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The Use of a Laser Diffractometer to Analyze the Particle Size Distribution of Selected Organic Soils

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Abstract: This study was conducted to verify the usefulness of the laser diffractometer method for determining the particle size distribution of selected organic soils from the Podkarpacie region in Poland. The soil selected for this research represented three main classification groups, namely, low-organic, medium-organic and high-organic soil, in accordance with the standard criterion. Particle size distribution was determined using two types of laser diffractometers: the Helos laser diffractometer manufactured by Sympatec GmbH (Clausthal-Zellerfeld, Germany) and the laser particle size analyzer Analysette 22 MicroTech plus manufactured by Fritsch GmbH (Idar-Oberstein, Germany). The standard mechanical and sedimentation methods, which are perfect for testing mineral soils, are not applicable to organic soils; therefore, a serious problem was found and examined. A reference method that could verify the test results obtained using the laser diffractometer methods was required. After analyzing the literature, the hydrometric (sedimentation) method was adopted as the reference method. Currently, there are no reliable and fully verified methods for testing soils with such a complex skeleton structure, and the resources, standards and guidelines concerning the issues discussed are extremely limited; therefore, new research methods are being sought to fill this gap, and this work is a step in this direction. The results of the conducted studies and analyses have shown that laser diffractometry methods can be useful for determining the particle size distribution of organic soils, but to a limited extent, depending mainly on the quantity of organic substances. The highest agreement was obtained by comparing the results of the sedimentation method with those obtained using the diffractometer analyzer Analysette 22 in the group of highly organic soils.

Keywords: particle size distribution; organic soils; soft soils; hydrometer method; laser diffraction

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1. Introduction

Particle size distribution is one of the basic soil tests for geotechnical engineering purposes. In the case of soils considered suitable for construction, i.e., mineral soils, both plastic (cohesive) and non-plastic (non-cohesive) [1–3], several methods are known and commonly used, including two main groups: mechanical method screening (e.g., sieve analysis) and sedimentation (e.g., hydrometric analysis) [3–5]. These methods are characterized by the ability to determine the grain composition of a material whose soil skeleton is composed exclusively of mineral grains. If organic matter is identified, it is treated as a pollutant and removed by various methods at the sample preparation stage. Generally, in the case of mineral soils, this is a correct procedure which does not significantly change the geotechnical properties of the soils tested. In the case of soil classified as organic, the situation is different because the removal of organic matter from the soil skeleton dramatically changes its properties [6–13].

Soils containing more than 2% organic matter content are classified as a completely separate group, i.e., organic soils, which are characterized by properties different from those of mineral soils [1–3,13]. Most often, these are soils with high water content, up to several hundred percent, and low bulk density, which translates directly into the fact that they are extremely compressible and their strength parameters are not impressive compared to mineral soils. In the case of soils defined as organic, particle size distribution research is performed extremely rarely, primarily due to the lack of precise guidelines and standard procedures. This is due to the fact that organic soil, as a research material, is not composed of individual grains, as is the case with mineral soils, but of mineral–organic aggregates, which should be tested only in their natural form.

Depending on local soil and water conditions, with low organic matter content and water content, there may be sufficient load-bearing capacity to solve a specific, individual geotechnical problem. This is becoming an increasingly interesting topic for engineers, mainly because areas with high soil load-bearing capacity are becoming increasingly difficult to access, especially near large urban agglomerations. Currently, increasingly advanced work and research are being carried out on the successful use of load-bearing soils, used as a substrate for various types of construction investments (e.g., residential, industrial, warehouse) and linear infrastructure elements in a given area. The key to decision making regarding their potential suitability for geotechnical purposes is the correct identification and determination of reliable geotechnical parameters. Therefore, for this purpose, standard and proven research methods are being increasingly verified, and completely new methods, including artificial intelligence, are being employed [14–21].

In the case of the necessity of determining the particle sizes of soils for the purposes of broadly understanding geotechnics or geology, the situation is exceptionally complicated because the research material, depending on its genesis, may be in the form of non-cohesive or cohesive soil, which can additionally contain organic matter, often strongly associated with the mineral skeleton. One of the factors that has a decisive influence on the real result of granulometric analysis is the method of preparing samples for testing, at which stage, due to the high sensitivity of the research material, they may be damaged or even destroyed, to which organic soil samples are particularly susceptible. Taking into account the need for extremely careful handling of the research material, a method is sought that will meet these requirements, which, due to its potential and specificity, may be the laser diffractometer method.

The laser diffractometer method has been successfully used for many years, among other uses, in various branches of industry in order to measure the size of various particles of regular and irregular shape [22–26], and even for such unconventional purposes as forensic investigation [27,28], which is why attempts are also made to use its capabilities in the field of geotechnical engineering. In practical research, various types of diffractometers are used, differing in their type of particle size measurement, and in their range and dispersion possibilities [29]. A sample for diffractometric research is most often prepared in the form of a suspension made of a representative part of the soil in distilled water, which, due to the preservation of a complete soil skeleton, makes it a fully reliable as a research material [11,30]. Unfortunately, due to the diverse and often extremely complicated structure of soils, depending on their type and origin, no single, binding research procedure has been developed to date for research using the laser diffraction phenomenon. In general, works published on this subject most often present a complete case study, including the results of diffractometer tests and their verification using the selected method [31–38]. These works mainly concern the study of mineral soils, while the information on organic soils in the literature is fragmentary or includes information on the study but only after removing organic substances from the soil skeleton, which ultimately deprives the study of practical application, because this process not only changes the structure and composition but also the geotechnical properties of the soil [6–8,11,39]. Research using laser diffractometers has been conducted for many years, and the overall results are encouraging, but it should be borne in mind that this method also has limitations and the research conducted today is aimed at their precise identification and, if possible, their reduction or elimination in the future [40–45].

This work presents research that uses two types of laser diffractometers: the Helos laser diffractometer manufactured by Sympatec GmbH (Clausthal-Zellerfeld, Germany) [46,47] and the laser particle sizer analyzer Analysette 22 MicroTech plus by Fritsch GmbH (Idar-Oberstein, Germany) [48,49], the results of which were referred to the hydrometric method [4,5] and classified as sedimentation methods, the effectiveness of which has been confirmed by many years of practice in soil research for the purposes of agricultural soil science, with agricultural soil often containing significant amounts of organic substances.

2. Materials and Methods

The subject of this work is the study of local organic soils in which the dominant parameter is organic matter content [1–3,13]. The most commonly used method for determining the amount of organic matter in the soils there is the loss on ignition method, which can be used in principle for all natural and anthropogenic soils [3,50,51], and based on the authors' own experiences and studies in relation to a method for the determination of the loss on ignition of local organic soil, the optimal roasting temperature was assumed [52,53].

