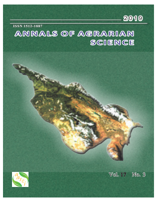
Annals of Agrarian Science

Journal homepage: http://journals.org.ge/index.php



Laser diffraction spectra's deconvolution in the study of soil texture

Yu. N. Vodyanitskii[a], A.T. Savichev[bc], E.G. Morgun[a]

[a]Lomonosov Moscow State University, Faculty of Soil Science, 1, Leninskiye Gory, Moscow, 119234, Russia
[b]Geological Institute, Russian Academy of Sciences, 7, Pyzhevskiy Per., 119017, Moscow, 119017, Russia
[c]Dokuchaev Soil Science Institute, 7, Pyzhevskiy Per., 119017, Moscow, 119017, Russia

Received: 06 January, 2020; accepted: 07 February, 2020

ABSTRACT

The deconvolution procedure for separating the original particle size distribution spectrum by laser diffraction (LD) gives a different share of the fractions compared to the common method, when the number of particles is determined in the vertical walls at the group boundaries according to the FAO classification or other classification. In addition, a new indicator provides information on the properties of individual fractions by particle size: the LD fractions dispersion (D). Vertisols have a harder texture after deconvolution, especially in the Stavropol Vertisols, and the less extent in the Texas Vertisols. Vertisols have relatively low dispersion $D < 0.7$, which means relatively homogeneity of LD-fractions. Andosols dispersion (D) is very high; as a result of small number LD fractions, less than the number of FAO fractions. Very high D values can be attributed to preservation aggregates due to incomplete dispergation of soil. A strong disturbance in the distribution of LD-fractions and a high degree of their superposition makes it possible to assume that micro aggregates will remain in them after not adequate chemical preparation for LD analysis.

Keywords: Deconvolution procedure, Splitting of LD-spectrum, Soil texture, Vertisols, Andosols. Dispergation of soil.

*Corresponding author: Yuri Vodyanitskii; E-mail address: yu.vodyan@mail.ru

Introduction

At present, there are two common methods for analyzing the grain size composition of soils and sediments. The older method, a sieve analysis combined with a thin-particle pipette analysis, is based on determining the mass fraction of individual fractions [1]. Later, a method of laser diffraction analysis (LDA) was developed, based on the determination of the volume part of fractions [2]. Laser diffractometer provides fast analysis and does not require a greater sample mass. Another important advantage of LDA is to obtain an almost continuous spectrum of particle content.

The main task of any analysis of grain size, including LDA, is to determine the texture of the soil / sediment, that is, the proportion of each fraction, expressed as a percentage. Is this problem solved now, in relation to the LDA?

Now the concept of an "impenetrable vertical" boundary between fractions is conditional. So, we see that the usual schemes for decrypting LD-spectra entail serious errors due to the assumption of vertical barriers on the border between fractions [3-5]. But in reality, the particles are distributed statistically, forming a series of independent sets, as a rule, in accordance with the law of normal distribution [2, 6]. Therefore, between the boundaries of the factions taken in any classification, there are no rigid vertical boundaries.

To circumvent the error in determining the soil / sediment texture, a different approach to deciphering continuous LD-spectra is needed.

Objective: to propose a universal methodology for decrypting LD-spectra.

Characteristics of LD-fractions

Nine classic size fractions of the dominant particles with different mineralogy are allocated in the range 0-2000 μm according to FAO Classification. Thus, each of the groups: clay (C), silt (Si) and sand (S) are subdivided into 3 fractions: fine (f), medium (m) and coarse (c), total 9 classic fractions. Each fraction is marked by the index (Table 1). Usually, the each FAO-fraction content is determined by area under the LD-curve limited by vertical walls at the borders between the factions. In other words, content of FAO-fraction (S_{FAO}) is determined by the area (S), limited by top LD-spectra, and the side verticals in the points with the low and high limits fraction in a classification. For example, the contents cSi fraction is determined by the vertical walls at 20 and 63 µm.

But in fact, using the vertical boundaries between the FAO fractions is a very blatant admission. The normal distribution of the particles does not take into account the specified vertical borders between the fractions. As this problem is solved when analyzing soil LD-spectra?

The simplest form of statistical particles distribution is described by gauss form [7]. The meaning of deconvolution (with respect to decoding the energy dispersive X-ray fluorescence spectrum) is described by Savichev and Stepanov [7] and in relation to texture composition, defined by the LDA, is described earlier [8].

The main indicator of particle size distribution is the content of some fraction in the soil. It is possible to solve the problem of grain-size composition characteristics using the maximum position of LD-fraction on the axis diameter: $d_{max} = d_{average}$, placing it between the fraction bounds. In other words, we will name the LD-fraction, on the basis of the conditions: $d_1 < d_{average} < d_2$, where d_1 and d_2 are the lower and upper grain diameter of this fraction. For a precise definition of the centre faction $d_{average}$ is need to “deconvolute” or to split LD-spectrum.

