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Effects of Igneous Intrusion on the Mineralogical Content of Irati Formation, Paraná Basin, in Sapopema (PR), Southern Brazil

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Abstract

Igneous intrusions in sedimentary basins are commonly related with mineralogical association changes in host-rock. At Sapopema region (Paraná State, southern Brazil), an extensive diabase sill (associated to Serra Geral Formation) was emplaced in pelitic-carbonate succession during post-Triassic. The sedimentary host-rock association includes mostly shale, siltstone and carbonate of the Permian Irati Formation. X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) data revealed that heat transfer was not enough to cause modifications in mineral assemblage of the Taquaral Member (quartz + albite + muscovite + illite + kaolinite + chlorite). However, mineralogical content from Assistência Member presented changes probably caused by the intrusion of diabase sill (talc + pyrophyllite + calcite). Talc and calcite were formed due to the reaction between dolomite and quartz, while pyrophyllite was the product of reaction between kaolinite and quartz.

Keywords: Sedimentary Basin. Igneous Intrusions. Sediment Metamorphization. Mineralogical Reactions. XRD. SEM/EDS.

1. Introduction

Igneous intrusions are common in many sedimentary basins worldwide (e.g. Jones et al., 2007; Thomaz-Filho et al., 2008; Delpino and Bermúdez, 2009; Miranda et al., 2016), including the intracratonic Paleozoic Paraná Basin (southern Brazil; Fig. 1). During magmatic intrusion, temperatures above 1100 °C generally cause melting and the sills can affect the sedimentary host rock by developing contact metamorphic aureoles (e.g., Jaeger, 1959; Simoneit et al., 1978; Aarnes et al., 2010; Senger et al., 2014). Heat transfer related to igneous intrusions in sedimentary basins will produce variations in rock properties, such as changes in mineralogy, organic geochemistry, and rock microstructure (Senger et al., 2014; Li et al., 2016; Illgen et al., 2017).

In its stratigraphic record, the Paraná Basin exhibits igneous rocks related to the rupture of the Gondwana Supercontinent, which gave rise to the opening of the Atlantic Ocean ca. 120 Ma (Mizusaki and Thomaz Filho, 2004). The Serra Geral Formation comprises a thick pile of lava flows, which can reach thicknesses of 2.000 m in some regions (Milani and Zalan, 1999). This formation is characterized by a network of dikes, cutting the entire Paleozoic-Mesozoic sedimentary succession, and multiple diabase sills that intrude into the stratification planes of Paleozoic sediments, preferentially along the Irati Formation horizons (Mizusaki and Thomaz Filho, 2004).

The effects of magmatic intrusions on physical properties and evolution of organic matter in shales have received much attention in the Paraná Basin (Milani, 1997; Milani and Zalán, 1999; Araújo et al., 2000; Correa and Pereira, 2005; Costa et al., 2016, among others). However, few researches have addressed the changes in mineralogical

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content of the Irati Formation (Girardi et al., 1978; dos Anjos et al., 2010). This work describes the impact of igneous intrusion in the Permian Irati Formation, in Sapopema region, and discusses changes in the mineralogical content.

2. Study Area

The study area is part of the tectonic-stratigraphic context of the Paraná Basin (Fig. 1), a vast region composed of sedimentary and magmatic rocks, which extends from the southern part of Brazil to Eastern Paraguay and the northeast of Argentina and northwest of Uruguay, covering a total area of approximately 1.4 million square kilometers (Milani, 1997).

Irati Formation (Permian) lies within the tectonic sequence of Gondwana I, corresponding to the basal unit of Passa Dois Group, and is subdivided into the Taquaral (lower) and Assistência (upper) members (Fig. 1). Taquaral Member is composed of grey siltstones and shales with parallel lamination (Santos Neto, 1993). The Assistência Member is composed of bituminous black shales intercalated with cream-colored to grey carbonatic horizons (Hachiro, 1996). According to Schneider et al. (1974), the lithological and paleontological characteristics of Irati Formation indicate, for Taquaral Member, deposition in a marine environment with calm waters, below the level of wave action. For Assistência Member, a shallow marine environment under basin restriction (low circulation and oxygenation), which allowed the deposition of black carbonatic shales, which developed on shelf areas (Araújo et al., 2000).

Costa et al. (2016) demonstrated, through 2D seismic interpretation, that the Irati Formation contains post-Triassic, basic igneous bodies, composed of dykes and sill with thicknesses that vary in different parts of the basin. In the study area, the isopach map of Serra Geral Formation shows the occurrence of thick intervals of igneous rocks in contact with Irati Formation (Fig. 2).

3. Materials and Methods

X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analyses were carried out in Irati Formation core samples from the well SP-58- PR (Coordinates Córrego Alegre, Zone 22S: 7383382N/549372E). This well is located in Sapopema, northeastern Paraná state, and was drilled by the Geological Survey of Brazil (CPRM) in the 1970s.

3.1. X-ray diffraction (XRD)

All the preparation procedures for the samples and XRD analyses were carried out in the Laboratório Multiusuários de Caracterização Tecnológica (LMCT) of the Centro de Tecnologia Mineral (CETEM), Rio de Janeiro (Brazil). The XRD method was applied to 16 samples from different depths, encompassing the Taquaral and Assistência members.

