

MAGNETIC SEPARATION AS A METHOD TO ASSIST MINERALOGICAL CHARACTERIZATION OF ROCKS BY X-RAY DIFFRACTION

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ABSTRACT

The X-ray diffraction (XRD) corresponds to one of the main techniques for characterization of structures in crystalline materials widely used in the identification of minerals in samples of geological materials such as rocks. However, the large number of mineral phases present in a rock sample can generate excess peaks in the diffractogram, and it can promote overlapping peaks and induce erroneous identification. The purpose of this study was to perform magnetic separation of minerals from rock samples in order to enable the identification of the minerals by XRD. For this magnetic separation, two samples of rock were selected: a sample of high silica content and a sample with low silica content. The magnetic separation of minerals from each sample was performed using the magnetic separator isodinamic Frantz. Posteriorly, the fractions obtained in magnetic separations were analyzed by XRD. In the sample with high silica content, it was obtained a fraction where was identified the accessory mineral epidote, which had not been identified in the total sample diffractogram. In the sample with low silica content, the magnetic separation into several mineral fractions made possible to obtain diffraction patterns with fewer peaks and peaks with higher relative intensities, which allowed its mineralogical characterization. The results showed that the mineral separation by the magnetic separator Frantz made the identification of accessory minerals by XRD and the characterization of samples which have many mineral phases possible, which proves that magnetic separation by Frantz is a method which can assist analyses by XRD.

1. INTRODUCTION

The X-ray diffraction (XRD) corresponds to one of the main techniques for characterization of structures in crystalline materials widely used in the identification of minerals in samples of geological materials, such as ores and rocks.

The phenomenon of X-ray diffraction results from a process in which X-rays are scattered by electrons of atoms without changing the wavelength [1], whenever certain geometric conditions are satisfied.

Upon reaching a material, X-rays can be scattered without loss of energy by the electrons of the atoms (coherent scattering or Bragg scattering). After collision with the electrons of the sample, the X-ray beam changes its propagation direction keeping, however, the same wavelength of incident radiation [2,3].

When such dispersions are generated by electrons of a set of atoms arranged in a systematic way as in a crystalline structure, it can be seen that the phase relations between the scatterings become periodic and that the phenomenon of X-ray diffraction is observed at various angles of incidence of the beam, since its wavelength λ is of the order of magnitude of the interplanar distances [4,5].

If it is considered two or more planes of a crystalline structure, the conditions for the occurrence of X-ray diffraction will depend on the difference of the path of the X-ray beam and the wavelength λ of the incident radiation. This condition is expressed by Bragg's law, $n\lambda = 2d \sin\theta$, in which λ corresponds to the wavelength of the incident radiation, n to an integer, d to the interplanar distance for a set of planes $\{hkl\}$ (Miller index) of the crystal structure and θ to the incident angle of X-rays (measured between the incident beam and the crystal planes) [5,6].

Although XRD is a technique for characterization of monophasic materials (single substance), multiphasic materials are often required to be analyzed by XRD. However, it is observed that the higher the number of phases of a multiphasic material, more difficult is the interpretation of the diffractograms. Examples of these multiphasic materials are the rocks.

A rock corresponds to a solid aggregate of natural occurrence, which consists, generally, of various mineral phases [7]. The X-ray diffraction has limitations in the analyses of rocks which are composed of several minerals. The large number of mineral phases present in a rock sample can generate excess peaks in the diffractogram, and it can promote overlapping peaks and induce erroneous identifications. It can also be more difficult to identify accessory minerals in a rock, which are present in rocks in amounts lower than 5% [7].

For this reason, mineral separations are required to facilitate the mineralogical characterization of rock samples. A very used method of mineral separation is magnetic separation.

In a study by Silva Junior *et al.* (2008), a rock sample was submitted to magnetic separation with the main purpose to separate epidote. The mineral fractions obtained were analyzed by XRD, where it was revealed that the diffraction pattern of the fraction of epidote showed great similarity to the diffraction pattern of a sample of monocrySTALLINE epidote. This result showed that the magnetic separation can be employed to help the mineralogical characterization of rocks by XRD [8].

The magnetic separation based on magnetic susceptibility of minerals. Magnetic susceptibility is the property of a material that determines their response to a magnetic field. Based on this property the minerals are classified into three categories:

- Ferromagnetic: strongly attracted by the magnetic field;
- Paramagnetic: weakly attracted by the magnetic field;
- Diamagnetic: show weak repulsion magnetic fields of external nature [9].

