DOMÍNIOS MINERALÓGICOS POR DRX E ANÁLISE ESTATÍSTICA MULTIVARIADA DA MINA DE SANTA RITA, BRASIL, EM APOIO AO MODELO GEOMETALÚRGICO *

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Resumo

Este estudo foi realizado em amostras de uma zona disseminada de minério de sulfeto de Ni e Cu em uma intrusão acamada máfico-ultramáfica no Estado da Bahia, Brasil. Esta intrusão foi posteriormente metamorfoseada em fácies granulito. Dados da plantas industriais mostraram uma fraca relação entre a composição química do minério e seu comportamento no processamento mineral. Portanto, o conhecimento da variabilidade mineralógica no depósito é crucial para o planejamento de lavra. Mais de 300 amostras de testemunhos de sondagem, dos estudos de viabilidade e plano de mineração de curto prazo, foram agrupados em domínios geológicos por análise estatística multivariada aplicada à difratogramas de raios-X. Amostras representativas dos principais domínios mineralógicos pré-definidos foram posteriormente submetidas a estudos mineralógicos detalhados por microscopia eletrônica de varredura (MEV) e análise automatizada de imagens (MLA - Mineral Liberation Analyzer). Estes resultados validaram os domínios mineralógicos pré-definidos, concernentes à mineralogia dos sulfetos e suas associações, características fundamentais para o modelo geometalúrgico e processamento mineral. É possível concluir que a área de estudo da região de depósito de Santa Rita pode ser classificada em sete domínios mineralógicos com base nos principais minerais (serpentina, olivina e piroxênios).

Palavras-chave:difração de Raios X, estatística multivariada, minério de níquel, geometalurgia.

MINERALOGICAL DOMAINS BY XRD AND MULTIVARIATE STATISTICAL ANALYSIS OF STA RITA MINE, BRAZIL, IN SUPPORT TO GEOMETALLURGICAL MODEL

Abstract

This study was carried out on samples from a disseminated Ni and Cu sulfide ore zone emplaced in a mafic-ultramafic layered intrusion at Bahia State, Brazil. This intrusion was latter metamorphosed into granulite-facies. Industrial plant benchmark data have showed a poor relationship between ore chemical composition and its mineral processing behaviour. Therefore, the knowledge of mineralogical variability in the deposit is crucial for mine planning. More than 300 samples from drill core samples from the feasibility studies and short term mining plan were grouped into geological domains by multivariate statistical analysis (MSA) of X-ray diffraction data (XRD). Representative samples of the main mineralogical domains pre-defined by XRD-MSA ore-types were later subject to detailed mineralogical studies by scanning electron microscopy (SEM) and automated image analysis (MLA – Mineral Liberation Analyser). These results validated the pre-defined mineralogical domains, concerning about sulfides mineralogy and their associations, which are key features for the geometallurgical model and mineral processing. It is possible to conclude that studied area from the Santa Ritadepositregioncan be classified into seven mineralogical domains based on themain minerals (serpentine, olivine, and pyroxene) content.

Keywords: *X-ray diffraction; cluster analysis; nickel ore; geometallurgy;*

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1INTRODUCTION

The Santa Rita mineis a mafic-ultramafic intrusion with nickel and copper sulfides mineralization located in FazendaMirabela complex, in the southeast of Bahia state, Itagibá municipality. The nickel sulfide deposit comprises a portion of a mafic-ultramafic layered complexundergranulitemetamorphismfacies. Disseminated Ni and Cu sulfides form a stratiform body parallel to the lithostratigraphic contacts extending upwards essentially from the harzburgite unit and through the olivine orthopyroxenite unit (BARNES et al., 2011).

The deposit mineralogy is mainly composed by magnesium silicates (olivines, pyroxenes and serpentines). The Ni bearing mineral is essentially pentlandite and a minor portion of Ni is associated with magnesium silicates and spinels, despite other minor sulfide minerals.

