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APLICAÇÕES DO MÉTODO DE RIETVELD NA PETROGRAFIA DE ROCHAS KIMBERLÍTICAS

MATHEUS ANDRADE NASCIMENTO

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APLICAÇÕES DO MÉTODO DE RIETVELD NA PETROGRAFIA DE ROCHAS KIMBERLÍTICAS

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Aos meus pais e Mulher, com muito carinho.

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RESUMO

A análise qualitativa através da difração de raios X se iniciou a partir de estudos de William H.Bragg, que observou que este fenômeno, causado quando um feixe de raios X monocromáticos incide sobre um material cristalino, ocorre o fenômeno da difração que pode ser utilizado para a identificação mineralógica. Assim, para analisar as fases cristalinas que estão presentes em um material pulverizado analisado, é feito um difratograma, que é um gráfico de intensidade observada em relação ao ângulo de espalhamento. Com o difratograma são calculados os picos de máxima intensidade. O método de Rietveld foi criado em 1969 por Hugo M. Rietveld, foi inicialmente idealizado por Hugo. M Rietveld, foi inicialmente idealizado para o refinamento da interpretação de dados dos difratogramas gerados a partir da difração de nêutrons e posteriormente aplicado a difração de raios X. O método permite calcular o difratograma ponto a ponto de uma determinada amostra através de um algoritmo com um padrão difratométrico adequado à fase que se pretende estudar, aplicando para isto o método matemático dos mínimos quadrados. Assim, comparando os picos experimentais obtidos na amostra desconhecida e o padrão de pontos calculados é possível se identificar e obter os valores das dimensões da célula unitária. Alguns destes valores podem ser encontrados em artigos que reportam estruturas similares. O estudo petrográfico clássico em kimberlitos é dificultado pela presença da mineralogia exótica, xenólitos, xenocristais, e minerais de origem mantélica pouco conhecidos. Além disto a geoquímica destas rochas, com riqueza de voláteis, favorece os processos tardios de serpentinização, carbonatização e oxidação, transformando as rochas expostas em superfície e impossibilitando muitas vezes a correta identificação das fases minerais presentes na amostra. Por estes motivos, o refinamento das análises por difratometria de raios X através do método de Rietveld é uma poderosa ferramenta na interpretação petrográfica de rochas de mineralogia exótica e alterada. Tais informações são essenciais para uma melhor compreensão da natureza e gênese deste importante prospecto mineral. Este estudo tem o objetivo de aplicar o método de quantificação de Rietveld através de análises por difratometria de raios X no método do pó-total para ampliar o entendimento sobre a petrografia do Lamproíto Aroeira. Para isso foi utilizado o software que DIFRAC.TOPPAS no laboratório de tecnologia mineral de raios X (LAPAG- UFBA). A metodologia de trabalho desenvolvida neste estudo focou na seleção de duas amostras representativas do Lamproíto Aroeira. Estas duas amostras foram laminadas em três direções ortogonais, obtendo-se assim 6 lâminas polido-delgadas e 6 tabletes destas mesmas frações, os quais foram moídos e geraram o pó analisado por DRX. Assim, o estudo da petrografia juntamente com o a aplicação do método de Rietveld no seu respectivo pó trouxe uma confiabilidade maior as descrições petrograficas e a identificação de fases exóticas e serpentinizadas.

Palavras-chave: Petrografia: Método de Rietveld; difratometria de raios X; Kimberlito

ABSTRACT

The use of X-rays began in 1895, by Wilhelm Conrad Roentgen. Initially it was used for medical studies, until in 1912 Max Von Laue applied the technique on crystals, discovering X-ray diffraction. Soon a powerful tool for scientific studies was obtained, which guided the discoveries of all structures known to date. Qualitative analysis by X-ray diffraction started from Bragg's studies, where it was determined that when a monochromatic X-ray beam falls on a crystalline material, the phenomenon of diffraction occurs. Thus, to analyze the crystalline phases that are present in an analyzed powder, a diffractogram is made, which is a graph of observed intensity. With the diffractogram the peaks of maximum intensity are calculated. The Rietveld method was created in 1969 by Hugo. M Rietveld, was initially designed to refine the interpretation of diffractogram data generated from neutron diffraction. The method allows the calculation of the standard diffractogram of a given sample through an algorithm with a diffraction pattern suitable for the phase to be studied, applying for this the method of least squares. Thus, comparing the experimental peaks obtained in the unknown sample and the pattern of calculated points, it is possible to identify the unit cell of the crystallographic structure. Some of these values can be found in articles that report similar structures. The classical petrographic study of kimberlites is hampered by the presence of exotic mineralogy, xenoliths, xenocrystals, and little-known minerals of mantle origin. Furthermore, the geochemistry of these volatile rocks favors the late processes of serpentinization, carbonatization and oxidation, transforming the exposed rocks on the surface and often making it impossible to correctly identify the mineral phases present in the sample. For these reasons, the refinement of X-ray diffraction analysis using the Rietveld method is a powerful tool in the petrographic interpretation of exotic and altered mineralogy rocks. Such information is essential for a better understanding of the nature and genesis of this important mineral prospect. This study aims to apply the Rietveld quantification method through X-ray diffraction analysis in the total-powder method to broaden the understanding of the petrography of Lamproíto Aroeira. For this, the software that DIFRAC.TOPPAS will be used in the laboratory of X-ray mineral technology (LAPAG). The work methodology developed in this study focused on the selection of two representative samples of Lamproíto Aroeira, these two samples were laminated in three orthogonal directions, thus obtaining 6 thin-polished blades and 6 tablets of these same fractions, which were ground and generated the powder analyzed by DRX. Thus, the study of petrography together with the application of the Rietveld method in its respective powder gave a greater reliability to petrographic descriptions and thus help the identification of exotic and serpentinized phases.

Keywords: Petrography; X-Ray Diffraction; Rietveld Refinement; Kimberlite

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CAPÍTULO 1
INTRODUÇÃO GERAL

A utilização dos raios X começou em 1895, por Wilhelm Conrad Roentgen (1845-1923). Inicialmente os raios X eram utilizados unicamente para estudos médicos até que, em 1912, Max Von Laue (1879-1960) aplicou a técnica em cristais descobrindo a difração de raios X. Rapidamente a técnica de raios X se transformou em uma poderosa ferramenta para estudos científicos, guiando as descobertas da maioria das estruturas cristalinas conhecidas até hoje (Schmal, 2011). Os pontos medidos da Difratometria de Raios X (DRX) resultam em um diagrama, o difratograma, que é um conjunto de picos (ou planos) de intensidade e distância interplanares características dos cristais. A interpretação dos picos é facilitada por uma base de dados que contém, para cada estrutura presente na amostra, as energias e a intensidade das raias de difração que as produziram. Isto permite a sua correta identificação já que é possível localizar, para cada pico de difração, a lista de planos cristalinos que possuem picos de difração nestes ângulos (Rietveld, 1967,1969).

O método de Rietveld é um método matemático que permite calcular um "difratograma padrão" para determinada amostra através da aplicação de um algoritmo com padrão difratométrico adequado à fase que se pretende estudar e ajustando os resultados através do método dos mínimos quadrados (Rietveld, 1967, 1969). Além de refinamento estrutural, o método de Rietveld também permite a identificação das fases presentes na amostra, através da indexação dos picos de Bragg. Adicionalmente, o comprimento e a largura dos picos de incidência permitem a quantificação das fases presentes no material estudado (Kinast, 2000).

Assim, comparando os picos experimentais obtidos na amostra desconhecida com o padrão de pontos calculado para seus espectros é possível se identificar a célula unitária da estrutura cristalográfica. Atualmente, a maioria destes valores podem ser encontrados em bancos de dados automatizados que reportam estruturas parecidas com a da amostra em estudo, tal como o *Powder Diffraction File* (PDF, CoD, ICSD, CSD). Estes bancos de dados são continuamente atualizados e podem ser adquiridos pelos laboratórios de Difratometria de Raios X para otimizar os seus resultados. Desde então, com o avanço tecnológico, os bancos de dados elaborados por Rietveld e outros pesquisadores foram agrupados e transformados em bancos de dados digitais, a partir dos quais foram criados softwares de processamento.

O objetivo principal desta dissertação de mestrado é – a partir da criação de um protocolo analítico envolvendo descrições petrográficas e análises litoquímicas, em associação com a técnica de DRX e o método de Rietveld – caracterizar fases minerais características de rochas kimberlíticas, utilizando o dique lamproítico de Aroeira, como um estudo de caso para a aplicabilidade destas técnicas em estudos geológicos. Adicionalmente, espera-se com isto avançar na compreensão do magmatismo kimberlítico do Nordeste da Bahia.
Os objetivos específicos deste trabalho incluem:

(i) Formar pessoal qualificado para o estudo de magmas kimberlíticos e seus minerais indicadores.

(ii) Dar continuidade à formação do discente como pesquisador através do treinamento em técnicas analíticas específicas, tais como a Difração de Raio-X (DRX), método de refinamento de Rietveld, Fluorescência de Raio-X (FRX) e a Microscopia Eletrônica de Varredura (MEV).

(iii) Desenvolver e adaptar rotinas e metodologias laboratoriais na UFBA para a classificação de kimberlitos através do estudo de minerais indicadores.

(iv) Criar protocolos analíticos e dar suporte na implementação de técnicas que permitam ampliar a capacidade analítica e interpretativa do Laboratório de Tecnologia Mineral – Raios X – do Complexo Laboratorial de Pesquisas em Análises Geoquímicas (LAPAG) do IGEO/UFBA.

Kimberlitos (s.l.) são atualmente objeto de grande atenção econômica, pois constituem a principal fonte primária de diamantes (Mitchell, 1986). Cerca de 6.400 pipes de kimberlitos já foram descobertos no mundo, sendo que apenas 400 deles foram classificados como diamantíferos (Robb, 2005), ainda que nem todos sejam economicamente viáveis.

Apesar de ser uma rocha muito estudada, a definição e classificação de rochas kimberlíticas é muito complexa, devido à sua diversificada e exótica composição mineral. Esta característica é agravada pela riqueza emvoláteis e xenólitos do manto e das rochas encaixantes, carreados e assimilados pela rocha durante a sua ascensãodas zonas mantélicas profundas, onde este magma se origina, até à superfície terrestre. Isto resulta em uma grande susceptibilidade a processos de alteração tais como a serpentinização, carbonatização e oxidação que dificultam o reconhecimento dos minerais primários e alteram os resultados de suas análises químicas.

Historicamente, a classificação das rochas kimberlíticas (s.l.) tem como base sua mineralogia, petrologia e geoquímica, levando à criação de dois grandes grupos: o grupo 1 ou olivina-kimberlitos que incluem rochas similares às ocorrências inicialmente reportadas em Kimberley (Mitchell, 1986); e o grupo 2, onde as rochas foramdenominadas de orangeítos (Mitchell, 1995), ou flogopita-kimberlitos, e que atualmente também incluem os lamproítos ou kimberlitos lamprófiricos (Le Maitre, 2002). Scott Smith et al. (2018) sintetizaram todas estas definições e classificações históricas, estabelecendo critérios progressivos para a interpretação e nomenclatura destas rochas. Estes autores levaram em consideração a descrição petrográfica da rocha, classificação petrogenética, aspectos genéticos e texturais, o contexto espacial de colocação do corpo e a interpretação do processo genético a ela relacionado. Além destes parâmetros, a análise de rocha total, desenvolvida por Williams Jones et al., 2004, faz parte da metodologia mais recente para a classificação de kimberlitos (Scott Smith et al., 2018).

A rocha selecionada para o estudo de caso nesta dissertação de mestrado é o lamproíto Aroeira (Santos et al., 2019), um corpo diamantífero localizado na Província Kimberlítica Nordestina, no nordeste do estado da Bahia, a cerca de 380 km de Salvador, entre os municípios de Itiúba e Queimadas. O acesso é feito pela BR 324, durando cerca de 6 horas de viagem, partido da cidade de Salvador.

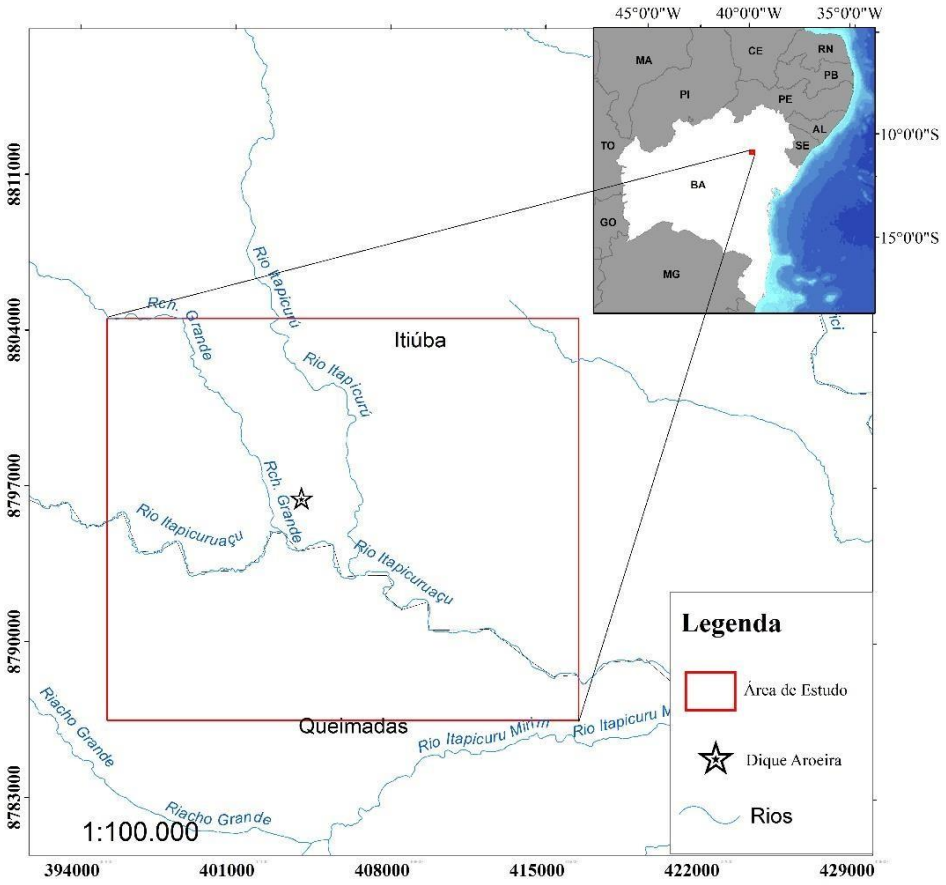

Figura 1. Mapa de localização da área de estudo (em vermelho).

A região é uma zona de clima semiárido, com menor pluviosidade no inverno e média pluviométrica de 656 mm ao ano. A temperatura média é de 25,3ºC, banhada pela bacia hidrográfica do Rio Itapicuru, e cortada pelo rio Itapicuru-Açu. O relevo é plano a suavemente ondulado.

Geologicamente, encontra-se associada à macro unidade geotectônica do Cráton do São Francisco (CSF), a mais exposta e bem estudada unidade tectônica do embasamento da Plataforma Sul-americana (Barbosa, 2003). O corpo Aroeira está inserido na zona de limite entre o Orógeno Itabuna-Salvador-Curaçá (OISC) e o Núcleo Serrinha. A principal unidade litológica do OISC é o Complexo Caraíba, composto por ortognaisses migmatíticosde natureza TTG. Além do Complexo Caraíba destaca-se o Sienito de Itiúba (2,1 Ga). Coberturas quaternárias areno-argilosas, de coloração bege, recobrem boa parte das rochas do Complexo Caraíba. O lamproíto Aroeira é um dos diques descritos na Província Kimberlítica Nordestina (PKN). A PKN integra vários pequenos corpos, diques e pipes, descobertos pela De Beers durante a década de 90 (Pereira, 2007) e aos quais foram adicionadas novas descobertas nos anos mais recentes. A província estende-se pelos municípios de Nordestina, Queimadas, Itiúba, Cansanção, Monte Santo e Uauá. Os corpos de maior destaque e melhor estudados formam o Campo Kimberlitico de Braúna (Pereira, 2007, Donatti-Filho, 2013, Santos et al., 2021), onde se localiza a única mina dediamantes em kimberlitos da América Latina (Nanini et al., 2017). Além do lamproíto Aroeira e dos corpos do CKB, há outras intrusões menores tais como os diques de Asa Branca 1 e 2, Icó 1 e 2, Umbú.

O Lamproíto Aroeira está localizado no município de Itiúba (Figura 1), intrudindo rochas do Complexo Caraíba. Em campo, reflete um relevo arrasado, de solo areno-argiloso, quase sem vegetação. O dique possui orientação geral NNW-SSE, sendo descrito como rochas ricas em flogopita e serpentina na matriz e macrocristaisde olivina, granada e espinélio, sendo reportados a presença de cristais de diamante (Nanini et al., 2017; Santos etal., 2019).

O detalhamento e a correta descrição e classificação deste corpo é de fundamental importância para maior compreensão dos limites e do potencial diamantífero da PKN. Sua importância reside no fato deste corpo apresentar características transicionais entre kimberlitos e orangeítos e na sua associação com rochas de naturezasienítica e ultramáficas de idade 0,9Ga (Espanta Gado, Huttner et al., 2019) a cerca de 2 km de distância.

Desta forma, as motivações para o desenvolvimento desta pesquisa, várias questões ainda não respondidas sobre as rochas da PKN, e mais especificamente, para o corpo Aroeira. Em especial, buscamos respostas para as questões relacionadas às variações composicionais e mineralógicas que este corpo apresenta. Buscamos com isto contribuir para o melhor entendimento dos corpos que ocorrem na PKN e sua contextualização na evolução geotectônica deste importante segmento do CSF.

Os resultados preliminares das pesquisas realizadas no contexto desta dissertação foram apresentados na forma de resumo em evento da área e encontram-se listados abaixo. Cópia destas publicações compõem o **AnexoA:**

Nascimento MA, Rios DC (2019) Aplicações do Método de Rietveld na petrografia de rochas Kimberlíticas saprolitizadas: Expectativas In: Anais do 28º Simpósio de Geologia do Nordeste, Aracaju, 415.

Oliveira Junior MR, Rios DC, Nascimento MA (2019) Técnicas de identificação e concentração de minerais indicadores de Kimberlitos e lamproíto -Estudo de caso do lamproíto transicional Aroeira. In: Congresso pesquisa,ensino e extensão UFBA, Salvador, 286.

Nascimento MA, Rios DC (2018) Aplicações do Metodo de Rietveld na Petrografia do corpo kimberlitico Aroeira.In: VI Oficina de Avaliação e Acompanhamento Discente do Programa de Pós-graduação em Geologia, 54.

De acordo com as normas do Curso de Pós-Graduação em Geologia da Universidade Federal da Bahia optou-se por apresentar esta dissertação de mestrado em formato artigo. Os trabalhos culminaram com a elaboração e apresentação deste volume.

O volume aqui apresentado contém 3 capítulos ilustrados com figuras e tabelas, e contendo a listagem das referências bibliográficas consultadas; o apêndice e os anexos.