The instrument used for the separations is the isodynamic magnetic separator Frantz. This instrument consists in a vibratory chute with variable slopes in vertical and horizontal directions. This chute is located between two electromagnets of adjustable current, which define the flux density of the magnetic field. The separation of minerals is primarily a function of magnetic field, however factors as side and forward slope of the chute, the chute vibration intensity (flow rate of particles) and the amount of material fed into the hopper influence the separations. The magnetic separation of minerals is performed by applying various intensities of electric current to Frantz, thereby generating different values of magnetic field to attract minerals. Literature presents tabulated values of electric current and their minerals [10].

The purpose of this study was to perform magnetic separation of minerals from rock samples in order to enable the identification of unidentified accessory minerals by X-ray diffraction and mineralogical characterization of samples which diffractograms show an excessive amount of peaks. The samples analyzed for this work are metamorphic rocks from Itapé and Itororó Cities, located in the South and Southwest of Bahia State respectively.

2. MATERIALS AND METHODS

2.1. Magnetic Separation of Minerals

Two rock samples were selected for magnetic separation: a sample with high silica content (ITO-03) and another with low silica content (ITP-04), which were analyzed by XRD before the magnetic separation of the minerals (total samples) and after that separation.

For the analysis of total samples, they were ground in a ring mill for 1.45 minutes, yielding particles of 200 mesh. For magnetic separation by Frantz, the samples were ground for 15 seconds, yielding particles from 40 to 60 mesh.

Initially, a previous separation of ferromagnetic minerals (mainly magnetite) was performed using a hand magnet. After this step, the separations were performed using the Frantz isodynamic magnetic separator.

The samples were introduced in Frantz only once to each value of electric current. Separation settings for the selected samples were defined by previous separations performed with the samples themselves, observing the conditions that favored more efficient separations visually for each sample. Such settings are shown in Table 1.

Table 1: Settings for Frantz magnetic separation of the samples

Side Slope		Electric Current (A)
Sample ITO-03 (High silica content)	Sample ITP-04 (Low silica content)	
5°	10°	0.1
		0.3
		0.5
		0.7
	5°	1.0
		1.3
3°	5°	1.7
1°		
0°		

Note: forward slope fixed at 7°.

2.2. Analysis by X-ray diffraction

The equipment used to perform the analyses was the PANalytical X'Pert PRO diffractometer with configuration θ - θ . The samples were irradiated by an X-ray tube with copper anode which emitted $K\alpha$ radiation of wavelength 1.5418\AA and operated at 45mA and 40kV. The radiation beam diffracted by the sample was collected by a proportional detector [11], where there was the formation of electrical pulses that were sent to a computer for processing and data storage. Scans of the samples were performed in a time step of the tube-detector system of 0.5 seconds and the angle step size equal to $0.02^\circ 2\theta$, which generated analyses that lasted 35 minutes each test.

The samples characterization was done from the High Score Plus 2.0 (software from PANalytical) by comparing the generated diffractograms with reference standards organized into sheets available in the database PDF2 of the ICDD - *International Center for Diffraction Data*, which maintains crystallographic information such as the interplanar distances. In this procedure, the software performed a search for the most intense peaks of each crystalline compound which was kept in the database and compared them with the peaks of the sample diffractogram. After the process of searching and comparison, the results values were expressed in increasing order of rank. The coincidence of values or a major proximity of values led to the identification of the mineral.

The magnetic separation of minerals and the analyses of X-ray diffraction were performed at magnetic separation laboratory and X-ray laboratory respectively, both located at the Centro de Pesquisa Professor Manoel Teixeira da Costa, Instituto de Geociências, Universidade Federal de Minas Gerais - CPMTC-IGC-UFMG.

3. RESULTS AND DISCUSSION

3.1. Sample with high silica content

The mineralogical characterization of the total sample ITO-03, as expected, showed only the major mineral constituents of the sampled rock, as it can be seen in Figure 1.

