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STUDY OF SOILS BY THERMAL ANALYSIS

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The study of soils is very important in the geological and geological engineering researches. A study of ten samples of soils was carried out by thermal analysis, and X-Ray Fluorescence Spectrometry to understand soil evolution in Angra dos Reis region, Rio de Janeiro State, Brazil. The sample collection sites were chosen based on geological characteristics, the soil layer thickness, the soil composition pattern, and whether or not it was moved either by erosion or by gravitational shifts. Because of the humid tropical climatic condition, natural soils tend to show great thickness of weathered mantles with formation of saprolites and saprolite soils. Kaolinite is an important secondary mineral which can be formed from many different minerals, like *k*-mica and *k*-feldspar and can be weathered to gibbsite. The results from TG/DTG and DTA indicated which soils had more weathering, and the same results were obtained by XRF, when silica/aluminum ratios from samples are compared with thermal analysis results.

Keywords: *soil, thermal analysis, weathering*

Introduction

Thermal analysis and X-ray fluorescence spectrometry were used to understand soil evolution in Angra dos Reis region, Rio de Janeiro State, Brazil. This area is a humid tropical climate zone, where the natural slopes tend to show great thickness of weathered mantles with formation of saprolites and saprolite soils, in which the weathering mechanisms are controlled by the weather and the anisotropic character of the rock massifs, along with several discontinuities which may be filled by clay material. In these regions, laterization processes may conduce to macrostructures and particular characteristics of resistance, plasticity and texture, which also conduce to differentiated behavior of soils in relation to those of temperate climates. According to some authors [1–3] tropical and subtropical regions are associated with the occurrence of clay bed zones and common occurrence of lateritic soil and latosols. These oxisols exhibit, as major diagnostic characteristics, the presence of an oxic horizon in which Al or Fe or both are enriched. Gibbsite frequently occurs in old soils of advanced weathering, and the various polymorphs of FeOOH and Fe_2O_3 commonly exist together. Kaolinite is an important secondary mineral which can be formed from many different minerals, like *k*-mica and *k*-feldspar, and can be weathered to gibbsite [4]. In young soils, with 10000 years or less, such as the glaciated and loess areas, the kaolinite is present. So, it shows that relatively short time is necessary to produce soil with kaolinite.

Experimental

Sites selection and sampling

In the sense of homogenizing the parameters that could influence the evolution of soils, all the sites selected for sampling presented similar conditions of landform, geological characteristics, slope, aspect and residual nature of soil. About 2 kg of clay material samples were collected from each site, with depths varying from 0.5 up to 1.5 m. A number of ten samples from different sites were selected for this study.

Samples preparation

All samples were dried in air for a week, but samples MH3, MH4 and MH5 were not, because it was necessary to determine their water content. The samples were reduced to quarter, repeatedly, and mechanically separated by sieve analysis until we obtained the ideal volumes. The particles less than 0.062 mm (250 meshes) were subjected to thermal analysis and XRF.

Methods

TG/DTG/DTA measurements

Thermogravimetry (TG) and derivative thermogravimetry (DTG) was used to analyze the clay material samples. The thermal runs were performed on a Mettler Toledo TG/SDTA. About 20 mg of material for each analysis were used, at N_2 atmosphere and heating rate of 10°C min^{-1} from room temperature to

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1100°C. TG/DTG and DTA analysis of samples were performed by TA SDT 2960, about 10 mg per sample in a N_2 atmosphere and heating rate of 10°C min^{-1} from room temperature to 1000°C.

X-ray fluorescence spectrometry

It was performed by the fusion method using 7 g of lithium tetra/metaborate. The samples were performed at an X-ray fluorescence spectrometer PW 2400 Phillips/sequential, with an Rh tube of 3 KW, six crystal analyzers and two detectors. The software used for these analyses was the SemiQ, developed by Phillips.

Results and discussion

DTA and TG of samples show kaolinite and gibbsite even present, except for MH8 sample. Figure 1 shows TG/DTG and DTA curves for MH3 clay sample in nitrogen atmosphere, with three decomposition stages. Water loss (~6.6%) in the first stage at 100°C, the second stage at 300°C, related to the loss of hydroxyl (~5.0%) from gibbsite, and the third decomposition range at 510°C, with mass loss of hydroxyl from kaolinite (~6.3%). The second stage of decomposition was associated to gibbsite because of DTA curve [5], which shows an endothermic event, while the TG curve shows a mass loss event. Associated to the chemical analysis results, was possible to correlate the presence of gibbsite.