The mineral processing in Santa Rita mine started with rock breakdown by explosives. Trucks then transport the ore for two primary conic and jaw crushers. The primary crusher product is sent to stock pile by conveyor belt. The stack material goes to the milling circuit, which is composed of a SAG mill, two secondary cone crushers and two ball mills generating a product with P80 of 125 μm which is pumped to a desliming stage with designed for the removal of very fine fibrous and plate like silicates responsible for slime coating in the flotation concentration.

After deslimed, the underflowis pumped to the concentration circuit by direct flotation of nickel and copper sulfides, with the rougher, scavenger, cleaner and recleaner stages, using xanthate-based collectors. The plant metallurgical recovery varies according to mineralogical ore characteristics, mainly regardingnickeldistributioninto silicates. The average overall nickel recovery is 58% and the final concentrate has nickel grades ranging from 13% to 15% (Santos Júnior&DelboniJúnior, 2016).

Industrial plant benchmark data have been showing a weak relation between ore chemical composition and its mineral processing behaviour. This study presents the classification of the mineralogical domains of the Santa Rita mine, in the region of the deposit planned for mining from 2013 to 2017, followed by detailed mineralogical studies of the main domains representative samples. The present work aimed to contribute to Santa Rita short-medium term mine plan, improving the knowledge of ore characteristics and its mineralogical variability in the deposit. These characteristics are relevant for mineral processing purposes and prediction of ore behavior in concentration stage, thereby a useful data for the geometallurgical mining plan.

2 MATERIAL AND METHODS

2.1 Samples

Seventeen EW sections, spaced about 100 m apart, were defined to cover a wide spatial distribution along the deposit, considering georeferenced position of drill holes from feasibility studies of Santa Rita mine. The holes were visualized three-dimensionally in the Surpac 6.2 software, concomitantly with the pit areas of 2013 and the 2017 pit planned, in addition to sulphide mineralization layer for sample selection. Only the drill holes intervals belonging to planned mine period of 2013-2017 containing sulphide mineralization were considered for sampling; therefore, 264 ore samples were collected from drill cores.

Afterwards, 55 short-term mining plan samples from blasting drilling were also sampled. A total of 319samples were utilized at XRD-MSA analysis. The 264 drill core samples were not considered to perform SEM image analysis studies as they could present partially oxidized sulphides due to prolonged atmosphere exposed storage, masking their natural characteristics. Therefore, only representative samples of the main mineralogical domains pre-defined by XRD-MSAwere selected from 55 blasting drilling samples for further SEM image analysis studies.

2.2 XRD and MSA analysis

The experimental procedures in XRD and MSA analyses were:

- sample preparation for XRD analysis comprising the pulverization in planetary ball mill in standard conditions and manual backloading pressing in a proper sample holder;
- diffractograms acquisition;
- MSA analysis with groups definition by mineralogical similarity;
- virtual composition of average samples representative of each group, followed by mineral composition estimation by RIR (Reference Intensity Ratio) method;
- 3D spatial visualization of samples classified into groups in the mine at SURPAC ® 6.2 software;
- definition of mineralogical domains, considering mineral composition similarity and space correlation in the deposit.

The diffractograms were collected with a diffractometerX'PertPro coupled to position-sensitive X'Celerator detector (PANalytical) with Cu kα radiation, 45kV / 40mA, in the angular range (2θ) from 5 to 70°, with step of 0.02° at 10 seconds. Cluster analysis was performed with HighScorePlus software tools for diffractograms classification by hierarchical correlation of mineralogical composition similarities (comparison of position and intensity of diffracted peaks). The conditions used in cluster analysis were: source of profile data, peaks, position and intensity, 10% criterion in threshold comparison, Euclidian distance measure, average linkage method and manual cut-off definition. The identification of the minerals for the virtual average samples of each groups was obtained by comparing the diffractograms with the database of the International Center for Diffraction Data (ICDD, 2003) and semiquantitative mineralogical estimation was done by RIR - Ratio Intensity Reference method, using crystal structures of the ICSD - Inorganic Crystal Structures Database (2007).