The current rules for classifying and marking organic soil for geoengineering purposes based on the organic matter content, included in the standards, recommend the following distinction: low-organic soils (2–6%), medium-organic soils (6–20%), high-organic soils (more than 20% organic matter). In addition, it also recognizes peat, gyttja, dy or humus, based on macroscopic analyses [1,2].

2.1. The Characteristics of Selected Organic Soils

The 12 samples of different organic soils that qualified for testing, representing the full classification spectrum, came from 7 locations in the Podkarpacie region in Poland, the basic characteristics of which are presented in Table 1.

Table 1. The basic properties of selected organic soils in the study areas.

Sample No.	Location	Soil [1]	Organic Content (%)	Water Content (%)	Bulk Density ($t\ m^{-3}$)
1	Iskrzynia	low-organic	4.35	25.51	1.96
2	Rzeszow 2	low-organic	5.73	22.37	1.98
3	Rzeszow 1	medium-organic	6.95	25.11	1.70
4	Czarna	medium-organic	7.71	24.49	2.00
5	Rzeszow 3	medium-organic	8.92	81.90	1.45
6	Rzeszow 3	medium-organic	13.1	58.24	1.61
7	Rzeszow 2	medium-organic	19.64	69.59	1.67
8	Rzeszow 2	high-organic	33.79	87.66	1.21
9	Mielec	high-organic	48.71	70.12	1.59
10	Rzeszow 1	high-organic	65.89	250.07	1.08
11	Rzeszow 4	high-organic	70.84	325.84	1.13
12	Rzeszow 2	high-organic	84.14	322.58	1.22

[1] Classification according to standard [1,2].

The content of organic matter in organic soils is most often assumed to be the leading parameter, so the soils selected for study represent a wide range of this parameter: from 4.35% to 84.14%. They are accompanied by closely related parameters such as natural water content (from 22.37% to 325.84%) and bulk density (from 1.08 $t\ m^{-3}$ to 2.00 $t\ m^{-3}$) [54,55].

2.2. The Research Methods

Three test methods were used to determine the particle size distribution of local organic soils: hydrometric [4,5] and two laser diffractometer methods: the Helos laser diffractometer manufactured by Sympatec GmbH [46,47] and the Analysette 22 MicroTech plus by Fritsch GmbH [48,49]. The hydrometric method was chosen as the reference method because it is effectively used in soil research for the purposes of agricultural soil science in Poland. The methods used are briefly described below.

2.2.1. Method No. 1—Hydrometer Method

In this study, hydrometer analysis was utilized to determine the particle size distribution in organic soil samples. The hydrometer method is a sedimentation method that is accepted as the standard method of soil particle size distribution [4,5]. The sedimentation procedure can be used for fine-grained soils. All sedimentation methods are based on the relationship between particle size and settle velocity in a fluid medium. Sedimentation methods include the hydrometer method and the pipette method [56]. The hydrometer method for the grain size analysis of fine-grained soils was originally proposed by Bouyoucos (1926) and later modified by Casagrande (1931) [57,58]. Since the method has been used for many years and has been described many times in standards, scientific publications and books, its detailed description has been omitted. The typical test set up for the hydrometer method is shown in Figure 1.

Figure 1. Typical test set up for hydrometer method.

The hydrometer method, as modified by Casagrande [57,58], is most commonly used in soil science. Due to the similarity of the research material, which is organic soil, a hydrometer was used for geoengineering purposes in this work. The planned tests were carried out in accordance with the standard guidelines [4,5].

2.2.2. Method No. 2—The Analysette 22 MicroTec Plus Laser Diffractometer

The laser particle sizer Analysette 22 MicroTec plus laser (MicroTec, Freiburg, Germany) diffractometer is a versatile laser with a measuring range of 0.08–2000 µm (Figure 2) [48,49].

Each Analysette 22 MicroTec plus laser diffractometer consists of a measuring unit that can be quickly and easily combined with different dispersion units for dry or wet measurements. The laser diffractometer performs most measurements in less than a minute and the instrument is then immediately ready for use. The Fritsch Analysette 22 MicroTec plus diffractometer uses a green-light semiconductor laser for small particle measurements and an infrared semiconductor laser for large particle size ranges. Both lasers can be aligned quickly, automatically and independently by lateral movement, resulting in the determination of wavelengths for each particle size and an ideal combination of wide measuring ranges. By redirecting the beam of the long-wavelength red laser, the instrument can measure even large particles with maximum precision in a compact unit, and to switch to the measuring of small particles with the short-wavelength green laser, the detector and the laser source are simply moved as a unit—the measuring cell remains in place. The optical system used in the Analysette 22 MicroTec plus it is presented in Figure 3 [47].

Figure 2. The Analysette 22 MicroTec plus laser diffractometer (right to left): wet dispersion unit, measuring unit and dry dispersion unit.

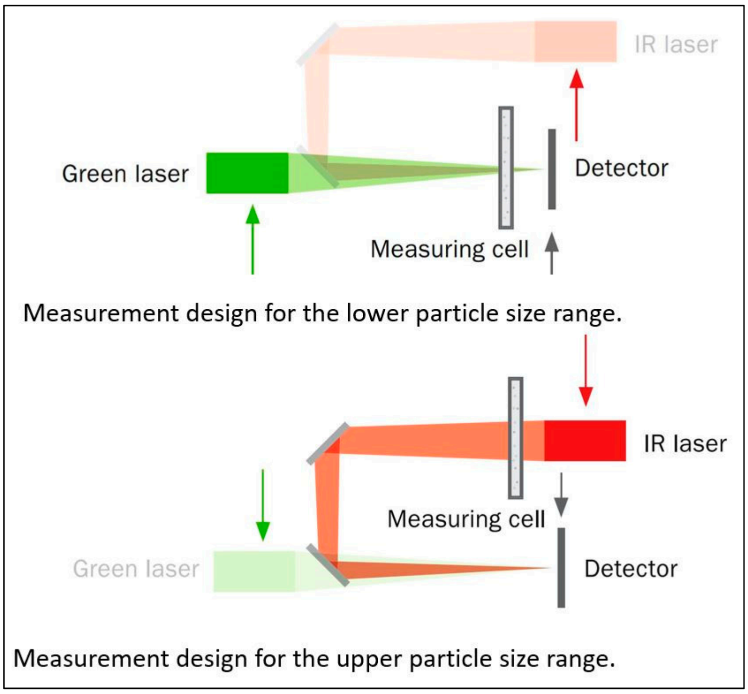

Figure 3. The optical system used in the Analysette 22 MicroTec plus laser diffractometer.