O **capítulo 1** traz uma introdução geral que abrange uma breve descrição sobre o método de Rietveld para o refinamento de dados da Difratometria de RaiosX. Inclui uma descrição do objetivo geral e específicos, motivaçõese justificativas. O corpo kimberlítico Aroeira, nosso estudo de caso, é caracterizado e localizado. Este capítulo apresenta ainda a estruturação do volume.

O **capítulo 2** traz o artigo científico com os resultados da pesquisa, submetido ao *Contributions to Mineralogy and Petrology Journal (https://www.springer.com/journal/410)* uma revista internacional da Editora Springer, classificado com Qualis CAPES A1.

O **capítulo 3** contêm as considerações finais, principais conclusões, e recomendações para estudos futuros.

O **Apêndice A** discorre sobre as contribuições de cada um dos co-autores. O **Apêndice B** contempla os dadosanalíticos produzidos na forma de tabelas e gráficos. O **Apêndice C** apresenta as cópias das publicações dos resultados parciais.

O **anexo A** traz as regras de formatação do periódico selecionado para a publicação dos resultados. O **anexo B** apresenta cópia do comprovante de submissão do artigo.

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CAPÍTULO 2

ARTIGO: INSIGHTS INTO THE PETROGENESIS OF KIMBERLITES: APPLICATIONS OF THE SCANNING ELECTRON MICROSCOPY, X-RAY FLUORESCENCE AND DIFFRACTION / RIETVELD REFINEMENT BASED ON THE AROEIRA DIAMONDIFEROUS LAMPROITE STUDY OF CASE

CONTRIBUTIONS TO MINERALOGY AND PETROLOGY

INSIGHTS INTO THE PETROGENESIS OF KIMBERLITES: APPLICATIONS OF THE SCANNING ELECTRON MICROSCOPY, X-RAY FLUORESCENCE AND DIFFRACTION / RIETVELD REFINEMENT BASED ON THE AROEIRA DIAMONDIFEROUS LAMPROITE STUDY OF CASE

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Abstract

The classical petrographic study of kimberlites is hampered by the presence of exotic mineralogy, xenoliths and xenocrystals. A robust petrographic knowledge including the correct identification of essential and accessory/exotic minerals of kimberlitic rocks is an important step to better understand the geotectonic environment and the metallogenetic potential for the kimberlitic rocks and will reduce the costs associated with the research during the green-field development of new deposits. This study applied the Rietveld quantification method through X-ray diffraction analysis in the total-powder method to broaden the understanding of the petrography of lamproite Aroeira. The work methodology developed in this study focused on the selection of two representative samples of Aroeira Lamproite, these two samples were laminated in three orthogonal directions, thus obtaining 6 thin-polished section and 6 tablets of these same fractions, which were crushed and generated the powder analyzed by DRX. Thus, the study of petrography together with the application of the Rietveld method in its respective powder gave a greater reliability to petrographic descriptions and thus help the identification of exotic and serpentine phases. The SEM analysis made possible to identify the olivine as forsterite, three pyroxene phases: augite, enstatite, hedenbergite. Additionally accessory minerals as a silica phase, chromite, barite and titanite in the pyroxene-rich areas of the groundmass. The XRD analysis identify nine minerals more abundant than 1%, these were: tetraferriphlogopite, vermiculite, enstatite, chabazite, forsterite, hedenbergite, whitlockite, cristobalite and ilmenite. The results quantitative mineralogical analysis by the Rietveld method demonstrated the unique information that this method can provide compared to petrographic microscope or another conventional method of quantitative analysis.

Keywords: PETROGRAPHY; X-RAY DIFFRACTION; RIETVELD REFINEMENT; KIMBERLITE

1. INTRODUCTION

Kimberlites are exotic ultrapotassic mafic volatile-rich magmas, which are responsible for carrying diamonds from mantle deeps to Earth's surface. Currently, kimberlitic rocks are responsible for more than 90% of the World diamond production (Robb 2005). These primary diamond sources have been the object of many studies since their discovery in Kimberley (Roberts 1976), although most bodies are barren. Their economic potential as a source rock for diamonds can only be assessed through a detailed study of the so-called kimberlite indicator minerals (KIM), in special the correct identification and classification of their olivine, garnet, chromium-pyroxenes, chromite, and ilmenite essential minerals (Mitchell 1986). However, kimberlitic rocks, due to their high volatile contents, and especially when exposed in tropical climates, are extremely altered in surface, making it difficult to identify and correctly describe the mineral phases present.

The detailed mineral identification and quantification, essential for understanding of the nature and genesis of any mineral prospect, involves classical petrological studies, in which the use of a microscope is fundamental. However, particles below silt size are imperceptible to the naked eye in the spectrum of visible light and optical microscopes also have limitations. Nowadays, most petrological studies combine different techniques – such as X-Ray Diffractometry (XRD), X-Ray Fluorescence (XRF), and Scanning Electron Microscopy (SEM) – to solve many of these problems.

The technological improvements in association of XRD resulted in potent mineral databases. Computer software allowed the development of data processing tools, as the Rietveld refinement technique (Young 1993; Schmal 2011), a powerful tool to the petrographic interpretation of rock modal compositions, even for samples with rare, exotic and/or modified mineralogy. The Rietveld refinement creates a calculate diffractogram of a given sample using a mathematic algorithm, resulting in a diffractometric pattern, suitable for the phase to be studied, applying the least squares method. Thus, by comparing the experimental peaks obtained in the unknown sample and the calculated points pattern, it is possible to identify the unit cell of the crystallographic structure.

The main aim of this paper is to test the applicability of a combined set of classical petrological tools (microscope, SEM, XRF and XRD) in association with the Rietveld refinement technique (Rietveld 1979) aiming for a more precise identification and quantification of KIM in fresh and weathered rocks to create an analytical protocol that simplifies the identification and preliminary economic evaluation of surficial kimberlitic samples exposed in tropical weathering environments. This study applied Rietveld quantification methods through total-powder XRD to surface samples (<2 m deep) collected in the outcrops of the diamond-bearing Aroeira lamproite in San Francisco Craton, aiming to a broaden understanding of its petrography and to create a new, robust, and low-cost method, applicable to the prospection and exploration of such rocks.

2. GEOLOGICAL FRAMEWORK

The Aroeira Lamproite is part of the Nordestina Kimberlitic Province (NKP), that intrudes the Archean Serrinha Nucleus (SerN; Rios et al. 2009; Nunes et al. 2021), in the semi-arid region of Northeast Bahia, São Francisco Craton (SFC), Brazil. The SFC was first described by Almeida (1977), been bordered by polymetamorphic folding belts (Brasília Belt, Rio Preto Belt, Sergipana Belt, Brasília Belt, and Aracuaí Belt). In this geological context three Archean cratonic nuclei, including SerN, collided during the Paleoproterozoic resulting in the formation of mobile belts – as the Itabuna-Salvador-Curaçá Orogen (ISCO) – that consolidated during the Paleoproterozoic (Mascarenhas et al. 1978; Martins de Souza et al. 2020; **Figure 1**).

Figure 1. A simplified geological map for the area of study. (A) Sao Francisco Craton, Archean nuclei and mobile belts. (B) Serrinha Nucleus and the Nordestina Kimberlitic Province.

The ISCO was first described and mapped by Delgado & Souza (1975). The main lithological unit is the Caraíba Complex, composed of tonalite, granodiorite, trondhjemite (TTGs), and quartz-monzonitic migmatitic orthogneisses. Paleoproterozoic post-tectonic igneous intrusions are the first evidence of ultrapotassic lamprophyric-lamproitic magmas (~2.11 Ga; Plá Cid et al. 2006; Rios et al. 2007) in the area, among them the Itiúba Syenite (2.09 Ga; Conceição et al. 1996, Conceição et al. 2003), which forms a prominent 1800km^2 mountain standing out in the region's relief, and a complex Archean/Paleoproterozoic evolution that resulted in a pervasive NNW-SSE oriented structural lineation (Brito Neves et al. 1980; Rios et al. 2008, 2009; Nunes et al. 2021).

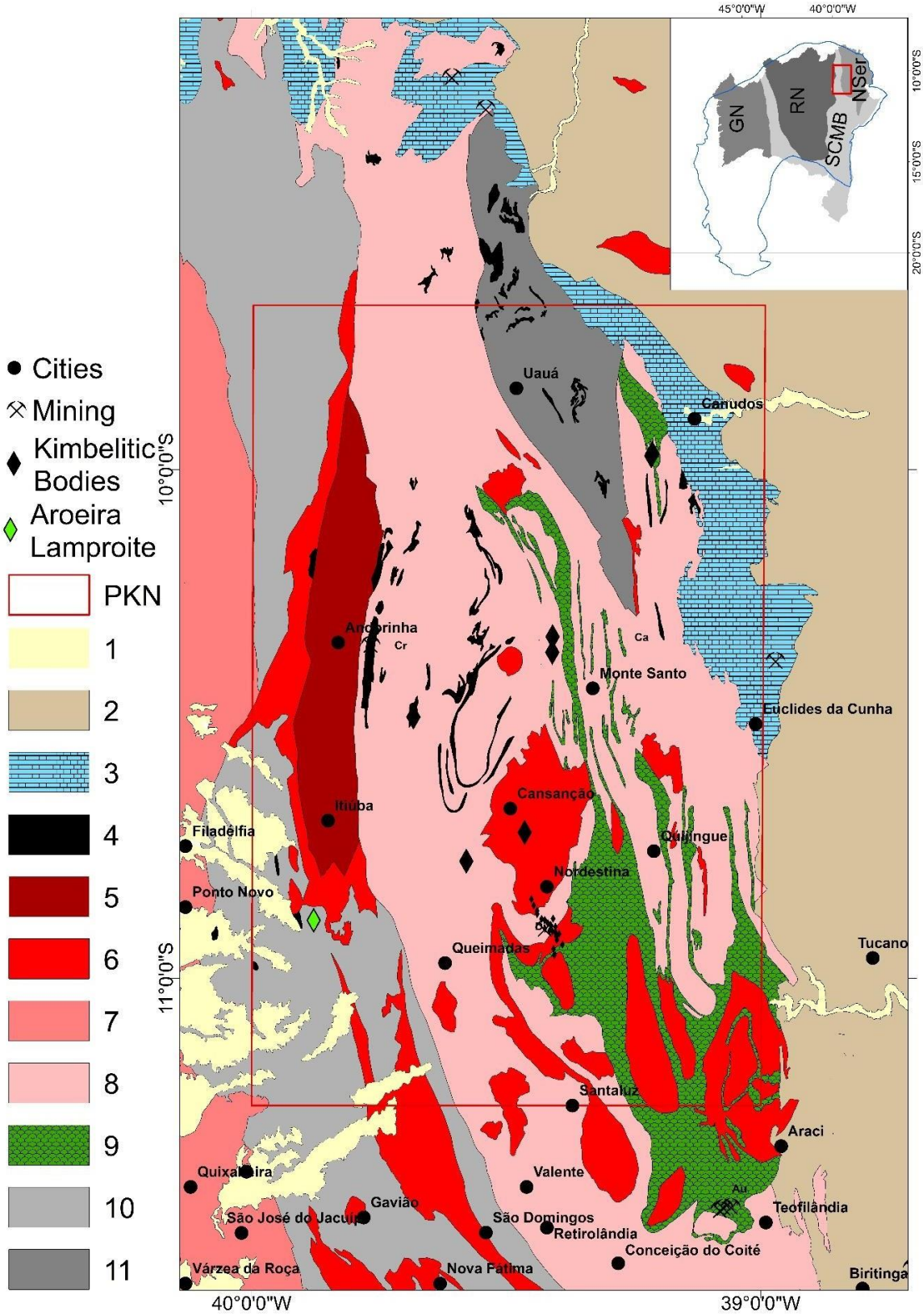
45°0'0"W
40°0'0"W
10°0'0"S
15°0'0"S
20°0'0"S
GN
RN
SCMB
NSer
Cities
Mining
Kimbelitic Bodies
Aroeira Lamproite
PKN
1
2
3
4
5
6
7
8
9
10
11
Uauá
Canudos
Monte Santo
Euclides da Cunha
Cansanção
Quijingue
Nordestina
Ponto Novo
Queimadas
Tucano
Santaluz
Araci
Valente
Teofilândia
Retirolândia
Conceição do Coité
Biritinga
Nova Fátima
Várzea da Roça
São José do Jacuipe
Gavião
Quixabeira
Filadélfia
Itiúba
Cr
Ca
Au
11°0'0"S
10°0'0"S
40°0'0"W
39°0'0"W

Figure 1

Almost 20 small kimberlite bodies, dikes and pipes were discovered by De Beers during the 90's (Pereira 2007) and currently other kimberlites were discovered in the NKP such as the dikes of Asa Branca 1 and 2, Icó 1 and 2, and Aroeira (Nannini et al. 2017, Nascimento et al. 2018, Santos et al. 2019, 2021, **Figure 1**). The most prominent intrusions, and best known by the scientific community, are still those that form the Braúna Kimberlitic Field, including the B3 pipe, that holds the only kimberlitic diamond mine in Latin America (Leroux et al. 2010; Donatti-Filho et al. 2013a, b, Santos et al. 2021). There is a controversy as some researchers in economic geology believe that the first diamond mine in kimberlites of Latin America is in the Juína Kimberlitic Field, Amazon Craton (PMP Garcia, oral communication). Conceptually, a diamond mine in kimberlites (Field et al, 2008) is restricted to a diamond deposit that has seen sustained mining for at least two years. Considering this definition, Brauna 3 is the first and only diamond mine that has been developed from a kimberlite deposit in South America (Svisero et al. 2017).

3. METHODOLOGICAL ASPECTS

The proposed analytical protocol (**Figure 2**) aims to associate detailed petrographic analyses with litochemical and mineral chemistry studies to optimize the results in kimberlite weathered samples, thus creating a viable analytical protocol for the identification and quantification of the varied and exotic mineralogy present in these peculiar igneous rocks and/or sampled by them on their journey to the surface.

Figure 2. Flowchart of the proposed analytical protocol, explaining the methodology applied in this study.

The diamond-bearing Aroeira Lamproite was selected as a case study. Relatively fresh rocks were obtained on site, in a 1m x 1m trench opened and dug approximately up to 2-3m deep pit aiming sample collection. About 40kg of fragmented rock were recovered from the 1.5m to 2m deep interval. Three visually best-preserved hand size sample fragments were randomly selected for analyses: NS3385A, NS3385B, and NS3354. Three other samples (NS3254, NS3255, and NS3256) provided additional information to sustain the discussions.

At the laboratories of the Geological Survey of Brazil (CPRM) – SUREG Salvador – these samples were cut for thin sections in three directions considering an apparent orientation:

Cut 1 is parallel to the main rock foliation, following the macroscopically visible direction of mineral alignment in the samples. NS3385-A1 and NS3385-B1 descriptions are quite similar, however there are slight differences in the degree of mineral weathering.

Cut 2, orthogonal to the visible mineral alignments, resulted in similar descriptions, been remarkable that this cut direction made possible to observe euhedral olivine mega and macrocrysts, apparently less altered than in cut 1.

Cut 3 are perpendicular to the rock foliation. This cut allowed similar descriptions for samples NS3385-A3 and NS3385-B3, however there is a greater number of "cavities" in these thin-sections. We believe these "holes" correspond to olivine and/or garnet crystals – maybe even microdiamonds – removed during polishing, as we can still see some olivine macrocrysts coming out of the sections.

Polished-thin sections were prepared for petrography and SEM. Polished slabs from the residual samples were reserved for macroscopic studies and later reduced to a <200# powder in a tungsten pan shatter box at the Laboratories of Petrography and Geochemistry (LAPAG) of the Geosciences Institute, Universidade Federal da Bahia (UFBA).

3.1. Petrography and Digital Imaging

Macroscopic description of hand samples and polished slabs were performed for NS3385A and NS3385B. These samples do not present distinction to the naked eye. Otherwise, the polished thin sections show remarkable differences related to the position of the cuts. Further descriptions of textures, structures and mineral relations under binocular microscope and petrographic polarizing microscope – Olympus BX-51 – were performed at the laboratory of petrography of the Program of Graduate Studies in Geology (PPGG/ IGEO/UFBA).

NS3354 polished thin section was digitally imaged using a Keyence Digital Microscope VHS 6000, at the Department of Chemical and Physical Sciences at the University of Toronto Mississauga. Images were automatically recorded in a multi-scan auto-mode with a medium focus moving rate, 2700K, a record size

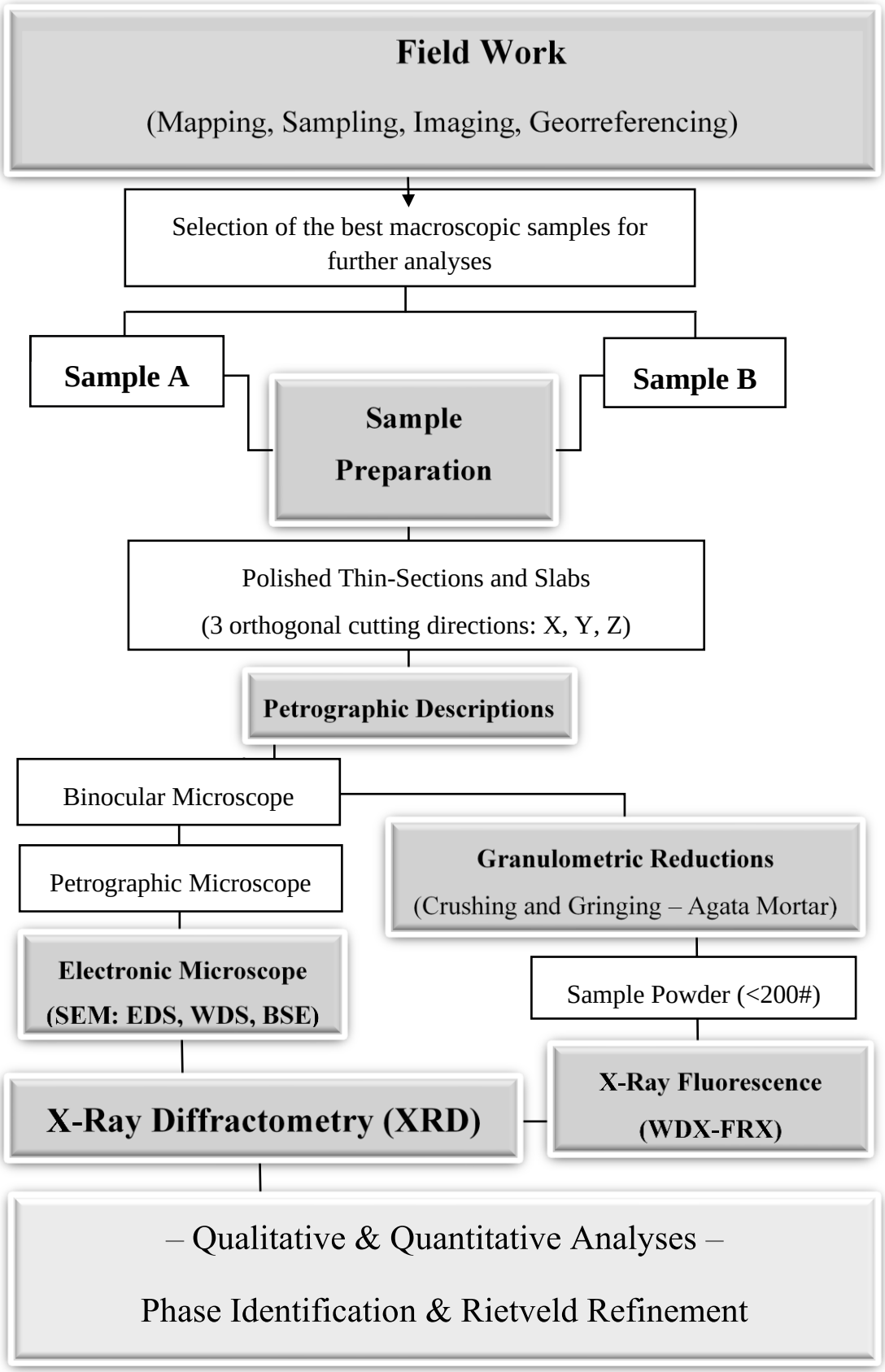
Field Work
(Mapping, Sampling, Imaging, Georreferencing)
Selection of the best macroscopic samples for further analyses
Sample A
Sample B
Sample Preparation
Polished Thin-Sections and Slabs
(3 orthogonal cutting directions: X, Y, Z)
Petrographic Descriptions
Binocular Microscope
Petrographic Microscope
Electronic Microscope
(SEM: EDS, WDS, BSE)
Granulometric Reductions
(Crushing and Gringing – Agata Mortar)
Sample Powder (<200#)
X-Ray Fluorescence
(WDX-FRX)
X-Ray Diffractometry (XRD)
– Qualitative & Quantitative Analyses –
Phase Identification & Rietveld Refinement

Figure 2

Standard of 1600x1200 and saved as high-resolution HDR tiff format. Multi sequential images with overlaps used a 20x ocular and 100x objective lens for coverage and later stitching together to form a full image of the thin section. For detailed views of additional features, splits of individual areas were performed with an 200x objective. Images were performed applying transmitted light, reflected light, transmitted, and reflected polarized lights, and cross-polarized transmitted light.