2.3 Mineralogical detailed studies by SEM image analysis

The experimental procedures for mineralogical detailed studies were:

- wet size sieving of the material at 0.15 mm sieve aperture;
- grinding of 0.15 mm oversize product in a planetary mill in closed circuit until P_{95} = 0.15 mm (equivalent to industrial plant grinding size after comminution circuit);

- the 0.15 mm undersize material was then homogenized with the oversized material grinded and it was wet sieved in apertures of 0.105 mm, 0.053 mm e 0.020 mm;
- sampling of an aliquot of each sieved fraction above 0.020 mm for polish section preparation, followed by SEM-MLA image analyses.

The mineralogical study of fractions above 0.020 mm was performed in an automated way, using MLA software - Mineral Liberation Analyzer (FEI) version 3.1 coupled to Quanta 650 FEG (FEI) scanning electron microscope andEDS microanalysis system Esprit (Bruker).

3. RESULTS

3.1 Cluster analysis by XRD-MSA

The conditions used to define clusters allowed samples discrimination into 17 groups, as shown in the dendrogram (Figure 1) and Principal Component Analysis (PCA) diagram (Figure 2). The PCA diagram is just a way to visualize the correlation matrix and cluster utilized: each point represents one sample; dots placed near each other represent similar samples and each group has a distinct color.