The most important selected technical parameters of the measuring and dispersion units are presented in Table 2.

Table 2. Selected technical parameters of Analysette 22 MicroTec plus laser diffractometer [49,59].

Unit	Technical Parameters
Measuring unit	Measuring range: 0.08–2000 µm (wet dispersion); 0.1–2000 µm (dry dispersion). Two semiconductor lasers: green (λ = 532 nm, 7 mW); IR (λ = 940 nm, 9 mW). Number of particle size classes: max 108. Optical arrangement: inverse Fourier design; movable measuring cell; Fritsch patent [48,49]. Fourier lenses: 260 mm and 560 mm focal length (green or infrared); 10 mm diameter of the laser beam in the Fourier lens. Automatic laser beam alignment: Sensor: 2 segments, 1× for vertical and 1× for horizontal direction of the laser light polarization; 57 elements. Typical measuring time: 5–10 s (measurement value recording of a single measurement); 2 min (entire measuring cycle).
Wet dispersion unit	Suspension volume: 300–500 cm^3; Radial pump with adjustable speed; Ultrasonic with adjustable output (max 60 Watt);
Dry dispersion unit	Sample volume: <1–100 cm^3; High-frequency feeder; Annular gap Venturi nozzle; Required compressed air supply: min 5 bar, 125 L/min; Oil-, water- and particle-free; External exhaust system required;

2.2.3. Method No. 3—The Helos Laser Diffractometer

The second measuring device used to determine the particle size distribution of organic soils in the experiment, the Helos laser diffractometer manufactured by Sympatec GmbH, adapted to standard procedures [29,47], is presented Figure 4.

Figure 4. The Helos laser diffractometer from Sympatec GmbH.

The optical system used in this diffractometer is schematically shown in Figure 5 [46,47]. A Helos diffractometer can work with dry dispersion (RODOS, dispersion in compressed air) and wet dispersion (QUIXEL, dispersion in liquid), and if necessary, it is also possible to combine both methods of dispersion (Oasis system). The comprehensive information and details regarding the construction of the device itself, measurement theory and practice, capabilities and technical parameters can be found on the manufacturer's website and in certain source materials [30,46,47].

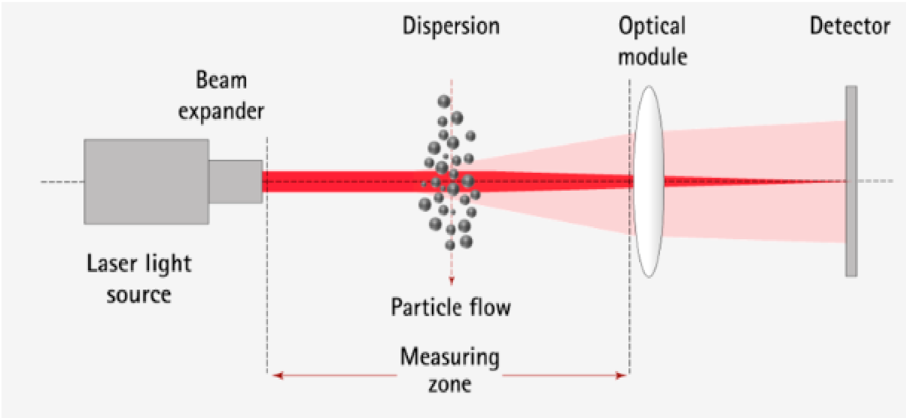

Figure 5. The optical system used in the Helos laser diffractometer.

The most important selected technical parameters of the measuring and dispersion units are presented in Table 3.

Table 3. Selected technical parameters of HELOS laser diffractometer [48].

Unit	Technical Parameters
Measuring unit	Overall measuring range Helos: BR: 0.1–875 µm; KR: 0.1–8750 µm; KR/VARIO: 0.1–875 µm. Measuring range modules (R1–R8): HELOS/BR: 5; HELOS/KR: 8; HELOS KR/VARIO: 8. Measurement: multielement frequency detector, 31 semicircular elements, 2000/s, continuous focusing. Dispersion: adaptable modules: air, sprays, suspensions, emulsions. Laser diffraction: forward scattering in parallel beam: classic optical Fourier set up (ISO 13320) [29]; open measuring zone offering unique work distance. Light source: helium-neon laser; λ = 632.8 nm (red); <5 mW; 3D with open dispersions units; 1 with closed dispersions units; automatic adjustment to measuring range; diameter of the laser beam (R1–R8) from 2.2 mm to 35 mm. System: PAQXOS version 4.1.0.
Wet dispersion unit	Sensor: Helos QUIXEL Analyzing volume: 300–1000 cm^3 Dispersing range: 0.1–3500 µm Closed loop flow-through cell for suspensions and emulsion, even with coarser, high density particles; built-in sonication (0–72 W); heatable
Dry dispersion unit	Sensor: Helos RODOS Analyzing volume: <1 mg–1000 g Dispersing range: <0.1–3500 µm

2.3. The Samples and Sample Preparation

The presented organic soil samples were obtained as part of our own research and provided by cooperating professional companies from the geotechnical industry. A total of 12 samples from different origins, locations and soil-water conditions were tested. Unfortunately, in the case of organic soils, there is very high variability in their parameters, even within one research site, so due to the nature of the research conducted, the priority was to maintain the natural grain size in the obtained soil samples. At every stage of sampling and transport, every effort was made to ensure that the research material was not mechanically damaged. Under laboratory conditions, the samples were tested using the hydrometric method and laser diffractometers in accordance with the standard guidelines and recommendations applicable to this method.

2.3.1. The Hydrometer Method

The soil samples had previously been air-dried, crushed, and sieved through a 2 mm mesh sieve. About 40 g of sieved soil samples were mixed with 25 mL 10% sodium hexametaphosphate (Calgon solution), diluted to 300 mL with distilled water and stirred for 30 min [36]. After stirring, the soil samples were placed in a 1 L sedimentation glass cylinder and diluted with distilled water to a 1000 mL final volume. After that, the samples were left overnight to reach a constant temperature. The research was continued using appropriate standard procedures [4,5].

2.3.2. The Analysette 22 MicroTec Plus Laser Diffractometer Method

In our study, a wet dispersion unit was used. A total of 10 g of air-dried soil sample, previously sifted through a sieve with a mesh diameter of 2 mm, was placed in an ultrasound bath for 5 min. The homogenized soil samples were then placed on a measurement cell in the Analysette 22 MicroTec plus laser particle analyzer. Measurements were taken over the full measurement range of the device (0.08–2000 µm), and the results were estimated using Fraunhofer's theory and automatic calculation model. The pump device ensured a smooth suspension flow and dispersed the sample into the ultrasonic bath and the measuring cell. Each sample was analyzed three times, the suspension was discharged from the diffusion unit to the outlet, and the diffusion unit was cleaned and prepared for the next measurement [48,49].