TG/DTG curves, from MH4 sample, show three decomposition stages (Fig. 2). The first one related to water loss (~10.0%); the second, associated to loss of hydroxyl from gibbsite (~2.3%) and the last one due to mass loss of hydroxyl from kaolinite (~7.5%). DTA curve shows three endothermic events. The first associated to water loss, at 100°C; the second, about 280°C associated to loss of hydroxyl from gibbsite [6]

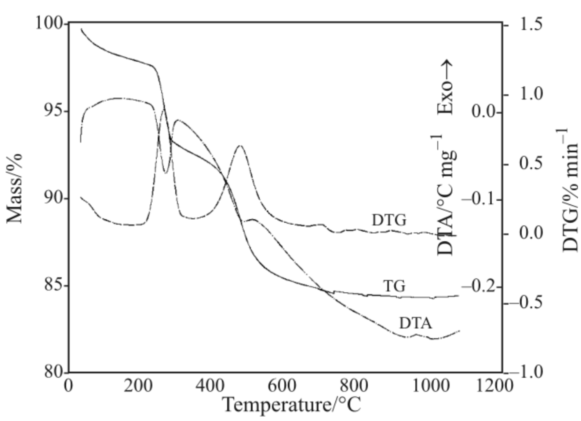

Fig. 1 TG/DTG/DTA curves for MH3 sample in nitrogen atmosphere

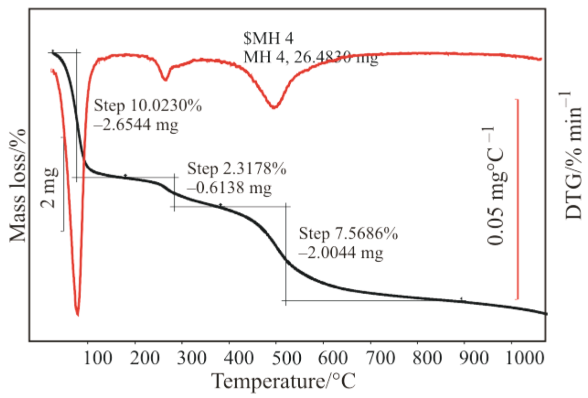

Fig. 2 TG/DTG curves for MH4 sample

and the last one, at 500°C, relates to mass loss of hydroxyl from kaolinite. The exothermic event occurs about 970°C, and it was associated to mullite prenucleation.

Figure 3 depicts a comparison of DTA curves, in N_2 atmosphere, from MH3, MH4 and MH5 samples. It is possible to see three endothermic and one exothermic events. The first was associated to water loss at 80°C, the next at 270°C corresponding to hydroxyl loss from gibbsite and the last one with peak temperature around 480°C indicating degradation of kaolinite. The exothermic event occurs at 960°C and it was associated to mullite prenucleation.

Figure 4 shows five DTA curves. MH8 sample curve shows one endothermic event at 510°C, decomposition of kaolinite and one exothermic event at 1000°C related to mullite prenucleation.

MH6a, MH7, MH8a and MH9 samples curves show three events: two endothermic and one exothermic. The first endothermic event is for the decomposition of gibbsite around 270°C, the second one corresponding to the degradation of kaolinite at 500°C while the third event (exothermic) between 940 and 1000°C was associated to mullite prenucleation.

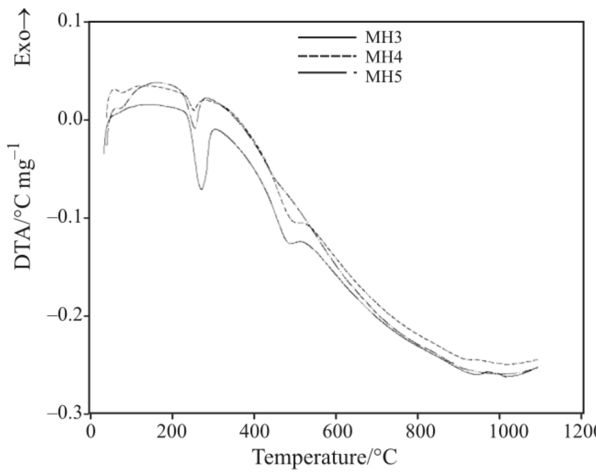

Fig. 3 DTA curves obtained from MH3, MH4 and MH5 samples

Table 1 SiO_2/Al_2O_3 ratio from 8 soil samples

Sample	MH3	MH4	MH5	MH6a	MH7	MH8	MH8a	MH9
SiO_2/Al_2O_3	1.39	2.0	2.46	1.48	1.21	1.57	0.76	1.28