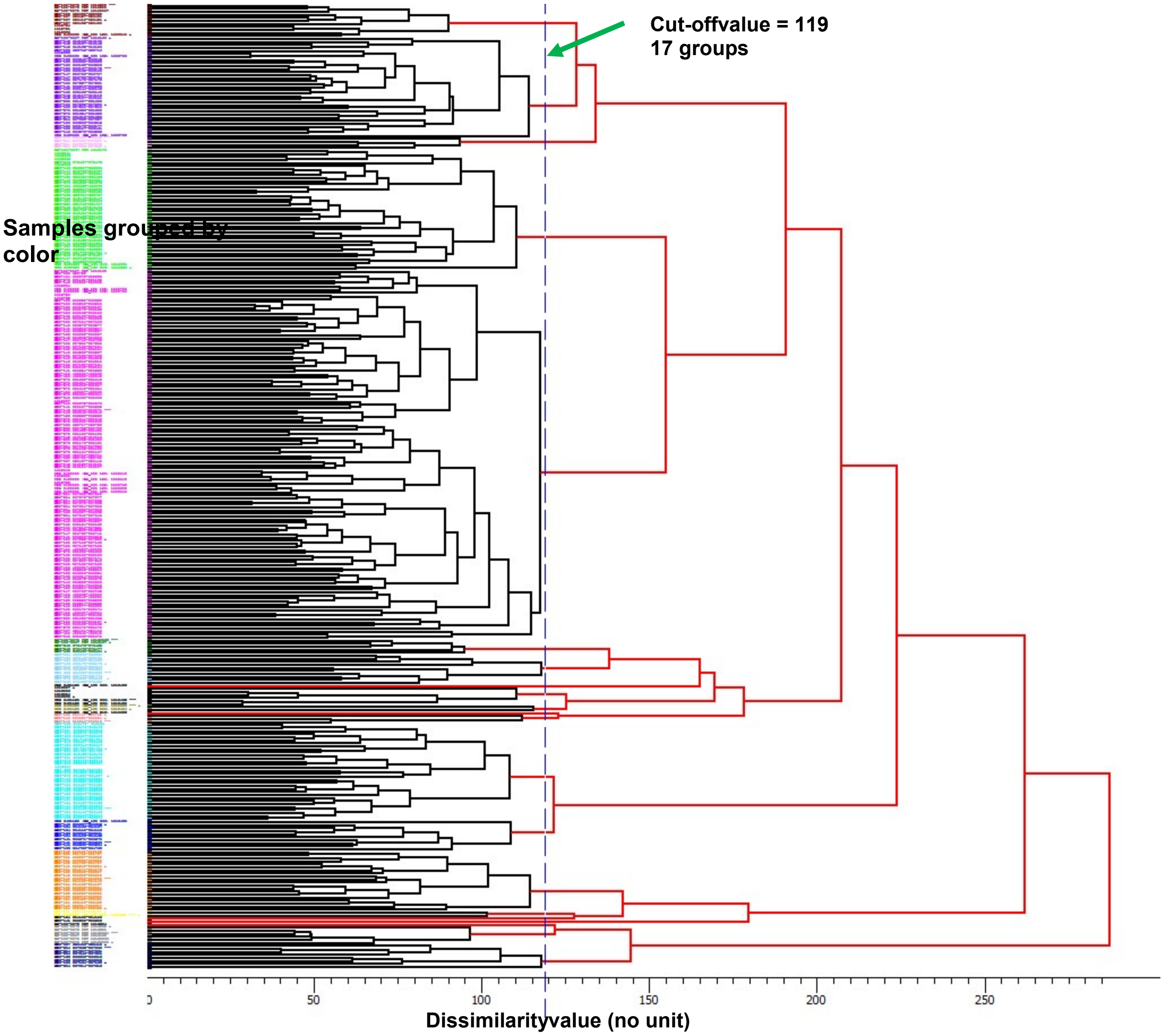

Figure 1 - Dendrogram. Group colours: 1 = dark blue, 2 = light blue; 3 = magenta; 4 = beige; 5 = orange; 6 = purple; 7 = light orange; 8 = green; 9 = black; 10 = blue; 11 = cyan; 12 = yellow; 13 = grey; 14 = dark green; 15 = light pink; 16 = brown; 17 = red

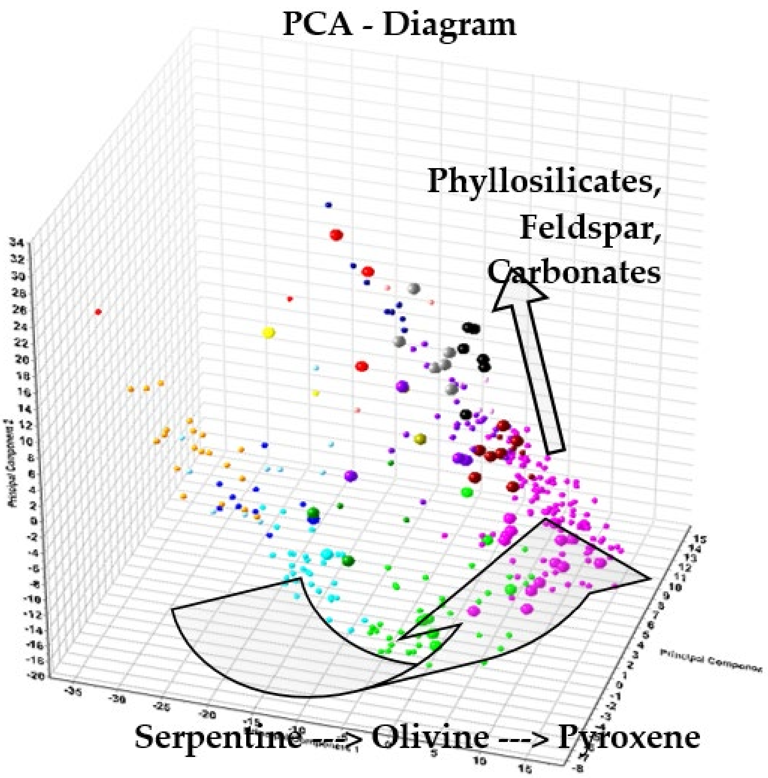

Figure 2 - PCA diagram. Larger spheres refers to samples from blasting drilling.Narrowsindicates increasing in the content of the specified minerals. Group colours: 1 = dark blue, 2 = light blue; 3 = magenta; 4 = beige; 5 = orange; 6 = purple; 7 = light orange; 8 = green; 9 = black; 10 = blue; 11 = cyan; 12 = yellow; 13 = grey; 14 = dark green; 15 = light pink; 16 = brown; 17 = red