The details of the measurement conditions for the Analysette 22 MicroTec plus laser diffractometer are presented in Table 4.

Table 4. The measurement conditions using an Analysette 22 MicroTec plus laser diffractometer.

System	
Instrument	ANALYSETTE 22 MicroTec plus
Software	MaScontrol v. 1.035
Dispersing Method	
Dispersant	water
Pump	7
Ultrasonic	10
Scans	500
Shots per scan	0
Measurement Range	Full (0.08–2000 µm)
Calculation model narrow	(Trade Off 0.001)
Theory	Fraunhofer

2.3.3. The Helos Laser Diffractometer Method

Due to the specific structure of organic soils, which create mineral–organic conglomerates that are not really soil grains in the sense of mineral soils, the determination of the grain composition of organic soils is not covered by the standard test procedure, which is why we are constantly looking for new and more efficient methods.

The pre-preparation of samples in the lab was relatively simple, because a representative part was separated from the soil material, which was air dried, and then, distilled water was poured over 20 g samples of the dry organic soils and the suspension was boiled until the sample disintegrated. After cooling, the suspension was poured into 100 mL samplers and distilled water was added if necessary. The actual measurement was carried out in the Helos diffractometer with QUIXEL dispersant in the wet dispersion system, with a reference measurement carried out on several lenses, using three measurement ranges, R3 (focal length 100 mm), R5 (focal length 500 mm) or R7 (focal length 2000 mm), depending on the situation [30,46,47]. Details of the measurement conditions are given in Table 5.

Table 5. Details of the measurement conditions using a laser diffractometer.

System	
Instrument	HELOS (H3324) and QUIXEL, R3 + R7
Software	PAQXOS 5.1.1
Dispersing Method	
Dispersant	Water
Cuvette size	2 mm
Fill level	High
Temperature	20 °C
Sonication	60 s at 100% power, 30 s pause
Pump speed	1000 rpm

3. Results and Discussion

The samples of local organic soils representing the full range of classifications, i.e., low-organic, medium-organic and high-organic soils, were selected to determine the particle size distribution. As some the samples were collected in person and others were supplied in varying quantities by co-operating geotechnical contractors from sites of current projects, their availability and quantity were limited in some cases, which was reflected in the number of tests carried out. The main objective of this review was to determine the granulometric composition of organic soils using laser diffraction, which appears to be the best method in this case due to the minimal impact on the structure of the soil at the preparation stage. Organic soils have an exceptionally complex structure, as they are mostly composed of mineral–organic aggregates, which are very sensitive to damage and require different methods than those that have proven effective for non-plastic (e.g., sand, gravel) and plastic minerals soils (e.g., silt, clay).

It should be noted that the grains were classified as clay, silt and sand fractions, but these are conventional terms that refer only to the grain size range of the fraction, and not to the type of soil. Thus, the "clay fraction" was assigned mineral–organic aggregates up to 0.002 mm, the "silt fraction" from 0.002 mm to 0.05 mm and the "sand fraction" from 0.05 mm to 2 mm [1,2].

The situation is further complicated by the fact that currently, the specialist literature lacks practical guidelines on conducting and interpreting organic soil research using the laser diffraction method; therefore, the presented results and methods of their interpretation are, in a way, pioneering and open to further discussion. Two types of laser diffractometers (Analysette 22 and Helos) were used for this research, characterized by different types of sample dispersion and measurement methods, which are described in detail in Section 2.2. This allowed for a comparison of results that theoretically should have been similar. Although the laser diffractometer method has long been known and used in various aspects of geotechnical research, it is not considered a leading method, mainly due to the difficulty of relating the results to methods recognized as standard.

The results of all studies were considered from different angles, giving them a multi-dimensional character, and were addressed in the following manners:

- Individually, via independent reference to each test method separately (Section 3.1), where the percentage of individual fractions in a given sample was analyzed and attempts were made to determine the regularity (tendency) of the occurrence of individual fractions, e.g., whether the fraction proportions are always the same or whether one fraction is always dominant, of course, within a given classification group, e.g., highly organic soils.
- Collectively, with reference to all research methods used simultaneously (Section 3.2), where the percentages of the three main fractions obtained by laser diffractometers and the hydrometric method were compared within each sample separately and additionally in comparison with the remaining samples of a given classification group.

- Individually, in terms of the considered grain size, i.e., within a given fraction (Section 3.3), determined by three independent research methods for all samples in a given series of tests.

In interpreting the research results in this complex way, it became apparent that the above aspects often had some common features, so that it was only necessary to consider them together rather than separately, thereby increasing the reliability of the conclusions.

3.1. The Determination of Particle Size Distribution

The results of testing the granulometric distribution of the tested organic soils (percentage content) depending on the research method used are presented in the corresponding charts. Figure 6 shows the test results obtained using the hydrometer method, while Figures 7 and 8 show the results obtained using the Analysette 22 and the Helos laser diffractometer. The samples marked with numbers 1 and 2 represent low-organic soil, samples from No. 3 to No. 7 represent medium-organic soil and the remaining ones (No. 8–12) represent high-organic soil.