3.2. Scanning Electron Microscopy

Scanning electron microscopy (SEM) used a Tescan-Vega3 equipment, coupled with Energy Dispersive (EDS) and Wavelength Dispersive (WDS) spectrometers, a Secondary Electron (SE) beam, and a Backscattering Spectrometer Energy (BSE), located at the Multi-users Laboratory of the Program of Graduate Studies in Basin Analyses of the University of Sergipe (UFS), Brazil. SEM was used to image and perform additional characterization of the principal and accessory mineralogy. Analytical conditions involved beam voltages of 15-20 kV, current of 1.6nA and a spot size of 1100 µm.

Chemically, there are no significant differences for the mineral phases present in samples 3385A and 3385B, reason why we opt for describe the results grouped only by phase. Otherwise, the SEM analyses were essential to confirm the nature of the mineral phases. Punctual analyses of selected areas resulted in a total of 326 spots analyzed: NS3385-A1 (67), NS3385-A3 (82), NS3385-B1 (65), and NS3385-B3 (116).

3.3. X-Ray Analyses

Total powder X-ray diffraction (XRD) and fluorescence (XRF) analyses were performed at the X-Ray laboratories of the UFBA (LAPAG, LABMUT, IQ).

Fluorescence analyses were performed in a high-performance sequential Wavelength Dispersive X-Ray Fluorescence Spectrometer (WDXRF), Bruker S8 Tiger equipment. Analytical conditions include a high sense X-ray Rh tube with 4kW, high resolution LiF 220 analyzer crystal, 75µm Be window, 170mA excitation current, 300µm spot size and 100nm step for sample mapping. No standards were available and a semi-quantitative approach for analyses of major and minor oxides, as well as some trace elements was applied. Pressed pellets were prepared mixing 2.7g of sample with 0.3g of starch ($C_6H_{10}O_5$), using a dilution ratio of 9:1. The starch was added so that the tablet does not break during the pressure imposed on the powder. The homogeneous mixture was then poured into a hydraulic pressing die and a pressure of 10GPa was applied during 5 (five) minutes.

WDXRF was followed by non-destructive whole-rock powder X-Ray Diffraction (XRD), further enhancing the identification, characterization, and quantification of the mineral phases. The equipment was a Shimadzu XRD-6000. Samples were flat placed directly in the sample holder of the diffractometer without any additional preparation. The specifications of the XRD equipment and settings are shown in **Table 1**.

Table 1. XRD Equipment specifications.

4. AROEIRA LAMPROITE – PETROGENETIC REMARKS

The petrographic knowledge of kimberlitic rocks is an essential tool to their correct classification and resources estimates (Scott Smith et al. 2018). Aroeira lamproite was selected as a case study for the application of the analytical protocol here proposed because there are no drill samples available for these ancient and mineralized rocks and due to the semi-arid tropical weather in the SerN area, there is a substantial amount of soil and saprolite rocks covering the outcrops. Unfortunately, due to the volatile rich, volcanic, mafic nature of kimberlitic rocks, these features are common to most occurrences worldwide. They restrict the access to fresh, no weathered samples, limiting low-cost petrographic studies and result in controversial and confuse nomenclatures and classifications.

The Aroeira Lamproite belongs to the Nordestina Kimberlitic Province (**Figure 3**), that holds the first – and up to know the only – diamond mine on kimberlites of Latin America, Braúna 3 Diamond Mine. Artisanal exploitation of diamonds occurred in the nearby Itapicuru River since the end of the 18th century and recently the source of diamonds in this area is attributed to kimberlitic magmas of the PKN.

Figure 3. Simplified geological map of Aroeira Lamproite intrusion.

Table 1

Instrument	Shimadzu 6000
Radiation	Cu Kα
Temperature	25º C
Specimen	Vertical goniometer
Power Setting	40Kv. 30mA
Soller slit	1°
Divergence slits	1°
Receiving slits	0.30°
Monochromator	Secondary. graphite
Detector	Scintillation counter
Range of 2θ	5º - 80º 2θ
Step width	0.02º 2θ
Time per step	1sec

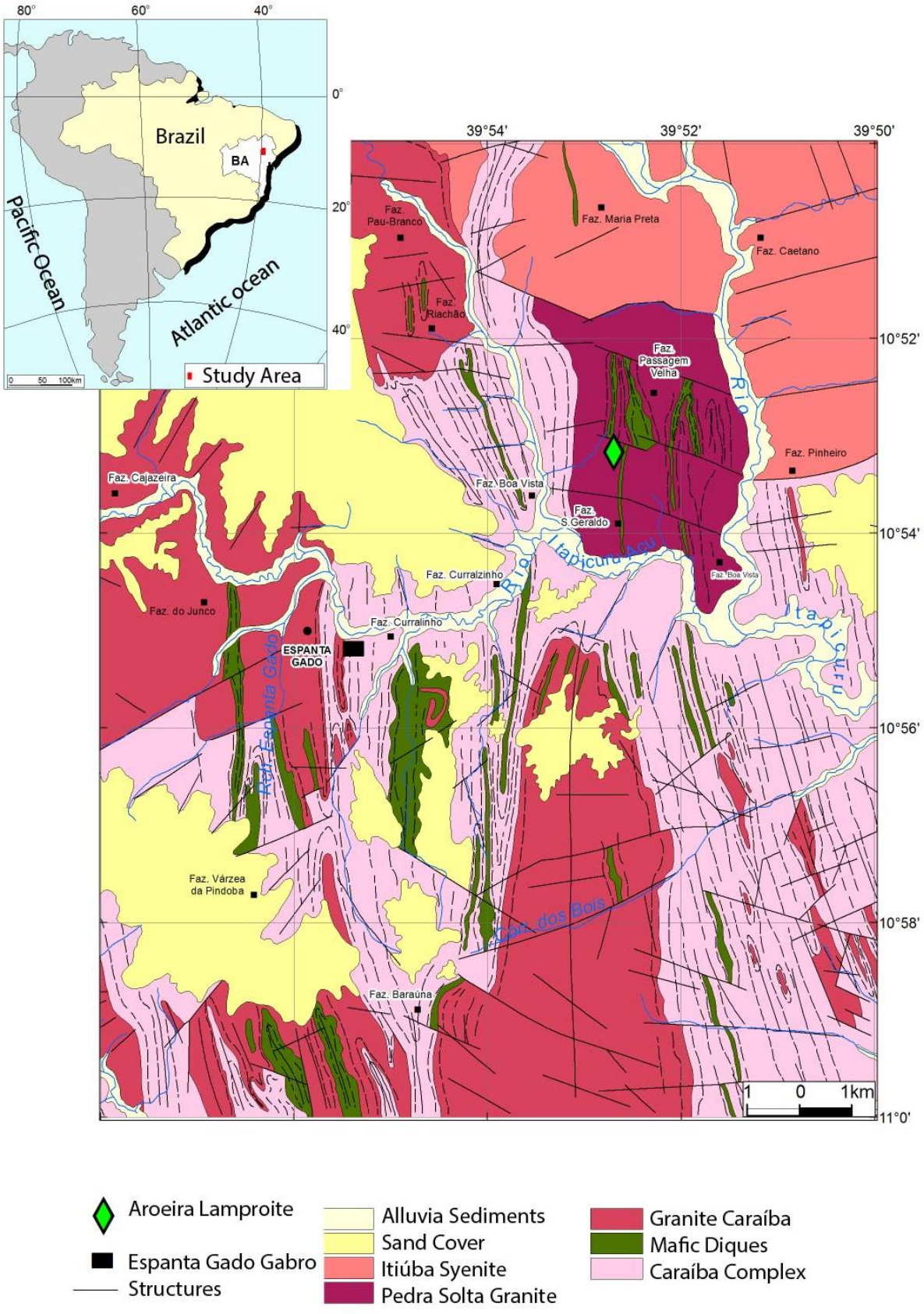
80°
60°
40°
0°
20°
40°
Brazil
BA
Pacific Ocean
Atlantic ocean
0 50 100km
Study Area
39°54'
39°52'
39°50'
10°52'
10°54'
10°56'
10°58'
11°0'
Faz. Pau-Branco
Faz. Maria Preta
Faz. Caetano
Faz. Riachão
Faz. Passagem Velha
Faz. Pinheiro
Faz. Cajazeira
Faz. Boa Vista
Faz. S.Geraldo
Faz. Curralzinho
Faz. do Junco
Faz. Curralinho
ESPANTA GADO
Rio Espanta Gado
Rio Itapicuru Acu
Rio Itapicuru
Córr. dos Bois
Faz. Várzea da Pindoba
Faz. Barauna
1 0 1km
Aroeira Lamproite
Espanta Gado Gabro
Structures
Alluvia Sediments
Sand Cover
Itiúba Syenite
Pedra Solta Granite
Granite Caraíba
Mafic Diques
Caraíba Complex

Figure 3

Aroeira is classified as an olivine-megacryst lamproite of hypabyssal-facies (Santos et al., 2019) and intrudes the Paleoproterozoic shoshonite of Pedra Solta (~2.1Ga, Otero, 2005) in the tectonic context of the SCMB, almost 7-8km far from the Neoproterozoic Espanta Gado Mafic Dykes (~0.9Ga, Huttner et al., 2019), which is part of the precursor dykes for the kimberlitic magmas of PKN.

Petrographically, Aroeira lamproite are porphyritic rocks with mega and macrocrystals of olivine and phlogopite immersed in a medium to fine grained kimberlitic matrix composed dominantly by phlogopite, pyroxene and opaque minerals (Santos et al. 2019). A Brazilian national exploration program for diamonds carried by the Brazilian Geological Survey (CPRM, Projeto Diamantes Brasil, Nannini et al., 2017) reported kimberlite indicator minerals garnet, ilmenite, spinel, clinopyroxene, perovskite and diamonds (5 microdiamonds, ~0.06 ct. /in 1000 l. of sample), as well as crustal and mantle xenoliths, for Aroeira rocks. Applying XRD method, Nascimento et al. (2018) identified Mn-rich ilmenites, advocating a high-deep mantle source or Aroeira Lamproite, comparable to the Juina Kimberlite in the Amazon Craton (Kaminski et al., 2008).

Descriptions for the size of lithic compounds in kimberlitic rocks were presented by Clement et al. (1989) and Mitchell (1986), summarized by Mitchell (1995), and revised by Scott Smith et al. (2013, 2018). In this work, we applied the following definitions (i) megacrysts, if >1cm and (ii) macrocrysts, 0.5-10mm, which sometimes are merely fragments of the megacrysts. The term must be included in the name of the rock when the amounts of these crystals are greater than 5 vol.%.

Aroeira lamproite display inequigranular porphyritic texture. Macro and megacrysts are set in a fine-grained kimberlitic groundmass, consisting of a densely packed aggregate of pyroxene prisms, opaque minerals, and small plates of phlogopite. Many of the phlogopite nucleate olivine megacrysts.

Veinlets filled with barite, carbonates silica and phosphate phases crosscut olivine megacrysts and groundmass. These veinlets are transparent in transmitted light, showing a white color and black extinction under polarized lens, and the characteristic barite intergrowth texture, when this mineral is present. Locally, these veins also display vesicular textures. A single crystal of a colorless very fractured crystal – bordered by two phlogopite macrocrystals and displaying a wavelength extinction – been identified as a silica phase.

The main minerals sampled for SEM-EDS analyses correspond to phlogopite (18 analyses), olivine (51 analyses,) and pyroxene (67 analyses). Accessory phases identified included ilmenite, phosphate, silica, chromite, magnetite, barite and titanite. A summary of the chemical analyses for the mineral phases is presented in **Table 2** and the full analyses are made available in the **Supplement 1**.

Table 2. Average chemical analyses of SEM for the minerals identified in Aroeira samples. The complete SEM analyses are available in **supplement 1**.

The WDXRF results are shown in **Table 3**. The elemental composition range obtained for Aroeira lamproite was 59.0% < SiO_2 < 82.8%; 7.8% < MgO < 16.3%; 4.8% < Fe_2O_3 < 15.0%; 2.0% < Al_2O_3 < 4.9%; 0.8% < CaO < 2.7%; 0.06% < K_2O < 0.13%; 0.04% < P_2O_5 < 0.68%; and 0.11% < Na_2O < 0.13%.

Table 3. Whole rock X-Ray Fluorescence litochemical analyses of Aroeira Lamproite.

5. RESULTS AND DISCUSSIONS

5.1. Rietveld Refinement applied to Aroeira lamproite

The Rietveld refinement calculate diffractograms and the difference between the experimental data and calculated data is minimized by a least squares refinement. The refinement progress is set based on a series of statistical calculations. The most common used are the weighted profile Rwp index and the 'goodness of fit' (X) index which is the ratio of Rwp over the statistically expected Rp (Snellings et al 2010).

The treatment to identify the minerals phases was performed using the HighScore Plus software (version 4.9, Degen *et al*, 2014). Due to a software limitation, the maximum number of mineral phases allowed while processing samples is limited to 9 (nine). This restrict us to the main phases (> 1% volume) present and other accessory or minor phases, described using the SEM, were not found in this stage.

Table 2

Mineral	Olivine	Pyroxene	Phlogopite	Ilmenite	Phosphate	Barite	Cromite
Analyses	**#44**	**#60**	**#18**	**#42**	**#32**	**#3**	**#22**
SiO2 (%)	68.08	47.44	51.64	7.42	4.66	0.00	5.52
TiO2 (%)	0.19	0.61	2.23	75.64	0.00	0.00	5.82
Al2O3(%)	0.26	1.43	13.22	0.80	0.82	0.00	14.00
Cr2O3 (%)	0.07	0.01	0.37	0.22	0.00	0.00	37.30
FeO (%)	5.62	18.00	6.57	10.03	1.08	0.00	24.48
Fe2O3 (%)	0.00	3.48	0.00	0.00	0.00	0.00	0.00
MnO (%)	0.02	0.00	0.00	4.47	0.00	0.00	0.00
MgO (%)	25.61	3.48	24.47	0.76	0.54	0.00	11.98
CaO (%)	0.12	24.40	0.56	0.16	47.75	0.00	0.90
Na2O (%)	0.01	0.06	0.06	0.01	0.01	0.00	0.00
K2O (%)	0.00	0.04	0.54	0.21	0.00	0.00	0.00
BaO (%)	0.00	0.09	0.31	0.00	0.00	66.80	0.00
SO3 (%)	0.00	0.00	0.00	0.00	0.00	33.20	0.00
P2O5(%)	0.00	0.00	0.00	0.00	35.11	0.00	0.00
C (%)	0.00	1.02	0.00	0.00	7.25	0.00	0.00
V2O5(%)	0.00	0.00	0.00	0.09	0.00	0.00	0.00
Total	99.99	100.06	99.97	99.82	97.21	100.00	100.00

Table 3

Element	NS3385-A1	NS3385-A2	NS3385-A3	NS3385-B1	NS3385-B2	NS3385-B2
SiO_2 (%)	58.98	59.17	65.00	79.44	82.75	82.23
TiO_2 (%)	2.43	2.70	1.97	1.22	0.96	1.12
Al_2O_3 (%)	3.85	4.47	3.33	2.83	2.00	1.97
Fe_2O_3 (%)	15.00	14.42	9.37	5.81	4.83	4.93
MgO (%)	14.09	13.77	16.29	9.21	7.78	8.04
MnO (%)	0.75	0.66	0.63	0.05	0.08	0.05
CaO (%)	2.65	2.39	1.83	0.75	0.81	0.82
Na_2O (%)	0.11	0.13	0.13	0.14	0.12	0.12
K_2O (%)	0.06	0.07	0.07	0.12	0.12	0.13
P_2O_5 (%)	0.68	0.56	0.46	0.04	0.05	0.06
Cr_2O_3 (%)	0.34	0.38	0.25	0.14	0.13	0.12
NiO (%)	0.26	0.26	0.16	0.10	0.08	0.08
BaO (%)	0.18	0.30	0.13	0.05	0.14	0.16
SO_3 (%)	0.03	0.08	0.03	0.03	0.06	0.09
Cl (%)	n.a.	n.a.	0.03	n.a	0.01	n.a
Total (%)	**99.68**	**99.36**	**99.65**	**99.93**	**99.92**	**99.92**
As_2O_3 (ppm)	3	28	20	18	n.a	14
CoO (ppm)	700	700	400	n.a	n.a	n.a
CuO (ppm)	98	100	81	34	34	44
Nb_2O_5 (ppm)	500	500	400	200	200	200
Pd (ppm)	67	n.a.	n.a.	69	45	n.a
PbO (ppm)	44	n.a.	n.a.	n.a	27	n.a
SrO (ppm)	200	200	100	n.a	48	48
ThO_2 (ppm)	200	200	100	96	n.a	n.a
V_2O_5 (ppm)	300	300	200	54	n.a	n.a
ZnO (ppm)	100	200	100	100	90	96
ZrO_2 (ppm)	600	600	400	200	54	95
La_2O_3 (ppm)	1000	1400	500	n.a	n.a	n.a
CeO_2 (ppm)	1300	2000	900	n.a	n.a	n.a
Nd_2O_3 (ppm)	300	n.a.	n.a.	n.a	n.a	n.a
Gd_2O_3 (ppm)	22	n.a.	n.a.	n.a	n.a	n.a
Ho_2O_3 (ppm)	0.01	0.01	n.a.	n.a	n.a	n.a
Y_2O_3 (ppm)	200	200	88	33	36	31

To process the Rietveld refinements, the General Structure Analysis System (GSAS, Toby 2001) software was applied (**Table 4).** The background was a manual fitted. After, that the intensity scale was refined, lastly add the unit cell parameters (Young 1993). Unfortunately, the minor phases, under 1% volume – chromite, augite, barite, titanite, hematite and carbonates – could not be included during this stage of data processing firstly because of the XRD HighScore Plus Software, that limits the maximum number of phases to nine. However, a larger number would increase the statistic discrepancy values as goodness-of-fit (X^2), and R factors values, lowering the precision and accuracy of the interpretation results.The X-ray diffraction (XRD) results for Aroeira lamproite are depicted as XRD scans in **Figure 4,** while additional diffraction information is presented in **Table 4**.