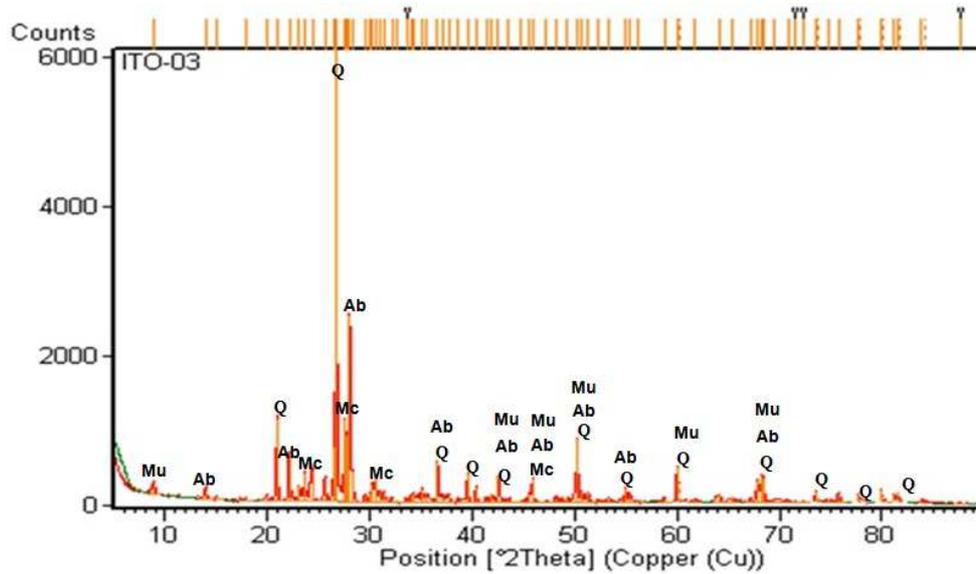


Figure 1: Diffractogram presenting the main mineral constituents of the sampled rock: quartz (Q), albite (Ab), microcline (Mc) and muscovite (Mu).

In Frantz magnetic separation, all obtained fractions were characterized by XRD and only in the fraction obtained at 0.3 A of electrical current an accessory mineral was identified, the epidote (Figure 2).

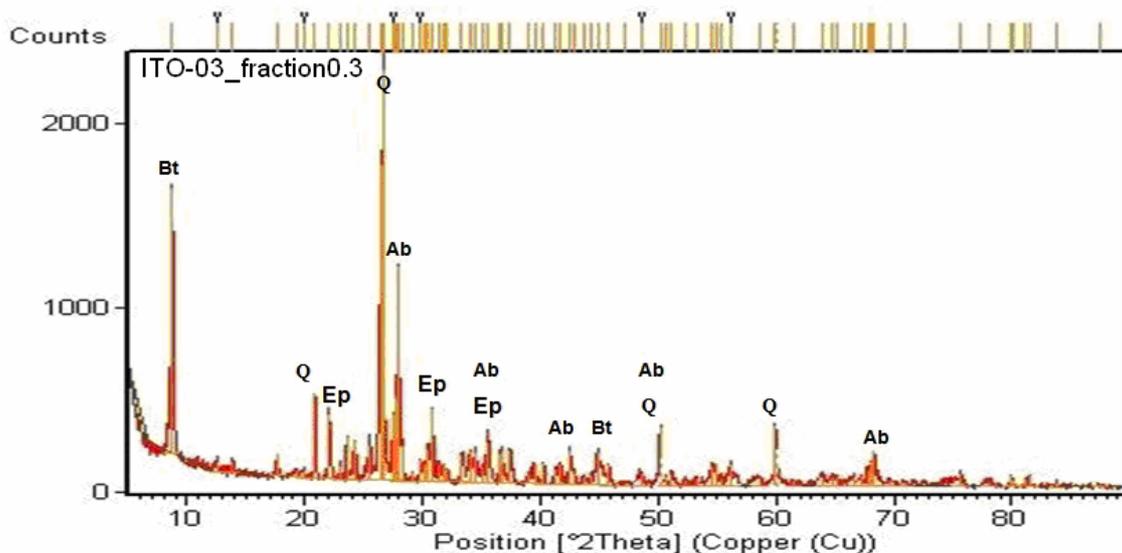


Figure 2: Mineralogical characterization of the fraction obtained at 0.3 A of current, which presents accessory mineral epidote (Ep), in addition to the mineral biotite (Bt), quartz (Q) and albite (Ab).

The other fractions showed the same minerals characterized in the total sample, as shown in Figure 3.