Considering the interplanar distances (d) relative to the diffractometric reflections, different mineral species were identified in the < 63 μm and < 2 μm fractions. Utilizing the powder method, the XRD analyses were carried out in a Bruker-AXS D8 Advance Eco equipment, with Cu Kα (40 kV/25 mA) radiation.

Randomly oriented samples of whole-rock powders were prepared by filling front-loading XRD mounts (Moore and Reynolds, 1997). The samples were scanned at a rate of 1°2θ/min from 5° to 50°2θ. For the analysis of the oriented samples (< 2 μm), non-clay minerals were removed through standard chemical treatment, based on Stokes' Law (Moore and Reynolds, 1997). Thus the <2 μm fraction were prepared by air drying a small amount of dispersed suspension on a glass slide. To help identify the clay minerals, ethylene glycol-solvated (12h) samples and samples heated at 510°C for 1h were also prepared using well known methods (Alves, 1987; Moore and Reynolds, 1997; Martins et al., 2007). Theses samples were scanned from 4° to 30° 2θ.

Qualitative and semiquantitative mineralogical analysis followed the method described by Melo et al. (2018). The qualitative interpretation of the spectrum was performed with a Bruker-AXS Diffrac.EVA 4.0 or 4.1 software and PDF04+ database. The first step was mineral identification, and then the peaks of each mineral were scaled manually to give the best fit to the observed XRD diffraction. The semiquantitative analysis was carried out considering the X-ray powder diffraction and the minerals in the International Centre for Diffraction Data database (ICDD, 2017).

3.2. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

Sample preparation for analysis in the SEM followed the norm established by the Laboratório de Análise Mineralógica of CENPES/PETROBRAS. Based on XRD mineralogical interpretation, four samples were selected to SEM/EDS analyze: first ground, in order to offer a fresh and irregular surface, then; glued to a brass conductive support and; finally covered by a thin layer of gold-palladium with the EMITECH K750X metallizer, to make it electrically conductive. Then, it was glued to an aluminum conducting support and analyzed in the ZEISS EVO LS-15 scanning electron microscope, with images produced by backscattered electrons, operating at high vacuum, at 20 kV, and using a working distance of 12.50 mm.

The image produced by backscattered electrons represents, in its shades of grey, the variation of the average atomic composition of the feature that was imaged,

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meaning that materials of heavier atomic weight will produce images in lighter shades of grey, while lower atomic weight materials will produce darker images.

