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Use of X-ray diffractometer technique for studying minerals in thin sections of soils and clays

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The X-ray diffractometer technique finds now a wide and manyfold use in soil and clay studies, including the structural mineral analysis proper (especially of clay minerals), the phase analysis of polymineral systems [2] and the control of structural changes in minerals when they undergo some treatments [3]. A considerable part of the above mentioned studies has been made in the U.S.S.R. by using the apparatus of the УРС-1 type and its improved modifications, providing precize experimental data (interlayer spacing and intensity values) to be obtained. The problems of soil mineralogical analysis and the clay mineral structure examination by means of УРС-5 U.M. apparatus were recently considered by one of the authors [2].

The X-ray diffractometer method is based on the interaction of the primary X-ray beam with the flat surface of a volume [1]. It is evident, therefore, that the flat-parallel thin sections of soils are ideal objects for the study by the X-ray diffractometer method. Aim of this paper is to discuss the optimum conditions of the work with the flat-parallel thin sections of soils and the main results, which can be obtained by the X-ray diffractometer technique of the flat-parallel thin sections.

The УРС-5 U.M. apparatus with Cu k_2 radiation, Ni filter was used. The angular velocity of the quantum counter (МСТР — 4 type) was 1° per 1 mm; the potential — 35 kv, the current — 12 mÅ. An optimum set of diaphragms was tested in two sensibility variants of the reflected quantum fixing arrangement. The most suitable conditions for primary mineral (such as quartz, felspars) determination were the following: the front slits 1 and 0.5 mm and the back slits — 0.5 mm; sensibility — 1,000; the time constant — III. The determination of clay minerals required the same width of the front slits but the width of the back slits was 0.25 and 0.5 (or 0.25) mm; the sensibility — 200, and the time constant — IV.

Open thin sections were prepared of soils and clays preliminarily fastened with colophony. The thickness of the thin sections was about 0.03 mm. If the specimen, from which the thin section was prepared, was

monolithic, without large pores no maxima due to the presence of cementing material were recorded on the X-ray diffractograms. And *vice versa*, if the specimens were loose, with large pores, or composed of several structural separates the cementing material produced broad diffraction maxima in the region of 15-20° with Cu k_2 radiation (Fig. 1c).

The thin sections of frozen-taiga and sod-podzolic soils were studied. Mineralogy and the grain-size of the soils varied widely, due to differences in the composition of the soil forming rocks. The open thin sections were mounted in the X-ray diffractometer on a holder for flat specimens. As the thin section plane (at least from one side of the surface) and the working plane of the holder had to coincide strictly it was necessary to prepare the thin sections so, as to allow the distance between the edge of the slide and the edge of the specimen plate to be no more than 3-4 mm or even less. The dimensions of the slide in three other directions did not affect the quality of the work. On working with a powder specimen holder — the device ГП — 4 [2] — the thin section fit to the size of the quartz cuvette could be fastened by means of plasticine. In this case no correlation between the edges of the slide and the specimen plate was necessary.

RESULTS

It was found that the X-ray diffractometer technique could be successfully used at least in two cases:

(a) for the determinations of predominant coarse-grained primary and secondary minerals;

(b) for the determination of clay minerals.

(a) DETERMINATION OF COARSE-GRAINED MINERALS

X-ray diffractograms of flat-parallel thin sections obtained for a sod-podzolic soil developed on cover-loam (Moscow region) and for a frozen-taiga soils (Kolyma province) are shown in Fig. 1a-e. Reflections with spacings 4.25 Å; 3.34 Å; 2.46Å; 2.28 Å and others belong to quartz; reflections with spacings 4.05 Å; 3.75 Å; 3.64 Å; 3.18-3.24 Å and others belong to felspars. These minerals are represented by the alkaline felspar group.

The presence of two maxima — one with d/n = 3.24 and the other with d/n = 1.53 provides grounds for a supposition that two felspar minerals are present in the specimen of the frozen-taiga soil. Mineralogy according to the X-ray data of the coarse-grained part of both soils coincides in general with the data of the optical microscope. Only single grains of hornblende, biotite and accessory minerals were not detected by the X-ray method.