The estimation of the average mineralogical composition in each group, obtained by RIR method,is presented in Table 1. The adopted group nomenclature considered the order of abundance of the minerals in each group. For example, the olivine / pyroxene group with serpentine>10% (OPS) has olivine as the most abundant mineral, followed by pyroxene and serpentine. To facilitate writing, the names were condensed into acronyms, each letter representing the initials of the minerals (O = olivine, P = pyroxene, S = serpentine, A = amphibole and C = carbonates).

The mineral grades and spatial correlation of the samples were considered to define the mineralogical domains, as an example shown in Figure 3.

Table 1 -Mineralogical composition of groups (RIR%, semiquantitative)

Domain	OPS				POS		PO	P			PAF		CS		C		*
Group	5	10	2	11	8	14	3	16	6	13	1	15	12	4	9	7	17*
Mineral	Weight%																
serpentine	25	25	15	20	10	10	<5	<5	<5	<5			25	10	<5	<5	
olivine	50	45	45	50	35	40	10	5	5	5				10			
pyroxene	20	20	35	25	50	45	75	80	85	85	65	60	5	20		10	
amphibole	5	10	5	<5	<5	5	5	5	5	<5	10	10	10	<5	<5	<5	
feldspar				5		<5	5	5	<5	5	15	25	5	15	65	45	
phyllosilicates	<5	<5	<5	<5	<5	<5	<5	5	<5	5	<5	5	30	15	10	10	
quartz							<5	<5	<5		5		10	15	10		
carbonates	<5	<5		<5	<5	<5					<5		15	15	15	35	
pentlandite	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5							

Note: * = outliers

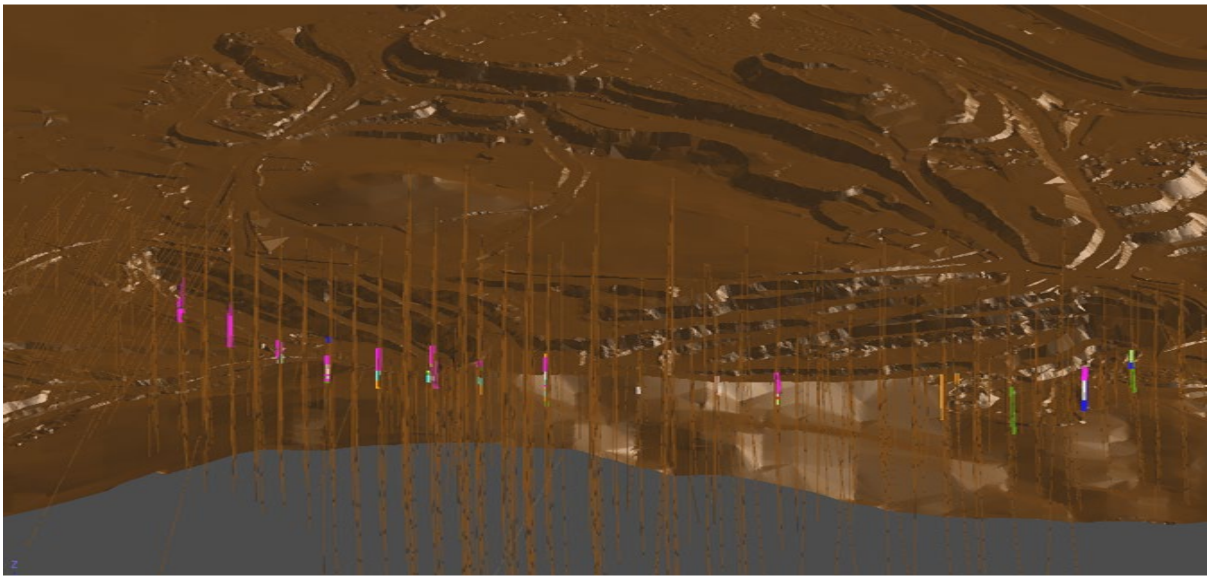

Figure3 - Spatial groups correlation evaluated to mineralogical domains definition. Lower view of Santa Rita pit mine with the portion of the drill cores holes sampled; each colour represents a group as described in Figure 1 and, in brown, the surface with the 2011 minepit and the drilling holes.

