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Clay Minerals in Soils Formed from Loess *

Глинистые минералы в почвах образовавшихся из лёссов

Minerały ilaste w glebach wytworzonych z lessów

The loess formations have been long an object of interest, both with regard to their origin and to their composition and properties. The physical and chemical properties of loess and of soils formed from loess are closely connected with their mechanical, mineralogical and chemical composition. One of the factors responsible for the formation of these properties is the colloidal fraction, and especially the so-called clay minerals, which are the main component of this fraction.

The purpose of the present paper was to investigate the composition of the clay minerals of the colloidal fraction of soils formed from loess. The material was furnished by 12 profiles (altogether 43 samples) representing various stages of the development of the soil and various loess areas of the country. The profiles concern the so-called deep loess, i. e. areas with a loess cover of considerable thickness.

Profiles I, II, III and IV come from the Lublin Upland, profiles V and VI from the Sandomierz Upland, profiles VII and VIII from the Cracow — Częstochowa Upland, profiles IX, X, and XI from the Carpathian Foreland, and profile XII from the Trzebnica Hills. Most of the profiles represent brown soils and chernozems, the remaining profiles belong to podsolic and buried soils. The excavations were made on arable land, but in some cases soils of wooded or turfy areas were also studied. Soil samples were taken from the separate genetic horizons of the soils as well as from various levels of loess rock.

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METHODS

1. Separation of the fraction $< 0,001$ mm

The investigations concerned the fraction of particles smaller than 0.001 mm. The clay fraction, i.e. $< 0,001$ mm, was separated by sedimentation according to the modified method of Gorbunow (3), after the samples had been freed from exchangeable cations by washing with distilled water (by decantation). If carbonates were present, they were removed at the beginning of the laboratory investigations with 0.2 n HCl. Most of the organic matter occurring in samples of clay from the humus horizons was removed with 6—8 per cent H_2O_2 .

2. Roentgenographic analysis

Prior to roentgenographic studies, the clay fraction was subjected to a special treatment, the first step of which consisted in removing from the studied samples „free” iron oxides by the method of Mitchell and Mackenzie (8), with the use of $Na_2S_2O_4$. Then the samples were divided into two parts. One of them was saturated with magnesium according to Jeffries and Jackson (5) and with glycerin according to MacEvan (7). The other part was saturated with potassium and ignited at $600^\circ C$ according to the methods of Kunze and Jeffries (6) and Dixon and Seay (2). The methods mentioned above were checked by Pavel (10). The removal of iron produces better, undimmed roentgenograms. Further operations facilitate the identification of minerals, especially in interstratified complexes, i.e. minerals of a mixed structure.

The clay fractions prepared by the method described above were studied roentgenographically by means of the microstructural apparatus Mikro-Meta according to the method given by Pavel (9). The samples, contained in gelatin capillaries, 0.4 mm in diameter, were placed in Debye-Scherrer cameras (Chirana), 64 mm in diameter, adapted to register small angle reflexes. The rays were filtered through a Ni-filter; the exposition lasted 1.5 hour.

The roentgenograms were measured on a comparator; a 10 degree scale was used to determine the intensity. The identification of the minerals was carried out by comparing the obtained values d and the relative intensities I of the separate lines with analogous values for „pure” minerals, and by using tables (4).

3. Thermal analysis

Samples destined for thermal analysis were saturated with magnesium and kept for some days in an atmosphere of which the relative

humidity was about 50%. The purpose of this operation was to saturate all samples with the same cation and to obtain uniform humidity, which facilitates the comparison of the results of thermal analysis.

Thermal analysis was carried out by means of an apparatus constructed by Pavel on the principles given by Berg and Rasonsk a.(1). Samples of the clay fraction in quantities up to 200 mg were placed in a quartz tube provided with a thermocouple Pt—Pt + 10% Rh. This standard sample was placed in another quartz tube. The quartz tubes were put into a ceramic block and placed in a preheated oven. The increase of the temperature of the oven was almost linear, and its rate was up to 100°C per minute. Thermal curves were registered on paper by means of a self-recording galvanometer. This is a rapid method; one determination can be accomplished in about 10 minutes.