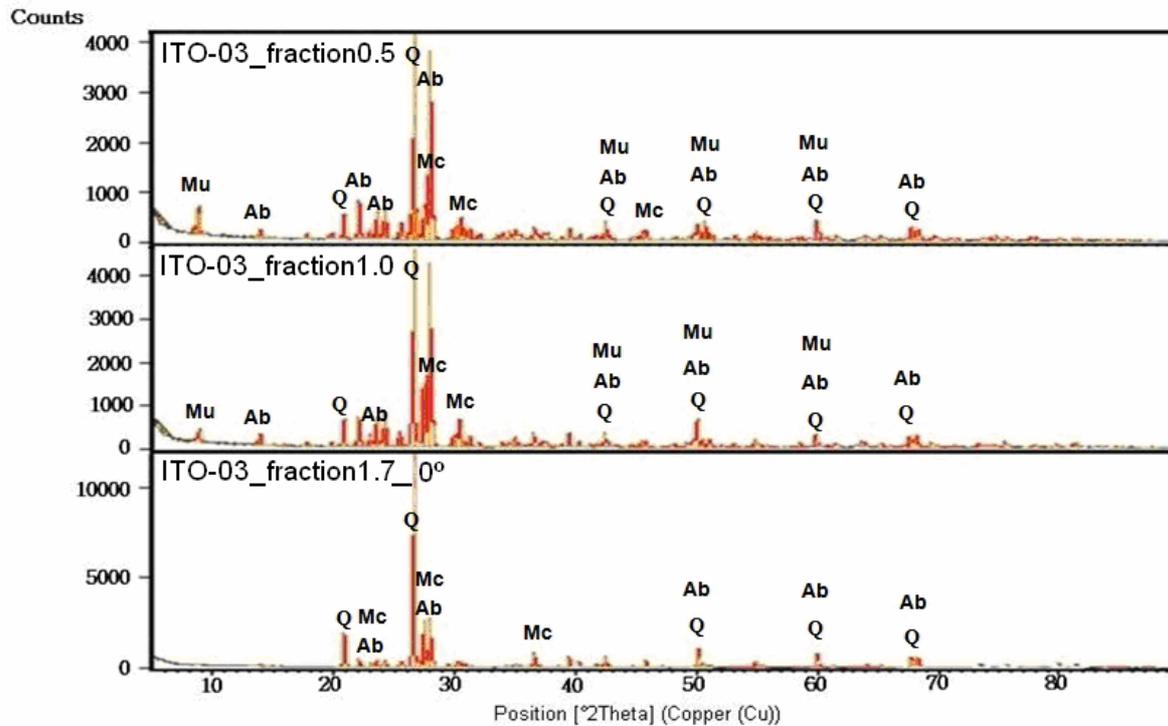


Figure 3: Diffractograms of the fractions 0.5, 1.0 and 1.7 A of the sample showing the minerals present in the total sample: quartz (Q), albite (Ab), microcline (Mc) and muscovite (Mu).

As it can be seen in the diffractograms of the sample ITO-03, Frantz magnetic separator was not able to separate the minerals in the sample, but concentrating them. The inability to mineral separation can be attributed to many factors, such as: side and forward slope of the chute, chute vibration intensity (flow velocity of the particles), vibration intensity of the feed hopper, the particle size and range of current intensities selected. Even though, it was possible to identify an accessory mineral (the epidote) in one of the fractions.

3.2. Sample with low silica content

In the case of sample ITP-04, it was not possible to perform the mineralogical characterization of the sample, because the diffractogram of ITP-04 showed an excessive number of peaks (Figure 4). Moreover, such excess peaks favored the indication of minerals unlikely to the constitution of the samples by the database ICDD PDF2, as it can be seen in Figure 5.