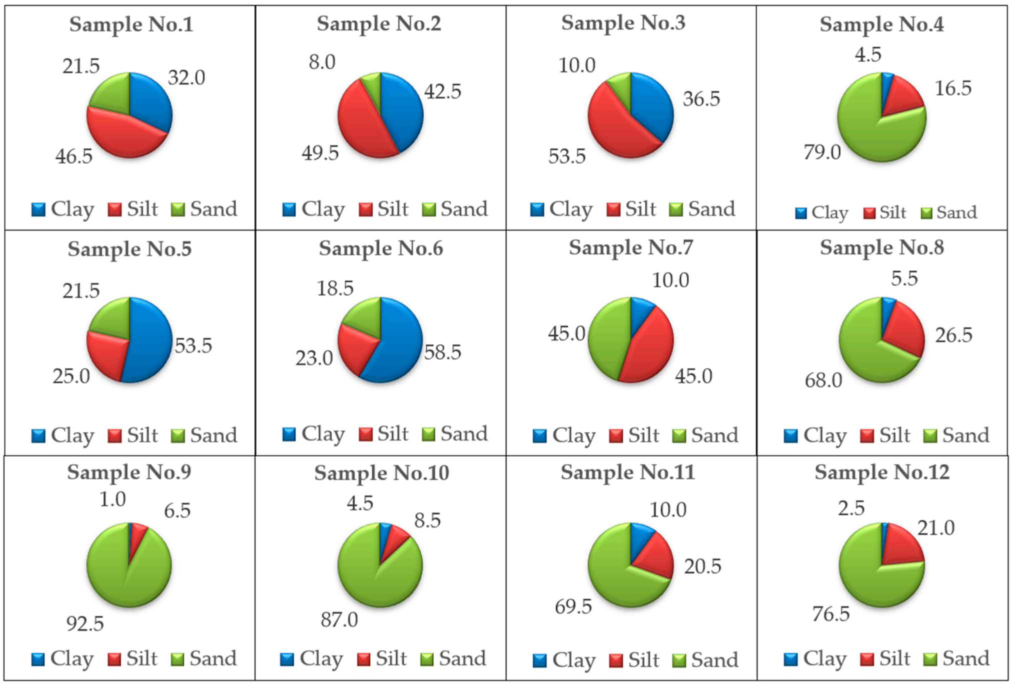

Figure 6. The content of soil fractions determined using the hydrometer method.

In this part of the discussion, the focus is mainly on presenting the percentage content of the individual fractions representing the sizes of the mineral–organic aggregates examined, called grains for the purpose of this work. The repeatability of the relationship between the fractions is also discussed, i.e., whether the same fraction always dominates and whether the remaining fractions (secondary and tertiary) occur in the same order, and the difference is only in the percentage content, i.e., whether the same tendency occurs in all methods.

The tests of the particle size distribution using the hydrometer method (Figure 6) showed that in the case of soils classified as low-organic (sample No. 1 and No. 2), the primary fractions were mineral–organic agglomerates, which, in terms of size, belonged to the silt fraction (46.5–49.5%). The smallest amount of grains (the tertiary fraction) was assigned to the sand fraction (8.0–21.5%). The grain size of medium-organic soils (sample Nos. 3–7) is difficult to classify clearly: sand fraction (10.0–79.0%), silt fraction (16.5–53.0%) and clay fraction (4.5–58.0%). The high-organic soils (samples 8–12) were clearly dominated by particles with a size corresponding to sands (68.0–95.5%), which are the primary fraction.

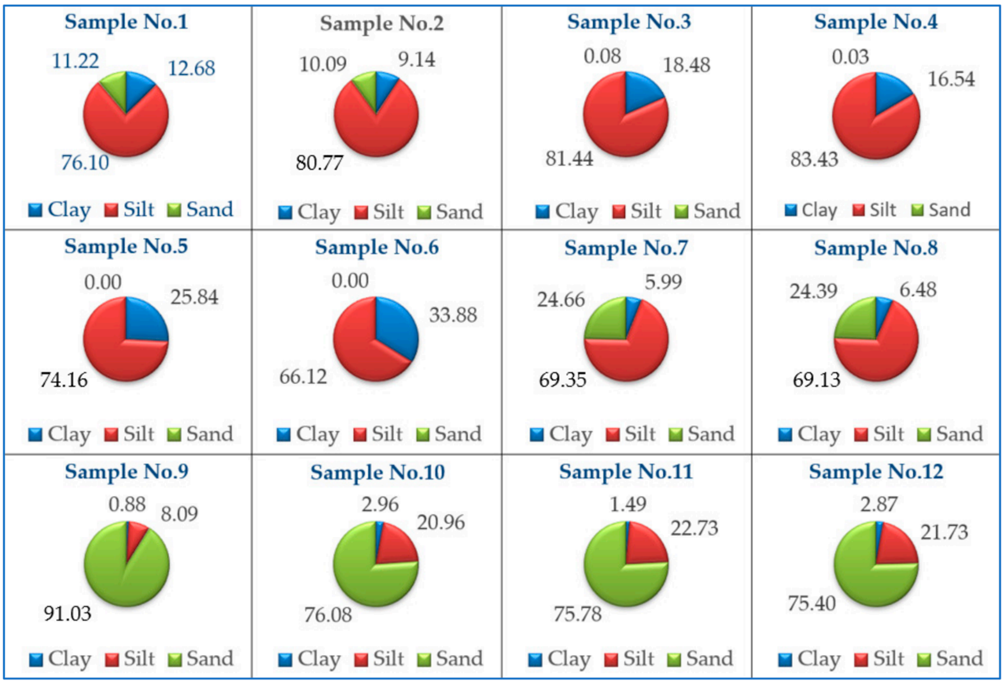

Figure 7. The content of soil fractions determined using the Analysette 22 laser diffractometer.

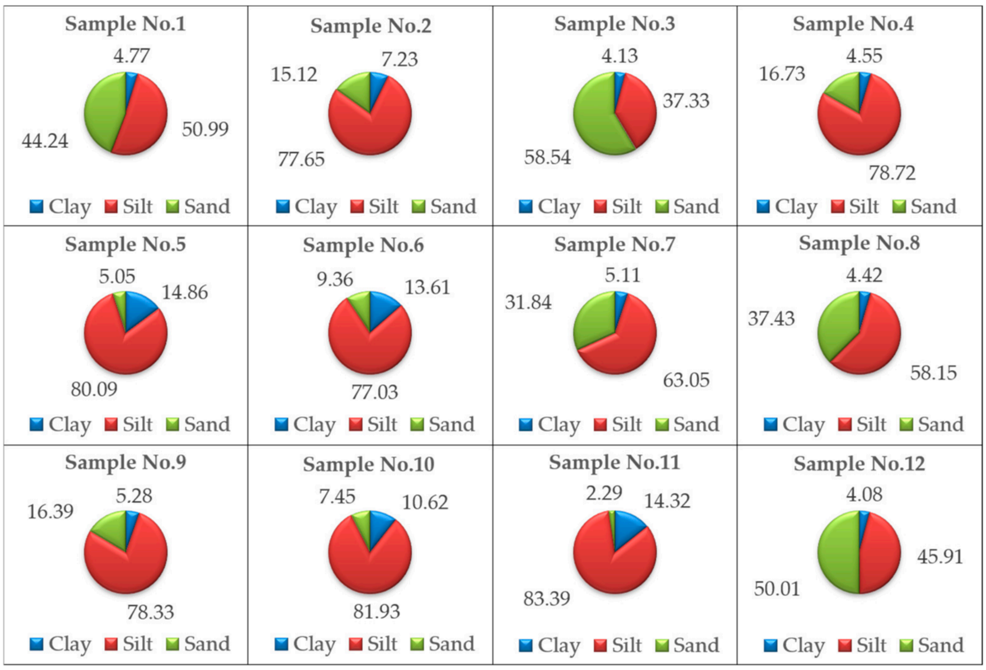

Figure 8. The content of soil fractions determined using the Helos laser diffractometer.

