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**Centro de Tecnologia Mineral (Center for Mineral Technology)**  
**Brazil's Ministry of Science, Technology, and Innovations**  
**Mineral Analysis Division - DIAMI**  
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## **Mineralogical and Technological Characterization of Clay Samples**

Final Report

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## 1. Introduction

This technical report aims to show the results of the mineralogical characterization study in connection with the project called "Mineralogical and Technological Characterization of Clay Samples".

## 2. Sample receipt and preparation

Five (05) clay samples were received at CETEM, sent by Mr. Thiago Almeida, for a mineralogical analysis (Figure 1). Initially, the samples were dried in an oven at 50 °C for a period of approximately two (2) days (Figure 2).

*[Figure]*

**Figure 1. Clay samples received at CETEM.**

*[Figure]*

**Figure 2. Sample clays prepared for drying in the oven.**

Then, the samples were disaggregated, homogenized, and quartered to take out 1-kg aliquots for the further mineralogical characterization tests (Figure 3).

*[Figure]*

**Figure 3. Samples T10-P03 and T18-P05 used in the study.**

Following the homogenization stage, the samples were ground dry in the grinding mills (Figure 4). Afterwards, the samples were sorted wet through vibrating screens (Figure 5).

*[Figure]*

**Figure 4. Grinding mills used to grind the clay samples.**

*[Figure]*

**Figure 5. Wet grain size sorting of the samples.**

## 3. Materials and Methods for the Mineralogical Characterization

The five samples were characterized using X-ray diffraction analysis (XRD), scanning electron microscopy (SEM), and a binocular magnifier.

The X-ray diffraction patterns of the samples were obtained on a Bruker-D8 Endeavor under the following operating conditions: CuK $\alpha$  radiation (40 kV/40 mA), goniometer speed at 0.02° 2 $\theta$  per step with a counting time of 0.5 seconds per step and collected from 4 to 80° 2 $\theta$ , with a LynxEye position-sensitive detector. The qualitative interpretations were carried out based on a comparison to patterns contained in the database PDF02 (ICDD, 2006) using the software Bruker DiffracPlus.

The thicker fractions were described using a binocular magnifier aiming to identify the mineral distribution by grain size ranges and degree of release of the mineral of interest.

Mineral identification, composition, and the textural relations of the +100#, +270#, and -325# fractions were determined on the scanning electron microscope (SEM) Hitachi Model TM3030 Plus equipped with an energy-dispersive chemical microanalysis system (EDS) Bruker Quantax. The samples were

metalized with silver and analyzed in the secondary electron modules, back-scattered detector, and EDS.

The -325# fractions of the samples were subjected to 2- $\mu$ m suspension sorting to separate the clay fraction (Moore and Reynolds Jr., 1989). In this process, a 100-mL test tube was used. The sample quantity was 5 g. Sodium hexametaphosphate was used as a dispersion agent for the ratios of 3 mg/g of sample, and an ultrasonic processor was used to disperse the clay minerals under the conditions of 50 amplitude for 1 minute. In addition to the method proposed by Moore and Reynolds Jr. (1989), the ultrasound method was also used. This stage showed to be important, since it allowed the clay minerals to concentrate, which enabled better identification using X-ray diffraction.

#### 4. Results of the Mineralogical Characterization

Presented below are the results of the stages of mineralogical characterization of the clay samples in terms of X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM).

##### 4.1 Clay T10-P03

##### Grain Size Classification

The information on the grain size distribution of sample T10-P03, following its disaggregation in a grinding mill for 10 minutes, is shown in Table 1. The sample exhibits a very fine grain size, as around 99.50% of the material lies below 325#.

**Table 1. Grain size classification of sample T10-P03 following 10-minute grinding in the grinding mill.**

Project: Characterization Study					
Material: T10-P03					
Grain Size Fraction		Mass (g)	Mass (%)	Mass Retained (%)	Mass Passing (%)
Mesh	mm				
6 #	3.360	0.00	0.00	0.00	100.00
8 #	2.380	0.00	0.00	0.00	100.00
10 #	1.680	0.00	0.00	0.00	100.00
14 #	1.190	0.00	0.00	0.00	100.00
20 #	0.840	0.00	0.00	0.00	100.00
28 #	0.590	0.00	0.00	0.00	100.00
35 #	0.420	0.00	0.00	0.00	100.00
48 #	0.297	0.00	0.00	0.00	100.00
65 #	0.210	0.00	0.00	0.00	100.00
100 #	0.149	0.00	0.00	0.00	100.00
150 #	0.105	0.00	0.00	0.00	100.00
200 #	0.074	0.00	0.00	0.00	100.00
270 #	0.053	0.00	0.00	0.00	100.00
325 #	0.044	4.55	0.50	0.50	99.50
400 #	0.037	897.45	99.50	100.00	0.00
-400 #	-			-	-
Total	-	902.00	100.00	-	-