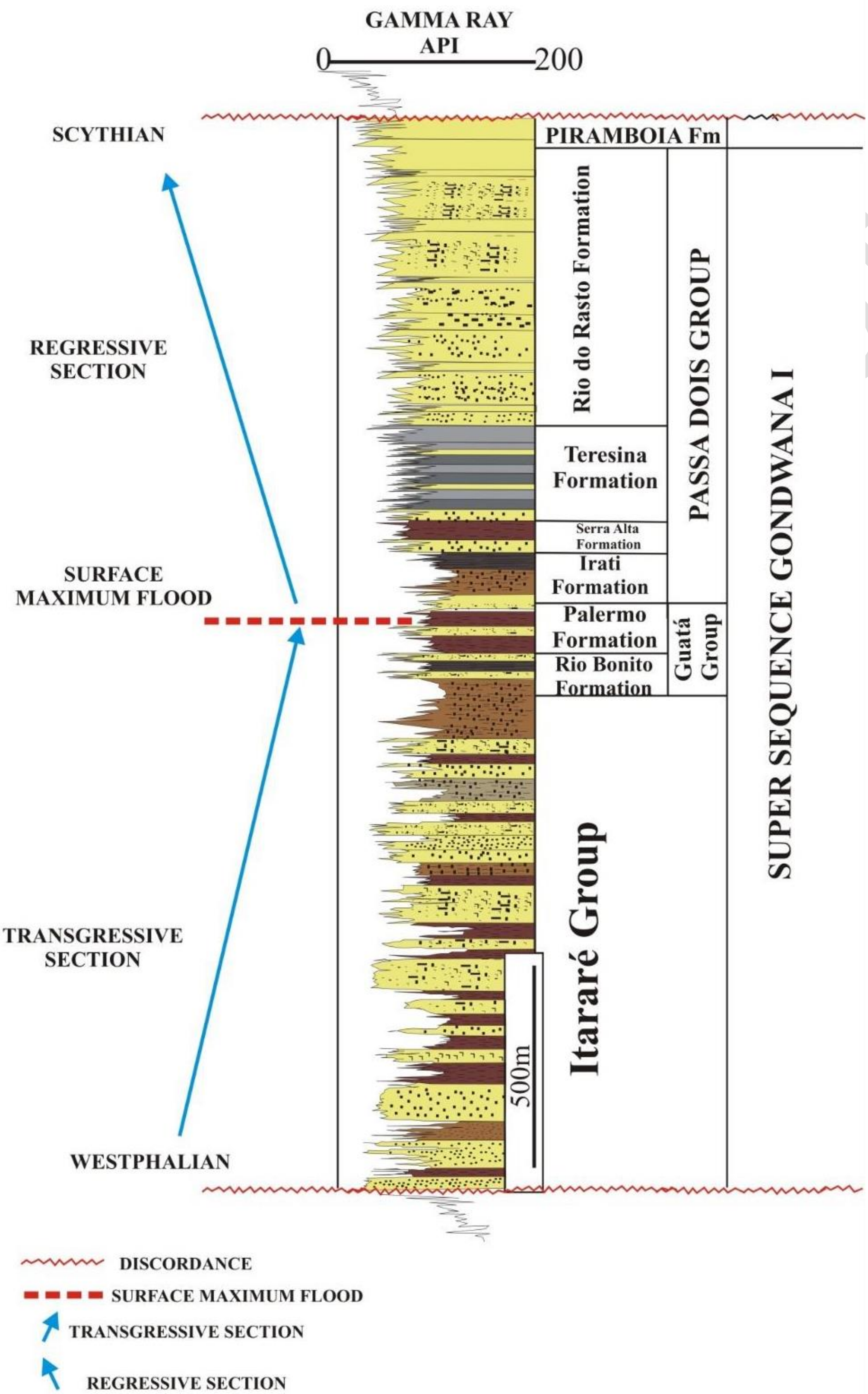

Fig. 1. The stratigraphic lithological pattern of the tectonic sequence Gondwana I, based on the Gama Ray well log (modified from Euzébio et al., 2016).

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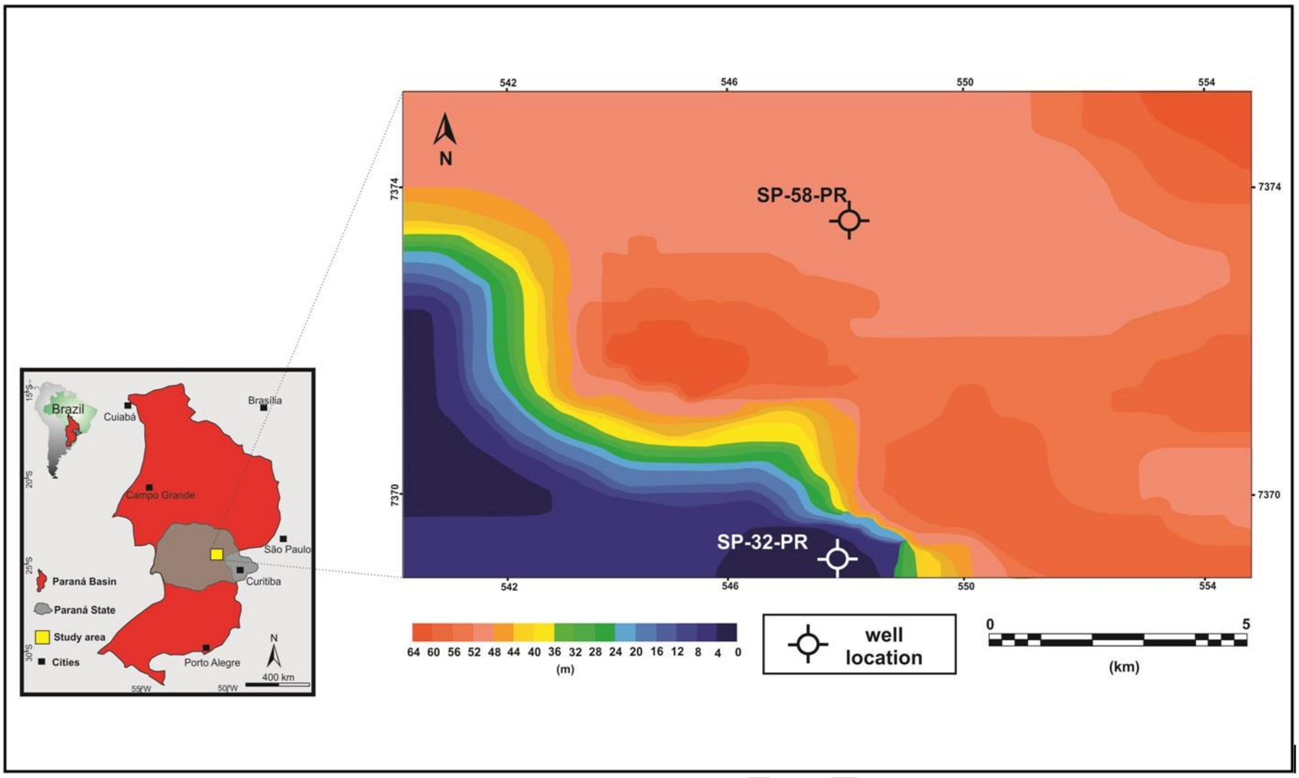

Fig. 2. Location of the study area in Paraná Basin. Isopach map of Serra Geral Formation in the study area, in Sapopema region (PR), southern Brazil. Well SP-58-PR used in the current study and SP-32-PR studied by Holanda et al. (2018).

The EDS microanalyses were obtained with the OXFORD Inca-AZtec Microanalysis System, coupled to the SEM, which provide the compositional (semiquantitative) tables of the chemical elements identified as oxides (calculated stoichiometrically). The EDS detector does not detect the elements H, He, Li and Be.