In the case of the method of the Analysette 22 laser diffractometer (Figure 7), a very similar grain size composition was observed in the low-organic soil samples (sample Nos. 1 and 2), where 76.10–80.78% of the grains corresponded in size to silts—the primary fraction—10.09–11.22% to clays and 9.14–12.68% to sands. The medium-organic soils (sample Nos. 3–7) were dominated by silts (66.12–83.43%), which were the primary fraction, sand was practically absent (0.00–0.08%) and the secondary fraction was clay, which ranged from 16.54 to 33.88%.

The particle size distribution tests using the Helos laser diffractometer are presented in Figure 8. The low-organic soils (sample No. 1 and No. 2) were characterized by the highest content of aggregates, corresponding in size to silt (50.99–77.65%), which constituted the primary fraction, followed by sand (15.12–44.24%)—the secondary fraction. The tertiary fraction included grains classified as clays, which had marginal significance (4.77–7.23%). In medium-organic soil (sample Nos. 3–7), the primary fraction consisted of grains belonging to the silt fraction (37.33–80.09%), except for sample No. 3, where sand predominated (58.54%).

The least numerous fraction (the tertiary fraction) turned out to be the clay fraction, ranging from 4.13 to 9.36%. In the last classification group, high-organic soils (samples 8–12), the primary fraction was silts particles (45.91–83.39%). For the other fractions (clays and sands), it was difficult to clearly identify whether the fraction was secondary or tertiary, as the content of grains corresponding to clays ranged from 2.29 to 14.32%, and for sands from 2.29 to 50.01%.

In addition, detailed results of the organic soil tests using the Helos laser diffractometer are presented in the form of particle size distribution curves. Figures 9–11 shows the particle size cumulative distribution curves of the tested low-organic soils (No. 1, 2), medium-organic soils (No. 3–7) and high-organic soils (No. 8–12).

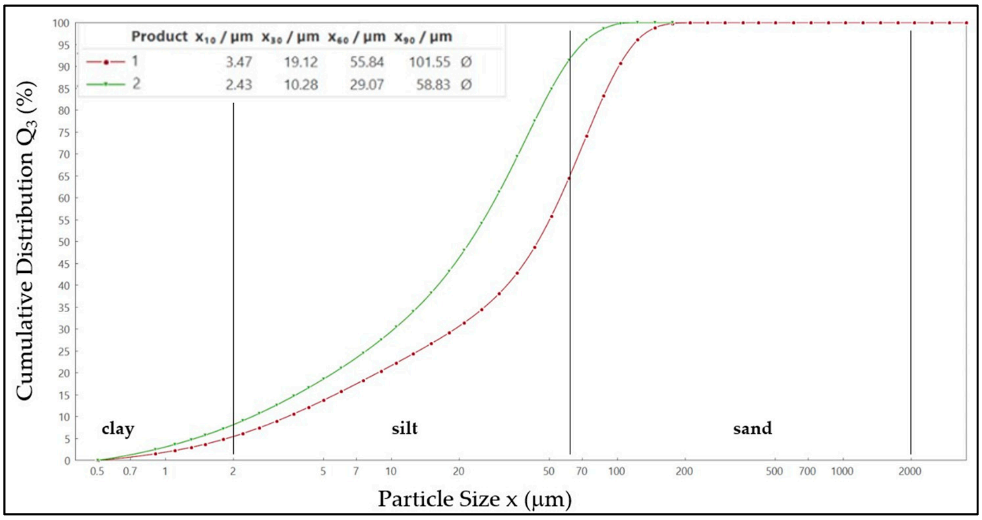

Figure 9. The particle size cumulative distribution density for low-organic soils (sample No. 1, 2).

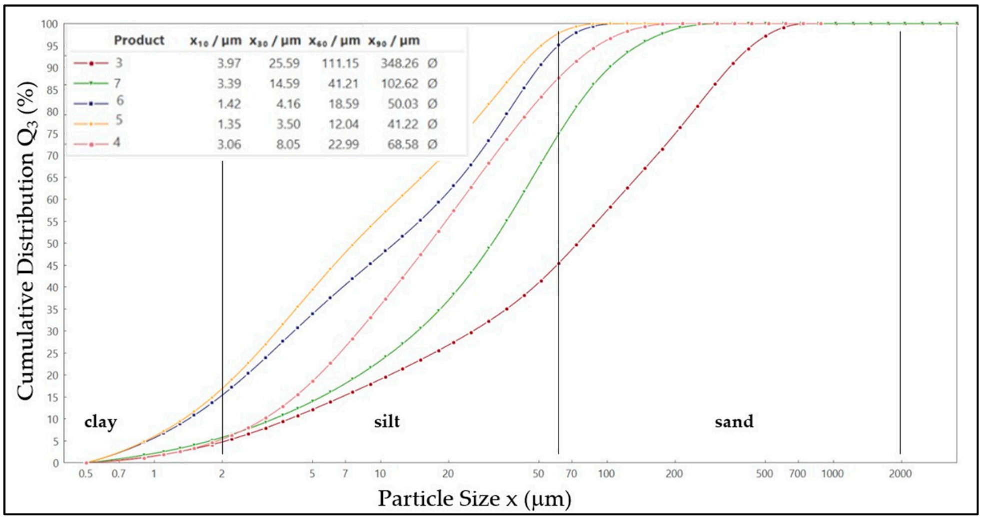

Figure 10. The particle size cumulative distribution density for medium-organic soils (sample Nos. 3–7).

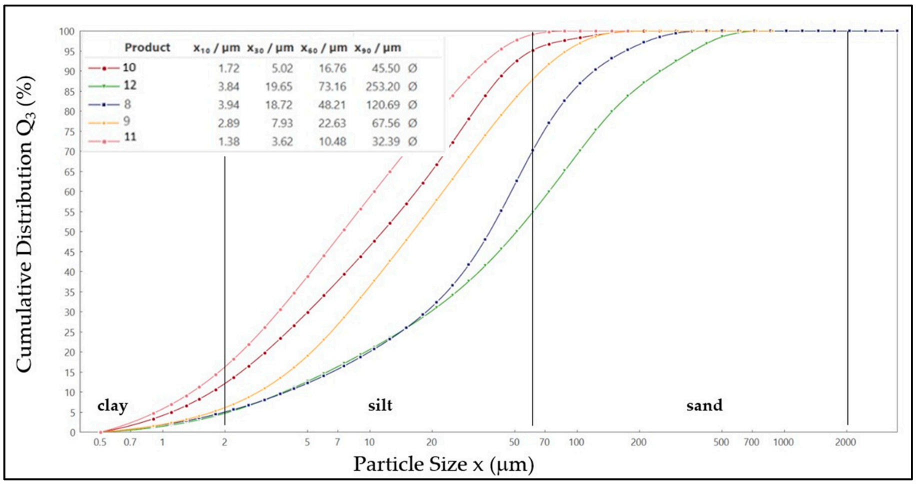

Figure 11. The particle size cumulative distribution density for high-organic soils (sample Nos. 8–12).