##### X-Ray Diffraction Analysis

Below are the results of the X-ray diffraction analysis of samples T10-P03 (Table 2, Figures 6 through 11). The raw fraction is made up of quartz, kaolinite, illite, and muscovite. The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction patterns. The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the clay fraction of the sample is made up of the clay minerals illite and kaolinite.

**Table 2. Mineral composition of the sample fractions.**

<b>Fraction</b>	<b>Mineral Composition</b>
<b>T10-P03 Raw</b>	Quartz, kaolinite, illite, and muscovite
<b>T10-P03 -325#</b>	Quartz, kaolinite, illite, and muscovite
<b>T10-P03 clay</b>	Illite and kaolinite

*[Figure]*

**Figure 6. X-ray diffraction pattern of sample T10-P03 Raw.**

*[Figure]*

**Figure 7. X-ray diffraction pattern of sample T10-P03 fraction -325#.**

*[Figure]*

**Figure 8. X-ray diffraction pattern of sample T10-P03 Natural Clay fraction.**

*[Figure]*

**Figure 9. X-ray diffraction pattern of sample T10-P03 Glycol-treated clay fraction.**

*[Figure]*

**Figure 10. X-ray diffraction pattern of sample T10-P03 Calcined clay fraction.**

*[Figure]*

**Figure 11. X-ray diffraction pattern of sample T10-P03 Natural, Glycol-Treated, and Heated.**

### Scanning Electron Microscopy

The fractions of sample T10-P03 were analyzed on the scanning electron microscope. The analyzed fractions are made up of the clay minerals illite, kaolinite, and quartz (Figures 12 through 15). Few grains of iron oxides/hydroxides and titanium oxide were identified. The illite occurs in the form of aggregates in all of the analyzed fractions, whereas the kaolinite occurs in the form of pseudo-hexagonal particles.

The results of the EDS analysis indicate that the samples are essentially composed of oxygen, silicon, aluminum, potassium, and secondarily of magnesium, iron, and titanium.

*[Figure]*

**Figure 12. Image of sample T10-P03 fraction +100#. Grains of quartz and clay minerals.**

*[Figure]*

**Figure 13. Image and EDS analysis of sample T10-P03 fraction +100#. Grains of quartz and clay minerals.**

*[Figure]*

**Figure 14. Image of sample T10-P03 fraction -325#. Aggregates of illite and kaolinite.**

*[Figure]*

**Figure 15. Image and EDS analysis of sample T10-P03 fraction -325# Aggregates of illite and kaolinite.**

## 4.2 Clay T18-P05

### Grain Size Classification

The information on the grain size distribution of sample T18-P05, following its disaggregation in a grinding mill for 10 minutes, is shown in Table 3. The sample exhibits a very fine grain size, as around 92.48% of the material lies below 325#.

**Table 3. Grain size classification of sample T18-P05 following 10-minute grinding in the grinding mill.**

<b>Project:</b> Characterization Study <b>Material:</b> T18-P05					
Grain Size Fraction		Mass (g)	Mass (%)	Mass Retained (%)	Mass Passing (%)
Mesh	mm				
6 #	3.360	0.00	0.00	0.00	100.00
8 #	2.380	0.00	0.00	0.00	100.00
10 #	1.680	0.00	0.00	0.00	100.00
14 #	1.190	0.00	0.00	0.00	100.00
20 #	0.840	0.00	0.00	0.00	100.00
28 #	0.590	0.00	0.00	0.00	100.00
35 #	0.420	0.00	0.00	0.00	100.00
48 #	0.297	0.00	0.00	0.00	100.00
65 #	0.210	9.05	1.13	1.13	98.87
100 #	0.149	10.90	1.36	2.50	97.50
150 #	0.105	0.00	0.00	2.50	97.50
200 #	0.074	17.10	2.14	4.64	95.36
270 #	0.053	8.70	1.09	5.73	94.27
325 #	0.044	14.35	1.80	7.52	92.48
400 #	0.037	738.90	92.48	100.00	0.00
- 400 #	-			-	-
Total	-	799.00	100.00	-	-

### X-Ray Diffraction Analysis

Below are the results of the X-ray diffraction analysis of samples T18-P05 (Table 4, Figures 6 through 21). The raw fraction of the sample is made up of quartz, kaolinite, illite, and muscovite. The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction pattern of Figure 17.