Figure 4. XRD Diffractograms with GPhaser software adjusted curves for the studied samples from Aroeira Lamproite.

According to the XRD (figure 4) analysis, these rocks are mainly composed of, in volumetric order, enstatite ($MgFeSi_2O_6$), vermiculite [$(Mg,Al)_3(Si,Al)_4O_{10}(OH)_2.4H_2O$], tetraferriphlogopite [$(K_{0.92}Na_{0.08}).(Mg_{2.88}Fe_{0.12}).(Si_3Fe_{0.88}A_{l.12})O_{10}(OH)_2$], forsterite ($Mg_2SiO_4$), hedenbergite ($Ca_{0.79}Al_{0.06}Fe_{0.08}Mg_{0.47}Fe_{0.60}Si_2O_6$), chabazite [$Ca_{1.85}(Al_{3.7}Si_{8.3}O_{24}).(H_2O)_{7.6}$], ilmenite ($Fe_{1.10}Ti_{0.90}O_3$), whitlockite [$Ca_{18.16}Fe_{0.4}H_{1.68}Mg_{1.6}(PO_4)_{14}$], and cristobalite ($SiO_2$).

Table 4. Rietveld refinement results for Aroeira Lamproite samples.

5.2. Forsterite

The olivine is identified as a Mg-rich phase is forsterite is restricted to the mega/macrocrysts. They are represented by moss green to light brown prismatic subhedral-to-anhedral crystals that range in size from 4mm to 30mm. Contacts with other phases are generally reentrant and curved (**Figure 5**). In cross-polarized light they show second to third order yellow interference color (**Figure 5A, B**).

Inclusions of euhedral chromite and of eye- and ripple-shaped minerals – identified as phosphates – were observed (**Figure 5A, B**). Olivine shows a fracture pattern also known as “mesh structure”, typical of weathered grains and most crystals are surrounded by serpentine (**Figure 5D**) and/or bordered by the pyroxene-rich groundmass Some crystals exhibit ragged and embayed margins suggesting resorption prior to serpentinization, corresponding to the dog’s tooth texture, characteristic of orangeitic rocks (Mitchell, 1995).

Figure 5. Microscopic images of the studied rocks. (A) Macrocryst of olivine with phosphate and chromite inclusions, transmitted light. (B) Olivine macrocrysts with phosphate and chromite inclusions, cross-polarized light; (C) Olivine macrocrysts weathered to serpentine and surrounded by a fine-groundmass, pyroxene-rich, with serpentine (Srp) and phlogopite (Phl), cross-polarized light; (D) macrocrysts of phlogopite in polarized light; (E) Macrocryst of olivine and fine phlogopite in matrix in transmitted light; (F) Macrocryst of olivine and fine phlogopite in matrix in cross-polarized light.

Fifty-one SEM analyses (51, **Supplement 1**) were performed in macrocrysts identified as olivine during the classical petrography. The weathering effects affected the ratios (Fe+Mg)/Si due to the serpentinization of the olivine affecting the cations/anions proportions and relations. We considered in the classification of olivine all results with total cations sum above 2.85, sum of cations minus [Si (Ti,Al)] > 1.3, and T2 > 1.3. The number of anions range from 4.51 to 4.72. **Table 2** presents the average values for #44 analyses in olivine. MnO was not detected during analyses. CaO was below 1% and present in 14 analyses and a high amount of Cr_2O_3 (3.7%) or Al_2O_3 (2.7%) was detected in one of the crystals. Regardless of the effects of weathering in increasing the amounts of Si and decreasing Mg and Fe contents, there is still an apparent proportion for these elements and analyses resulted in Fo (80.0 to 93.7%), Fa (0.0 to 19.1%), and Mo (0.0 to 1.2%) allowing to classify the olivine as a Mg-rich forsterite (~ 88.2% Fo, **Figure 7A**). Some olivine pseudomorphs commonly contain opaque cores mantled by vermiculite and chabazite. The degree of alteration is reflected in variable amounts of silica contents (43.8% to 86.4% SiO_2), that also tends to reduce the MgO, showing a progressive transformation of the olivine into vermiculite.

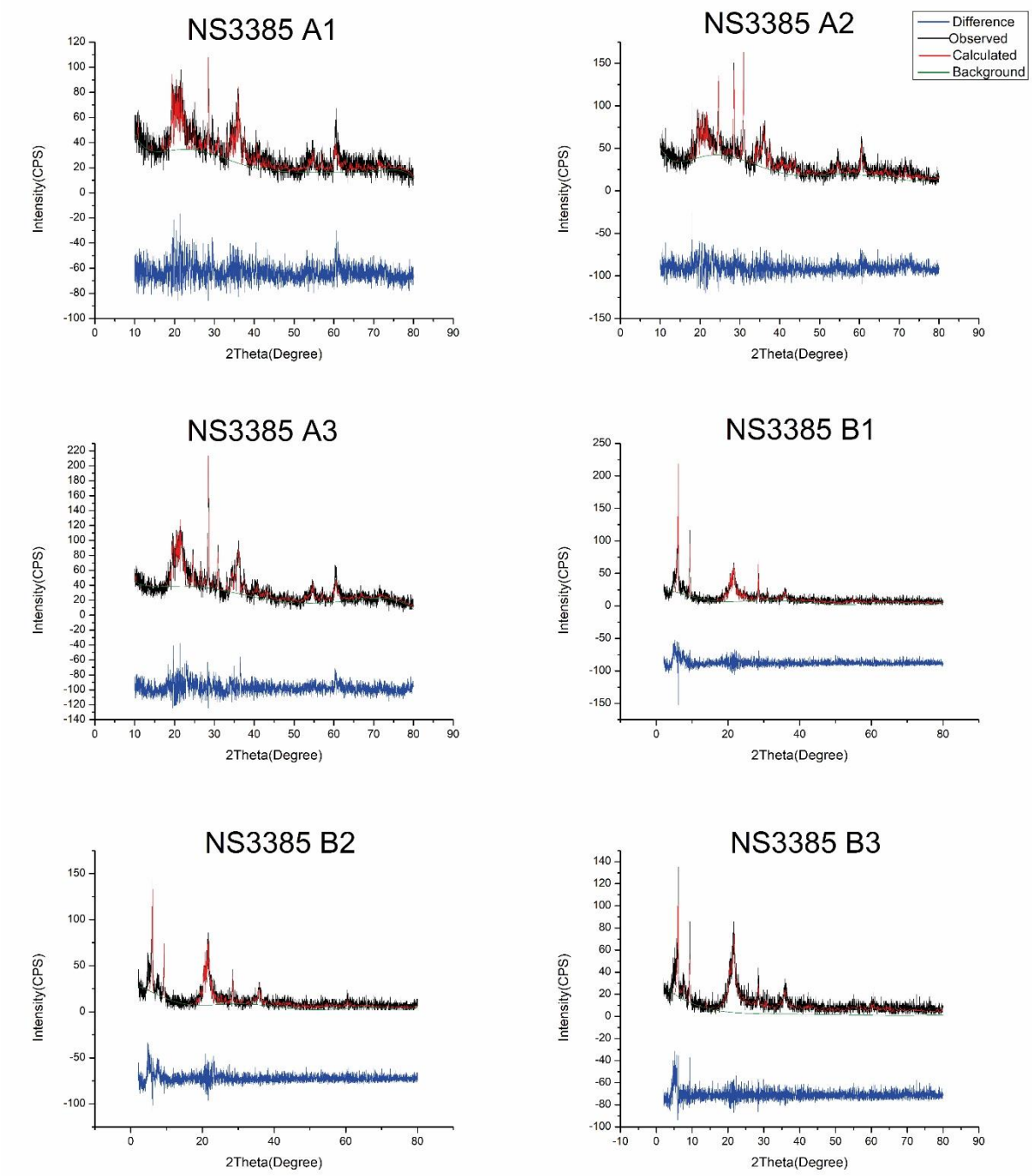
NS3385 A1
NS3385 A2
NS3385 A3
NS3385 B1
NS3385 B2
NS3385 B3
Difference
Observed
Calculated
Background
Intensity(CPS)
2Theta(Degree)

Figure 4

Table 4

	NS3385-A1	NS3385-A2	NS3385-A3	NS3385-B1	NS3385-B2	NS3385-B3
X^2	1.49	1.38	1.60	1.75	1.81	1.51
wRP	20.69	19.14	20.65	35.07	35.43	32.76
RP	16.02	14.52	15.36	27.59	27.55	25.59
Enstatite (%)	26.04	29.26	28.241	10.18	13.05	14.00
Tetra-ferri-phlogopite (%)	17.48	14.83	18.385	24.21	33.68	27.31
Vermiculite (%)	13.29	19.20	16.573	31.01	21.5	27.02
Forsterite (%)	10.39	8.43	9.4227	6.33	6.32	3.41
Hedenbergite (%)	9.28	12.32	14.243	8.61	5.33	5.96
Chabazite (%)	8.92	4.72	2.8897	9.83	10.67	18.14
Whitlockite (%)	6.45	2.67	4.1105	7.42	5.17	0. 22
Ilmenite (%)	4.54	5.84	3.0986	0.02	1.54	0.03
Cristobalite (%)	3.60	2.74	3.036	2.42	2.71	4.17

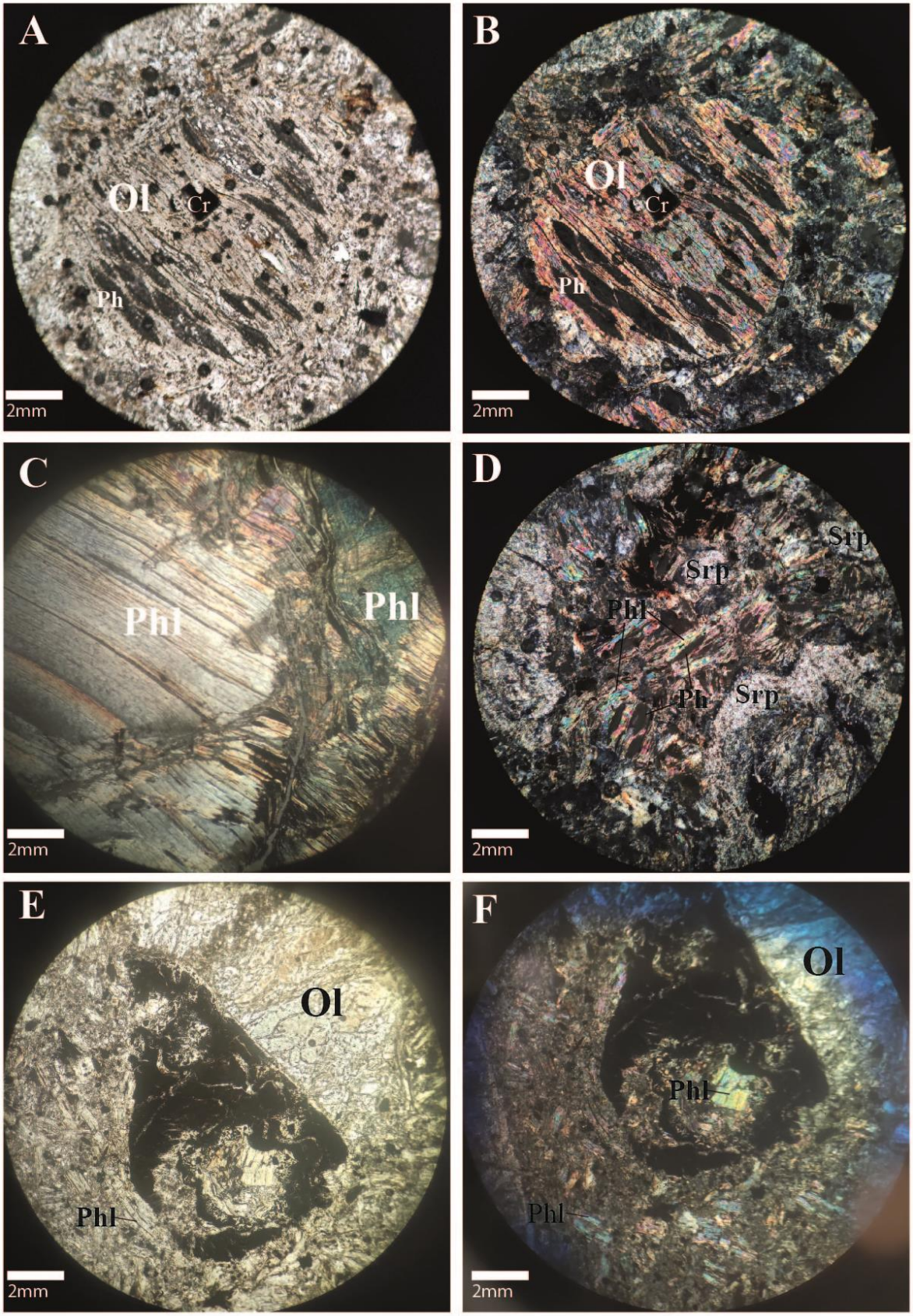
A
Ol
Cr
Ph
2mm
B
Ol
Cr
Ph
2mm
C
Phl
Phl
2mm
D
Srp
Srp
Phl
Ph
Srp
2mm
E
Ol
Phl
2mm
F
Ol
Phl
Phl
2mm

Figure 5

Figure 6. SEM Images of the studied mineral phases, Aroeira Lamproite. (A) BSE image of forsterite (Fo) and phlogopite (Phl) macrocrysts showing inclusions of phosphates (Ph). (B) The alpha radiation elemental map for Mg, highlighting the macrocrysts and the richness of Mg phases in the groundmass. (D) Backscattered-Electron (BSE) image of the groundmass crosscut by Barite (Bar) veins and showing Hedenbergite (Hd) microcrysts. (D) The alpha radiation elemental map for Ba highlighting the barite veins – in yellow – in the are showed in figure (C). (E) The alpha radiation elemental map for P highlighting the phosphates (same area as image A). (F) The alpha radiation elemental map for Ca – in green – showing the distribution of the Ca-pyroxenes and phosphates in the groundmass and as inclusions in the macrocrysts (same area as image A).

Figure 7. Compositional diagrams for mineral phases of Aroeira lamproite. (A) Olivine; (B) Mica; (C) Pyroxene.

5.3. Tetraferriphlogopite

The XRD analyses allowed to identify the mica as a tetraferriphlogopite. It is present as subhedral lamellar crystals mega/macrocrysts and in the groundmass, showing bird's eye extinction and sizes ranging in average from 2mm to 12mm (**Figure 4C, D**), although a crystal larger than 40mm was observed. It shows light brown color and interference colors from first order yellows to third order green. Contacts of the mega/macrocrysts with groundmass result in curved to reentrant borders, surrounded by serpentine and/or enstatite.

When comparing Aroeira' mica macro/megacrysts and microcrysts in the groundmass no significant chemical variation was observed. Chemically, they are characterized by high amounts of magnesium (10.1% to 27.5% MgO), iron (3.6% to 23.6% FeO) and titanium (0.9% to 4.8% TiO_2). The high iron content of these phlogopites is a remarkable feature of Aroeira rocks (**Figure 5A**), which belong to the phlogopite-tetraferriphlogopite series (**Figure 7B**). Weathering resulted in the tetraferriphlogopite been at least partially converted in vermiculite (13.3 to 31 wt.% vol.), resulting in higher amounts of silica (62 wt.% < SiO_2) in comparison with similar contents of MgO (19-26 wt.%) and relatively lower Fe_2O_3 (3-6 wt.%).

Kargin *et al.* (2019) related low TiO_2 and Cr_2O_3 concentrations (<1 wt.%) to primary phlogopites found in garnet lherzolite xenoliths otherwise, titanium enrichment (>2 wt.% TiO_2) is a secondary feature in phlogopites derived from mantle xenoliths (Carswell 1973), modified during kimberlite ascent and/or a feature of the phlogopites in kimberlite groundmass (Giuliani et al. 2016). Aroeira tetraferriphlogopites are Ti- and Cr-poor, suggesting they originated from garnet-lherzolites, although, some analyses show higher amounts of Ti indicating a possible modification of these micas during the weathering.

Although SEM gave some indications that the mica in Aroeira is tetraferriphlogopite, the XRD was essential to confirm that. Farmer and Boettchern (1981) reported tetraferriphlogopite in kimberlites and associated them to ultramafic xenoliths. In Aroeira they have been interpreted as both: a product of magmatic crystallization and a post magmatic product.

5.4. Pyroxenes / Serpentine / Zeolite

Pyroxene is identified as the most abundant mineral in these rocks, occurring as microphenocrystals or microlites restricted to the groundmass. Therefore, due to the fine and weathered nature of this groundmass, it is impossible to identify the type of the pyroxene(s) during the classic petrographic descriptions. In transmitted light colors range from brown to moss-pale-green, and in cross-polarized light it varies from first order yellow to light brown. Contact of the groundmass with mega/macrocrysts of olivine and mica are curved and/or reentrant. Poiquilitic texture, with inclusions of pyroxene needles in olivine mega/macrocrysts was observed.

SEM-EDS chemical compositions resulted in four (4) distinct phases: Hedenbergite (8 analyses, **Figure 6C**), Enstatite (4 analyses), Ferrosilite (6 analyses) and Augite (2 analyses), plus 47 analyses for pyroxenes that were grouped as "undefined", probably a result of their partial alteration. Representative compositions of these pyroxenes are given in **Table 2** and **Supplement 1**. XRD allowed us to identify only two main crystallographic forms of pyroxenes: enstatite (10.2 to 29.3% vol) and hedenbergite (5.3 to 14.2% vol.).