3.2 Mineralogical detailed studies

Pentlandite texture

Some pentlandite particles shows regions with different gray levels at backscattered electrons (BSE) images (Figure 3), reveling variations in their composition. The lighter gray levels corresponds to pentlandite, intermediate gray levels are violarite and the darkestgray levels are iron oxide.The SEM-MLA resolution applied could not segmented violarite from the iron oxide presented at micro veins. Therefore, theses phases were grouped and called as viol + ox-Fe (Figure 4).

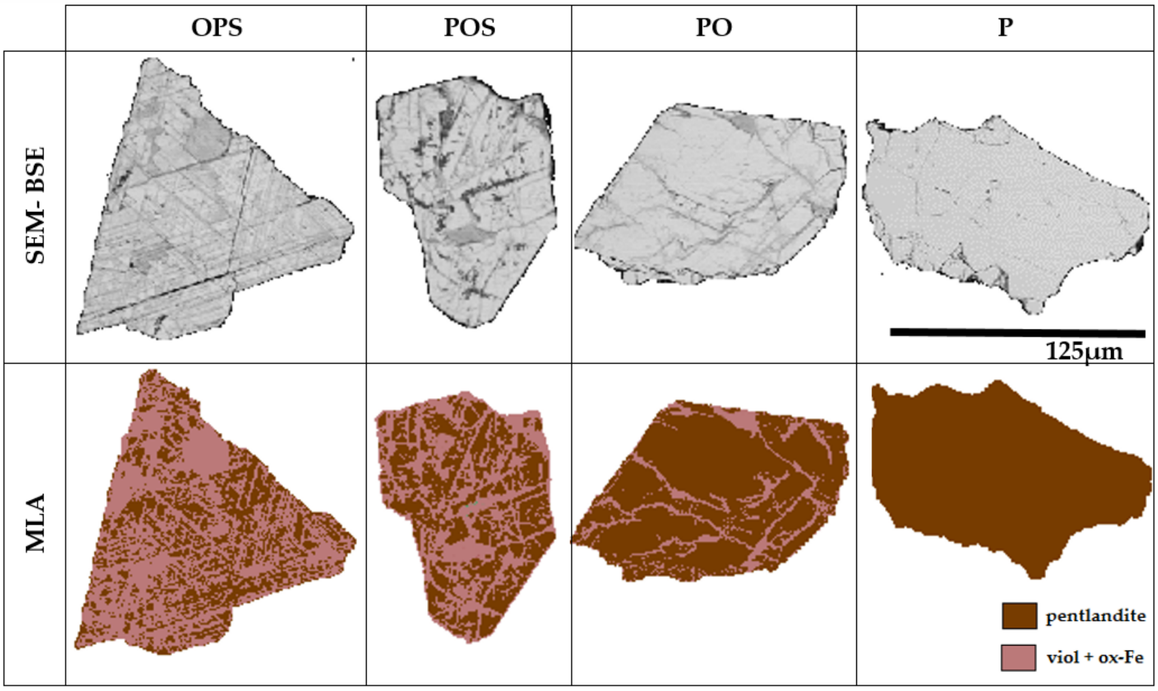

Figure 4 –Pentlandite alteration texture.Classified SEM-MLA images per mineralogical domain

SEM-MLA automated image analysis

The modal mineralogy of the main domainsis shown in Table 2.