Since laser diffraction is not able to detect fine clay of 0–0.2 µm, we use the eight fractions in the calculations. Thus, the average diameter ($d_{average}$) of the LD-fractions can be used to identify LD-fractions by placing it in the classical borders of the 8 groups (clay, silt and sand) of FAO Classification [1].

It is obvious that the vertical borders between the fractions are an abstraction, not taking into account the statistical grain probability distribution within the fraction. This assumption would be true if the minima were reduced to zero. But, as can be seen from Figures 1-3, particle content is never reduced to zero.

The main indicator of LD-fraction is the position of its center on the axis of diameters: $d_{average}$. The second indicator is the area under the Gauss-line, which defines the share fraction S_{LD} in %. The third indicator is the fraction dispersion – D.

The deconvolution procedure allows solving two problems. First, it is possible to clarify the usual indicators, which are determined from the initial dependence of the particles on their diameter. In addition, the share of neighboring fractions can be distorted due to their superposition on the initial curve. The overlap effect is revealed as a result of the deconvolution of the original spectra. At the same time, it is possible to correct the ratio between the LD-fractions.

Secondly, perhaps the most important advantage of deconvolution is the ability to study the fraction dispersion D. If each fraction were completely separated from the neighboring fractions, then on the initial curve the minimum concentration would decrease to zero. Usually this does not happen, which means the superposition of fractions. It is clear that the greater the dispersion D, the lower its homogeneity and the higher the likelihood of the presence of microaggregates.

Dispersion D is lower in fully isolated fractions without superposition (a partial contribution of particles of a given fraction to the content of particles of neighboring fractions) and increases with an increase in the effect of superposition. We calculated the dispersion of completely isolated fractions on the basis of the ideal distribution of the grain size fractions of rocks [6]. Their dispersion is low D = 0.26-0.38, on average 0.32. This value is taken as the reference dispersion of completely isolated LD-fractions.

High values D > 1 can be associated with two factors. Stable particles are considered the first cause of the weak weathering fraction. The second reason is to save some micro-aggregates before LD-analysis.

Table 1. *Index and borders (µm) between the soil size fractions [1]*

Index	fC	mC	cC	fSi	mSi	cSi	fS	mS	cC
d, µm	0-0.2	02-0.63	0.63-2.0	2.0-6.3	6.3-20	20-63	63-200	200-630	630-2000

Objects and Methods

1. *Vertisols from different regions*. We studied two soil complexes with gilgai microrelief in the temperate and subtropical climates. The various elements of the microrelief differ significantly in the amount of precipitation flow.

Vertisol soil complex of temperate climate was described and sampled in the southeastern European part of Russia (44°38’17”N, 42°15’04” E) in the Stavropol region, North Caucasus. Soils are formed on marine clay under native steppe vegetation. According to Soil Taxonomy [9], this soil complex consists of Sodic Haplusterts (microhigh) and Typic Epiaquert (microlow).

The second Vertisol soil complex is located in the southern part of the United States, in Texas. 12-m-long trench has been formed on the second terrace of the Brazos River, Burleson County, near city of College Station (30°29’21”N, 96°28’44”W). The soils are developed from clayey alluvium, consisting of derivatives of the red-earth Permian and Triassic clays. The soils have been classified as Typic Haplusterts (microhigh) and Udic Haplusterts (microlow).

Grain size soil composition was determined on the laser diffraction particle size Analyzer “Sizer Analysette 22 comfort” (FRITSCH, Germany). Before analysis soil samples in the form of suspension were treated by ultrasound without chemical processing. The original soil samples are mixed with a rubber pestle and sieved through a 0.25 mm sieve. The hitch (0.10-0.13 g) was added 30 ml of distilled water and soil was treated by ultrasonic dispenser Digital Sonifir 250 (Branson Ultrasonics) with a dispersive element probe type, operating on a frequency of 20 kHz, in non-impulse mode, the energy of ultrasound – 450 J/ml.

2. *Andosols. (Southern Italy)*. We studied recent soils and paleosols in quaternary fluvio-lacustrine sediments in the basin of Bozhano in Southern Italy, described by Colombo [4]. Recent soils and paleosols were formed as a result of pedogenesis on the products of interaction of pyroclastic material with alluvial clay sediments, some of which are enriched in carbonates.

Recent soils are called andosols, their thickness is 1.5 m. The parent rocks C-1 and C-2 at depths of 80-150 cm are strongly enriched in carbonates: the calcite content reaches 70-77%. Below, up to a depth of 11 m, paleosols are opened, which according to morphology are divided into four solums: Solum I-IV. Their parent rocks are lake sediments.