4. Results

Based on the interplanar distances (d) of the diffractometric reflections (Tab. 1), the following minerals were identified in the whole rock analysis for well SP-58-PR: quartz, albite, muscovite, calcite, dolomite, talc, pyrophyllite and pyrite (Fig. 3).

Tab. 1. Interplanar distance of the minerals interpreted in the current work.

Whole Rock	Primary peak (d)	Secondary peaks (d)
Calcite	3.03 Å	2.09 Å and 2.28 Å
Dolomite	2.88 Å	1.78 Å and 2.19 Å
Muscovite	3.32 Å	9.95 Å and 2.57 Å
Pyrite	1.63 Å	2.70 Å and 2.42 Å
Pyrophyllite	9.2 Å	4.6 Å and 3.07 Å
Albite	3.17Å	3.21 Å and 3.75 Å
Quartz	4.26Å	3.34Å and 1.82 Å
Talc	9.35 Å	1.53 Å and 4.59 Å

The diffractometric reflections interpreted under different conditions (samples without treatment, samples solvated with ethylene glycol for 12h and samples heated to 510ºC for 1h) indicated the following groups of clay minerals: illite, smectite, chlorite and kaolinite (Tab. 2). The occurrence of kaolinite was limited to the Taquaral Member, along with illite and chlorite, while in Assistência Member, the diffractometric peaks related to illite and smectite were most remarkable (Fig. 3).

Tab. 2. Primary peak of clay minerals under normal conditions (N), solvated with ethylene glycol (G) and heated (H). Modified from Neves (1968) and Albers et al. (2002).

Clay minerals	d (Å) - N	d (Å) - G	d (Å) - H
Illite	10 Å	10 Å	10 Å
Kaolinite	7Å	7Å	--
Chlorite	14 Å	14 Å	14 Å
Smectite	14 Å	17 Å	10 Å

Figure 4A shows the general appearance of the sample closest to contact with overlying diabase (approximately 40 cm from it), with polygons showing the different areas in which chemical analyses were performed with EDS (Table 3) to help identify minerals.

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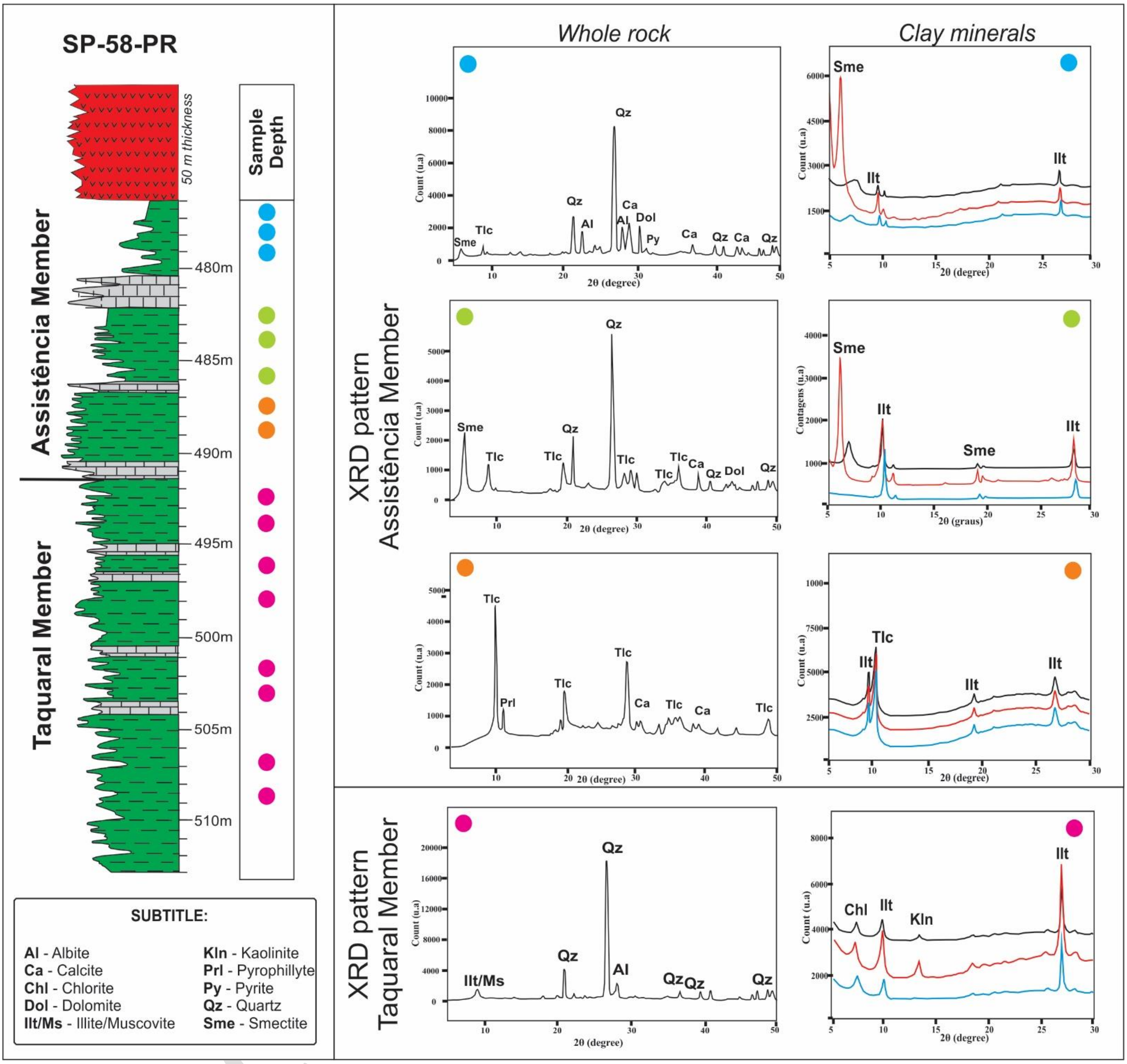