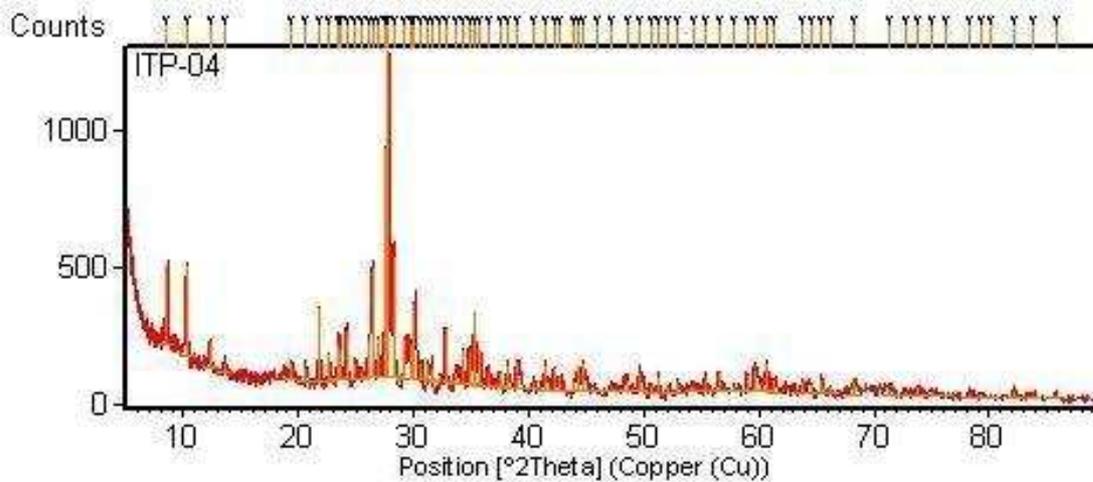


Figure 4: Diffractogram of the sample ITP-04 with excessive number of peaks

No.	Ref. Code	S.	Compound Name	Chemical Formula
1	00-023-0118	45	Ferroactinolite	Ca ₂ Fe ₅ Si ₈ O ₂₂ (OH) ₂
2	00-038-0448	38	Opal	SiO ₂ · xH ₂ O
3	00-031-0615	38	Sekaninaite, syn	Fe ₂ Al ₄ Si ₅ O ₁₈
4	00-017-0746	38	Pyrochlore	(Ca, Na) ₂ (Nb, Ti) ₂ O ₆ F
5	00-017-0525	38	Sekaninaite, syn	Fe ₂ Al ₄ Si ₅ O ₁₈
6	00-026-1373	36	Ferrichterite, syn	Na ₂ CaFe ₅ Si ₈ O ₂₂ (OH) ₂
7	00-014-0466	36	Boron phlogopite, syn	KMg ₃ B ₃ Si ₃ O ₁₀ (OH) ₂
8	00-031-0616	35	Sekaninaite	Fe ₂ Al ₄ Si ₅ O ₁₈
9	00-018-1202	33	Anorthite, sodian, intermed.	(Ca, Na)(Si, Al) ₄ O ₈
10	00-010-0361	32	Anorthoclase, syn	Na _{0.71} K _{0.29} AlSi ₃ O ₈
11	00-029-1258	32	Ferrohornblende, sodian	(Na, K)Ca ₂ (Fe, Mg) ₅ (Al, Si)...
12	00-040-0496	30	Esseneite	CaFeAlSiO ₆
13	00-042-1432	29	Efremovite	(NH ₄) ₂ Mg ₂ (SO ₄) ₃
14	00-020-0528	29	Anorthite, sodian, ordered	(Ca, Na)(Al, Si) ₂ Si ₂ O ₈
15	00-029-0865	28	Kanoite	(Mn, Mg) ₂ (Si ₂ O ₆)

Figure 5: Indicated minerals to the characterization of the total sample. Detail for the anorthite plagioclase as the ninth indicated candidate.

It was expected that the sample would present predominantly plagioclase in its constitution, based on results of mineralogical characterization of other samples from the same region. However, the indication of anorthite (mineral of the plagioclase series, therefore, likely to characterization) in the ninth place in database created doubts in relation to their choice.

Because of the uncertainty in the interpretation of the diffractogram, the sample was subjected to magnetic separation to assist the mineralogical characterization of the total

sample. Figure 6 shows the mineralogical characterizations of some of the obtained fractions in Frantz.