3.2. Collective Summary and Comparison

The test results of all selected organic soil samples with respect to the applied research methods were presented graphically and subjected to a comparative analysis within the adopted standard classification group. The results of testing the particle size distribution using the hydrometric method were considered the reference (standard) for the purposes of this work. The test results were analyzed both for agreement with the reference method and similarity to the test results obtained using the laser methods Analysette 22 and the Helos laser diffractometer for each sample separately. The general similarities and regularities between the test results of all samples within the group were also looked for [4,5].

3.2.1. The Low-Organic Soils

The test results for low-organic soils are from samples taken from two sites (sample No. 1 and No. 2.). In the case of sample No. 1, apart from the clear dominance of the silt fraction and its similar content in the case of the reference method and the Helos laser diffractometer, no other significant similarities were observed. The results of the Analysette 22 laser diffractometer method were not comparable to alternative methods (Figure 12). The share of the silt fraction determined by this method was more than 1.6 and 1.5 times higher than the hydrometric and laser methods using the Helos laser diffractometer, respectively. However, the proportions of sand and clay fractions determined by the reference method were lower, with the proportion of clay almost three times higher and the proportion of sand almost four times lower compared to the Helos laser diffractometer.

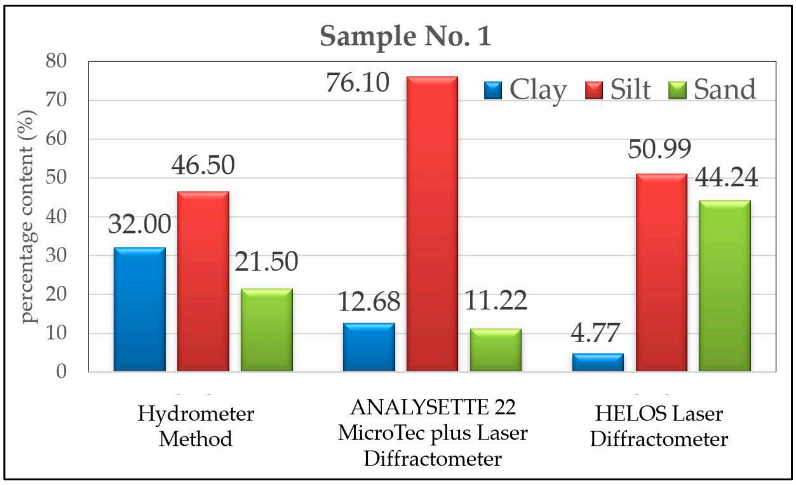

Figure 12. A summary of the results of sample No. 1.

The results of the granulometric tests of sample No. 2 confirmed the dominance of the silt fraction, the content of which was much higher than in sample No. 1 (Figure 13). Moreover, a visible similarity was observed in the results of the tests using the laser diffractometer methods (Analysette 22 and Helos), which, however, differed significantly from the hydrometric reference method. Taking both samples into account, relatively repeatable test results were only obtained with the Analysette 22 laser diffractometer.

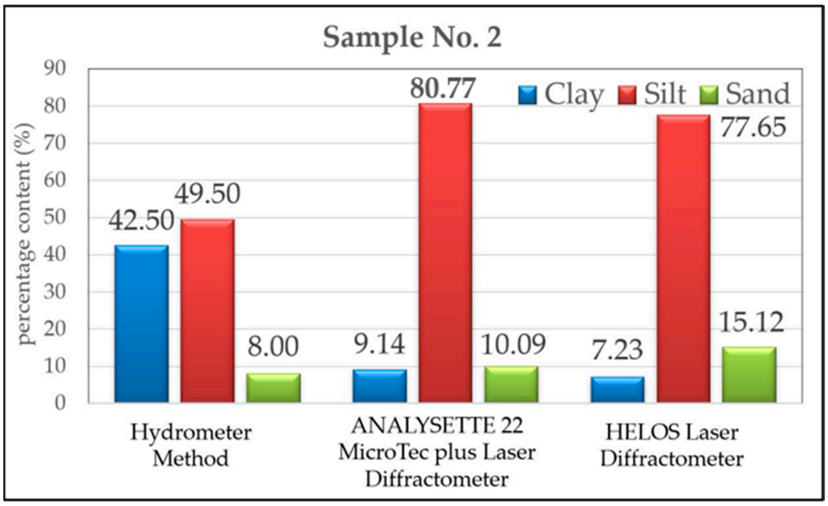

Figure 13. A summary of the results of sample No. 2.

3.2.2. The Medium-Organic Soils

The particle size distribution of medium-organic soils is represented by tests of sample Nos. 3–7. The tests of soil sample No. 3 presented in Figure 14 show some similarity between the results obtained using the hydrometric method and the Analysette 22 laser diffractometer, i.e., the quantitative trend (silt–clay–sand) is maintained, but the measured values themselves are very different and cannot be compared. This is particularly true for the sand fraction, which, in this case, is below 1%, which is a value over 125 times lower compared to the reference method and as much as 731 times lower than the Helos laser diffractometer. The analysis of the test results obtained using the Helos laser diffractometer did not show any similarity with the results obtained using the other methods.

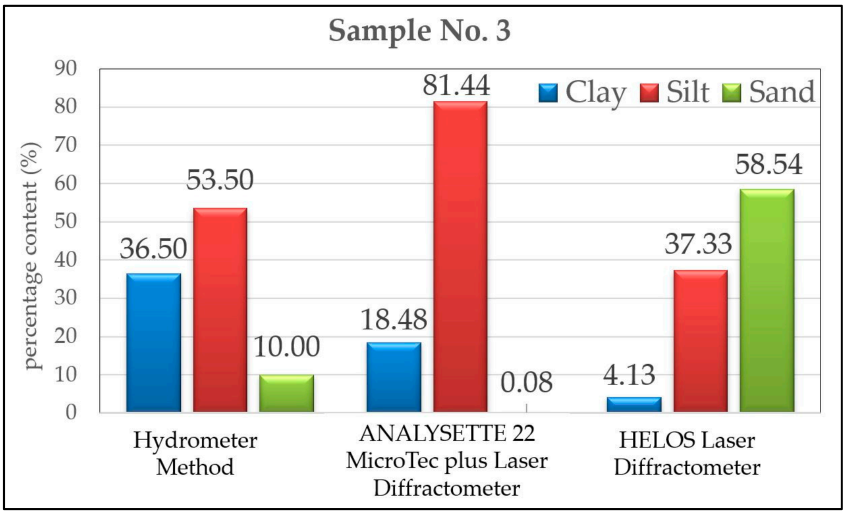

Figure 14. A summary of the results of sample No. 3.