The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the clay fraction of the sample is made up of the clay minerals illite and kaolinite.

**Table 4. Mineral composition of sample T18-P05.**

Fraction	Mineral Composition
T18-P05 Raw	Quartz, kaolinite, illite, and muscovite
T18-P05 -325#	Quartz, kaolinite, illite, and muscovite
T18-P05 clay	Illite and kaolinite

*[Figure]*

**Figure 16. X-ray diffraction pattern of sample T18-P05 Raw.**

*[Figure]*

**Figure 17. X-ray diffraction pattern of sample T18-P05 fraction -325#.**

*[Figure]*

**Figure 18. X-ray diffraction pattern of sample T18-P05 Natural Clay fraction.**

*[Figure]*

**Figure 19. X-ray diffraction pattern of sample T18-P05 Glycol-treated clay fraction.**

*[Figure]*

**Figure 20. X-ray diffraction pattern of sample T18-P05 Calcined clay fraction.**

*[Figure]*

**Figure 21. X-ray diffraction patterns of sample T18-P05 Natural, Glycol-Treated, and Calcined clay fraction.**

### Scanning Electron Microscopy

The fractions of sample T18-P05 were analyzed on the scanning electron microscope. The analyzed fractions are made up of the clay minerals illite, kaolinite, and quartz (Figures 22 through 27). Few grains of iron oxides/hydroxides and titanium oxide were identified. The illite occurs in the form of aggregates in all of the analyzed fractions, whereas the kaolinite occurs in the form of pseudo-hexagonal particles.

The results of the EDS analysis indicate that the samples are essentially composed of oxygen, silicon, aluminum, potassium, and secondarily of magnesium, iron, and titanium.

*[Figure]*

**Figure 22. Image of sample T18-P05 fraction +100#. Grains of quartz and aggregates of clay minerals.**

*[Figure]*

**Figure 23. Image and EDS analysis of sample T18-P05 fraction +100#.**

*[Figure]*

**Figure 24. Image of sample T18-P05 fraction +270#. Illite aggregates.**

*[Figure]*

**Figure 25. Image and EDS analysis of sample T18-P05 fraction +270#. Aggregates of illite and kaolinite.**

*[Figure]*

**Figure 26. Image of sample T18-P05 fraction -325#.**

*[Figure]*

**Figure 27. Image and EDS analysis of sample T18-P05 fraction -325#.**

### 4.3 Clay T11-P03

#### Grain Size Classification

The information on the grain size distribution of sample T11-P03, following its disaggregation in a grinding mill for 10 minutes, is shown in Table 5. The sample exhibits a very fine grain size, as around 91.16% of sample lies below 325#.

**Table 5. Grain size classification of sample T11-P03 following 10-minute grinding in the grinding mill.**

<b>Project:</b>		<b>Characterization Study</b>			
<b>Material:</b>		<b>T11 P03</b>			
<b>Grain Size Fraction</b>		<b>Mass (g)</b>	<b>Mass (%)</b>	<b>Retained Mass (%)</b>	<b>Passing Mass (%)</b>
<b>Mesh</b>	<b>mm</b>				
6 #	3.360	0.00	0.00	0.00	100.00
8 #	2.380	0.00	0.00	0.00	100.00
10 #	1.680	0.00	0.00	0.00	100.00
14 #	1.190	0.00	0.00	0.00	100.00
20 #	0.840	0.00	0.00	0.00	100.00
28 #	0.590	0.00	0.00	0.00	100.00
35 #	0.420	0.00	0.00	0.00	100.00
48 #	0.297	0.00	0.00	0.00	100.00
65 #	0.210	0.00	0.00	0.00	100.00
100 #	0.149	0.00	0.00	0.00	100.00
150 #	0.105	0.00	0.00	0.00	100.00
200 #	0.074	0.00	0.00	0.00	100.00
270 #	0.053	30.92	5.32	5.32	94.68
325 #	0.044	20.47	3.52	8.84	91.16
400 #	0.037	13.32	2.29	11.13	88.87
- 400 #	-	516.84	88.87	-	-
<b>Total</b>	-	<b>581.55</b>	<b>100.00</b>	-	-

### X-Ray Diffraction Analysis

Below are the results of the X-ray diffraction analysis of samples T11-P03 (Table 6, Figures 29 through 34). The raw fraction is made up of quartz, kaolinite, illite, and muscovite. The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction patterns.