The orthopyroxene enstatite (10 – 29% vol.), range from $En_{64}Fs_{35}Wo_{0.03}$ to – $En_{72}Fs_{26}Wo_{0.08}$ (**Figure 7C**); contains 40 to 41wt.% SiO_2 in comparison with 26 to 27 wt.% MgO, 4.7 to 4.8 wt.% Fe_2O_3, show variable Al_2O_3 (2.0 to 7.5 wt.%), and is typically poor in CaO (0.2 to 0.4 wt%) and Cr_2O_3 (undetectable). They occur as discrete

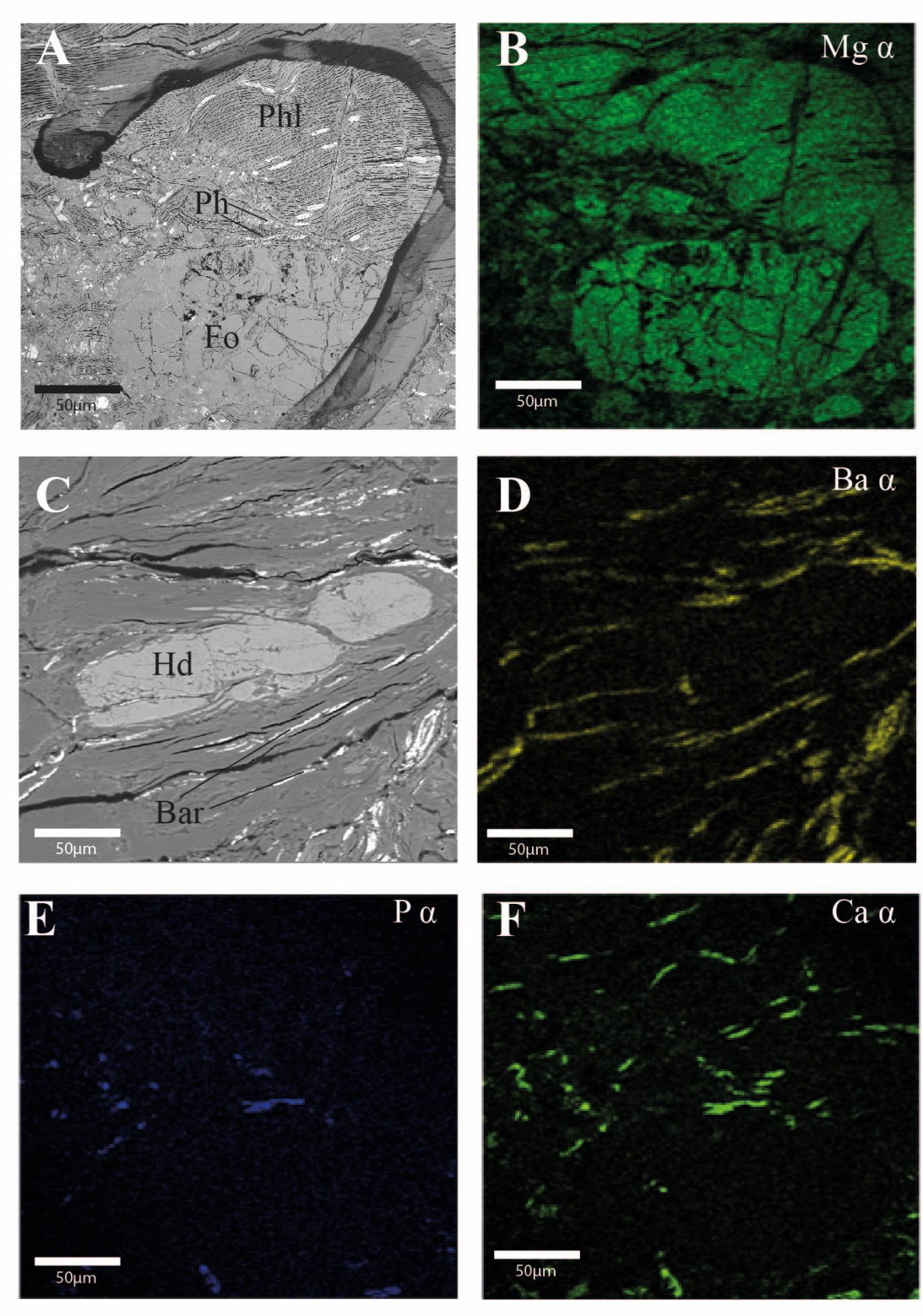
A
Phl
Ph
Fo
50µm
B
Mg α
50µm
C
Hd
Bar
50µm
D
Ba α
50µm
E
P α
50µm
F
Ca α
50µm

Figure 6

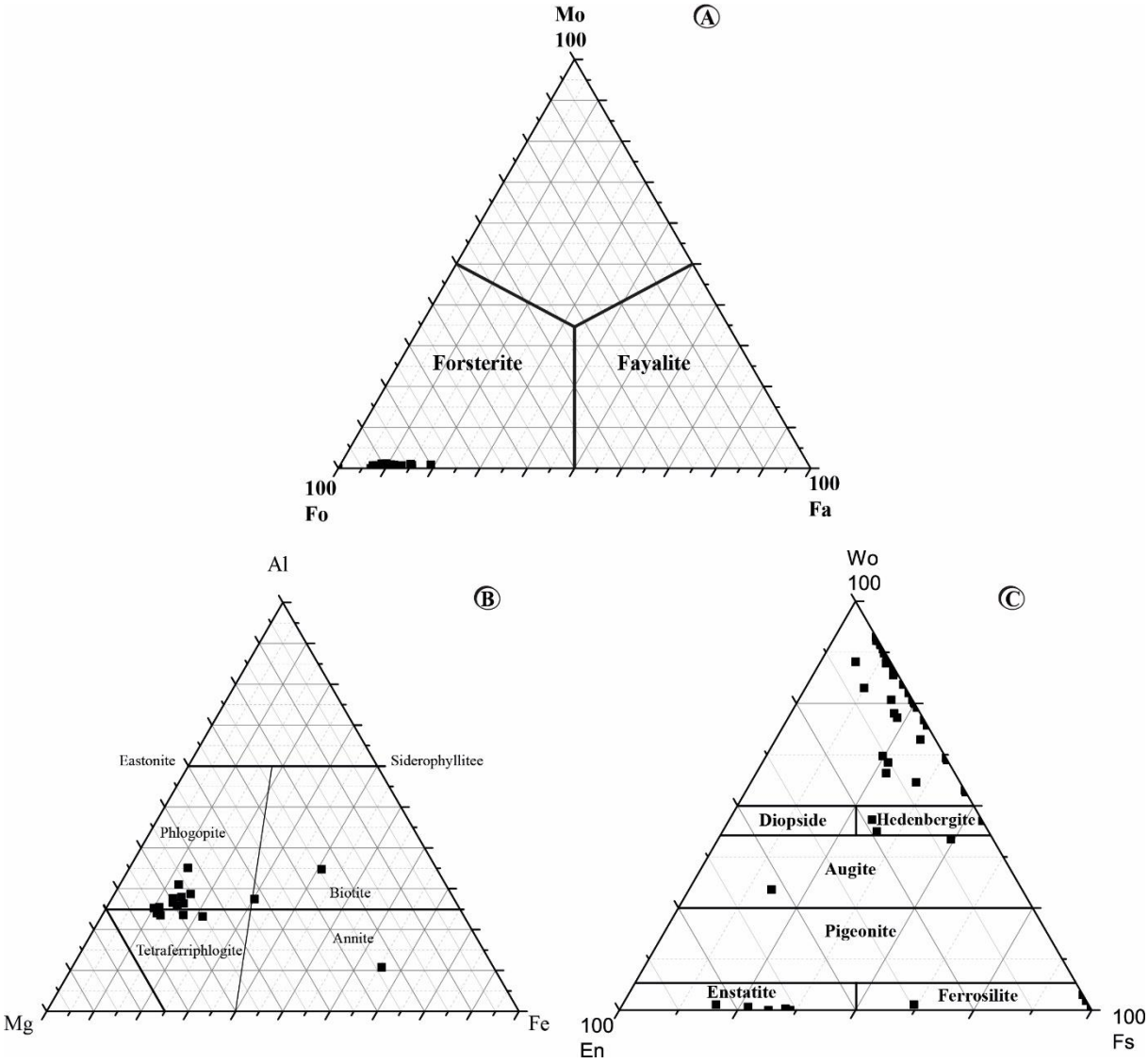
Mo
100
Ⓐ
Forsterite
Fayalite
100
Fo
100
Fa
Al
Ⓑ
Eastonite
Siderophyllitee
Phlogopite
Biotite
Annite
Tetraferriphlogite
Mg
Fe
Wo
100
Ⓒ
Diopside
Hedenbergite
Augite
Pigeonite
Enstatite
Ferrosilite
100
En
100
Fs

Figure 7

Subhedral–to–euhedral microphenocrystals and/or euhedral groundmass microlites. The weathering of samples NS3385A and NS3385B resulted in a large pyroxene modal variation (26% to 29% versus 10% to 14%). The weather effects implicit in this observation suggest that, if macro/megacrysts were originally present, they may have been completely transformed. The most probable candidate for replacing this enstatite is the serpentine or vermiculite, the most abundant secondary phase in these rocks. The alteration of orthopyroxenes to serpentine is a common feature of alkaline rocks (Fuster et al. 1967, Lopez & Rodriguez Badiola 1980).

The Ca-rich pyroxene hedenbergite has an average of 40 to 50% SiO_2 in comparison with 20 to 26% MgO; 4.4 to 7.8% Fe_2O_3; and 21.8 to 24.4% CaO (**Figure 6C, 7C**), been volumetrically significant in the XRD analyses (5.3 to 14.2% vol.). The secondary phase chabazite (2.9 to 18.1% vol.) – a zeolite – seems to be a pseudomorph after pre-existing hedenbergite.

Augite (2 analyses, 52.70% SiO_2; 21.1% MgO; 18.0% Fe_2O_3; 7.8% Al_2O_3) and ferrosilite (6 analyses, 61.8 to 93.0% SiO_2; 0.0 to 17.5% MgO; 3.7 to 24.2% Fe_2O_3; 0.0 to 3.30% CaO) are minor phase, identified only sporadically during the SEM mapping, and undetectable in the XRD, as their volumetric amounts are well below 1%. Augite is the pyroxene phase that occur as anhedral intergrown with olivine and tetraferriphlogopite and they are aluminous (> 3.6wt.% Al_2O_3), have low Na_2O and no detectable TiO_2 (**Figure 7C**).

The application of XRD technique associated to Rietveld refinement demonstrated that ferrosilite is not present in Aroeira, and the chemical changes perceived here in some pyroxene analyses are interpreted because of rock's alteration. It was possible for us to confirm the presence of three distinct pyroxenes in these rocks: enstatite, hedenbergite, and augite. Due to the limitation of phases in the refinement software used, augite was not included in the analysis because its modal value was close to 1wt.%.

The absence of an extensive compositional variation or zonation trends perceived for Aroeira pyroxenes (**Table 2**) is a remarkable feature of lamproitic pyroxenes (Mitchell & Bergman 1991). Diopside clinopyroxene is the typical pyroxene in the majority of lamproites. Although, diopside is absent in Aroeira lamproite. Instead, the most abundant phase in the studied rocks is the enstatite orthopyroxene that forms mesh or needle shaped microphenocrystals, reaching up to 0.2mm and restricted to the kimberlitic groundmass, suggesting magmatic origin. Few lamproites are known to have magmatic orthopyroxene, the most studied are the Spanish lamproites (Mitchell & Bergmann, 1991). Another rare texture described for Aroeira enstatite is the acicular quench shape of these pyroxene microphenocrystals, which are only common in olivine and madupitic lamproites. The absence of sanidine is a possible explanation for the Ca-rich nature of the pyroxenes in Aroeira phlogopite-lamproite (e.g. Smoky Butte Lamproite, Mitchell et al. 1987)

Augite, a rare minor phase in Aroeira, is reported in Leucite Hill (Mitchell & Bergman 1991) as a paragenesis related to olivine-biotite-piroxenite (OBP) suite of Holmes (1950). Their occurrence as intergrowns in olivines and tetraferriphlogopite macro/megacrystals in Aroeira lamproite suggest that at least part of them is of xenocrystic origin.

5.5. Opaque Minerals

Elongated, subhedral, ilmenite micro-crystals, ranging in size between 0.09mm to 0.1mm, were identified in the groundmass. This phase forms poikilitic textures, been included in some macrocrystals of olivine and phlogopite, in which cases they display ellipsoidal shapes.

Ilmenite has an average composition of 75.64% TiO_2; 10.03% FeO; 4.47% MnO; 0.81% Al_2O_3; 0.77% MgO; 0.22 % Cr_2O_3; 0.09% V_2O_5 (**Table 2, Supplement 1**) and demonstrate these are low-Mg and high-Mn, named manganoan-ilmenite. Nascimento et al. (2018) attributed these chemical signatures of Aroeira's ilmenites to their deep source, comparable to the inclusions of ilmenites in diamonds of the Juina kimberlites, Brazil (Kaminski & Belosouva 2009) and to ilmenites from the orogenic lamproites of Mucia-Almeria, Spanish (Mitchell & Bergmann 1991).

Microcrystals of a euhedral, Cr-rich, cubic shaped opaque mineral, up to 0.1mm in size – interpreted as chromite - were observed as inclusions in macro/megacrysts and/or immersed in the groundmass (**Figure 5A; Table 2**).

SEM analyses shows that part of the opaque minerals correspond to titanite. These are anhedral to subhedral crystals, <0.01mm in size, that are distributed in the groundmass, in association with other opaque minerals.

An oxidation area resulted in the identification by SEM of an iron oxide although was not possible to confirm the presence of hematite or magnetite in the XRD, due to the small amount of this phase and the maximum number of phases limitation while using the refinement methodologies. It is possible this iron-oxide be a secondary phase resulting of the weathering of hematite/magnetite/ilmenite.

5.6. Phosphates

Subhedral to globular shaped microcrystals – 0.01mm to 2.00mm – occur as inclusions in macro/megacrysts and/or immersed in the groundmass. They present a white color in transmitted light and show black extinction in cross-polarized light, as well as other features typical of apatite (**Figure 4A, B, D**). Phosphates have not been previously described in Aroeira rocks.

During the SEM analyses, these phosphates were one of the most intriguing mineral phases observed (**Figure 5A, E, F**). CaO contents range from 32.8 to 57.3% and P_2O_5 from 12.6 to 39.5%. Some of these phosphate grains also show high contents of Fe_2O_3 that reach up to 10.2%. Fluoride and chloride were not analyzed. These compositions could be associated to apatite.

XRD was essential to identify these phosphates. All our refinements went wrong when considering these phosphates to be apatite and led us to interpret their crystallographic phase as the monofluorophosphate whitlockite. Ionov et al. (2006) described this mineral phase in upper mantle peridotites (spinel harzburgites and lherzolites equilibrated at 890–950°C) and describe how complex it is to distinguish the two phases – apatite and whitlockite – only by mineral chemistry. Ionov et al. (2006) shows that whitlockite presents an increase in 50–70% for LREE and 20–40% of Y, Sr, Th and heavy to middle REE compared to apatite and argues that trace elements analyses were essential to distinguish between them. Although, our results shows that the low-cost XRD and less time-consuming analyses can be as effective as the more complex chemical methods and easily applicable even to saprolite samples during prospective work.

5.7. Other Mineral Phases

The barite occurs as fine aggregates forming veinlets in the groundmass (**Figure 5C, D**) and/or filling the fractures of olivine macrocrysts.

Cristobalite was first found in Aroeira with SEM analysis, and it was confirmed in the XRD data refinement step. The SEM analysis made possible to identify that silica phase is associated with pyroxene-rich areas of the groundmass. We also observed an anhedral and fractured cristobalite crystal (~2mm) surrounded by two mica megacrysts and this phase also shows micro-inclusions of whitlockite and opaque minerals (ilmenite/chromite). Silica is a common phase in kimberlites and lamproites, because of secondary silicification following weathering (Mitchell & Bergmann 1991). Therefore, the crystallographic form correspondent to the silica identified in this study is ‘low-cristobalite’. We need at least to consider that this cristobalite may be an earlier mineral phase, not necessarily related to the secondary silicification event that affected Aroeira’s rocks and resulted in the formation of vermiculite and chabazite, but xenocrysts inherited from the incorporation of granitic xenoliths during magma ascent and crustal emplacement and/or a mineral that probably resulted from hydrothermal fluids circulation.

6. A Proposed Analytical Protocol for Study of Whole Rock Samples

A robust petrographic knowledge including the correct identification of essential and accessory/exotic minerals of kimberlitic rocks is an important step to better understand the geotectonic environment and the explorational potential for the kimberlitic rocks saving time and allowing a significant reduction of the costs associated with the research during the green-field development of new deposits.

Since the second half of the past century, technological advances in the XRD methodologies and the increased amount of information in the digital databases allowed Rietveld (1961) and other researchers to propose methods to improve the interpretation of whole rocks diffractograms.

The analytical protocol here presented (**Figure 2**) is not new in essence. The proposed flow can be used for a large diversity of samples. However, it is even more useful in rocks that present a challenge for mineral

identification and quantification using classical methods as kimberlites, weathered/saprolites, volcanic ashes/tuffs, and when polymorphs and/or pseudomorphs are present.

The analytical protocol involves field work/sampling, classical sample preparation and combination of a set of simple analytical resources available in most academic settings, even in low-income countries, where research infrastructure are limited. An important step is to use the same fragment for petrographic and chemical analyses (thin-sections and slabs). Combining classical petrographic descriptions with XRF, SEM and XRD allows to get the best of each one of them, in special for the refinement of data, resulting in more precise identification and quantification of exotic, rare, weathered, and complex mineralogical phases (**Figure 9**). Some steps can be adjusted and modified, as requested, for local availability of resources. The XRD is the climax of the proposed protocol as it allows, applying the Rietveld methodology, for the identification and quantification of mineral phases. XRF analysis will help in the stoichiometric balance. Adding SEM images and semi-quantitative chemical punctual analyses of hard to identify minerals reinforce the interpretation, eliminating some bias and resulting in more precise data.

Figure 8. Diagram illustrating the potential of combining the results of petrographic analyses with chemical and structural information.

After obtaining the semi-quantitative data from the Rietveld method, the results may be organized in a stoichiometric **table 5**, which shows the percentages of each of the phases present, comparing them with the XRF analysis. This step adds chemical consistency to the results and allow for an easy visualization of structures and mineral chemistry.

Table 5. Stoichiometric data from the Rietveld method compared to XRF data.

It is important to point out that the quantification made by the Rietveld refinement is linked to the amount of limit phases, so the data does not correspond to the total sample. Therefore, to corroborate the data, a stoichiometric calculation was performed to compare the XRD and XRF data. In these calculations, some differences were observed between what was expected by the Rietveld method and what was found by the XRF. The explanations include the fact that the petrographic files do not exactly correspond to the chemistry of the mineral in the sample, for example, olivine which in their crystallographic record has about 43% of silica content, whereas in SEM analyzes the olivines vary their content of silica between 43.80% to 86.4%, this fact demonstrates that this stoichiometric calculation does not consider these natural variations. Another important fact to score is the lack of phases, due to software limitations. Therefore, contents such as chromium and nickel found in chromite were not calculated. Finally, it is good to remember that as this rock underwent various weathering processes, many of the mobile cations such as Mg, Ca and K were leached, and other elements were concentrated, especially Si.

6.1. Aroeira Lamproite Revisited

Even where the weathering effects are not evident in macroscopic samples or their polished thin sections, their impact can be perceived in changes on the chemical compositions of the studied rocks. In Aroeira lamproite, WDXRF analyses of sample NS3385B (**Table 3**) resulted in higher values of silica ($79.4<SiO_2<82.8\%$), much higher than what was expected for these lamproitic rocks or even presented by the sample counterpart (sample NS3385A, $59.0\%<SiO_2<65.0\%$).