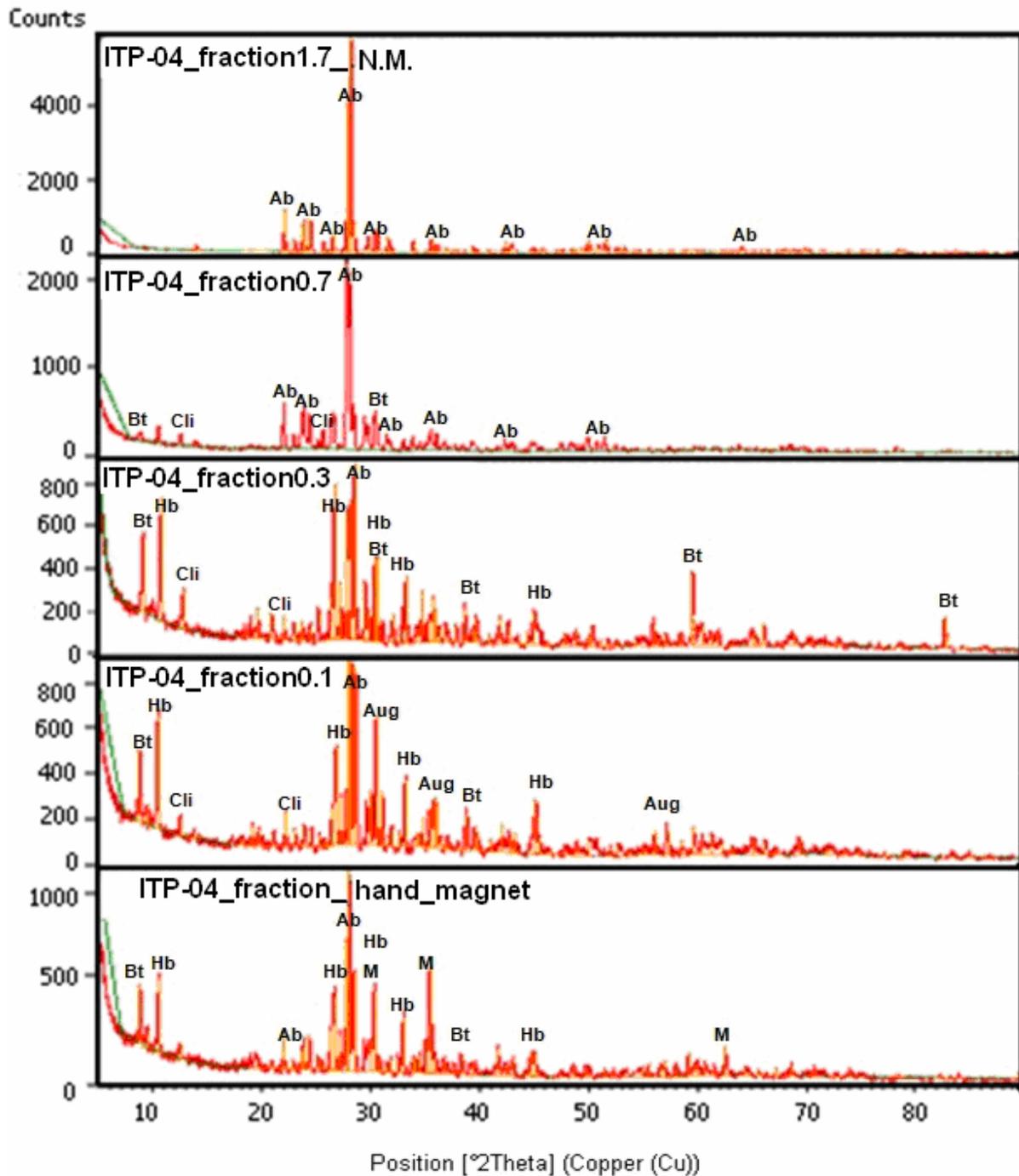


Figure 6: Diffractograms of the following fractions: 1.7 nonmagnetic (NM), 0.7, 0.3 and 0.1 A obtained in Frantz and by hand magnet. Diffractograms showing albite (Ab), biotite (Bt), clinocllore (Cli), magnesium-hornblende (Hb), augite (Aug) and muscovite (M).

The diffractograms of the fractions obtained by Frantz showed smaller number of peaks, better defined peaks and peaks with higher relative intensities, which favored the interpretation of the spectra and thus the mineralogical characterization.

As it can be seen in Figure 6, the fraction of 1.7 N. M., albite was identified as the constituent plagioclase rock, unlikely what occurred in the total sample analysis, in which the database suggested anorthite as likely plagioclase.

4. CONCLUSIONS

In the present work it was possible to prove that XRD is not the most appropriate method for the analysis of materials that have many phases to be identified - such as rocks - because the large number of mineral phases present in a rock sample generates excess peaks in the diffractogram, which can overlap them and induce erroneous identifications. However, some methods of mineral separation – as magnetic separation – can make the mineralogical characterization of rocks by XRD possible.

Although the Frantz magnetic separator was not able to completely separate the minerals in the sample, the results showed that the magnetic separation made the identification of accessory minerals by XRD possible and also the characterization of samples which have many mineral phases, which proves that magnetic separation by Frantz is a method which can assist analyses of rocks by XRD.

To facilitate the interpretation of diffractogram of rocks and thus obtain more accurate results of mineralogical characterization by XRD, a more efficient mineral separation is needed. For this, it should optimize the Frantz magnetic separator according to the minerals to be separated, besides using other methods of mineral separation, as separation by density. A larger number of samples to be analyzed it is also needed to obtain more conclusive results.

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