The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the clay fraction of the sample is made up of the clay minerals illite and kaolinite.

**Table 6. Mineral composition of sample T11-P03.**

<b>Fraction</b>	<b>Mineral Composition</b>
<b>T11-P03 Raw</b>	Quartz, illite, kaolinite, and muscovite
<b>T11-P03 +270#</b>	Quartz, illite, kaolinite, and muscovite
<b>T11-P03 +325#</b>	Quartz, illite, kaolinite, and muscovite
<b>T11-P03 +400#</b>	Quartz, illite, kaolinite, and muscovite
<b>T11-P3 -400#</b>	Quartz, illite, kaolinite, and muscovite
<b>T11-P03 clay</b>	Illite, kaolinite

[Figure]

**Figure 29. X-ray diffraction pattern of sample T11-P03 Raw.**

[Figure]

**Figure 30. X-ray diffraction pattern of sample T11-P03 +270#.**



[Figure]

**Figure 31. X-ray diffraction pattern of sample T11-P03 +325#.**

[Figure]

**Figure 32. X-ray diffraction pattern of sample T11-P03 +400#.**

[Figure]

**Figure 33. X-ray diffraction pattern of sample T11-P03 -400#.**

[Figure]

**Figure 34. X-ray diffraction patterns of sample T11-P03 Natural, Glycol-Treated, and Calcined clay fraction.**

### Scanning Electron Microscopy

The fractions of sample T11-P03 were analyzed on the scanning electron microscope. The analyzed fractions are made up of the clay minerals illite, kaolinite, and quartz (Figures 35 through 38). Few grains of iron oxides/hydroxides and titanium oxide were identified. The illite occurs in the form of aggregates in all of the analyzed fractions, whereas the kaolinite occurs in the form of pseudo-hexagonal particles.

The results of the EDS analysis indicate that the samples are essentially composed of oxygen, silicon, aluminum, potassium, and secondarily of magnesium, iron, and titanium.

[Figure]

**Figure 35. Image of sample T11-P03 fraction +270#. Aggregates of illite and kaolinite.**

[Figure]

**Figure 36. Image and EDS analysis of sample T11-P03 fraction +270#. Grains of quartz and clay minerals.**

[Figure]

**Figure 37. Images of sample T11-P03 fraction -400#.**

[Figure]

**Figure 38. Image and EDS analysis of sample T11-P03 fraction -400#. Aggregate of clay minerals, especially kaolinite.**

### 4.4 Clay T02-P04

#### Grain Size Classification

The information on the grain size distribution of the sample, following its disaggregation in a grinding mill for 10 minutes, is shown in Table 7. The sample exhibits a very fine grain size, as around 97.33% of the material lies below 325#.

**Table 7. Grain size classification of sample T02-P04 following 10-minute grinding in the grinding mill.**

Project: Characterization Study					
Material: T02 P04					
Grain Size Fraction		Mass	Mass	Retained Mass	Passing Mass
Mesh	mm	(g)	(%)	(%)	(%)
6 #	3.360	0.00	0.00	0.00	100.00



8 #	2.380	0.00	0.00	0.00	100.00
10 #	1.680	0.00	0.00	0.00	100.00
14 #	1.190	0.00	0.00	0.00	100.00
20 #	0.840	0.00	0.00	0.00	100.00
28 #	0.590	0.00	0.00	0.00	100.00
35 #	0.420	0.00	0.00	0.00	100.00
48 #	0.297	0.00	0.00	0.00	100.00
65 #	0.210	0.00	0.00	0.00	100.00
100 #	0.149	0.00	0.00	0.00	100.00
150 #	0.105	0.00	0.00	0.00	100.00
200 #	0.074	0.00	0.00	0.00	100.00
270 #	0.053	7.87	1.35	1.35	98.65
325 #	0.044	7.73	1.32	2.67	97.33
400 #	0.037	7.42	1.27	3.95	96.05
- 400 #	-	560.42	96.05	-	-
Total	-	583.44	100.00	-	-

### X-Ray Diffraction Analysis

Below are the results of the X-ray diffraction analysis of samples T02-P04 (Table 8, Figures 40 through 45). The raw fraction is made up of quartz, kaolinite, illite, and muscovite. The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction patterns.