Before analyzing the grain size of andosols, to dispergate microaggregates, samples < 2 mm were treated with H_2O_2 to oxidize organic matter, and then with dithionite-citrate-bicarbonate (DCB) to dissolve ferrous cement. In samples of carbonate rocks underlying recent soil (samples RS-CI and RS-C2), carbonates were removed by treatment with Na acetate, buffered at pH = 5 [4].

Particle size distribution was determined using an “Analysette 22 comfort” laser diffraction particle size analyzer (FRITSCH, Germany). The device uses the method of “reverse Fourier optics”, this is a system of a converging laser beam (helium is a neon laser with a wavelength of 632.8 nm). Soil samples were sonicated before analysis in the form of a suspension without additional chemical treatment [4].

After this preparation, the soil composition was determined by laser diffraction on a Malvern Mastersizer 2000 analyzer. The analysis parameters are the following: the pump speed is 2500-3000 rpm, the number of measurements is 6-10, the refractive index is 1.52, and the absorption index is 0.1 [4]. The particle size distribution was obtained using the full Mie scattering function for spheres, which provides a more accurate estimate of the particle size in the clay fraction than the Fraunhofer function [10].

The obtained particle distribution spectra were interpreted by deconvolution. Then a LD-fraction share as area of elementary contour obtained after deconvolution (S_{LD}) was compared with a share of FAO-factions (S_{FAO}). The most important fractions of soil/sediment difference was estimated: $\Delta S = S_{LD} - S_{FAO}$ for clay (<2 µm).

Results and discussion

Characteristics of the of Stavropol Vertisols’s LD-fractions

The content of the LD fractions differs from the content of the FAO fractions and it was calculated according to the old method, assuming that the vertical walls at the fractions boundaries (Fig. 1). Deconvolution procedure showed the Vertisol heavier texture. Clay content of LD-fractions with average diameter < 2 microns is increased to 10-11% in comparison with the calculation based on vertical borders between particles FAO-fractions.

The homogeneity of the particle size fractions was evaluated by the values of their dis-persions. The dispersion data of the Gaussians of each of the fractions are shown in Table 2.

In Vertisols of temperate climate common regularities were observed: the homogeneity of particles of fine fractions is higher than that of large fractions. Thus, the dispersion D is 0.50 for the three thinnest fractions, and D is 0.60–0.63 for the largest fractions. From this it follows that the coarse fractions are less homogeneous than the fine ones. Overall uniformity of LD-fractions of the Vertisols is high, because D < 0.7.

Characteristics of the Texas Vertisols LD-fractions

The LD-fractions contents are different from contents of FAO-fractions, calculated under the old methodology, assuming vertical walls at the fractions borders (Fig. 2). Deconvolution procedure is showed the Vertisol heavier texture. Clay content of LD-fractions with an average diameter < 2 microns is increased to 7-8% in comparison with the FAO-fractions (Table 2).

In the Texas Vertisols the homogeneity of clay and silt fractions is lower than in Vertisols of Stavropol region. The relatively high dispersion D of fine fractions of Texas Vertisols reflects their lower weathering compared to Vertisols of Stavropol region. Probably, in Texas Vertisols, the absence of coarse silt is explained by the lithological peculiarities of the parent material, but not by the preservation of aggregates after preparing the soil for laser diffraction.