RESULTS

Preliminary information on the mineralogical composition of the clay fractions of loess soils was given in the report of 1960 (16). The present paper deals with the detailed results of the investigations.

Table 1 contains data on hydrogen ion concentration, on the content of calcium carbonate and of clay fractions in soils and loess formations.

It results from this table that calcium carbonate occurs, as a rule, in the parent rock only. It is usually leached down to the depth of more than 1 m, often much deeper (profiles III and IX). The pH values usually depend on the distribution of carbonates, on the type of the soil and on its utilization. In the upper horizons of brown soils and chernozem it is usually slightly acid, in podsollic soils acid. The loess rock shows an alkaline reaction.

The content of particles smaller than 0,001 mm also varies in the studied soils. In the humus horizons it averages 10% or slightly more. The brown and illuvial horizons usually show an increased content of this fraction. In the loess rock it averages 10—15%. Occasionally considerable deviations from these regular findings can be observed.

The results of the determination of the mineralogical composition of the clay fraction of the studied soils are contained in Table 2. It appears that this fraction is composed of illite, montmorillonoid, interstratified complex of illite and montmorillonoid, and, in addition, of kaolinite and quartz. As the predominant minerals occur either illite or montmorillonoid, or their interstratified complex. Usually one of these predominates, the others belong to the so-called accompanying minerals. The occurrence of the minerals mentioned above varies in the se-

Table 1. Some properties of soils formed from loess.

Nos. of samples	Type of soil and mode of utilization	No. of profile and place	Depth in cm	CaCO ₃ %	pH KCl	pH H ₂ O	Content of fraction <0.001 mm %
I 1 2 3 4	Young brown soil (wooded)	U/46 Kazimierz	5-15 25-35 ~ 400 ~ 1200	0,0 12,0 6,9 0,0	5,9 7,2 7,4 6,9	6,7 8,1 8,2 7,3	13,1 15,5 10,5 21,0
II 5 6 7 8	Brown soil (turfed)	U/47 Lublin-Rury	5-20 40-50 180-200 ~ 800	0,0 0,0 13,9 10,1	6,1 6,0 7,3 7,6	6,9 6,7 8,2 8,5	9,7 16,9 9,0 7,1
III 9 10 11 12 13	Brown soil, buried (wooded)	U/40 Szczepieszyn	0-10 50-60 460-470 480-495 530-540	0,0 0,0 0,0 0,0 0,0	5,4 7,1 5,3 5,5 5,4	6,3 7,6 6,3 6,3 6,3	10,2 10,0 15,4 3,3 4,7
IV 14 15 16 17	Degraded chernozem (arable)	U/94 Hrubieszów	5-15 30-40 90-100 ~ 300	0,0 0,0 1,2 11,2	5,9 5,4 7,3 7,3	6,9 6,6 8,2 8,2	15,6 15,3 12,9 10,7
V 18 19 20	Degraded chernozem (arable)	U/35 Lipnik (Sandomierz Region)	5-15 30-40 90-100	0,0 0,0 1,1	6,6 6,5 7,2	7,3 7,1 8,1	13,6 17,1 17,1
VI 21 22 23 24	Brown soil (arable)	U/36 Sandomierz	5-15 30-40 ~ 300 ~ 1500	0,0 0,0 10,5 4,2	6,6 6,7 7,6 7,7	7,2 7,3 8,5 8,0	10,8 13,1 15,2 2,9
VII 25 26 27 28	Brown soil (arable)	U/38 Kraków — Cegielnia	5-20 40-50 ~ 500 ~ 650	0,0 0,0 7,1 4,7	6,0 6,3 7,2 7,2	6,5 7,1 8,2 8,4	10,8 17,0 13,2 17,7
VIII 29 30	Degraded chernozem (arable)	U/39 Słomniki	5-15 30-40	0,0 0,0	6,4 6,2	7,0 6,9	19,0 18,3
IX 31 32 33 34 35	Podsolich soil (arable)	R/471 Przybyszówka (Rzeszów Region)	5-15 20-35 70-80 ~ 250 ~ 350	0,0 0,0 0,0 0,0 9,7	4,9 5,0 4,7 4,8 7,1	6,1 6,2 6,3 5,9 8,0	8,9 8,5 20,9 14,3 13,8