Fig. 3. Whole rock and clay minerals XRD pattern from Taquaral and Assistência Members, in well SP-58-PR.

Tab. 3. Chemical composition, in oxides, of the polygons signed in Figure 2.

Polygon	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	TiO_2 %	FeO %	Total %
1				100						100
2	9.57		19.79	68.52		0.62	0.57		0.93	100
3				99.34					0.66	100
4	9.59		19.3	65.71	2.88	0.53			1.98	100
5				100						100
6				100						100

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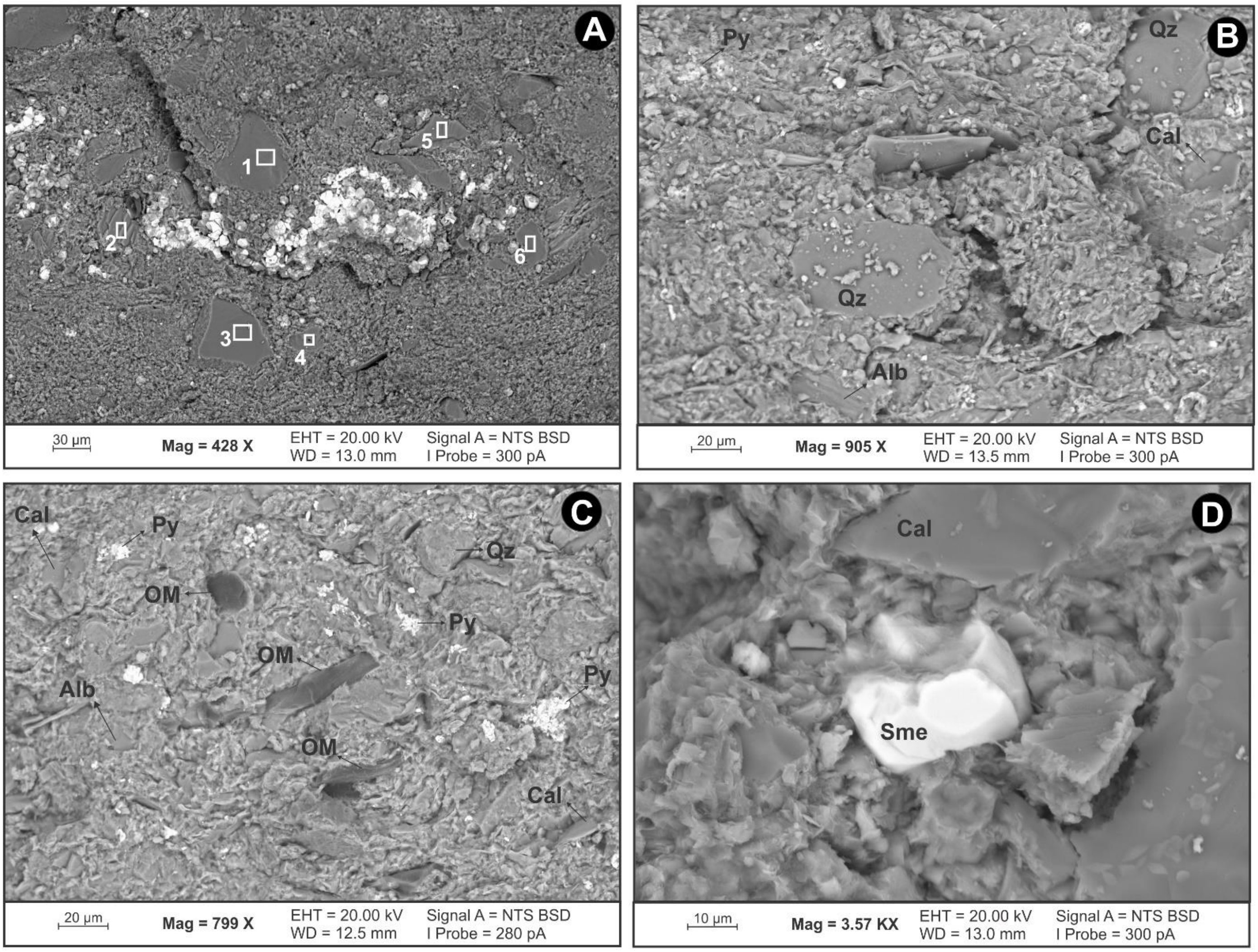

Fig. 4. SEM photomicrographs of sediment samples of the Irati Formation in well SP-58-PR, showing: (A) a general view of a sample of the Assistência Member; (B) quartz (Qz), albite (Alb), calcite (Cal) and pyrite (Py) grains; (C) quartz (Qz), albite (Alb), calcite (Cal), pyrite (Py) grains and organic matter (OM); (D) smectite (Sme) agglutinated flocs.