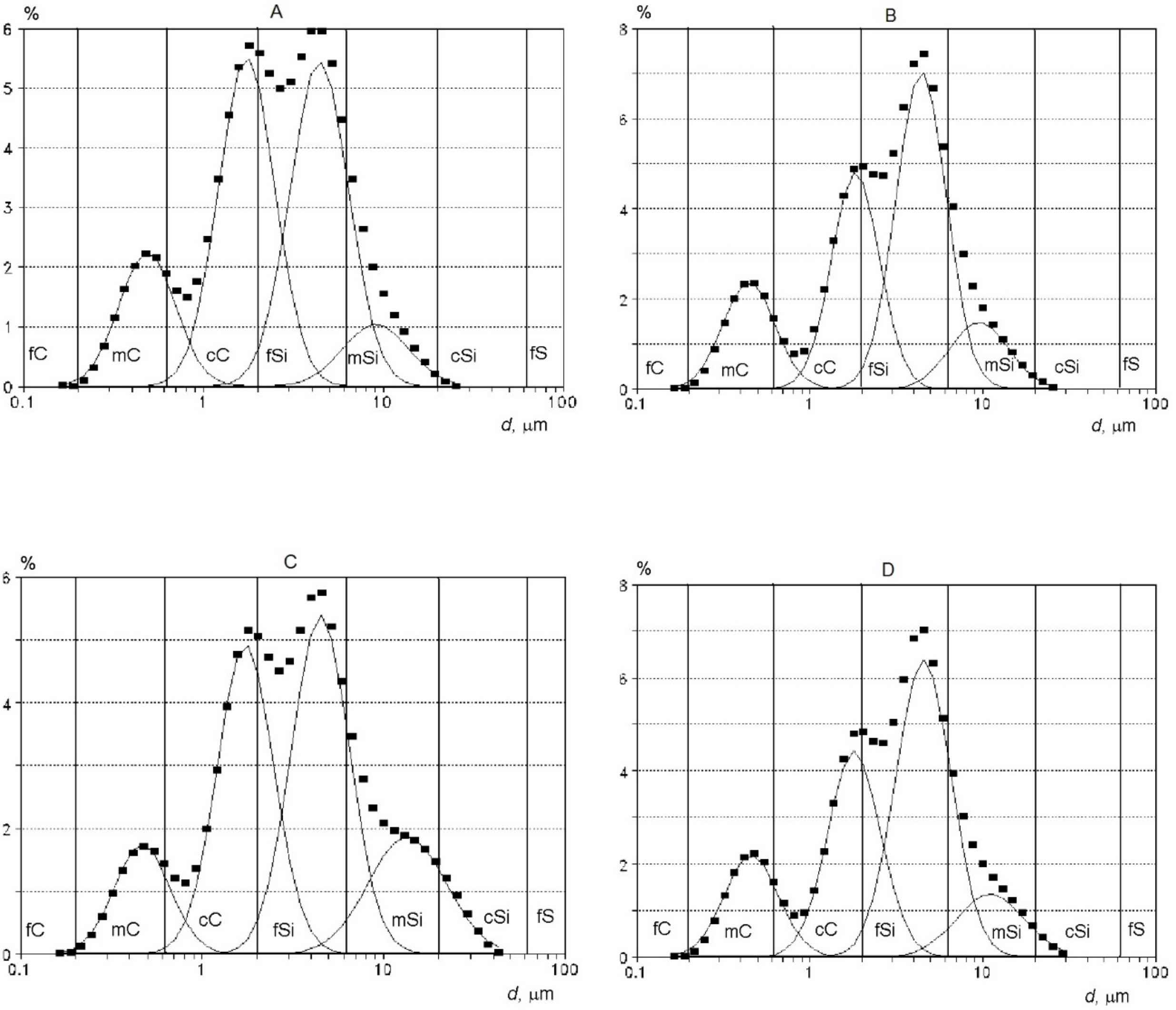


Fig. 1. *The distribution of particle diameters (Stavropol, Vertisols). The points are the initial differential distribution; thin lines are elementary contours. A- microhigh, AU-horizon; B - microhigh, Cv-horizon; C - microlow, AU-horizon; D - microlow, Cv-horizon.*