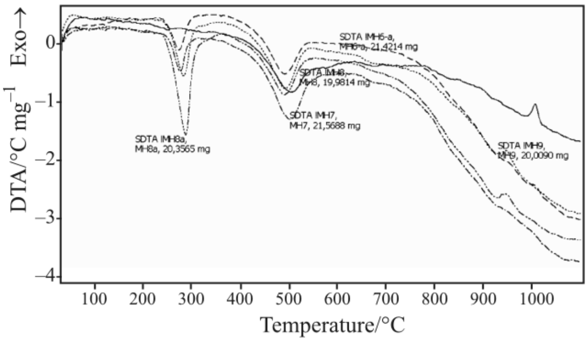

Fig. 4 DTA curves of samples MH6a, MH7, MH8, MH8a and MH9

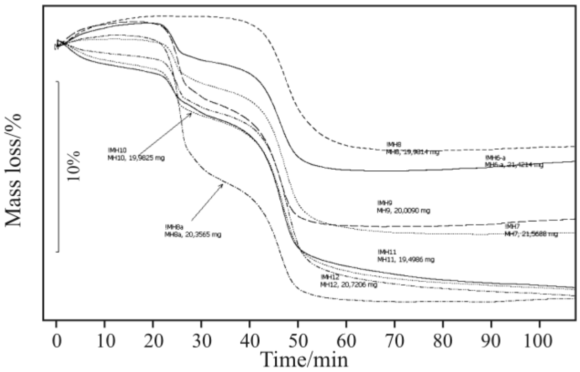

Fig. 5 TG curves of 8 soil samples

TG and DTA data suggest that sample MH8 is younger than others and these results confirm the field interpretations. The residual soil of MH8 was covered by the MH8a soil that was transported; hence they are limited by primary mineral debris and protected from weathering.

TG curves for soils from samples MH6a, MH7, MH8, MH8a, MH9, MH10, MH11 and MH12 are shown in Fig. 5.

These sites were chosen for sampling because they represent different landforms among themselves, considering the landform analysis from horizontal and vertical profiles on slopes. The samples were collected from profiles of residual soils on slopes with exception of the sample MH8a collected from coluvial material overlaying the profile of the sample MH8. All the TG curves except the curve of sample MH8 present two decomposition steps. The first step was correlated to hydroxyl loss of gibbsite in the 250–300°C temperature range and the second step was correlated to the kaolinite decomposition, between 420 and 580°C.

Sample MH8 shows one decomposition stage, related to hydroxyl loss of kaolinite with mass loss about 6% while the other samples show mass loss between 7 and 15%.

Analysis of MH8 sample showed 42.9% clay content, while sample MH8a, which presented the largest mass loss, approximately 16.4% of gibbsite and 50.1% of kaolinite was determined.

Chemical compositions considered [7]:

- Kaolinite – $Al_4Si_4O_{10}(OH)_8$: SiO_2 46.54%; Al_2O_3 39.50%; H_2O 13.96%;
- Gibbsite – $Al(OH)_3$: Al 51.4%, $(OH)_3$ – 48.6%.

XRF study of 8 soil samples shows values between 0.764 and 2.464, (data collected in Table 1), which can be or not correlated to development stages of soil. The preliminary results suggest that gibbsite can be present in young soil.

The MH4 and MH5 samples came from bordering gap between two strata of rocks blocks in fractures. They had much more water than soil on the surface and many plants and roots growing up there. These special sites can explain the SiO_2/Al_2O_3 ratio. XRF results confirm thermal analysis data and field research, which suggests MH8 sample as less developed soil, maybe because it is protected from weathering by the overlaying of MH8a soil profile.

Conclusions

Thermal analysis can be an effective tool to study soil formation and together with XRF they can improve soil research since thermal analysis is a convenient and rapid technique to provide information. The silica/aluminum ratio indicated which soils had more weathering [8] and the same results were obtained by TG/DTG and DTA methods. MH4 and MH5 samples had unusual development since they developed in a bordering gap between blocks of rocks and these special geological sites create different conditions to soil formation.

Despite of the different and special conditions to the development of soils the thermal analysis was capable to detect accurately all those variations related to the composition of the soils.

Formation of kaolinite in the first step of the weathering of the soils and of gibbsite in the subsequent step indicates the evolution degree of the soils. Thermal analysis could identify and quantify that rate in the different types of studied soils, which could be ‘in situ’ transported or between rock blocks.

Acknowledgements

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