The alteration effects are perceptible in the XRF analyses, in the high amounts of SiO_2 and lower contents of MgO and K_2O (**Table 3**). The mineral composition of Aroeira rocks here presented agrees with the characterization done by Naninni et al. (2017) and Santos et al. (2019). Rios et al. (in preparation) presents volatile content, determined by furnace loss on ignition (LOI) at 950 ◦C, ranging from 6.33-11.34wt%; considering the low contents of carbonates, such volatiles are attributable mainly to pre-existing hydrated silicates, metal hydroxides, and the weathering effects of a tropical environment.

Moreover, when analyzing the XRD results, a significant amount of secondary minerals, such as serpentine and chabazite, was observed represented by a distinct peaks pattern in the diffractograms of the studied samples (**Figure 4**). On sample NS3385A, the first peaks start at 10^o 2θ in clear contrast with sample NS3385B, in which remarkable peaks were found at ~5^o 2θ. These lower-degree peaks are associated to the presence of secondary mineral phases – vermiculite and chabazite – related to the weathering effects. Although it was not intentional, and not clue was found during the petrographic descriptions, the diffractograms clearly indicate that sample NS3385A was more preserved/fresh while sample NS3385B is more affected by surficial weathering processes.

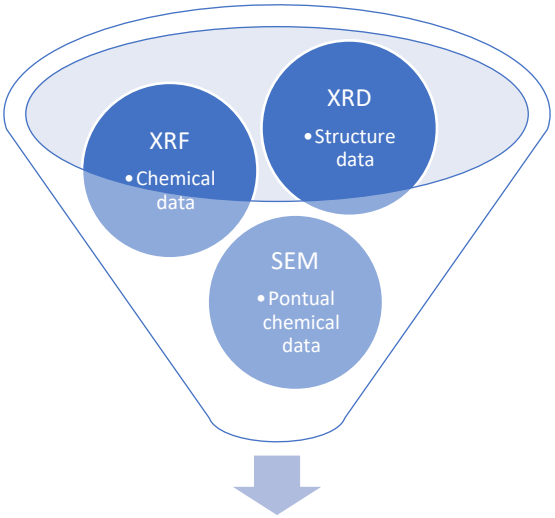
XRF
• Chemical data
XRD
• Structure data
SEM
• Pontual chemical data

Figure 8

Table 5

	NS3385 A1		NS3385 A2		NS3385 A3		NS3385 B1		NS3385 B2		NS3385 B3	
	XRD	XRF	XRD	XRF	XRD	XRF	XRD	XRF	XRD	XRF	XRD	XRF
SiO2	50.07	58.98	53.67	59.17	47.59	58.98	56.16	79.44	52.91	79.44	60.87	79.44
Fe2O3	8.84	15.00	8.64	14.42	11.45	15.00	4.33	5.81	5.55	5.81	4.65	5.81
MgO	24.92	14.09	25.36	13.77	28.81	14.09	24.66	9.21	26.85	9.21	23.70	9.21
Al2O3	2.69	3.85	1.54	4.47	0.33	3.85	3.01	2.83	3.29	2.83	5.32	2.83
CaO	7.60	2.65	5.13	2.70	6.86	2.65	8.24	0.75	6.12	0.75	3.96	0.75
TiO2	3.03	2.43	3.89	2.39	2.39	2.43	0.13	1.22	1.03	1.22	0.02	1.22
MnO	0.00	0.75	0.00	0.66	0.00	0.75	0.00	0.05	0.00	0.08	0.00	0.05
P2O5	1.68	0.68	0.69	0.56	1.11	0.68	1.93	n.a	1.34	n.a	0.06	n.a
Cr2O3	0.00	0.34	0.00	0.38	0.00	0.34	0.00	0.14	0.00	0.14	0.00	0.14
NiO	0.00	0.26	0.00	0.30	0.00	0.26	0.00	0.10	0.00	0.10	0.00	0.10
BaO	0.00	0.18	0.00	0.26	0.00	0.18	0.00	0.05	0.00	0.14	0.00	0.16
CeO2	0.00	0.13	0.00	0.20	0.00	0.13	0.00	n.a	0.00	n.a	0.00	n.a
Na2O	0.09	0.11	0.09	0.14	0.12	0.11	0.14	0.14	0.19	0.14	0.16	0.14
La2O3	0.00	0.10	0.00	0.13	0.00	0.10	0.00	n.a	0.00	n.a	0.00	n.a
CoO	0.00	0.07	0.00	0.08	0.00	0.07	0.00	n.a	0.00	n.a	0.00	n.a
ZrO2	0.00	0.06	0.00	0.07	0.00	0.06	0.00	0.00	0.00	0.00	0.00	0.00
K2O	1.08	0.06	0.98	0.07	1.35	0.06	1.60	0.12	2.23	0.12	1.81	0.12
Total	100.00	99.74	100.00	99.77	100.01	99.74	100.20	99.86	99.52	99.98	100.55	99.97

Rietveld refinement identified vermiculite as the second more abundant mineral phase in Aroeira rocks, as pseudomorphs replacement of pre-existing enstatite and forsterite. Lamproites saprolites are commonly rich in vermiculite and some intensely metamorphosed lamproites (e.g. Enoree Lamproite, South Carolina, and Hill Ponds Lamproite, North America Craton; Mitchel & Bergmann 1991) were previously misnamed as vermiculite bodies. This occurs because of weathering processes, hydrothermal events, and metamorphism, that may intensely modify the primary mineralogy of lamproites and made the correct identification of these rocks very difficult, even impossible in the absence of detailed mineralogical studies.

The very thin size of zeolites and serpentines does not allow their identification during classic petrography. At Aroeira lamproite, a significant quantity of the zeolite chabazite was identified in the XRD (> 2.8% up to 15% vol.), and SEM mapping demonstrated they occur filling cavities or as veins cutting the groundmass. These occurrences of zeolites are described by Mitchell & Bergmann (1991) as a typical feature of lamproites. This author also describes a Ba-bearing zeolite as a typical association for these alkaline rocks. Ba-rich zeolites were not identified in Aroeira lamproite, although, we also found barite, a secondary phase, and cristobalite, in these amygdales and veins. In this case we advocate that barite is replacing the preexisting barium-bearing zeolites.

The occurrence of zeolites – in special chabazite – has economic implications for the Nordestina Kimberlitic Province as the saprolite of these kimberlitic rocks may be considered as a source of this mineral, opening a new window of opportunities for the tailings of the diamond exploration in a more sustainable and environmentally friendly approach for the mining development of this semi-arid region of Brazil. Together, XRD and XRF analyses worked as a more powerful tool to elucidate chemical composition and correlate it to the diversity in mineral phases and amounts present in the two observed samples.

7. FINAL REMARKS

The analytical protocol (**Figure 2**) involves the combination of a set of analyses – Petrographic Microscope + SEM + WDXRF + XRD – to identify the mineralogical phases present in the studied samples and optimize the use of the Rietveld method (Rietveld 1961) to refine the results, resulting in a more precise identification and quantification of the mineral phases. The identification of Aroeira's mineralogy set a good example of the efficacy and accuracy of this protocol.

One of the strengths of this methodology is how easily it can be implemented and applied in several laboratories at a low investment cost. XRF analyses were essential to create a parameter for comparison with the quantitative analyzes of the Rietveld refinement. On the other hand, SEM analyses served as a guide to find the correct mineral phases in the XRD, as it gives clues to the chemical composition. SEM is the more expensive of the techniques used here and it was of fundamental importance for chemical mineral identification, but good results may be obtained even if it is absent.

The resulting quantitative mineralogical modal data by the Rietveld method has many advantages compared to the petrographic microscope alone. Moreover, the Rietveld method was essential to identify mineral phases such as whitlockite and tetraferriphlogopite, in addition to confirm and quantify the distinct pyroxenes phases present in the matrix. The benefit of using this technique provided countless results that would be difficult or even impossible to achieve without it.

We must emphasize the importance of combining these diverse low-costs and widely disseminated methods in an analytical protocol to provide a more detailed petrographic descriptions for kimberlitic rocks – in special in weathered tropical environments and developing countries of Latin-America, Africa and Asia. The protocol allows to identify certain minerals species that otherwise could only be identified with certainty as members of mineral groups, giving essential insights in the origin and paragenesis of these rocks. The association of SEM, XRF and XRD techniques empower the determinations of the primary and secondary phases and complement each other, been very useful for analysis of small size minerals in the groundmass. Also, the modal quantification enhances the interpretation. The most stressful limitation that needs a better approach is the restricted number of phases that can be added to the Rietveld refinement as kimberlitic magmas shows a large diversity of mineral phases and not all of them can be included in the analyses without also introducing interpretative bias. XRD analyses need to be carefully and critically reviewed to avoid errors that led to misinterpretations. Sample selection is also critical as these rocks may carry large xenoliths and inclusions and/or present a size heterogeneity that needs to be compensated in the amount of sample processed or eliminated during preparation to steer clear of incorrect mineralogical information.

The new analyses here presented corroborate the diamondiferous potential and confirm the high-deep mantle source of Aroeira rocks. In resume the applied methodology combined with the Rietveld refinement is a powerful tool for future works, thus giving a new perspective for the studies of saprolite and exotic rocks with basic analytical techniques.

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CAPÍTULO 3
CONCLUSÕES

A intenção desse estudo é avaliar e criar uma metodologia utilizando técnicas analíticas mais acessíveis. Inicialmente escolheu se os saprólitos kimberliticos do pipe Aroeira. A enorme complexidade desse corpo traria a dificuldade necessária para provar que esta metodologia seria muito útil. Após esse período de seleção de objeto de estudo, foi elaborado uma metodologia própria para estudar a petrografia e mineraloquimica. Esta metodologia sofreu várias adaptações devido à falta de infraestrutura da Universidade Federal da Bahia (UFBA), a inexistência de laboratórios analíticos abriu uma necessidade para parcerias com outras instituições, que por um lado foi muito positivo porque o aluno teve contato com profissionais gabaritados trazendo um aprendizado nas técnicas laboratoriais. Entretanto, o fato de essas instituições serem distantes da UFBA gerou custos adicionais e tempo gasto para obter os resultados. O período pandêmico foi outro grande empecilho para este trabalho. O isolamento diminui muito a produtividade do aluno, e isso demandou muito tempo para chegar nos mesmos resultados. Gostaria de evidenciar a importância da orientadora nesse período, que se empenhou e motivou o docente para a conclusão deste estudo.

Um dos principais desafios em rochas kimberlíticas é a identificação correta de minerais essenciais e acessórios / exóticos de rochas kimberlíticas. Este processo de identificação pode fornecer informações como o ambiente geotectônico e o potencial exploratório para as rochas kimberlíticas e irá reduzir os custos associados à pesquisa durante o greenfield desenvolvimento de novos depósitos. A combinação de estudos petrográficos, combinado a técnicas analíticas com microscopia eletrônica de varredura (MEV), difratometria de raios X (DRX) e a fluorescência de raios X (FRX), permitiu uma identificação tanto da química mineral, mas também da estrutura cristalina dos minerais.

Outro grande desafio foi a utilização do método de Rietveld, foi muito difícil de aprender refinamento de estruturas. Contudo o curso do Método de Rietveld lecionando pelo Professor Marcos Sasaki, no instituto de Química (fomentado pelo programa de Pós-Graduação), fez toda a diferença no desenvolvimento deste trabalho. Demonstrando a necessidade de trazer profissionais de outras áreas do conhecimento para os estudos em geociências.

Os resultados obtidos neste trabalho são extremamente promissores, a intenção de propagar o método de Rietveld e popularizar sua utilização nas geociências. O protocolo analítico aqui apresentado não é essencialmente novo, o fluxo proposto pode ser usado para uma grande diversidade de amostras, porém é ainda mais útil em rochas que apresentam um desafio para a identificação e quantificação de minerais por métodos clássicos, alguns exemplos são: Amostras intemperizadas / saprólitos, cinzas / tufos vulcânicos, polimorfos e / ou Pseudomorfos. Por fim os objetivos propostos neste estudo foram atingidos totalmente.

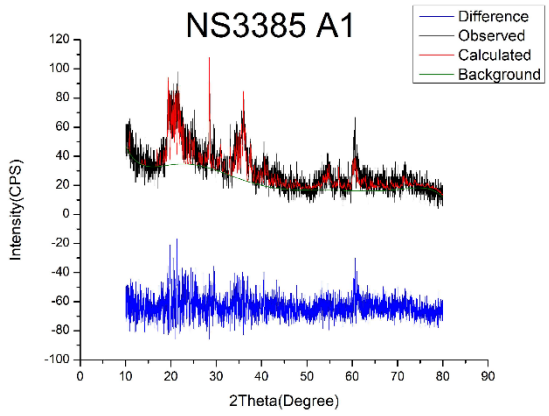
APÊNDICE A – JUSTIFICATIVA DA PARTICIPAÇÃO DOS CO-AUTORES

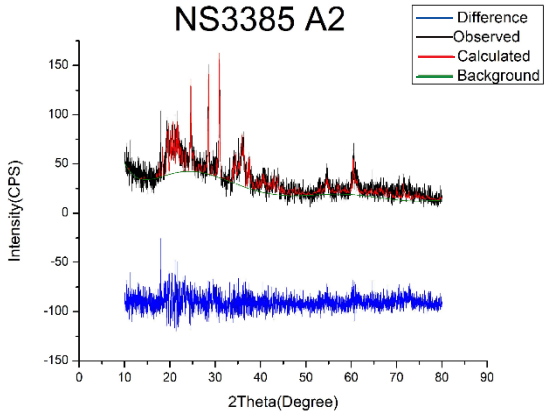
Matheus Andrade Nascimento desenvolveu esta pesquisa no contexto de seus trabalhos de mestrado. Foi responsável pela obtenção, tratamento e interpretação dos dados e pela redação do artigo, elaboração de figuras,tabelas e diagramas.

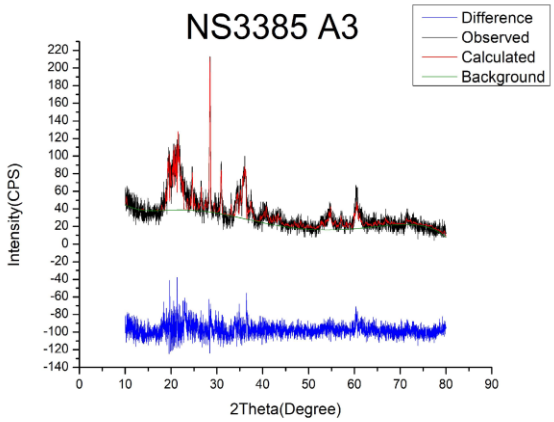
A professora **Débora Correia Rios** elaborou a proposta da pesquisa, sendo responsável pela obtenção dos recursos financeiros e acesso à infraestrutura e equipamentos necessários. Supervisionou os trabalhos de desenvolvimento da pesquisa e participou na redação e correção do presente texto.

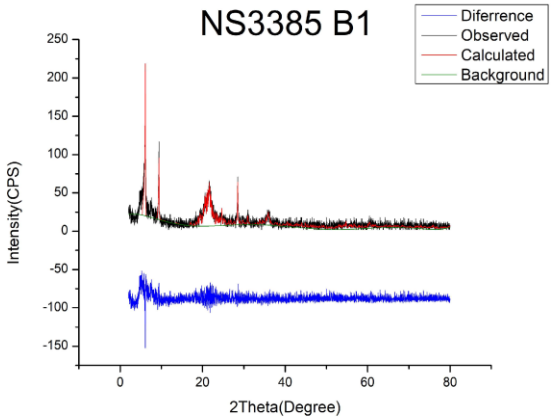
O professor **Herbet Conceição** realizou as análises e imagens de microscópio eletrônico de Varredura (MEV), supervisionou e auxiliou nas interpretações dos dados mineraloquímicos e contribuiu efetivamente na elaboração do artigo.

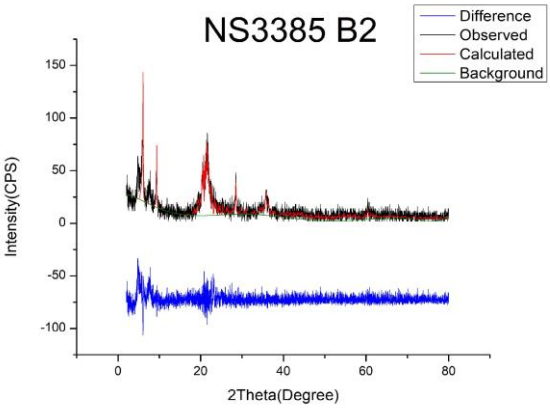
APÊNDICE B – DADOS BRUTOS

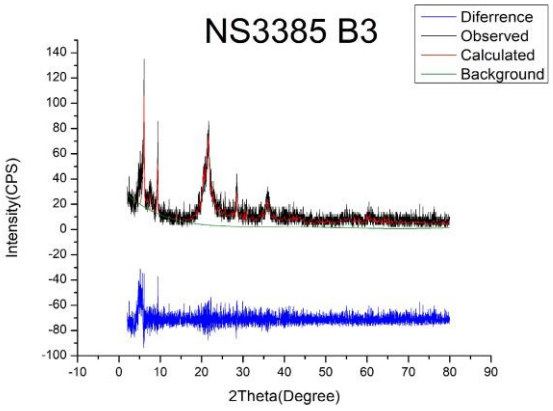












Lamina/Corte	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1
Campo	1	1	1	1	1	1	1	1	1	1
Análise	1	2	3	4	5	6	7	8	9	10
SiO2 (%)	40,90	40,10	42,20	41,10	47,80	43,90			64,80	96,70
TiO2 (%)									0,60	
Al2O3(%)	1,00								2,70	
Cr2O3 (%)										
FeO (%)	25,60	26,40	25,30	26,00	25,00	25,00			6,10	2,80
Fe2O3 (%)										
MnO (%)										
MgO (%)	0,10								25,60	
CaO (%)	32,50	33,50	32,50	32,90	31,10	31,10			0,20	0,30
Na2O (%)										
K2O (%)										0,30
BaO (%)							69,30	65,50		
SO3 (%)							30,70	34,50		
P2O5(%)										
C (%)										
V2O5(%)										
F(%)										
Cl(%)										
Ce(%)										
Y(%)										
Nd(%)										
La(%)										
Ta(%)										
Ni(%)										
Co(%)										
Rb(%)										
Sr(%)										
Total	**100,10**	**100,00**	**100,00**	**100,00**	**103,90**	**100,00**	**100,00**	**100,00**	**100,00**	**100,10**

NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1
1	1	1	1	1	1	1	1	1	1	1	1
11	12	13	14	15	16	17	18	19	20	21	22
98,60	97,90	97,00	41,00	43,50	40,10	42,90		4,00	42,50	1,80	
			0,20								
			0,60			1,10		1,30	7,40	2,60	
1,40	2,10	3,00	25,40	25,00	26,00	24,20	0,80				3,20
			0,30			1,30	1,20				
			32,60	31,50	33,80	30,40	0,10				34,80
							46,60	46,30	32,60	35,70	
							23,90	25,10	17,40	19,40	
											20,70
							27,40	23,40		40,40	41,30
100,00	**100,00**	**100,00**	**100,10**	**100,00**	**99,90**	**99,90**	**100,00**	**100,10**	**99,90**	**99,90**	**100,00**

NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1
1	1	1	1	1	1	1	1	1	1	1	1
23	24	25	26	27	28	29	30	31	32	33	34
62,90	61,20		68,20	81,40	67,00	57,20	67,30	82,70	100,00	100,00	100,00
	29,50										
		95,30				10,40					
5,60	4,20		5,70	4,80	6,50	5,60	5,70	3,60			
27,60			26,10	13,40	26,50	14,00	26,90	13,80			
0,30	5,10	1,00		0,30		0,60					
2,40		3,80									
1,10											
						12,30					
99,90	**100,00**	**100,10**	**100,00**	**99,90**	**100,00**	**100,10**	**99,90**	**100,10**	**100,00**	**100,00**	**100,00**

NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1
1	1	1	1	1	1	1	1	1	1	2	2
35	36	37	38	39	40	41	42	43	44	45	46
100,00	100,00	100,00	100,00	83,90	22,70	31,00	67,60	43,60	58,50		37,20
									21,30		
									0,70		
				4,70	76,60	69,00	7,80	31,10	4,30		
									0,00		
				11,10			24,30		2,60		
				0,40	0,70		0,30	25,10	12,20		
									0,20		
									0,10		
										65,60	40,50
										34,40	22,40
100,00	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**	**100,00**	**99,80**	**99,90**	**100,00**	**100,10**

NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1
2	2	2	2	2	2	2	2	3	3	3	3
47	48	49	50	51	52	53	54	55	56	57	58
24,90	42,00	42,10	41,80	41,30	98,80	85,80	52,80	100,00	100,00	100,00	99,60
						12,20	45,60				
	25,30	25,00	25,40	25,20	1,20	0,90					0,40
0,30	32,70	32,90	32,80	33,50		0,80	1,60				
						0,30					
37,10											
19,00											
18,60											
99,90	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385A1	NS3385 A3	NS3385 A3	NS3385 A3
3	3	3	3	3	3	3	3	3	1	1	1
59	60	61	62	63	64	65	66	67	28	29	30
100,00	100,00	100,00	30,20	36,90	40,50	44,30	50,10	48,50		5,30	50,80
									59,60	94,70	3,40
											13,10
									0,50		
									37,20		7,10
			20,10	17,60	26,10	24,70	20,40	23,50			
									0,70		
							7,80		2,00		24,00
			24,10	20,20	33,40	31,00	21,80	27,90			0,40
											0,30
											0,70
			25,50	25,20							
									0,00		
100,00	**100,00**	**100,00**	**99,90**	**99,90**	**100,00**	**100,00**	**100,10**	**99,90**	**100,00**	**100,00**	**99,80**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
1	1	1	1	1	1	1	1	1	1	1	1
31	32	33	34	35	36	37	38	39	40	41	42
66,10	51,40	100,00	99,80	91,80	51,40	8,40		51,60	49,40	40,00	
	1,90				1,90	81,90	53,10	2,50	2,50	0,10	71,40
	16,90				15,10			12,30	13,60	1,20	
	0,20				0,10	0,40	0,30				0,30
5,50	25,60			7,40	4,90	9,30	45,80	6,10	5,30	25,10	27,40
	0,20				0,10	0,00	0,80	0,00			0,90
28,40					25,50			26,10	27,50	0,10	
	3,00	0,20		0,80	0,40			0,50	0,50	33,40	
	0,90				0,40			0,20	0,30		
					0,00			0,20	0,50	0,20	
						0,00	0,00				0,00
								0,00	0,00		
								0,00	0,00		
100,00	**100,10**	**100,20**	**99,80**	**100,00**	**99,80**	**100,00**	**100,00**	**99,50**	**99,60**	**100,10**	**100,00**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
1	1	1	1	1	1	1	1	1	2	2	2
43	29	30	31	32	33	34	35	36	1	2	3
29,50	87,30		46,40	8,00	6,30	0,00	4,80	3,20	67,10	67,90	67,60
2,40	11,30				5,40	76,10	89,50	92,60			
9,50			4,00	8,70	7,90						
						0,30	0,30	0,90	3,70		
5,30		0,90	20,40	0,80	54,90	23,20	5,40	3,30		4,30	4,20
28,30						0,30	0,00	0,00			
21,00			4,40						29,20	27,80	28,20
3,40	1,40	57,30	24,40	39,40	0,90						
0,60			0,20								
			0,20								
		39,50		28,70							
				14,40	24,60						
						0,00	0,00	0,00			
		0,00									
		0,00									
		0,10									
		0,10									
		0,10									
		0,00									
		0,00									
		0,00									
100,00	**100,00**	**98,00**	**100,00**	**100,00**	**100,00**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
2	2	2	2	2	2	2	2	2	2	2	2
4	5	6	7	8	9	10	11	12	13	14	15
66,80	52,70				100,00		51,60	41,90	47,80	52,30	
			89,10	94,40				1,10	1,30	2,00	87,30
	6,80							25,40	16,10	12,00	5,00
			0,40	0,60							0,30
4,10	17,00	2,00	10,30	5,00		0,80		11,00	8,10	12,40	7,20
							36,70	0,30	0,60	0,20	0,00
28,20	3,10	1,50						17,50	23,90	19,30	
	20,10	32,80				53,70	11,70	1,60	0,90	0,60	
	0,30							0,40	0,30		
								0,40	0,20	0,10	
		19,90				40,10					
		43,80									
											0,00
								0,00	0,00	0,00	
								0,00	0,00	0,00	
						0,10					
						2,40					
			0,20			0,40					
99,10	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**97,50**	**100,00**	**99,60**	**99,20**	**98,90**	**99,80**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
2	2	2	2	2	2	2	2	3	3	3	3
16	17	18	19	20	21	22	23	1	2	3	4
	33,00	99,60	74,20	66,70				66,90	66,70	66,80	66,90
	0,30				4,80		4,90				
	16,10				2,50		0,90				
					0,60		0,30				
2,70	25,00		24,20	5,20				4,40	4,30	4,30	4,20
0,00					90,70		93,90	0,00			
1,20	1,10			28,20	0,80			28,60	29,00	28,70	28,90
36,20	24,30	0,40	1,60			49,20				0,30	
	0,20										
22,40						37,70					
37,50						10,00					
					0,00		0,00				
						0,80					
						0,00					
						0,00					
						0,00					
0,00						0,00					
100,00	**100,00**	**100,00**	**100,00**	**100,10**	**99,40**	**97,70**	**100,00**	**99,90**	**100,00**	**100,10**	**100,00**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
3	3	3	3	3	3	3	3	3	3	3	3
5	6	7	8	9	10	11	12	13	14	15	16
66,70	66,40	67,20	67,60								
						78,30	75,70	90,50	88,10	80,40	87,00
				12,00	19,00			4,60	8,10		
				45,40	32,80	0,40	0,30	0,30		0,00	
3,90	3,90	6,30	5,70	29,20	28,50	21,00	23,90	4,60	3,80	19,60	13,00
						0,30	0,00	0,00		0,00	
29,00	29,70	26,50	26,70	13,40	19,70						
0,30			0,40								
						0,00	0,00	0,00			
99,90	**100,00**	**100,00**	**100,40**	**100,00**	**100,00**	**100,00**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3	NS3385 A3
3	3	3	3	3	3	3	3	3	3	3	4
17	18	19	20	21	22	23	24	25	26	27	25
	7,90	2,60			93,00	19,50	5,80	41,20	34,50	24,50	
83,00							0,40	0,20			0,00
	13,20					4,10	1,30	6,10	10,20	4,40	
										0,20	0,30
17,00		0,70		10,20	3,70	1,20	1,30	23,40	25,30	35,60	
	60,50							0,00			71,70
							1,30	1,50	0,70	0,60	
	2,90	53,70	40,50	48,90	3,30	6,90	0,10	27,60	29,40	34,70	
		0,00									
	15,50						0,40				27,50
		40,70	26,10	37,60		4,70					
			30,50			63,70	89,50				
											0,50
		0,00		0,00							
		0,00		0,00							
100,00	**100,00**	**97,70**	**97,10**	**96,70**	**100,00**	**100,10**	**100,10**	**100,00**	**100,10**	**100,00**	**100,00**

NS3385 A3	NS3385 A3	NS3385 A3	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1
4	4	4	1	1	1	1	1	1	1	1	1
26	27	28	1	2	3	4	5	6	7	8	9
90,20	51,30	56,00	46,50	41,30	40,10	65,20	65,20	58,10	65,00	64,80	70,60
0,00	4,80	0,90			0,40			1,40	29,00	0,00	
3,10	13,20	5,70	1,70	0,70	0,80	2,00	2,00	7,50	0,60	0,80	
					0,00						
6,50	9,90	23,60	21,50	26,00	25,60	4,70	4,70	4,70	4,90	4,80	11,10
0,00				0,00	0,00	0,00	0,00	0,00	0,00		
	17,80	11,00	4,00	0,30		27,60	27,60	26,40		29,10	17,50
0,30	1,50	1,70	26,30	31,60	33,20	0,20	0,20	0,40	0,20	0,00	0,90
	0,10	0,60		0,00		0,40	0,40	0,90	0,40	0,30	
	1,30	0,40						0,50		0,10	
											0,00
100,10	**99,90**	**99,90**	**100,00**	**99,90**	**100,10**	**100,10**	**100,10**	**99,90**	**100,10**	**99,90**	**100,10**

NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1
1	1	1	1	1	2	2	2	2	2	2	2
10	11	12	13	14	15	16	17	18	19	20	21
50,80	61,80	6,50			38,90	50,40	40,50	51,00	48,70	46,30	43,70
3,80	8,90	89,90	93,80	83,50	0,10	0,00	0,00	0,10	0,10	0,00	
16,40				3,70	2,00	3,60	0,90	6,70	2,90	1,00	2,10
0,20		0,50	0,20	0,60		0,10	0,00		0,10	0,00	0,00
13,10	9,90	1,60	5,80	5,60	26,40	17,50	25,70	18,50	20,10	23,10	23,10
0,00			0,00			0,00	0,10			0,00	0,10
10,10	12,30	1,20		6,60	1,90	7,90	0,10	0,30	3,00	1,00	1,60
	0,50				30,60	20,30	32,50	23,10	24,90	28,60	29,20
						0,10		0,20	0,10	0,00	0,00
5,80	0,50				0,20	0,10	0,20	0,10	0,20	0,00	0,20
	6,00										
0,00											
		0,00									
0,00											
	0,10										
		0,30	0,20								
100,20	**100,00**	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**

NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1
2	2	2	2	2	2	2	2	2	2	2	2
22	23	24	25	26	27	28	29	30	31	32	33
52,60	47,40	41,70	45,70			73,00	70,00	71,00	73,60	70,70	69,70
		0,30	0,00	88,90	81,70	23,40					
			1,30								
		0,20	0,00	0,70	0,90						
21,00	29,00	25,20	22,70	4,10	4,90	3,10	5,20	5,40	4,70	5,40	5,60
		0,00	0,00								
		2,40	4,00	5,60			24,30	23,40	21,70	23,90	24,50
26,40	23,60	29,90	26,30			0,60	0,30	0,30			
		0,20	0,10	0,60							
		0,20			8,70						
					3,90						
							0,20				0,20
100,00	**100,00**	**100,10**	**100,10**	**99,90**	**100,10**	**100,10**	**100,00**	**100,10**	**100,00**	**100,00**	**100,00**

NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1
2	3	3	3	3	3	3	3	3	3	3	3
34	35	36	37	38	39	40	41	42	43	44	45
82,50		43,80	42,20	43,40	66,20	66,30	63,00	74,50	68,50	20,60	13,60
	4,40	4,70					0,80			79,40	78,50
	36,90	10,40									
	5,50										
4,20	12,50	20,00	26,40	26,10	5,20	5,30	10,70	7,00	7,40		4,90
		0,90									
13,30	40,60	19,00				28,40	25,10	18,00	23,70		3,00
		0,70	31,40	30,50	28,60		0,40	0,50	0,40		
		0,30									
		0,20									
100,00	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1
3	3	3	3	3	3	3	3	3	3	3	3
46	47	48	49	50	51	52	53	54	55	56	57
12,70	58,10	22,60	17,50	99,60	98,60	98,20	99,30	67,00	62,40	63,40	59,20
85,10	41,90	62,80	82,50					22,10	24,60	20,00	26,70
		8,20									
		4,80		0,40	1,40	1,60	0,70	3,00	4,40	4,30	4,60
2,20											
		1,60				0,20		7,90	8,70	12,30	9,50
0,10											
100,10	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**

NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B1	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
3	3	3	3	3	3	3	3	1	1	1	1
58	59	60	61	62	63	64	65	1	2	3	4
66,80	49,00	48,10	49,50	63,30	56,70	53,80	53,20	67,80	66,50	66,30	66,70
19,50	1,90	3,70	2,20	1,20	1,90	4,30	2,50				
	14,00	14,40	14,30	10,40	15,90	10,50	11,80				
		2,80									
2,80	5,70	5,50	4,90	3,30	3,80	5,20	7,40	5,20	5,20	5,30	5,10
	26,90	24,30	28,30	19,60	20,90	23,20	24,70	26,60	28,30	28,40	28,20
10,90	0,30	0,70	0,50	0,50	0,80	0,40	0,30	0,40			
	0,30	0,60	0,30	0,50							
	1,90			1,10		2,60					
100,00	**100,00**	**100,10**	**100,00**	**99,90**	**100,00**	**100,00**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
1	1	1	1	1	1	1	1	1	1	1	1
5	6	7	8	9	10	11	12	13	14	15	16
	51,60	52,10	48,00	50,10	51,70	100,00	100,00	70,10	98,30	100,00	99,00
2,50	1,00	0,90	1,40	1,20	0,70						
	13,50	13,30	13,40	13,10	13,00						
10,00	4,00	3,60	4,80	4,20	4,10			7,40	1,40		1,00
86,70	29,50	29,80	32,10	30,90	30,20			22,50			
0,90	0,40	0,30	0,30	0,50	0,30				0,40		
100,10	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
1	1	1	1	1	1	1	1	1	1	1	1
17	18	19	20	21	22	23	24	25	26	27	28
42,50	44,40	47,20	43,50	88,50	34,70	40,10	43,70	40,20		26,80	69,50
											30,50
		8,00							7,30		
25,90	26,20	15,10	25,70	11,20	21,50	27,40	26,00	26,30	0,50	1,40	
		16,80									
31,60	29,40	13,00	30,80	0,30	26,20	32,50	30,20	33,50	16,50	22,40	
									12,60	16,80	
					17,60				63,10	32,60	
100,00	**100,00**	**100,10**	**100,00**	**100,00**	**100,00**	**100,00**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
1	1	1	1	1	1	1	1	1	1	1	1
29	30	31	32	33	34	35	36	37	38	39	40
40,00	38,30	14,80	5,80	13,20	3,90	26,30	43,30	16,40	2,60		
4,10	1,80	78,10	92,10	74,80	19,10	64,10					
	4,10						6,90				
6,30	7,10	5,80		12,00	3,30	5,30	1,00	2,20			1,00
9,30	6,80					2,60		5,90			
1,60	0,70	1,30			0,70	1,60	19,40	40,80	51,70	55,40	54,60
									0,20		
2,90											
								32,70	39,60	42,10	41,30
35,80	41,10				72,70		29,40				
									0,00	0,00	0,00
									0,00	0,00	0,00
									0,00	0,10	0,10
									2,80	0,50	0,00
			2,10								
									1,40	0,00	
100,00	**99,90**	**100,00**	**100,00**	**100,00**	**99,70**	**99,90**	**100,00**	**98,00**	**98,30**	**98,10**	**97,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
2	2	2	2	2	2	2	2	2	2	2	2
41	42	43	44	45	46	47	48	49	50	51	52
94,90	100,00	96,30	86,40	66,30	66,60	69,10	66,60	66,60	15,30	39,70	62,40
									67,20	36,90	3,70
4,60		3,70	5,40	5,30	5,20	5,70	5,30	4,80	9,30	4,80	7,10
			8,20	28,20	28,30	25,20	28,10	28,20	8,30		26,40
0,60				0,20				0,50		18,60	0,30
									0,00		
100,10	**100,00**	**100,00**	**100,00**	**100,00**	**100,10**	**100,00**	**100,00**	**100,10**	**100,10**	**100,00**	**99,90**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
2	2	2	2	2	2	2	2	2	2	2	2
53	54	55	56	57	58	59	60	61	62	63	64
4,80	15,50		33,90		51,40						
86,40	64,00				6,30						
0,70						22,30	24,70	12,60	12,00	33,20	9,80
						64,20	36,90	37,10	42,90	34,50	37,90
6,70	6,40		1,60		40,50		19,60	39,90	33,60	18,80	43,70
	3,70					13,40	18,80	10,40	11,50	13,50	8,60
	4,40	55,00	36,20	55,40	1,80						
	5,80										
		42,20	28,30	41,20							
				0,00							
		0,00		0,00							
		0,10		0,20							
		0,70		0,00							
	0,10				0,00						
1,50											
		0,00		0,00							
100,10	**99,90**	**98,00**	**100,00**	**96,80**	**100,00**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
2	2	2	2	2	2	2	2	2	2	2	2
65	66	67	68	69	70	71	72	73	74	75	76
								47,70	47,20	5,10	34,80
								3,80	1,50	7,50	44,00
9,50		11,00	33,80	32,90	7,30			15,40	16,10	11,00	
43,90	55,90	43,20	32,80	50,00	78,30			3,70		40,10	
35,80	44,10	34,10	17,70			1,10	0,90	4,20	5,70	25,50	6,60
10,90		11,70	15,70	17,20	14,30			24,60	29,10	10,80	4,50
						46,70	55,50	0,60	0,40		10,00
						37,10	42,10				
						0,00	0,00				
						0,00	0,00				
						0,30	0,00				
						12,60	0,20				
							0,00				
100,10	**100,00**	**100,00**	**100,00**	**100,10**	**99,90**	**97,80**	**98,70**	**100,00**	**100,00**	**100,00**	**99,90**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
2	2	3	3	3	3	3	3	3	3	3	3
77	78	79	80	81	82	83	84	85	86	87	88
38,70	37,30	66,10	65,60	66,90	66,40	65,20	65,20	65,30	66,30	18,00	32,90
28,00	29,20										
8,30	7,00										
6,90	6,90	4,80	6,10	5,20	4,10	6,50	6,50	5,70	4,60	2,60	4,20
14,90	13,90	29,10	28,30	27,40	29,50	28,30	28,30	29,00	29,10	3,20	5,40
3,20	5,80			0,50						42,90	32,30
										33,20	25,20
100,00	**100,10**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**100,00**	**99,90**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
3	3	3	3	3	3	3	3	3	3	3	3
89	90	91	92	93	94	95	96	97	98	99	100
8,00	39,80			41,40	44,30	5,90	13,50	26,90	6,40	6,40	24,70
						74,30	77,20	63,40	80,70	80,70	63,00
10,30											
0,80	26,20			26,10	30,40	19,80	5,30	8,80	12,80	12,80	7,80
46,20	33,90	55,70	56,50	32,40	25,30			0,90			4,40
34,70		41,40	40,60								
		0,00	0,00								
		0,00	0,00								
		0,20	0,20								
		1,00	0,00								
							4,00				
		0,00	0,00								
100,00	**99,90**	**98,30**	**97,30**	**99,90**	**100,00**	**100,00**	**100,00**	**100,00**	**99,90**	**99,90**	**99,90**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
3	3	3	3	3	3	3	3	3	3	3	3
101	102	103	104	105	106	107	108	109	110	111	112
14,30	8,70		26,80				99,60				94,60
69,80	82,60	88,00	60,30		10,60	3,00					2,10
				9,20	6,40	12,00					
				75,10	32,30						
10,90	8,70	12,00	7,10		38,00	85,10	0,40				2,20
4,90			5,70	15,70	12,60						
								57,20	55,90	57,10	1,10
								40,20	41,10	39,30	
								0,00	0,00	0,00	
								0,00	0,00	0,00	
								0,00	0,10	0,10	
								0,00	1,20	0,00	
								0,00	0,00	0,00	
99,90	**100,00**	**100,00**	**99,90**	**100,00**	**99,90**	**100,10**	**100,00**	**97,40**	**98,30**	**96,50**	**100,00**