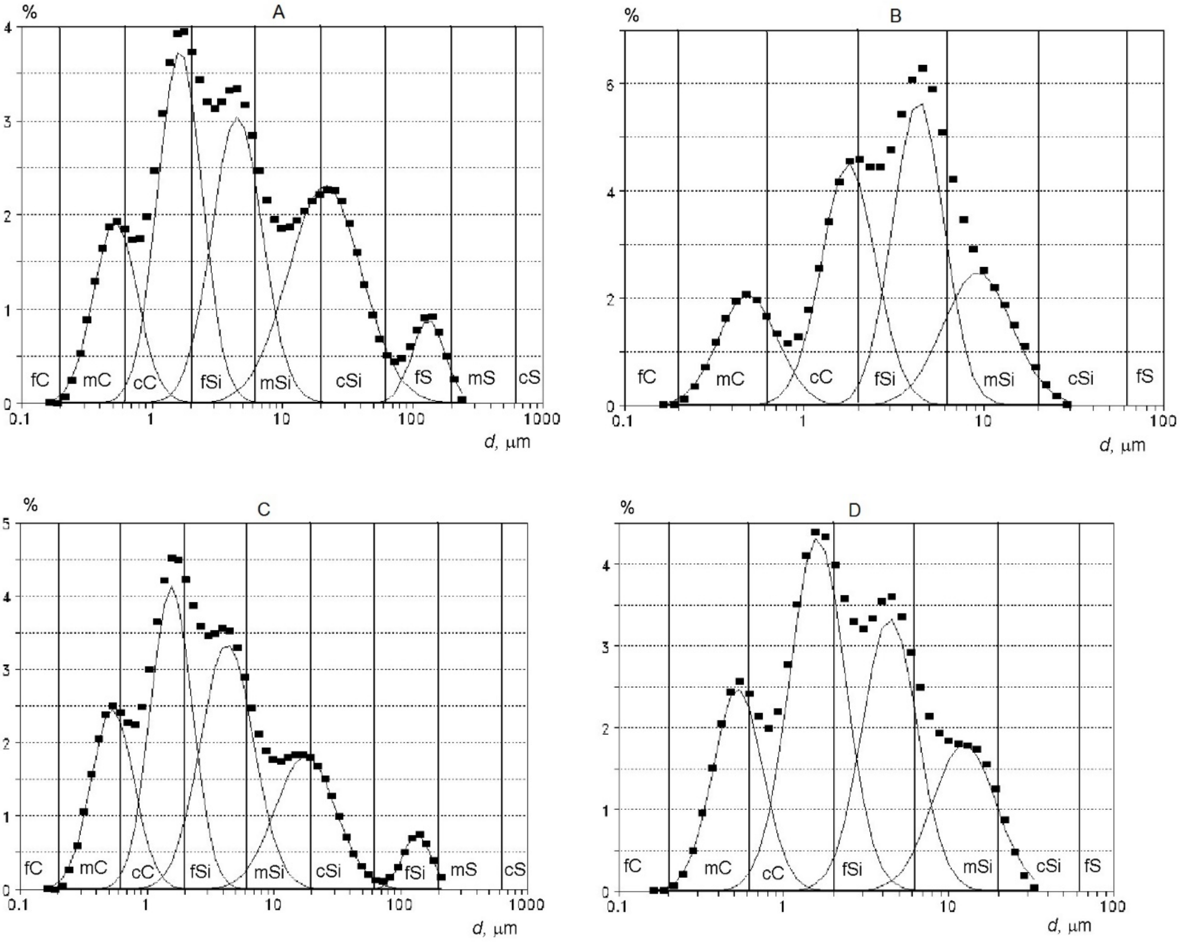


Fig. 2. *The distribution of particle diameters (Texas, Vertisols). The points are the initial differential distribution; thin lines are elementary contours. A - microhigh, AUca-horizon; B - microhigh, Cv-horizon; C - microlow, AUca-horizon; D - microlow, Bca-horizon.*

Table 1. *Index and borders (μm) between the soil size fractions [1]*

Group (μm)	Share S_{FAO}, %	After deconvolution			Share S_{FAO}, %	After deconvolution		
		Share S_{LD}, %	*d*, μm	*D*		Share S_{LD}, %	*d*, μm	*D*
Stavropol, Vertisols								
	Microhigh, AU-horizon				Microhigh, Cv-horizon			
mC (0.2-0.63)	12.9	15.3	0.49	0.51	13.7	15.6	0.44	0.49
cC (0.63-2.0)	28.4	37.7	1.73	0.51	20.6	29.1	1.84	0.45
fSi (2.0-6.3)	47.2	38.7	4.39	0.53	52.1	44.4	4.39	0.47
mSi (6.3-20)	11.3	8.3	9.02	0.60	13.4	10.9	13.6	0.64
ΣC, %	41.3	53.0	Δ=11.7		34.3	44.7	Δ=10.4	
	Microlow, AU-horizon				Microlow, Cv-horizon			
mC (0.2-0.63)	5.5	6.1	0.46	0.49	13.0	14.9	0.43	1.38
cC (0.63-2.0)	17.2	25.4	1.80	0.50	20.9	29.6		