The correlation between Figure 4 and Table 3 supports the illation that the areas represented by the polygons 1, 3, 5 and 6 represent anhedral quartz crystals, which stand out from the matrix due to their larger size (generally larger than 40 µm) and because their edges evidence recrystallization. The areas represented by polygon 2 and 4 are plagioclase crystals, which, due to their high NaO content, should be albite, and also stand out in the rock matrix. The area represented by polygon 7 exhibited a more complex chemical composition, compatible with biotite.

Figures 4B and 4C are 2.0 and 3.8 meters far from the contact with intrusive rocks, respectively. The mineralogy in these intervals is characterized by the presence of quartz, albite, calcite and pyrite. SEM/EDS analyses revealed the occurrence of clay minerals of the smectite group that occur mainly as agglutinated anhedral flocs approximately 2 µm in size (Fig. 4D).

Based on the MEV/EDS analysis (Fig. 5 and Tab. 4) the areas represented by polygons 1, 2, 3, 4, 6, 7, 8 and 10 represent the mineral talc, which seems to be responsible for the sample crenulated texture when observed at micrometric scale (Figs. 5A and 5B). The areas represented by polygons 5, 9, 11 and 12 represent large crystal of calcite (5B), with rhombohedral (5C) and tuberose (5D) habits.

5. Discussion

In this study, we have investigated the process of heat transfer from igneous (ca. 50 m thickness) to host-rock based on the mineralogical content pelitic rocks. Holanda et al. (2018) studied the mineralogical content of the Irati Formation in the Sapopema region, well SP-32-PR (Fig. 1), where the Irati Formation is not in contact with the igneous rock. Table 5 correlates the mineralogical content interpreted in both wells based on XRD patterns.

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Apparently, the heat transfer was not enough to cause modifications in the mineralogical assemblage of the Taquaral Member (quartz + albite + muscovite + illite + kaolinite + chlorite). However, the mineralogical content from Assistência Member presented changes probably caused by the intrusion of the diabase sill.

Through SEM/EDS analyses, crystals with chemical composition compatible with calcite and talc were observed. Dos Anjos and Guimarães (2008) proposed a chemical reaction (1) that would justify the presence of talc and calcite from dolomite and quartz in the northern region of the Paraná Basin:

$$3CaMg(CO_3)_2 + 2SiO_2 + 2H_2O \rightarrow Mg_3Si_2O_5(OH)_4 + 3CaCO_3 + 3CO_2\uparrow \text{.........................} (1)$$

Dos Anjos et al. (2010) described talc crystals as a metamorphic mineral according to their coherent scattering domain size values, relative quantities and texture (northern region of the basin). Besides that, the authors noted that metamorphic minerals were parallel to the bedding in the unmetamorphosed rocks but become major components in some layers near the sill where they are randomly oriented with respect to the bedding.

In the Perolândia quarry (northern region of the basin), a 13-m-thick basic sill intrudes black shales and limestones of the Irati Formation. A detailed investigation across the contact between the igneous and the sedimentary rocks revealed that near the contact with the sill, talc and serpentine formed in the limestone, while magnesium-bearing minerals such as pyroxene formed in the black shales. In the same region, Santos et al. (2009) modeled the variation of temperature across the contact zone using a one-dimensional transfer model, concluding that temperature of the sedimentary rocks reached 500°C in the vicinity of the contact and then decreased to 200°C at a distance of 18 m from the contact.

Based in thermodynamic model of Berman (1988) and Brady et al. (1998), we propose that stability of the assemblage talc + calcite is limited to temperatures below ≈450°C and pressure lower than 6 kbar (Fig. 6). Fig. 3 illustrates the presence of this mineral assemblage along 16 m of the Assistência Member thickness.

The analyses performed by XRD indicated diffractometric reflections related to pyrophyllite. Frey (1987) described pyrophyllite in the Helvetic nappes and the Switzerland Prealps. It has been discovered mainly in Jurassic black shale and in Jurassic and Eocene laterites. The mineral distribution pattern strongly suggests that kaolinite + quartz (Fig. 6) provides the starting material for the appearance of pyrophyllite by virtue of the reaction (2):

$$Al_2Si_2O_5(OH)_4 + 2SiO_2 \rightarrow Al_2Si_4O_{10}(OH)_2 + H_2O \text{..........} (2)$$

Fluid inclusion studies by Mullis (1979) have documented methane-rich fluids at such very low metamorphic grade, derived from the cracking of organic material. Assuming constant temperature and fluid pressure, an assemblage containing kaolinite + quartz + organic matter could have reacted to yield pyrophyllite. According to Euzébio et al. (2016) and Holanda et al. (2018), TOC ranges between 0.25 and 2.11% for the Assistência Member in SP-58-PR.