Nos. of samples	Type of soil and mode of utilization	No. of profile and place	Depth in cm	CaCO ₃ %	pH KCl	pH H ₂ O	Content of fraction <0.001 mm %
X 36	Degraded chernozem (arable)	Jar 446	5—15	0,0	6,5	6,8	11,5
37		Zadąbrowie	30—45	0,0	6,4	6,7	14,9
38		(Jarosław Region)	70—80	0,0	6,0	6,5	23,9
XI 39	Brown soil (pasture)	P/543	5—15	0,0	6,9	7,3	17,0
40		Nehrybka	35—45	0,0	6,8	7,1	20,2
41		(Przemyśl	75—85	9,0	7,3	7,7	14,8
42		Region)	~ 1000	0,0	7,1	7,3	15,9
XII 43		U/50 Brochocin (Trzebnica Region)	250—260	7,7	7,3	8,2	11,9

parate horizons. Kaolinite and quartz occur in all profiles as admixtures.

Attention should be drawn to the fact that a fairly common finding is the presence of minerals of a mixed structure, i.e. minerals which consist of alternate layers of illite and montmorillonoid. The investigations of Schroeder (13) on the loess of Germany and of Pavel (10) on the loess of Czechoslovakia, revealed the occurrence of similar illite-montmorillonoid complexes. Our investigations (11) point to the presence of an analogous complex in other silty formations of Poland.

Siedleckij (14) found a great variety of clay minerals in the loess which occurs in the basin of the river Don. He concludes that only eolian deposits can exhibit such great differences both in mineralogical composition and geographical distribution. It results from the studies of Scheffer, Meyer and Kalk (12) on the loess of southern Saxony, and of Stefanovits (15) on the Hungarian loess that the predominant mineral of these soils is illite.

The data contained in Table 2 suggest some tendency toward regularity in the distribution of clay minerals both in the profiles and in the separate areas where loess occurs.

In the profiles of the loess soils of the Lublin Upland, interstratified illite and montmorillonoid usually constitute the predominant mineral; montmorillonoid predominates in the parent rock. The loess soils and rock of the Małopolska Upland and of the Carpathian Foreland often contain illite as the main mineral. In some profiles the content of kaolinite decreases in deeper layers. The content of quartz in the clay fraction is usually small.

Table 2. Mineralogical composition of fraction < 0,001 mm of soils formed from loess.

Nos. of samples	Type of soil	No. of profile and place	Depth in cm	Mineralogical composition of fraction < 0,001 mm		Remarks
				Predominant minerals	Accompanying minerals	
I — 1 2 3 4	Young brown soil	U/46 Kazimierz	5—15	interstratified complex of illite and montmorillonoid	montmorillonoid, kaolinite, quartz	Small content of quartz
			25—35			
			~ 400	montmorillonoid	illite (partly interstratified with montmorillonoid), kaolinite, quartz	
			~ 1200			
II — 5 6 7 8	Brown soil	U/47 Lublin-Rury	5—20	interstratified complex of illite and montmorillonoid	kaolinite, quartz	Considerable admixture of kaolinite
			40—50			
			180—200	montmorillonoid	illite, kaolinite, quartz	
			~ 800			
III — 9 10 11 12 13	Brown soil, buried	U/40 Szezebrzeszyn	0—10	montmorillonoid, illite	kaolinite, quartz	Small content of quartz
			50—60			
			460—470	interstratified complex of illite and montmorillonoid		
			480—495			
			530—540			
IV — 14 15 16 17	Degraded chernozem	U/94 Hrubieszów	5—15	interstratified complex of illite and montmorillonoid	kaolinite, quartz	
			30—40			
			90—100	montmorillonoid	illite, kaolinite, quartz	
			~ 300			
V — 18 19 20	Degraded chernozem	U/35 Lipnik (Sandomierz Region)	5—15	illite	interstratified complex of illite and montmorillonoid, kaolinite, quartz	Small content of quartz
			30—40			
			90—100	montmorillonoid	kaolinite, illite, quartz	