Carbonates — calcite (by the spacings 3.85 Å; 3.03 Å; 2.5 Å; 2.28 Å and others) and dolomite (by the specings d/n = 2.87 Å; 2.67 Å; 2.40 Å; 2.19 Å

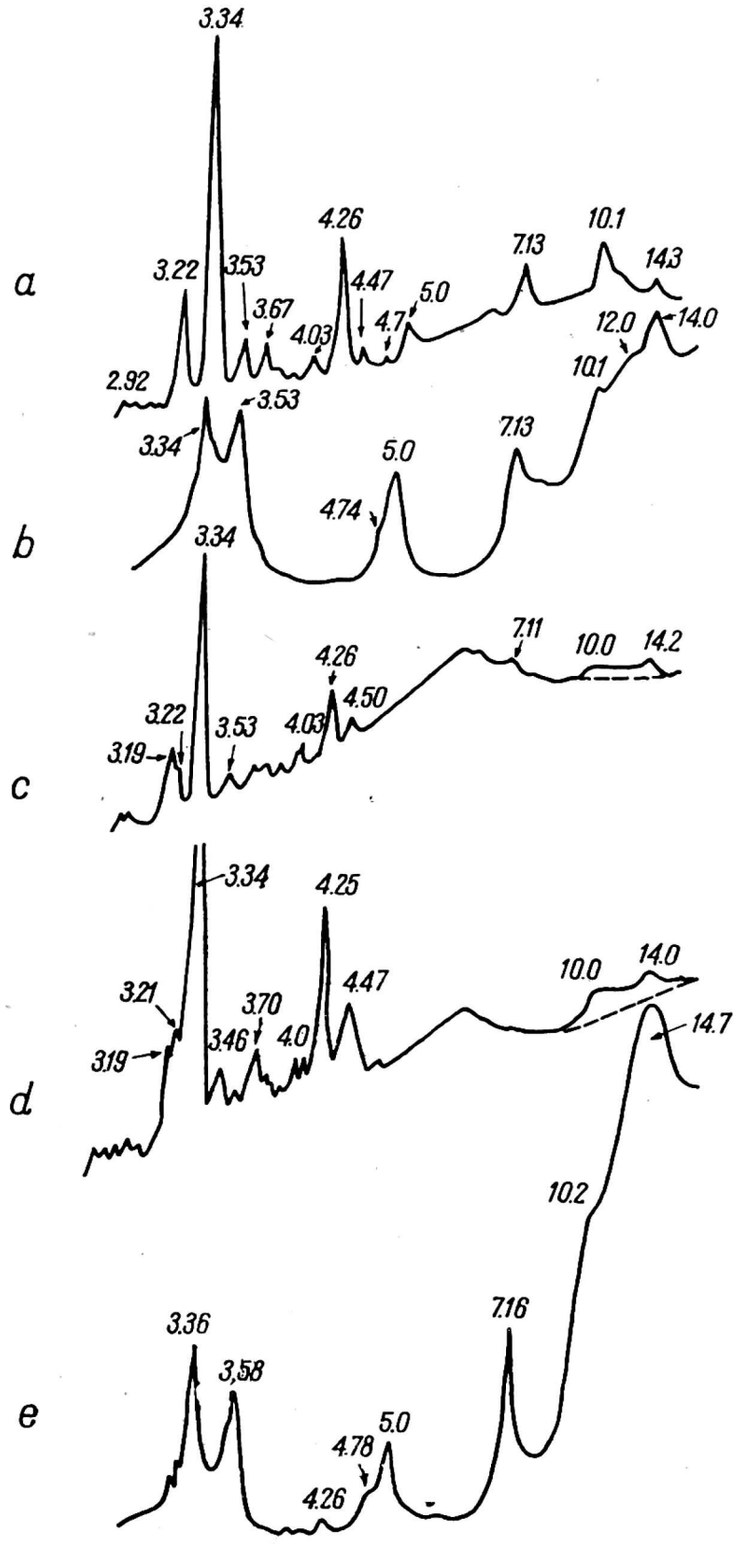

Fig. 1. X-ray diffractograms of thin sections and less than 0.001 mm fraction of soils. *a-c* — frozen-taiga soil (Kolyma province); *a* — depth 90-100 cm, thin section; *b* — the same depth, less than 0.001 mm fraction; *c* — depth 0-7 cm, thin section; *d-e* — sod-podzolic soil (Moscow region); *d* — depth 130-140 cm, thin section; *e* — the same depth, less than 0.001 mm fraction.

and others); gypsum (d/n = 7.56 Å; 3.80 Å; 3.06 Å; 2.87 Å and others) and the mineral-salts were readily determined in thin sections by the X-ray diffractometer technique.