Tab. 4. Chemical composition, in oxides, of the polygons in Figure 3.

Polygon	Na_2O %	MgO %	Al_2O_3 %	SiO_2 %	SO_3 %	K_2O %	CaO %	FeO %	Total %
1	1.11	30.44	2.95	62.11	1		2.39		100
2	1.14	31.09	2.84	61.24			2.38	1.31	100
3		26.72	3.42	61.97			2.9	4.98	100
4	1.13	27.61	3.4	64.83			1.15	1.88	100
5							100		100
6	0.58	15.34	0.92	79.58			1.29	1.32	100
7	0.85	25.16	210	69.59			0.97	1.34	100
8	0.73	25.5	1.67	69.52			1.16	1.42	100
9	1.08	1.57		3.08			94.27		100
10	2.71	31.22	6.21	56.54			1.72	1.62	100
11							100		100
12				0.95			99.05		100

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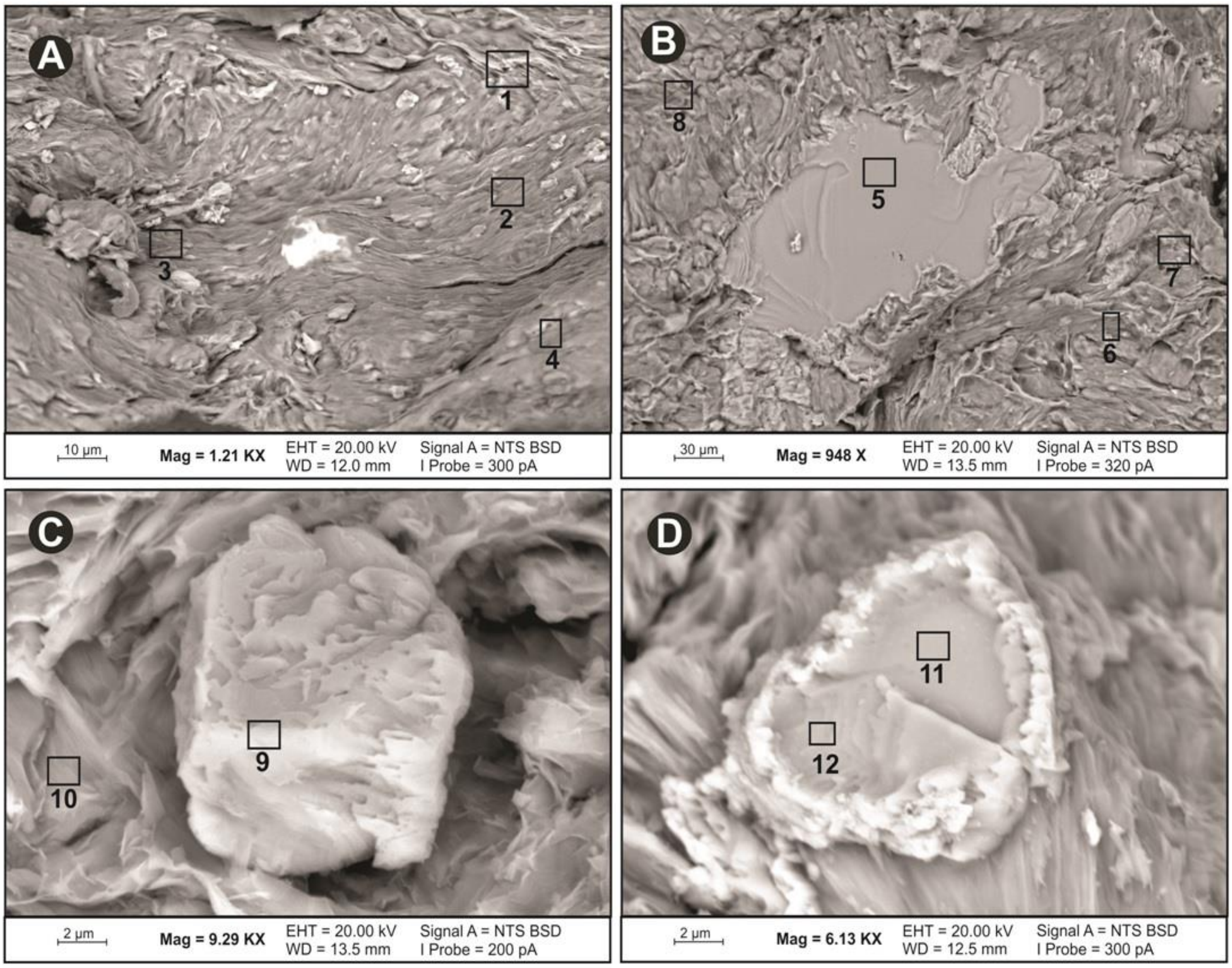

Fig. 5. SEM photomicrographs of the sample at 486.0 m of the Irati Formation in well SP-58-PR, showing: (A) crenulated texture of the rock, (B) crystal of calcite with rhombohedral (C) and tuberose (D) habits.

Tab. 5. Comparison of the mineralogical content of Irati Formation in the Sapopema region.

