCM). A more comprehensive assessment of the EQUAMP constituents was needed to define its timing and duration, and over 15 samples were selected for zircon separation collected from the two (E- and NE-) branches of the RCM swarm, as well as the Riacho do Cordeiro and Canindé dike swarms and the eastern Parnaíba Basin sills.

The CA-ID TIMS zircon U-Pb dating was made possible by the development of a novel method for zircon concentration from mafic (sub)volcanic rocks, which was built from bulk rock chemical dissolution using a combination of hydrochloric, nitric, and hydrofluoric acids. This technique proved successful in concentrating zircon crystals using ca. 1 kg of bulk rock sample. Firstly, zircon crystals were selected for a screening LA-ICPMS analysis to evaluate the chemistry of the grains, as well to provide a preliminary chronological result that was key to selecting the best samples for high-precision (CA ID-TIMS) dating. Nine samples in total (one from CAMP – DCE68, and eight from EQUAMP) were dated by the CA-ID TIMS U-Pb technique providing high-precision ages at the order of <0.05% 2σ errors. The age of the CAMP dike (201.464 ± 0.017 Ma) agreed well with other Brazilian CAMP high-precision ages (Davies et al., 2017; Heimdal et al., 2018), while the set of ages obtained from the EQUAMP dikes and sills spread in a time interval of less than 800 kyr – 133.805 to 133.071 Ma.

The ca. 201 and ca. 133 Ma dates are strictly comparable with global environmental changes that characterize the Jurassic/Triassic boundary and the Late Valanginian (Early Cretaceous) period. Following our proposal of recalculating the published dated in view of a strict Th-correction, we showed that the Brazilian CAMP magmas have had great influence on promoting the end-Triassic extinction, and that the current accepted 'global' timing for the extinction might be put to scrutiny.

Preserving particular geochemical characteristics, the EQUAMP and PEMP magmas have remarkable similarities (Ngonge et al., 2016a; Dantas, 2021; Macêdo Filho, 2021; Macêdo Filho and Hollanda, 2022) suggesting that mechanisms to induce mantle melting and sources may have been common. Age consistence between them is now proved, pointing out that rifting of the West Gondwana continent, from the south to the equatorial segments, did not exceed 5 myr. If we consider EQUAMP and PEMP as a 'single' event of continental-scale generation of mantle-derived magmas, they certainly played a key role as the main cause(s) of the 'Weissert' carbon isotope excursion (CIE). The present geochronological knowledge supports its synchronicity with the onset and peak of such global environmental changes. Issues concerning to how and what mechanisms were responsible for triggering this CIE remain open for debate.

To conclude, this thesis helped seal the gaps of knowledge concerning the precise geochronology of mafic rocks forming the multiple dike swarms and sill complex in NE Brazil, thus providing a timing constrain for two of the three Mesozoic LIPs recognized in South America. The project also made an important contribution in proposing, developing, and optimizing analytical and laboratorial methods that might have significant scientific impact when reproduced to other LIPs and/or major tectonomagmatic events.

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