VI-21 22	Brown soil	U/36 Sandomierz	5-15 30-40	illite	interstratified complex of illite and montmorillonoid, kaolinite, quartz	Small content of kaolinite, very small content of quartz
23 24			~ 300 ~ 1500		interstratified complex of illite and montmorillonoid, montmorillonoid, kaolinite, quartz	Small content of quartz
VII-25 26 27 28	Brown soil	U/38 Kraków — Cegielnia	5-20 40-50 ~ 500 ~ 650	illite	interstratified complex of illite and montmorillonoid, kaolinite, montmorillonoid, quartz	
VIII-29 30	Degraded chernozem	U/39 Słomniki	5-15 30-40	illite	interstratified complex of illite and montmorillonoid, quartz, kaolinite, mica	Great content of quartz
IX-31 32 33 34 35	Podsolich soil	R/471 Przybyszówka (Rzeszów Region)	5-15 20-35 70-80 ~ 250 ~ 350	interstratified complex of illite and montmorillonoid illite	kaolinite, quartz interstratified complex of illite and montmorillonoid, kaolinite, quartz	Small content of quartz
X-36 37 38	Degraded chernozem	Jar/446 Zadąbrowie (Jarosław Region)	5-15 30-45 70-80	illite	montmorillonoid, kaolinite, quartz	The content of kaolinite decreases with the increasing depth
XI-39 40 41 42	Brown soil	P/543 Nehrybka (Przemyski Region)	5-15 35-45 75-85 ~ 1000	illite, montmorillonoid	kaolinite, quartz	The content of illite increases and the content of kaolinite decreases with the increasing depth.
XII-43		U/50 Brochocin (Trzebnica Region)	250-260	interstratified complex of illite and montmorillonoid	kaolinite, quartz	

Remarks: 1) Montmorillonoid is an atypical montmorillonite, probably of the beidelite or nontronite type
2) The structure of kaolinite is not completely ordered, so that it approaches the fireclay type.

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Р Е З Ю М Е

Для исследования избраны 12 профилей (43 пробы) из почв, находившихся в разной стадии развития, отобранных в разных лёссовых районах Польши. Эти профили взяты из т. н. глубоких лёссов т.е. из районов покрытых мощным лёссовым покровом. Четыре из указанных профилей происходят из Люблинской возвышенности, два из Сандомирской возвышенности, два из Краковско-Ченстоховской возвышенности, три из Предкарпатия и один из Тшебницкой гряды.

Большинство почвенных профилей представляет буроземы и черноземы, остальные — подзолистые и ископаемые почвы. Они происходят, главным образом, из похотных почв, но некоторые также из-под древесных покровов и задернованных почв.

Исследования проводились в пределах фракции меньше 0,001 мм. Эта фракция выделена методом седиментации по Г о р б у н о в у, после предварительного удаления из проб обменных катионов промыванием дистиллированной водой (декантацией). В случае присутствия карбонатов они удалялись 0,2% HCl. Органическое вещество окислялось 6—8% H₂O₂.

Перед рентгенографическим анализом из приготовленного материала удалялись свободные окислы железа: часть пробы насыщалась магнием и глицеролом, вторая часть калием и производилось прокалывание в температуре 600°С. Таким путем можно получить лучше рентгенограммы и легче определить некоторые минералы, особенно со смешанными структурами. Для облегчения сравнения термических кривых все пробы, предназначенные к термическому анализу, насыщались одинаковым катионом (магнием), а также приводились к одинаковой влажности.

При исследованиях глинистых минералов в почвах применялся порошковой рентгенографический метод по Д е б е й - Ш е р р у и термический анализ.

Для рентгенографических исследований использовался микроструктурный рентген микрометра с малой камерой (64 мм.) и с медной анодной лампой. Пробы в желатиновых капиллярах (диаметра 0,4 мм.) экспонировались в течение 1,5 часа. Для термических анализов

употреблялась аппаратура Павеля, основанная на быстром методе Берга и Рассона (рост температуры печи почти линейный со скоростью около 100° Ц/мин.).