NS3385 B3	NS3385 B3	NS3385 B3	NS3385 B3
3	3	3	3
113	114	115	116
55,70	55,70	55,10	55,80
41,10	41,20	41,20	35,50
0,00	0,00	0,00	0,00
0,00	0,00	0,00	0,00
	0,00	0,20	1,10
0,00	0,60	0,30	0,00
0,00	0,00		0,00
96,80	**97,50**	**96,80**	**92,40**

APÊNDICE C – CÓPIAS DAS PUBLICAÇÕES DE RESULTADOS PARCIAIS

APLICAÇÕES DO MÉTODO DE RIETVELD NA PETROGRAFIA DO CORPO KIMBERLÍTICO AROEIRA

Aluno: Matheus Andrade Nascimento**Orientadora:** Débora Correia Rios **Nível:** Mestrado
Semestre de ingresso: 2018.2
Área de Concentração: Petrologia, Metalogênese e Exploração Mineral

As ocorrências da Província Kimberlítica de Nordestina (PKN; Santos *et al*., 2016,2018) foram inicialmente descritas por Pereira (2007), e posteriormente objeto deestudos de Donatti Filho (2011), Rios *et al*. (2011a,b), Donatti Filho *et al*. (2013), eSantos *et al*. (2014, 2016, 2018). Devido a implantação e exploração pela empresaLipari Inc. no campo de Braúnas a primeira mina em kimberlito da América Latinaos estudos destes corpos se tornaram necessários para maior compreensão deste depósito mineral. Sua importância reside no fato deste corpo apresentarcaracterísticas transicionais entre kimberlitos e orangeítos e na sua associação comrochas de natureza sienítica e ultramáficas de idade 0,9Ga (Espanta Gado). Esteprojeto tem como objetivo aplicar o método de quantificação de Rietveld através deanálises por difratometria de raios-x na compreensão da petrografia do corpokimberlitico Aroeira. Para isso será utilizado o software que DIFRAC.TOPPAS nolaboratório de tecnologia mineral de raio-x (LAPAG). O estudo de kimberlitos porlâminas petrográficas se torna complexo, devido a mineralogia exótica, pois háxenólitos e a minerais de origem mantélica pouco conhecidos. Além disto, processoscomo a serpentinização, carbonatização e oxidação são observadas na exposiçãoda rocha em superfície, torna difícil a identificação das fases minerais presentes na amostra. Por estes motivos, a análise por difratometria de raio-X, junto com ométodo de Rietveld se tornam uma poderosa ferramenta para analises de rochas de mineralogia exótica e alterada.

Palavras-chave: Petrografia; Método de Rietveld; Difratometria de raios-x; Kimberlito.

28º Simpósio de Geologia do Nordeste

4º Simpósio Sobre o Cráton de São Francisco e Orógenos Marginais

SGNE-10
415/537

APLICAÇÕES DO MÉTODO DE RIETVELD NA PETROGRAFIA DE ROCHAS KIMBERLÍTICAS SAPROLITIZADAS: EXPECTATIVAS

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A utilização dos raios- X começou em 1895, por Wilhelm Conrad Roentgen. Inicialmente era utilizada para estudos médicos, até que em 1912 Max Von Laue aplicou a técnica em cristais descobrindo a difração de raio- X. Logo se obteve uma poderosa ferramenta para estudos científicos, que guiou as descobertas de todas as estruturas conhecidas até hoje. A análise qualitativa por difração de raios - X se iniciou a partir de estudos de Bragg, onde se determina que, quando um feixe de raios- X monocromáticos incide sobre um material cristalino ocorre o fenômeno da difração. Assim para analisar as fases cristalinas que estão presentes em um pó analisado, é feito um difratograma, que é um gráfico de intensidade observada. Com o difratograma são calculados os picos de máxima intensidade. A interpretação dos picos é facilitada por uma base de dados que contém, para cada elemento, as energias e a intensidade das raias que as produziram. O método de Rietveld foi criado em 1969 por Hugo. M Rietveld, foi inicialmente idealizado para o refinamento da interpretação de dados dos difratogramas gerados a partir da difração de nêutrons. O método permite calcular o difratograma padrão de determinada amostra através de um algoritmo com um padrão difratométrico adequado à fase que se pretende estudar, aplicando para isto o método dos mínimos quadrados. Assim, comparando os picos experimentais obtidos na amostra desconhecida e o padrão de pontos calculados é possível se identificar a célula unitária da estrutura cristalográfica. Alguns destes valores podem ser encontrados em artigos que reportam estruturas parecidas. O estudo petrográfico clássico em kimberlitos é dificultado pela presença da mineralogia exótica, xenólitos, xenocristais, e minerais de origem mantélica pouco conhecidos. Além disto a geoquímica destas rochas, com riqueza de voláteis, favorece os processos tardios de serpentinização, carbonatização e oxidação, transformando as rochas expostas em superfície e impossibilitando muitas vezes a correta identificação das fases minerais presentes na amostra. Por estes motivos, o refinamento das análises por difratometria de raio-X através do método de Rietveld é uma poderosa ferramenta na interpretação petrográfica de rochas de mineralogia exótica e alterada. Tais informações são essenciais para maior uma melhor compreensão da natureza e gênese deste importante prospecto mineral. Este estudo tem o objetivo de aplicar o método de quantificação de Rietveld através de análises por difratometria de raios-x no método do pó-total para ampliar o entendimento sobre a petrografia do Lamproíto Aroeira. Para isso será utilizado o software que DIFRAC.TOPPAS no laboratório de tecnologia mineral de raio-x (LAPAG). A metodologia de trabalho desenvolvida neste estudo focou na seleção de duas amostras representativas do Lamproíto Aroeira, estas duas amostras foram laminadas em três direções ortogonais, obtendo-se assim 6 lâminas polido-delgadas e 6 tabletes destas mesmas frações, os quais foram moídos e geraram o pó analisado por DRX. , assim o estudo da petrografia juntamente com o a aplicação do método de Rietveld no seu respectivo pó dará uma confiabilidade maior as descrições petrograficas e assim ajudar a identificação de fases exóticas e serpentinizadas.

PALAVRAS-CHAVE: PETROGRAFIA, MÉTODO DE RIETVELD, DRX, KIMBERLITO

APOIO: CNPQ

morfológicas parecem ser controladas pelos diferentes litotipos que constituem o preenchimento da bacia sedimentar do Recôncavo. Deste modo as ilhas existentes na BTS atualmente são suportadas por litotipos mais arenosos enquanto as regiões baixas intervenientes correspondem a pelitos com menor resistência à erosão. Os principais lineamentos estruturais da bacia do Recôncavo também exerceram um controle durante a erosão determinando o desenvolvimento de feições erosivas como canais. Com a subida do nível do mar desde o último máximo glacial, este paleo-relevo foi inundado e passou a controlar a sedimentação Holocênica no interior da BTS. A utilização de sísmica rasa de alta resolução e a aplicação dos princípios da sismo-estratigrafia possibilitaram a realização de dois mapas, topo do embasamento rochoso e espessura de sedimentos. Estes mapas permitiram entender os controles litotípicos e estruturais da bacia do Recôncavo no desenvolvimento do paleo-relevo da BTS e deste paleo-relevo na sedimentação Holocênica da baía. Além disso foi possível reconstruir a paleo-rede de drenagem da BTS durante o Último Máximo Glacial e calcular o volume de sedimentos acumulados durante o Holoceno.

Palavras-chaves : EROSÃO DIFERENCIAL,SÍSMICA RASA,PALEO-RELEVO

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Area: CIÊNCIAS EXATAS E DA TERRA - GEOCIÊNCIAS

TRABALHO: TÉCNICAS DE IDENTIFICAÇÃO E CONCENTRAÇÃO DE MINERAIS INDICADORES DE KIMBERLITO E LAMPROÍTO - ESTUDO DE CASO DO LAMPROÍTO TRANSICIONAL AROEIRA

Autor(es) : MANOEL ROSA DE OLIVEIRA JÚNIOR, DÉBORA CORREIA RIOS, MATHEUS ANDRADE NASCIMENTO

Resumo : As técnicas de identificação de minerais indicadores de kimberlito e lamproíto se baseiam em duas etapas, primeiro em analises macroscópicas, através das propriedades físicas como brilho, cor, dureza, hábito, presença ou não de clivagem, e segundo, é necessário recorrer a métodos analíticos, através da química e física, como Difração de Raio X (DRX) e Microscopia Eletrônica de Varredura (MEV) para identificar e diferenciar com precisão os minerais presentes no concentrado. Nesse contexto, é que se baseia esse estudo de caso. O Lamproíto Aroeira é um dos corpos da Província Kimberlítica Nordestina. Ele aflora próximo ao povoado de Espanta Gado, município de Queimadas, e corresponde a um dique de natureza transicional, colocado em situação off-cráton, intrudindo as rochas do Maciço Pedra Solta, a sul do Batólito Sienítico Itiúba. Neste trabalho, o objetivo principal é caracterizar através das técnicas de identificação, os KIM do dique Aroeira. Para este estudo foram coletados cerca de 10 kg de amostra. A rocha encontra-se relativamente saprolitizada, apresenta coloração esverdeada, textura inequigranular, presença de macrocristais, sendo possível macroscopicamente identificar Cr-diopsídio, olivina/ serpentina e flogopita além de grãos menores de granada. Em campo a amostra sofreu um processo de pré-concentração dos minerais pesados, utilizando-se bateia do tipo garimpo, visando a redução de volume e eliminação da fração lama e de cristais/fragmentos maiores que 1cm. O processo de bateamento foi realizado no Rio Itapicuru–Açu, em água corrente, sendo recuperados cerca de 10% da amostra inicial, ou seja, reduzindo a aproximadamente 1kg de concentrado de minerais pesados. Na UFBA, em laboratório, este pré-concentrado foi peneirado gerando duas frações de granulometria distintas: (i) >60# e (ii) <60#. A fração mais fina foi reservada e a fração >60# foi estudada sob lupa binocular, sendo observados granada, diopsídio, ilmenita e olivina serpentinizada. Estes minerais foram catados com auxílio de uma pinça, e separados em frascos individuais. Por fim, as fases minerais mais abundantes foram selecionadas. Cerca de 20 (vinte) grãos de granadas, com tamanho variando de 0,5 - 1,0 mm, anédricos a subédricos, fragmentados, de brilho vítreo a gorduroso e 20 (vinte) grãos de diopsídios com tamanhos variando de 1,0 – 1,5 mm, com coloração verde escuro, opacos a translúcidos e brilho gorduroso, foram selecionados e dispostos em fita dupla face. Nos laboratórios da CPRM, Sureg Salvador, foi preparado dois mounts em resina, um de granada e outro de diopsídio, e por último, foi realizado o polimento. Essa classificação macroscópica dos minerais permite identificar as diferentes fases presentes no concentrado do saprólito de forma preliminar, além de dar subsídios à próxima etapa da pesquisa que envolve sua caracterização química. Nesse sentido, o objetivo final é caracterizar as granadas e os diopsídios através da Microscopia Eletrônica de Varredura (MEV). O estudo desses minerais é de extrema importância na exploração do diamante, sendo utilizados na caracterização da rocha primária (kimberlito, lamproíto, lamprófiro) e para a avaliação da potencialidade econômica do corpo.

Palavras-chaves : LAMPROÍTO MICROSCOPIA ELETRÔNICA DE VARREDURA

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Area: CIÊNCIAS EXATAS E DA TERRA - MATEMÁTICA

TRABALHO: A GEOMETRIA DOS QUATÉRNIONS

Autor(es) : CAUÃ PACHA DE CARVALHO VILTE, JAIME CHAMORRO

Resumo : Nesta apresentação vamos mostrar brevemente o que foi estudado durante o projeto de Iniciação Científica de título "A Geometria dos Quatérnions e dos Octônions". Durante o período de 1 ano neste projeto, estudamos os Números Complexos (C) e sua geometria, isto é, o grupo de Simetria S02 - que, a saber, determina todas as rotações em C - e, de maneira mais geral, o grupo O2, visto que o primeiro é um subgrupo do segundo - Os elementos de 02 que não pertencem a S02 determinam as reflexões em C. O estudo desses grupos se deu tratando os elementos deles como funções complexas para, assim, entendermos a Geometria deste corpo. Após isso, fizemos o estudo dos Quatérnions, começando pelas propriedades algébricas mais básicas e que se mostraram essenciais para o estudo do que veio a seguir. Depois da passagem na álgebra, buscamos entender como os Quatérnions (Q) podiam ser usados para estudar a geometria do espaço R^3, visto que Q tem dimensão 4 sobre R. A chave para isso eram os chamados Quatérnions Imaginários Puros, que são os elementos de Q cuja parte real é 0. Semelhantemente a C, o grupo S03 determina as rotações em Q, mas estas são sutilmente diferente às de C. Como os Imaginários Puros tem 3 dimensões, estas rotações fixam uma direção e rotacionam (como S02) no plano ortogonal à direção fixadas. A medida que o projeto caminha para o final, começamos o estudo da Geometria Esférica, desde a verificação da validade dos 4 primeiros Postulados de Euclides até o estudo de sua simetria, que agora foi facilitado pois, como a esfera mora em R^3, podemos estudar sua geometria utilizando as funções Quaterniônicas. O que faremos nesta apresentação é dar uma breve perspectiva

ANEXO A – INSTRUÇÕES AOS AUTORES CONTRIBUTIONS TO MINERALOGY AND PETROLOGY

Submission guidelines

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Instructions for Authors

Manuscript Submission

Manuscript Submission

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Always use footnotes instead of endnotes.

Acknowledgments

Acknowledgments of people, grants, funds, etc. should be placed in a separate section on the title page. The names of funding organizations should be written in full.

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References

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Cite references in the text by name and year in parentheses. Some examples:

- Negotiation research spans many disciplines (Thompson 1990).
- This result was later contradicted by Becker and Seligman (1996).
- This effect has been widely studied (Abbott 1991; Barakat et al. 1995a, b; Kelso and Smith 1998; Medvec et al. 1999, 2000).

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Reference list entries should be alphabetized by the last names of the first author of each work. Please alphabetize according to the following rules: 1) For one author, by name of author, then chronologically; 2) For two authors, by name of author, then name of coauthor, then chronologically; 3) For more than two authors, by name of first author, then chronologically.

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- Journal article

  Gamelin FX, Baquet G, Berthoin S, Thevenet D, Nourry C, Nottin S, Bosquet L (2009) Effect of high intensity intermittent training on heart rate variability in prepubescent children. Eur J Appl Physiol 105:731-738. https://doi.org/10.1007/s00421-008-0955-8

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- Book

  South J, Blass B (2001) The future of modern genomics. Blackwell, London

- Book chapter

  Brown B, Aaron M (2001) The politics of nature. In: Smith J (ed) The rise of modern genomics, 3rd edn. Wiley, New York, pp 230-257

- Online document

  Cartwright J (2007) Big stars have weather too. IOP Publishing PhysicsWeb. http://physicsweb.org/articles/news/11/6/16/1. Accessed 26 June 2007

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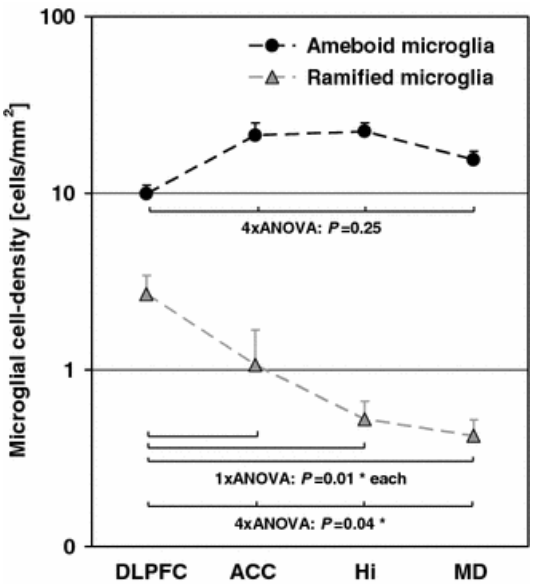

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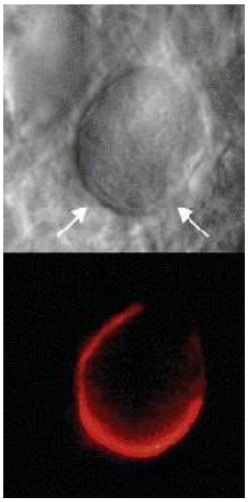

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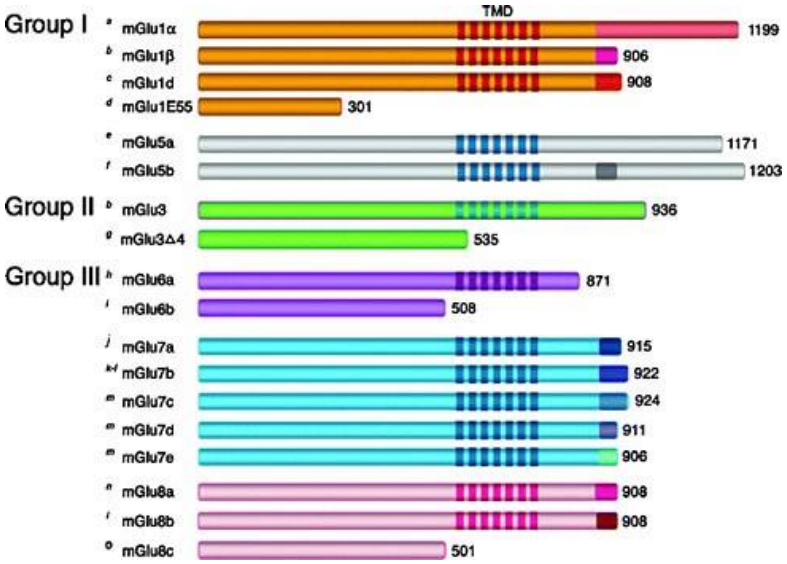

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