Lithostratigraphy	SP-58-PR (with thermal influence from sill)		SP-32-PR (without thermal influence from sill)	
	Whole rock	Clay Minerals	Whole rock	Clay Minerals
Assistência	Quartz	Illite	Quartz	Illite
	Albite	Smectite	Albite	Smectite
	Muscovite	Talc	Muscovite	Kaolinite
	Pyrite	Pyrophyllite	Pyrite	-
	-	-	Analcime	-
	-	-	Gypsite	-
	Dolomite	-	Dolomite	-
	Calcite	-	Calcite	-
Taquaral	Quartz	Illite	Quartz	Illite
	Albite	Kaolinite	Albite	Kaolinite
	Muscovite	Chlorite	Muscovite	Chlorite

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Alves and Rodrigues (1985) studied the effect of igneous intrusions on the mineralogy of the Devonian shale of the Amazonas Basin. Based on Winkler (1976), the authors proposed that pyrophyllite formation occurred from kaolinite + quartz reaction at temperature below 400°C.

The XRD and SEM analyses pointed to the presence of clay minerals of the smectite and illite groups in Assistência Member, while in Taquaral Member, besides illite, diffractometric reflections consistent with chlorite and kaolinite are observed. The relation between the thermal evolution of pelitic rocks and the occurrence of different types of clay minerals was the subject of several prior research works (Winkler, 1976; Frey, 1987; Weaver, 1989; Meunier, 2005).

There is a mineralogical order that reflects thermal evolution conditions ranging from smectite to mixed-layer smectite/illite to illite, where the smectite is associated with lower temperatures and the illite with higher temperatures. Despite the thermal halo generated by the contact with the overlying diabase, it is not possible to discern an ordering of the clay minerals in well SP-58-PR. The reaction of smectite to illite has been used as an empirical geothermometer or thermal maturity indicator in a wide range of geologic environments (e.g., Hoffman and Hower, 1979).

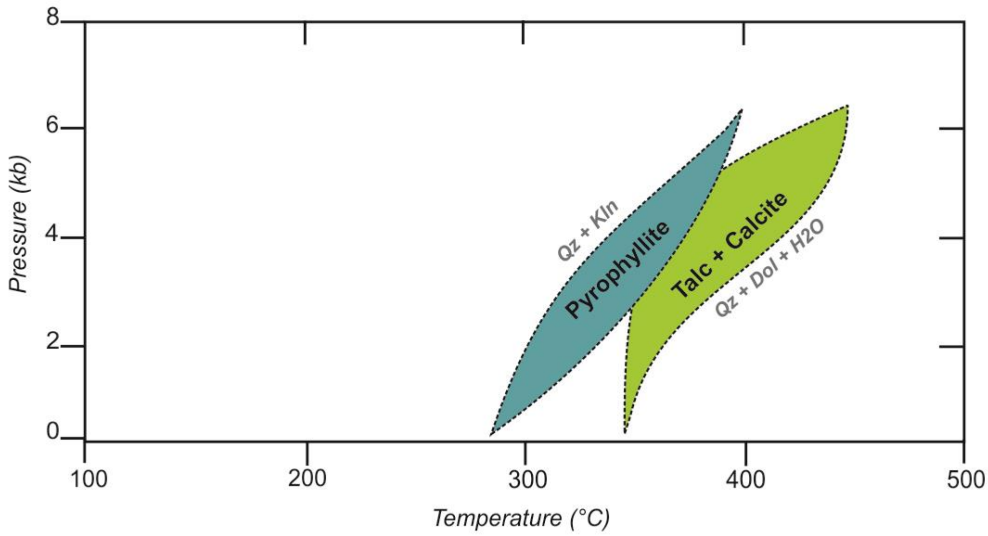

Fig. 6. Thermodynamic equilibrium curve of the pyrophyllite (Winkler, 1976) and talc (Brady et al., 1998).

Some studies have demonstrated that the illitization reaction is more complex than previously thought and that several chemical and physical variables may affect the rate of reaction (Inoue et al., 1988; Whitney and Northrop, 1988). Although a sixth-order kinetic expression effectively models of the overall reaction, the reaction undoubtedly represents a series of poorly understood processes which together constitute the total reaction (Pytte and Reynolds, 1989).

The results predicted by most published heat transfer models of igneous intrusions can match well with some geothermometers such as vitrinite reflectance and fluid inclusions. This partly attests the reliability of those models for reconstructing the thermal evolution of host rocks (Galushkin, 1997; Wang et al., 2010, 2011). Therefore, based on the study of chemical reactions and mineral modifications it is very difficult to stablish the overall heat transfer model of igneous intrusions within sedimentary layers. For that other analytical techniques besides mathematical models to get a more accurate result are required.

6. Conclusion

The investigation based on XRD and SEM/EDS analyses indicates that the mineralogical changes in pelitic rocks of Irati Formation were induced by thermal effects of diabase sill. However, these changes are restricted to the Assistência Member.

Talc and calcite were formed due to the reaction between dolomite and quartz, limited to temperatures below about 450°C. This assemblage was found along 16 m of Assistência Member thickness. However, pyrophyllite can be considered as product of the reaction between kaolinite and quartz limited to temperatures below 400°C. XRD pattern of these samples were restricted to samples farthest from the diabase sill.

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