The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the clay fraction of the sample is made up of the clay minerals illite and kaolinite.

**Table 8. Mineral composition of the fractions of sample T02-P04.**

Fraction	Mineral Composition
T02-P04 Raw	Quartz, illite, kaolinite, and muscovite
T02-P04- +270	Quartz, illite, kaolinite, and muscovite
T02-P04 +325#	Quartz, illite, kaolinite, and muscovite
T02-P04 +400#	Quartz, illite, kaolinite, and muscovite
T02-P04 -400#	Quartz, illite, kaolinite, and muscovite
T02-P04 clay	Illite, kaolinite

[Figure]

**Figure 40. X-ray diffraction pattern of sample T02-P04 Raw.**

[Figure]

**Figure 41. X-ray diffraction pattern of sample T02-P04 fraction +270#.**

[Figure]

**Figure 42. X-ray diffraction pattern of sample T02-P04 fraction +325#.**

[Figure]

**Figure 43. X-ray diffraction pattern of sample T02-P04 fraction +400#.**

[Figure]

**Figure 44. X-ray diffraction pattern of sample T02-P04 fraction -400#.**

[Figure]

**Figure 45. X-ray diffraction patterns of sample T02-P04 Natural, Glycol-Treated, and Calcined clay fraction.**

### Scanning Electron Microscopy

The fractions of sample T02-P04 were analyzed on the scanning electron microscope. The analyzed fractions are made up of the clay minerals illite, kaolinite, and quartz (Figures 46 through 49). Few grains of iron oxides/hydroxides and titanium oxide were identified. The illite occurs in the form of aggregates in all of the analyzed fractions, whereas the kaolinite in the form of pseudo-hexagonal particles.

The results of the EDS analysis indicate that the samples are essentially composed of oxygen, silicon, aluminum, potassium, and secondarily of magnesium, iron, and titanium.

[Figure]

**Figure 46. Images of sample T02-P04 fraction +270#. Aggregates of grains of quartz, clay minerals, and muscovite.**

[Figure]

**Figure 47. Image and EDS analysis of sample T02-P04 fraction +270#. Aggregate with a grain of quartz and clay minerals.**

[Figure]

**Figure 48. Images of sample T02-P04 fraction -400#. Aggregates of clay minerals.**

[Figure]

**Figure 49. Image and EDS analysis of sample T02-P04 fraction -400#. Aggregate of illite and kaolinite.**

### 4.5 Clay T01-P04

#### Grain Size Classification

The information on the grain size distribution of the sample, following its disaggregation in a grinding mill for 10 minutes, is shown in Table 9. The sample exhibits a very fine grain size, as around 94.89% of sample lies below 325#.

**Table 9. Grain size classification of sample T01-P04 following 10-minute grinding in the grinding mill.**

Project: Characterization Study					
Material: T01 P04					
Grain Size Fraction		Mass (g)	Mass (%)	Retained Mass (%)	Passing Mass (%)
Mesh	mm				
6 #	3.360	0.00	0.00	0.00	100.00
8 #	2.380	0.00	0.00	0.00	100.00
10 #	1.680	0.00	0.00	0.00	100.00
14 #	1.190	0.00	0.00	0.00	100.00
20 #	0.840	0.00	0.00	0.00	100.00
28 #	0.590	0.00	0.00	0.00	100.00
35 #	0.420	0.00	0.00	0.00	100.00
48 #	0.297	0.00	0.00	0.00	100.00
65 #	0.210	0.00	0.00	0.00	100.00
100 #	0.149	0.00	0.00	0.00	100.00
150 #	0.105	0.00	0.00	0.00	100.00