fSi (2.0-6.3)	39.1	32.6	4.53	0.48	50.0	44.5		
mSi (6.3-20)	27.0	14.4	10.7	0.58	15.6	11.6	8.5	1.44
cSi (20-63)	11.0	21.8	22.2	0.65				
ΣC, %	22.7	31.5	Δ=8.8		33.9	44.5	Δ=10.6	
Texas, Vertisols								
	Microhigh, AUca-horizon				Microhigh, Cv-horizon			
mC (0.2-0.63)	11.2	14.1	0.53	0.55	12.3	14.7	0.49	0.53
cC (0.63-2.0)	23.5	27.6	1.65	0.55	21.9	29.6	1.77	0.49
fSi (2.0-6.3)	28.7	25.6	4.53	0.63	46.9	35.0	4.31	0.46
mSi (6.3-20)	18.5				18.6	20.6	9.21	0.62
cSi (20-63)	12.2	28.1	21.3	0.91				
fS (63-200)	5.7	4.9	133.0	0.41				
ΣC, %	34.7	41.7	Δ=7.7		34.2	44.3	Δ=10.1	
	Microlow, AUca-horizon				Microlow, Bca-horizon			
mC (0.2-0.63)	14.0	18.0	0.54	0.55	15.8	18.7	0.53	0.49
cC (0.63-2.0)	27.9	29.3	1.57	0.53	30.3	36.5	1.60	0.55
fSi (2.0-6.3)	31.0	30.4	4.31	0.68	34.5	27.7	4.39	0.54
mSi (6.3-20)	16.9	18.7	17.8	0.77	18.0	17.2	12.4	0.62
cSi (20-63)	6.4				1.3			
fS (63-200)	3.4	3.7	134.0	0.37				
ΣC, %	41.9	47.3	Δ=5.4		46.1	55.2	Δ=9.1	
Italy, Andosols								
	Recently soil, RS-Bw1-horizon				Recently soil, RS-C-1			
mC (0.2-0.63)	2.2				2.1			
cC (0.63-2.0)	10.4				10.0			
fSi (2.0-6.3)	17.0	35.2	2.1	1.52	16.8	33.4	3.0	1.52
mSi (6.3-20)	25.1				19.8			
cSi (20-63)	29.1	59.6	27	1.24	17.4	31.4	21	1.24
fS (63-200)	9.4				19.9	29.5	129	0.96
mS (200-630)	6.1	7.0	372	0.68	13.1	7.3	412	0.62
ΣC, %	12.6	0.0	Δ=-12.6		12.1	0.0	Δ=-12.1	
	Paleosols, SI-1-horizon				Paleosols, SIV-3			
mC (0.2-0.63)	3.5				2.7			
cC (0.63-2.0)	19.9				15.3			
fSi (2.0-6.3)	34.4	80.3	3.6	1.46	23.9	53	3.1	1.52
mSi (6.3-20)	24.0				23.1			
cSi (20-63)	11.9	16.6	29.3	1.18	21.6	44	30.0	1.40
fS (63-200)	2.9				9.1			
mS (200-630)	2.9	6.1	428	0.70	3.7	3	388	0.52
ΣC, %	13.4	0.0	Δ=-13.4		18.0	0.0	Δ=-18.0	

Characteristic of the South of Italy Andosols LD-fractions

The main features of Andosols LD-fractions. The LD-fractions contents are different from contents of FAO-fractions. In addition, it is not individual LD-clay fractions, with an average diameter of <2 µm (Fig. 3).

Clay particles are allocated traditionally on vertical limits of only 13%. But in fact clay particles are belonging to "tail" of fine silt LD-faction. The "tail" of this faction is very long, as evidenced by the unusually high the fine silt fraction dispersion D = 1.5 (Table 2).

Secondly, there is no independent fine sand LD-fraction 63-200 microns. A small fine sand content by traditional analysis (<1%), is the result of superposition "tails" of neighbor large silt and medium sand LD-fractions.

Thus, grain size deconvolution is reduced drastically the number of LD-fractions by excluding as independent fractions, consisting long LD-fractions tails. The reason is the very high LD-fractions dispersion D = 1.5. Smooth (absent-mindedness) of grain size fractions can be linked to two factors: 1) weak weathering of minerals; 2) preservation aggregates due to incomplete dispersion of soil. The first reason is possible if there are many resistant to weathering particles of feldspar and quartz. Meanwhile, Andosols are dominated by X-ray amorphous minerals (allofanes and imogolites), distinguished by extremely low resistance. Therefore, the first assumption about the reason of weak weathering minerals for andosols is not suitable.

The second assumption remains an abnormally high dispersion, that indicates the preservation of micro-aggregates. One possible reason is preservation of aggregates due to incomplete dispersion of soil. This suggests the probable conservation of microaggregates after preparing the soil for laser diffraction.