Результаты исследований представлены в табл. 1 и 2.

Из проведенных исследований можно высказать следующие заключения:

1. В почвах, образовавшихся из лёссов, содержится иллит, монтмориллоноид, а также минералы со смешанной иллитомонтмориллоноидовой структурой, и кроме того каолинит и кварц.

2. Главными минералами являются иллит или монтмориллоноид или же их интерстратифицированный комплекс.

3. В почвенных горизонтах лёссов Люблинской Возвышенности преобладающим минералом является обычно интерстратифицированный иллит с монтмориллоноидом, а в материнской горной породе — монтмориллонит.

4. Почвы и лёссовые отложения Люблинской Возвышенности, а также Предкарпатия содержат часто как главный минерал иллит.

STRESZCZENIE

Do pracy wybrano 12 profilów (o łącznej liczbie 43 próbek), reprezentujących gleby w różnym stadium rozwoju i pochodzących z różnych obszarów lessowych kraju. Profile te dotyczą tzw. lessów głębokich, tj. terenów o dość znacznej miąższości pokrywy lessowej. Cztery profile pochodzą z Wyżyny Lubelskiej, dwa z Wyżyny Sandomierskiej, dwa z Wyżyny Krakowsko-Częstochowskiej, trzy z Przedgórze Karpackiego oraz jeden ze Wzgórz Trzebnickich. Większość profili reprezentuje gleby brunatne i czarnoziemy, reszta przypada na gleby biellicowe i gleby kopalne. Przeważnie pochodzą one z terenów uprawnych, ale reprezentowane są również gleby zadrzewione i zadarnione.

Badania przeprowadzono w obrębie frakcji mniejszej od 0,001 mm. Frakcję tę wydzielono metodą sedymentacji według Gorbunowa, po uprzednim usunięciu z próbek kationów wymiennych przemywaniem wodą destylowaną (przez dekantację). W przypadku obecności węglanów usuwano je na wstępie 0,2% HCl. Substancję organiczną w próbkach próchnicznych spalano 6—8% H₂O₂.

Przed analizą rentgenograficzną usunięto z przygotowanego materiału wolne tlenki żelaza, część próbek nasycano magnezem oraz glicerolem, a drugą część potasem i wyprażono w temperaturze 600°C. Dzięki tym zabiegom uzyskuje się lepsze rentgenogramy oraz łatwiej zidentyfikować niektóre minerały, zwłaszcza o mieszanych strukturach.

Dla ułatwienia porównania krzywych termicznych nasyceno wszystkie próbki do analizy termicznej jednakowym kationem (magnezem) oraz doprowadzono je do jednakowej wilgotności.

Do badania minerałów ilastych w glebach stosowano metodę rentgenograficzną proszkową według Debye-Scherrera i analizę termiczną.

W analizie rentgenograficznej posługiwano się mikrostrukturalnym rentgenem Mikrometa z małą komorą (\varnothing 64 mm) i lampą z miedzianą anodą. Próbki w kapilarach żelatynowych (o średnicy 0,4 mm) ekspozowano przez 1,5 godziny. Do analiz termicznych użyto aparatury Pavla, opartej na szybkiej metodzie Berga i Rassonskiej (wzrost temperatury pieca prawie liniowy z szybkością około $100^{\circ}\text{C}/\text{min.}$).

Wyniki badań przedstawione są w tab. 1 i 2.

Z przeprowadzonych badań można wyciągnąć następujące wnioski:

1. W glebach wytworzonych z lessów występuje illit, montmorillonoid oraz minerały o strukturze mieszanej illitowo-montmorillonoidowej, a ponadto kaolinit i kwarc.

2. Minerałami głównymi są illit bądź montmorillonoid, bądź ich interstratyfikowany kompleks.

3. W poziomach glebowych lessów Wyżyny Lubelskiej minerałem przeważającym jest zazwyczaj interstratyfikowany illit z montmorillonoidem, w skale zaś macierzystej — montmorillonoid.

4. Gleby oraz skały lessowe Wyżyny Małopolskiej, a także Przedgórze Karpackiego zawierają często jako minerał główny illit.