<b>200 #</b>	<b>0.074</b>	<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>100.00</b>
<b>270 #</b>	<b>0.053</b>	<b>12.65</b>	<b>2.66</b>	<b>2.66</b>	<b>97.34</b>
<b>325 #</b>	<b>0.044</b>	<b>11.68</b>	<b>2.45</b>	<b>5.11</b>	<b>94.89</b>
<b>400 #</b>	<b>0.037</b>	<b>11.70</b>	<b>2.46</b>	<b>7.56</b>	<b>92.44</b>
<b>- 400 #</b>	<b>-</b>	<b>440.31</b>	<b>92.44</b>	<b>-</b>	<b>-</b>
<b>Total</b>	<b>-</b>	<b>476.34</b>	<b>100.00</b>	<b>-</b>	<b>-</b>

### X-Ray Diffraction Analysis

Below are the results of the X-ray diffraction analysis of samples T01-P04 (Table 10, Figures 51 through 56). The raw fraction is made up of quartz, kaolinite, illite, muscovite, and feldspar (microcline). The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction patterns.

The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the clay fraction of the sample is made up of the clay minerals illite and kaolinite.

**Table 10. Mineral composition of sample T01-P04.**

<b>Fraction</b>	<b>Mineral Composition</b>
<b>T01-P04 Raw</b>	Quartz, illite, kaolinite, muscovite, microcline
<b>T01-P04 +270#</b>	Quartz, illite, kaolinite, muscovite, microcline
<b>T01-P04 +325#</b>	Quartz, illite, kaolinite, muscovite, microcline
<b>T01-P04 +400#</b>	Quartz, illite, kaolinite, muscovite, microcline
<b>T01-P04 -400#</b>	Quartz, illite, kaolinite, muscovite, microcline
<b>T01 - P04 clay</b>	Illite, kaolinite

[Figure]

**Figure 51. X-ray diffraction pattern of sample T01-P04 Raw#.**

[Figure]

**Figure 52. X-ray diffraction pattern of sample T01-P04 fraction +270#.**

[Figure]

**Figure 53. X-ray diffraction pattern of sample T01-P04 fraction +325#.**

[Figure]

**Figure 54. X-ray diffraction pattern of sample T01-P04 fraction +400#.**

[Figure]

**Figure 55. X-ray diffraction pattern of sample T01-P04 fraction-400#.**

[Figure]

**Figure 56. X-ray diffraction patterns of sample T01-P04 Natural, Glycol-Treated, and Calcined clay fraction.**

### Scanning Electron Microscopy

The fractions of sample T01-P04 were analyzed on the scanning electron microscope. The analyzed fractions are made up of the clay minerals illite, kaolinite, and quartz (Figures 57 through 60). The illite occurs in the form of aggregates in all of the analyzed fractions, whereas the kaolinite occurs in the form of pseudo-hexagonal particles.

Few grains of iron oxides/hydroxides and titanium oxide were identified. The results of the EDS analysis indicate that the samples are essentially composed of oxygen, silicon, aluminum, potassium, and secondarily of magnesium and iron.

[Figure]

**Figure 57. Images of sample T01-P04 fraction +270#. Grains of quartz and clay minerals.**

[Figure]

**Figure 58. Image and EDS analysis of sample T01-P04 fraction +270#. Kaolinite aggregate.**

[Figure]

**Figure 59. Images of sample T01-P04 fraction -400#. Aggregates of kaolinite and illite.**

[Figure]

**Figure 60. Image and EDS analysis of sample T01-P04 fraction -400#. Aggregates of kaolinite and illite.**

## 5. Conclusions

All five clay samples analyzed are similar in terms of both composition and grain size. The samples exhibit a fine grain size, as around 99.50% (T10-P03) and 91.16 (T11-P03) lie below 325#.

The raw fractions are made up of quartz, kaolinite, illite, and muscovite. The grain size sorting through a 325# sieve reduced the quartz concentration, as can be seen in the diffraction patterns and scanning electron microscope analyses. Sample T01-P04 exhibits darker coloring, probably due to the presence of iron hydroxides; the results of the x-ray diffraction analysis indicated the presence of feldspar (microcline) in the thicker fractions.

The treatment of the clay fraction, according to the method proposed by Moore and Reynolds Jr. (1989), confirmed that the fine fractions (clay) of the samples are made up of the clay minerals illite and kaolinite.

The x-ray diffraction and scanning electron microscope analyses did not reveal any fibrous minerals present in the samples.

The mill grinding followed by wet grain size sorting through a 325# sieve indicated that some of the quarts present in the sample can be removed by screening the clay.

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Nothing else. I have checked and found conforming, which I certify.  
São Paulo, February 3, 2023.



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