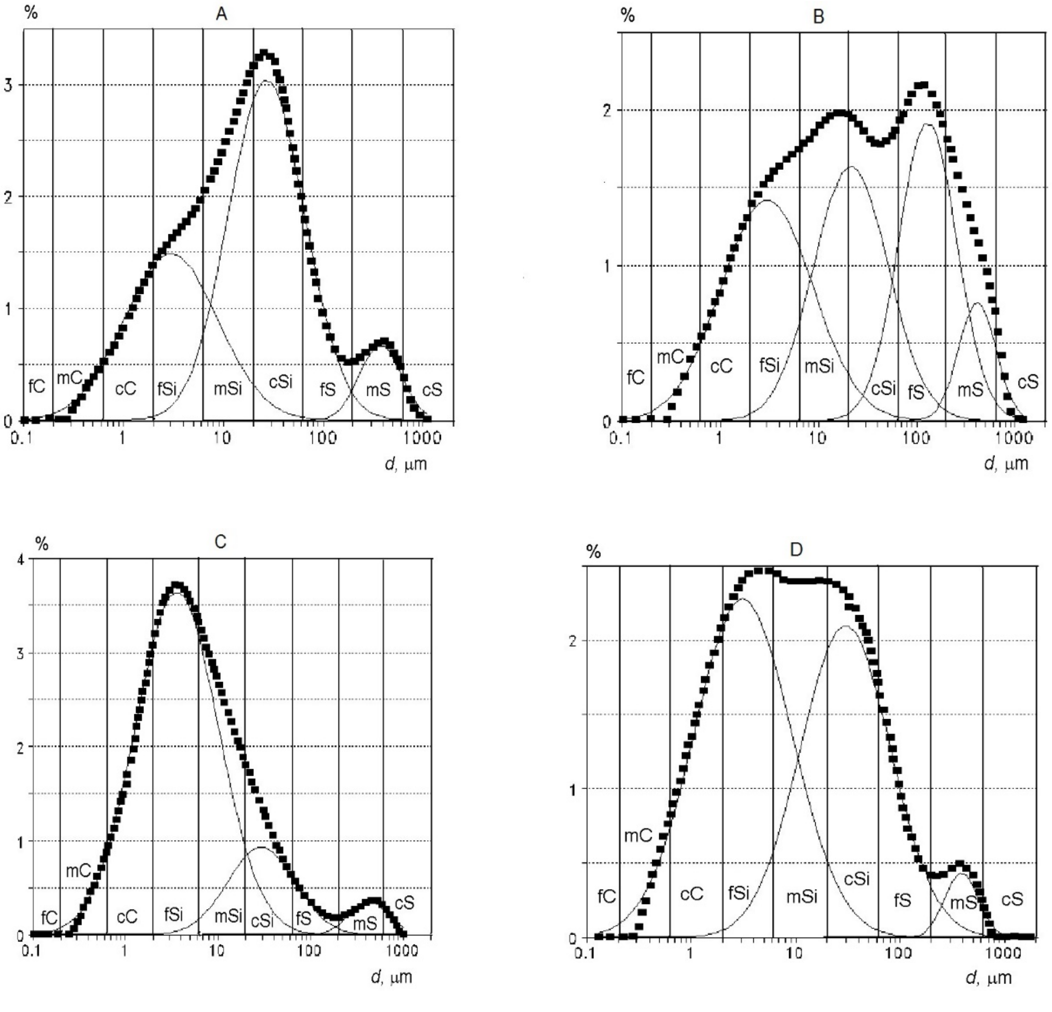


Fig. 3. *The distribution of particle diameters (Italy, Andosols). The points are the initial differential distribution; thin lines are elementary contours. A - recently soil, RS-Bw1; B - Recently soil, RS-C-1; C - paleosols, SI-1; D - paleosols, SIV-3.*

The mineralogical composition of Andosols in a neutral medium is determined by the hydrolysis of volcanic glass [11]. This leads to the formation of typical minerals of Andosols: clay minerals (allophane and imogolite) and Fe-hydroxide: ferrihydrate $2Fe_2O_3 \cdot FeOOH \cdot 4H_2O$) [11]. The role of mineral cement in Andosols is performed by active particles of allophone, imogolite and ferrihydrate, connected with organic matter [12].

Allophane and imogolite are nano-sized aluminosilicate with hollow spherule and tube structures with the diameter 3-5.5 nm; the minerals have high cation/anion exchange capacity as well as extensive, variable-charged surfaces [12, 13]. Together with Fe-containing minerals of short-range order, which can easily dissolve and precipitate during oxidation-reduction changes, these nano-sized minerals of short-range order and the organic matter associated with them can act as a strong binding agent for the formation of aggregates [14]. In addition, Al-organic (and, to less extent, Fe-organic) complexes formed via covalent bonding between monomeric Al and Fe ions with organic functional groups are also relatively abundant in Andosols [12, 13].

DCB effectively dissolves Fe-cement in strongly ferruginized soils, for example, in ferrallitic soils [15]. Feature of these soils is high content of gross iron, much more above the Clarke of the lithosphere - 6.2% [16]. But the total iron content in the Italian Andosols and paleosols is very low: 0.7-2.8%. This already indicates a weak ferrigination of Andosols. The most of thin microaggregates of Andosol have very high strength. It is obvious that DCB is not able to completely destroy solid micro-aggregates of Andosols.

A thorough study using the fine absorption structure of near X-rays showed that organic matter forms a weak bond with the smooth surface of large crystals [13]. This explains the reason not to attend such minerals like kaolinite, hematite, goethite in the formation of micro-aggregates in Andosol. But organic matter is formed a strong connection with unordered minerals such as allophone, imogolite and ferrihydrite [13].

Colombo [4] randomly used a reagent (DCB) that is not very selective reagent to Andosols. But this allowed us to obtain new information on the composition of soil microaggregates. In the future, it is possible to apply deliberately soft processing to obtain information about firmly connected soil microaggregates, not only in Andosol.