(b) DETERMINATION OF CLAY MINERALS

In the thin section of loamy and clayey soils, as well as of soils developed on shales, general clay mineral reflections are always recorded on X-ray diffractograms with following spacings: 4.3-4.5 Å; 2.50 Å; 1.48-1.53 Å. In the thin sections of soils on loams general reflections are either not accompanied by distinctly recorded clay mineral basal reflections or have a higher intensity in any direction of the thin section plate

relative to the structural separate. This is evidence of the predominance of an irregular arrangement of clay mineral crystals versus one another (Fig. 1a-e). The difference in intensities of the general clay mineral reflections at 4.5 Å and the quartz reflection (for example at 4.2 Å) throughout the soil profiles may serve as an indication of the impoverishment or enrichment of the clay phase of soil horizons with contrast texture (for example, sod-podzolic soils).

In some cases (soils on shales and on clays, thin sections of desintegrated shale and strongly cemented clay separates) even basal reflections can be recorded. The X-ray diffractograms in Fig. 1 may serve as an example. Reflections with spacings 14.2 Å; 7.1 Å, 4.75 Å, 3.53 Å and others belonging to chlorite are seen on the X-ray diagram of the soil forming shale thin section. The relation between the intensities of the reflections with d/n = 4.75 Å from one side and those with d/n = 3.53 Å and 7.1 Å from the other confirms the trioctahedral character of the mineral. The reflections with the spacings 10.2 Å and 5.0 Å and others belong to hydromica. It may be found by the comparison of the (002) and (001) basal reflection intensities that the hydromica is dioctahedral. A gentle decline of the intensity towards small angles of the 10 Å diffraction maximum reveals the presence of small amounts of swelling packets in the mineral. An X-ray diffractogram of the less than 0.0001 mm fraction separated from the fine earth of the frozen-taiga soil is presented in the same Fig. 1b. It is seen that hydromica and chlorite are common minerals in both specimens, and, at the same time, that there are much more minerals with mixed layer structure of mica and montmorillonite packets in the clay fraction from the fine earth. Moreover, the content of swelling packets is higher in the latter than in minerals of the thin section. An X-ray diffractogram of the thin section from the A horizon of the same soil is shown in Fig. 1. It is seen that the intensities of the clay mineral basal reflections considerably decreased as compared with the records of the thin section from the soil forming rock. It is characteristic, however, that the intensity of the general reflection at 4.5 Å in relation to the reflection with d/n = 4.2 Å of quartz in the thin section of the upper horizon is higher than where the decrease in basal reflection intensities in the clay minerals of the upper horizon is due not to the decrease in the amount of these minerals but to the lower regularity in the position of clay mineral particles as compared with the thin section from the soil forming rock. This coincides with soil mechanical analysis data showing a higher content of clay particles in the upper horizon than in the underlying horizon.

Basal reflection of the soil developed on loam (Moscow region) are recorded less distinct. As it is shown in Fig. 1d there are clay minerals in the thin section plane which form a broad diffraction maximum from 10 to 15 Å. It may be regarded as a summary reflection of hydromica,

mixed-layer mica-montmorillonite and chlorite. This is confirmed by the X-ray diffractogram of the less than 0.001 mm fraction separated from the same samples (Fig. 1e).

Of a special interest is the possibility to use the X-ray diffractometer analysis of the soil plate fragments extracted from the thin sections after the examination under the microscope. To these belong the incrusted clay material, characteristic of many soils and single mineral grains which are being substituted by secondary minerals. But this is not subject of the present paper.

It is doubtless, at any rate, that the combination of micromorphological examinations with the X-ray diffractometer study of thin sections and their fragments will present a series of new informations concerning the secondary changes of minerals during soil formation, formation of clayey separations, distribution of minerals in different soil horizons etc.

CONCLUSIONS

1. The X-ray diffractometer technique is perspective for the determination of soil minerals in thin sections.

2. In all cases the predominant coarse-grained minerals (primary: quartz, felspars; secondary: carbonates, gypsum, mineral-salts) may be precisely identified by means of the X-ray diffractometer technique.

3. Depending on the nature of the material studied there can be recorded by means of the X-ray diffractometer eighther only general or general and basal clay mineral reflections. This presents more information on the amount of the clay phase, the character of clay particle regularity and the mineralogical composition of the soil clays.

SUMMARY

The X-ray diffractometer technique finds now a wide and suitable application in soil and clay studies including the structural mineral analysis and the control of structural changes in various minerals. Aim of the paper is to present the optimal conditions of the work with flat parallel thin sections of soils and the main results that can be obtained by the X-ray diffractometer technique of flat parallel thin sections. These investigations show that it is doubtless that the combination of micromorphological research with the X-ray diffractometer study of thin sections and their fragments will present a series of new informations concerning the secondary changes of different minerals during soil formation and the development and distribution of clayey mineral separation in different soil horizons. Thus one can state that the X-ray diffractometer

technique is perspective for the determination of soil minerals in thin sections.

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