Table 2 – Modal mineralogy of the main domains by SEM-MLA (wt%) – Total +0,020 mm

Minerals / domains	**OPS**	**POS**	**PO**	**P**
olivine	37.8	18.5	9.99	8.78
serpentine	37.8	16.1	3.88	5.03
orthopyroxene	6.49	49.5	72.6	69.7
clinopyroxene	1.77	3.86	6.11	5.45
amphibole	2.00	2.42	1.79	4.50
otherssilicates	0.78	1.78	2.20	1.76
Cr-spinel	3.38	1.47	0.78	1.34
iron oxide	2.14	0.39	0.34	0.47
micas	0.25	1.24	0.30	0.35
carbonates	3.11	1.72	0.37	0.54
others	0.29	0.24	0.12	0.14
Sulfides				
pentlandite	1.67	1.37	0.80	0.90
viol + ox-Fe	1.71	0.31	0.10	0.20
chalcopyrite	0.25	0.35	0.19	0.23
pyrite	0.48	0.58	0.34	0.29
pyrrotite	0.04	0.15	0.11	0.26
arsenopyrite	<0.01	<0.01	0.01	<0.01
FeCoNisulfide	0.09	0.02	<0.01	0.01
~ratio (pentl: viol + ox-Fe)	***1:1***	***4.4:1***	***8:1***	***4.5:1***

The pentlandite + viol + ox-Fe were grouped into one phase called "total pentlandite" for mineral associations. Due its intrinsic relationship, it could not be separated by physical comminution. Therefore, the particles were classified inliberated, locked in binary (2 phases) or locked in ternary (3 or more phases) particles for the total >0.020mmas shown in Table 3, for the adopted comminutions conditions

Table 3 – Mineral locking for total pentlandite by domain (P_{95} of 0.15 mm) - Total +0,020 mm

Total pentlandite	**OPS**	**POS**	**PO**	**P**
Liberated (wt%)	22	46	43	48
Locked in binary (wt%)	40	30	41	30
Locked in ternary (wt%)	38	24	16	22

The Table 4 presents the nickel partition per mineral in each main domain. The nickel content in the bearing minerals followed the values adopted by Fróes (1993) and Purvis (2005).

Table 4 – Nickel partition in the bearing minerals by domain (%) - Total +0,020 mm

Mineral	**OPS**	**POS**	**PO**	**P**
pentlandite	43	72	76	78
viol + ox-Fe	30	9	9	5
FeCoNisulfide	3	1	1	<1
orthopyroxene	<1	2	4	4
olivine	12	9	7	9
serpentine	11	7	4	3
% Ni in sulfides	***77***	***82***	***86***	***83***
% Ni in silicates	***23***	***18***	***14***	***16***

4 DISCUSSION

The cut-off valuedefinesthe number of samples to be grouped in each cluster and represents the dissimilarity in mineralogical composition. This value can be defined by statistical methods, without verifying the physical meaning for the proposed grouping. Thereby, the cut-off value at this study were defined by observing similarities and dissimilarities of the diffractograms and to split the samples at feasible number of groups (17 groups) for possible future studies. Then, to verify grouping procedures, mineral composition (Table 1) and samples distribution in the deposit (Figure 3) were considered to define mineralogical domains.

The main differences between the XRD-MSA groups are mainly based on the relative content of serpentine, olivine, pyroxene and, secondly, phyllosilicates, feldspars and carbonates. The samples inserted in groups 2, 5, 10 and 11 are characterized by high contents of olivine and serpentine. The groups 8 and 14 also present high content of olivine, with less serpentine. Therefore, the groups 2, 5, 10, 11 and 8, 14 were considered as two different mineralogical domains, OPS and POS, respectively.

The samples inserted in groups 3, 6, 13 and 16 are basically composed of enstatite, with content variations in the minor constituents: olivine, amphibole, plagioclase, mica, among others. It is noteworthy that group 3 is the most representative of the sampled universe, encompassing 38% of the total samples analyzed, thus the group 3 was considered as a single domain (PO), while groups 16, 6 and 13 were clustered in other domain (P).