Conclusion

The deconvolution procedure for splitting the original spectrum of particle size distribution gives another fractions share compared with usual method, when the number of particles is determined within the vertical walls at the borders of groups according to the FAO classification or other classification. In addition, a new indicator provides information on the properties of individual particle size fractions: dispersion of LD-factions (D). Dispersion value ranges from 0.32 in the complete absence of LD-fractions superposition of up to 1.8 from Andosols with very long “tails” of the distribution.

After deconvolution vertisols have the harder texture, especially in the vertisols of Stavropol and, to a lesser extent at Texas vertisols. Vertisols have relatively low dispersion D < 0.7, which means relative homogeneity of LD-fractions.

In Northern Italy Andosols dispersion D of LD-fraction is very high; resulting in the real fraction number is less than the number of FAO-fraction. Very high values of the dispersion can be attributed to preservation aggregates due to incomplete dispersion of soil. A strong disturbance in the distribution of LD-fractions and a high degree of their superposition makes it possible to assume that microaggregates will remain in them after not adequate chemical preparation for laser diffraction, in particular, this applies to paleosols.

Acknowledgement

The authors thank Yu.A. Zhukova and A.V. Yudina for his research assistance.

References

[1] FAO, Guidelines for soil description, 4th ed. Food and Agriculture Organization of the United Nations, Rome, 2006.

[2] M. Konert, J. Vandenberghe, Comparison of laser grain size analysis with pipette and sieve analysis: a solution for the underestimation of the clay fraction, Sedimentology 44 (1997) 523-535.

[3] F. Lehmkuhl, Ph. Schulte, H. Zhao, D. Hüllec, J Protze, G. Stauch, Timing and spatial distribution of loess and loess-like sediments in the mountain areas of the northeastern Tibetan Plateau, Catena 117 (2014) 23–33.

[4] C. Colombo, G. Palumbo, E. Di Iorio, F. Russo, F. Terribile, Z. Jiang, Q. Liu, Soil development in a Quaternary fluvio-lacustrine paleosol sequence in Southern Italy, Quaternary Int. 418 (2016) 195-207.

[5] Ph. Schulte, F. Lehmkuhl, The difference of two laser diffraction patterns as an indicator for postdepositional grain size reduction in loess-paleosol sequences, Paleogeography, Paleoclimatology, Paleoecology 509 (2018) 126-136.

[6] M.A. Sadovsky, L.G. Bolkhovitinov, V.F. Pisarenko, On the properties of the rocks discreteness, Izvestiya of the USSR Academy of Sciences, series of Earth Physics, 12 (1982) 3-18 (in Russian).

[7] A.T. Savichev, S.S. Stepanov, 2007. Allowance for superimposition of lines and approximation of background radiation in X-ray fluorescent and microprobe energy-dispersive analyzes, Surface: X-ray, synchrotron and neutron studies, 2 (2007) 85-89.

[8] Yu.N. Vodyanitskii, E.Yu. Milanovskii, E.G. Morgun, A.T. Savichev, Deconvolution of the differential particle size distribution curves of Vertisols, Eurasian Soil Sci. 52 (7) (2019) 1112-1121.

[9] Soil Survey Staff, Soil Taxonomy, a Basic System of Soil Classification for Making and Interpreting Soil Surveys, USDA Agricultural Handbook, 436 (1999).

[10] G.B. de Boer, C. de Weerd, D. Thoenes, H.W. Goossens, Laser diffraction spectrometry: Fraunhofer diffraction versus Mie scattering, Particle and Particle Systems Characterization 4 (1999) 14–19

[11] Referentiel Pedologique, Inst. Nat. Resch, Agron, Paris, 1995.

[12] M. Asano, R. Wagai, Evidence of aggregate hierarchy at micro- to submicron scale in an allophonic Andosol, Geoderma 216 (2014) 62-74.

[13] M. Asano, R. Wagai, N. Yamaguchi, et al., In search of a binding agent: nano-scale evidence of preferential carbon associations with poorly-crystalline mineral phases in physically-stable, clay-sized aggregates, Soil Systems 2(2), (2018) 32.

[14] K. Alary, D. Babre, L. Caner, F. Feder, M. Szwarc, M. Naudan, G. Bourgeon, Pretreatment of soil samples rich in short-range-order minerals before particle-size analysis by the pipette method, Pedosphere 23(1), (2013) 20-28.

[15] Yu.N. Vodyanitskii, The effect of dithionite-containing reagents on soil minerals, Eurasian Soil Sci. 35(5): (2002) 552-563.

[16] N.N. Greenwood, A. Earnshaw, Chemistry of the Elements, Elsevier Sc. Kidlington, England, 1997.