In groups 1 and 15, the contents of enstatite and diopside (respectively 40-45% and 20% in the average of each group) are significant, therefore it was classified as another domain (PAF). Group 12 also includes serpentine-rich specimens which are practically free of olivine and pyroxene, with other phyllosilicates (~ 30%, mainly chlorite and, secondarily, mica and talc) and magnesite (~ 15%) contained.

In the samples from groups 4, 7 and 9, the contents of carbonates, predominantly dolomite, are highlighted. The first is still composed of intermediate proportions of enstatite, serpentine, olivine, plagioclase, quartz, mica and talc, while in groups 7 and 9, the proportions of feldspars (plagioclase and / or K-feldspar) are high. However, by spatial correlation, the groups 12 and 4 were considered as a domain (CS), while 9 and 7 other (C).

Therefore, the seventeen groups primarily defined by XRD-MSA were combinedin seven mineralogical domains. Only the groups OPS, POS, PO and P were forwarded to MLA image analysis study, since they represent the main domains in the spatial distribution in studied deposit.

The modal mineralogy of each main domain showed that OPS has the highest content of pentlandite, violarite and sulfides, when compared to other domains. There is a decrease in the olivine, serpentine and sulfide contents from OPS to POS, PO and P groups. The textural analysis (Figure 3) and ratio pentlandite: violarite+iron oxide (Table 2) shows that the pentlandite has more alteration to violarite and more intrinsic relation with micro iron oxide veins from OPS to P domain.

The mineral association studies showed that in all samples, the percentage of free total pentlanditeparticles does not exceed 50% for the grinding conditions used. The sample OPS presents between 19-28%ofpentlandite in free particles,and the locked particles are predominantly associated with serpentine. The samples POS, PO and P have around 43-48% ofpentlanditeinfree particles and the locked particles are mainly associated with orthopyroxene.

Regarding the nickel partition, 77-86% of the total Ni occurs in sulfide phases and there is an increase of nickel portion in silicates(23% of the Ni content) in the sample OPS, comparing it to the other samples.

There are relevant information regarding minerals associations and elucidated by this research that may interfere negatively in mineral processing nickel recovery, such as:

- alteration of pentlandite: it can significantly decrease pentlandite recovery by the presence of violarite and iron oxides;
- nickel partition in silicates: the amount of Ni that occurs in silicates cannot be recovered by mineral processing, since the processing of the Santa Rita mine concentrates only sulphfides;
- associations of pentlandite and sulfides: locked particles have lower potential recovery in flotation; if those particles float, they can reduce sulphfide content in final concentrate and also carry undesirable elements, depending on their mineral associations.

According to Lamberg (2011), geometallurgy, in its basics, combines geological and metallurgical data in order to create a spatially-based predictive model for mineral processing plant. This model must be useful in production management. Therefore, knowing the spatial distribution of mineral domains is one of the key data to input in the geometallurgical model.

5CONCLUSION

Based on this study, the Santa Ritadepositcanbe initially classified into seven mineralogical domains based on the content of the main minerals (serpentine, olivine, and pyroxene) and the presence of feldspathic and carbonategangue. The groups OPS and POS have more peridotite composition while the PO and P show a pyroxenite composition. Mineralogical studies indicate that groups with more peridotite have a higher serpentine content, greaterpentlandite alteration, higher proportion of the total nickel associated with silicates andlowerpentlanditeliberationdegree, compared to the groups enriched in pyroxene. Even though, the XRD detection limit does not allowed the quantification of sulphfide minerals, but it could be demonstrated the correlation between the gangue minerals assembly and sulfides characteristics, such as mineral association and degree of pentlandite alteration. Therefore, XRD-MSA diffractogramsanalysiscan be used to define mineralogical domains and for mineralogical data acquisition for geometallurgical model of Santa Rita mine.

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