In the case of sample No. 4, the test results presented in Figure 15 do not show any significant similarities between the methods used, apart from the similar percentage of silt fraction for the laser methods used (Analysette 22 and Helos). These results cannot be directly compared with the reference method, where the sand fraction was clearly dominant.

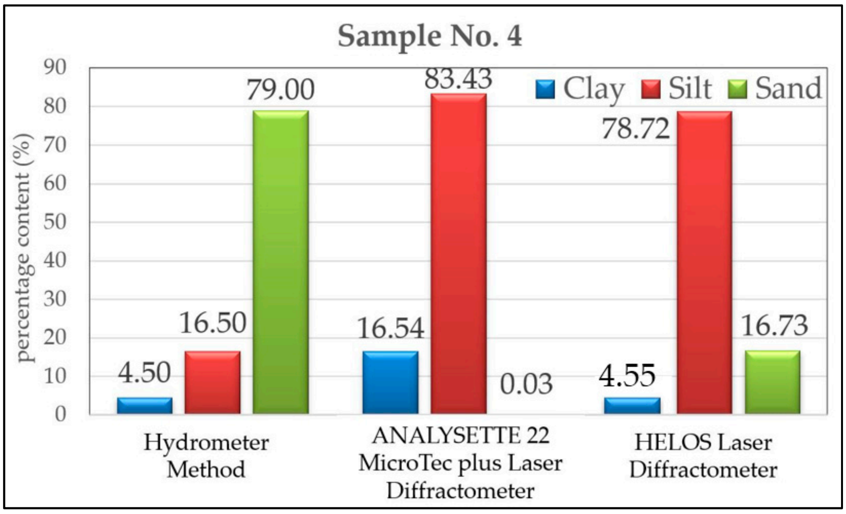

Figure 15. A summary of the results of sample No. 4.

When analyzing the test results of sample No. 5 (Figure 16), it was found that in terms of the percentage of individual fractions, there was a noticeable similarity between the test results obtained using the laser diffractometer only in terms of the dominant silt fraction. In the case of the reference method, the results obtained were different from those obtained using laser methods, and the largest number of grains was assigned to clay in terms of size.

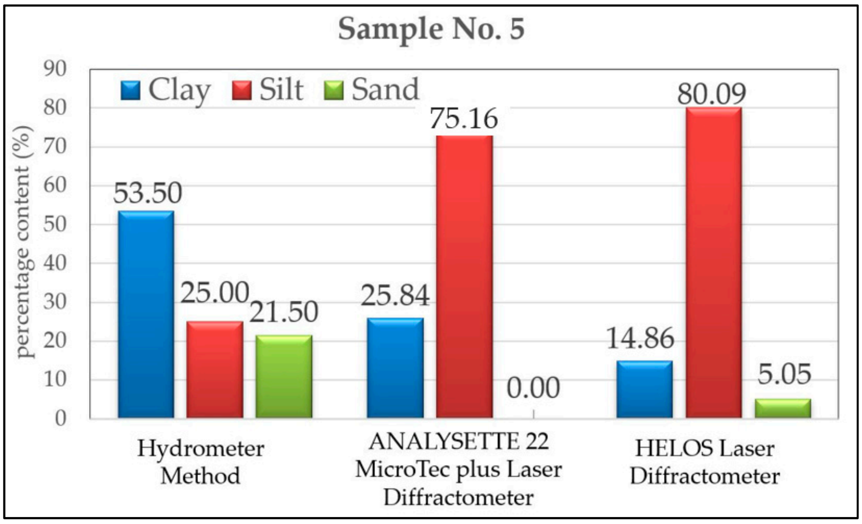

Figure 16. A summary of the results of sample No. 5.

The research results for sample No. 6 presented in Figure 17 are surprisingly consistent with the test results shown earlier for sample No. 5 (Figure 16), not only in terms of the presented trends, but also in terms of their percentage content. This applies to both the hydrometric and laser test results, which, again, cannot be compared with the reference method.

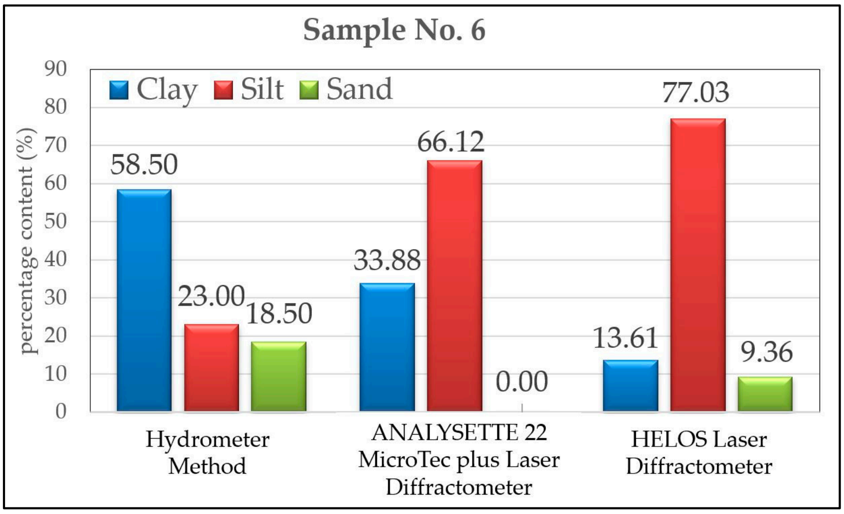

Figure 17. A summary of the results of sample No. 6.

In the case of the last sample of medium-organic soils (No. 7) presented in Figure 18, the test results confirmed the dominance of the silt fraction for the laser diffraction methods. A reversal of the trend in the size of the fraction observed in the remaining samples was also noticed, as the content of the sand fraction increased significantly at the expense of the clay fraction. The proportions between the fractions also changed in the case of the reference method, where the highest content of the clay fraction was most often observed, which, in this case, turned out to be the lowest, with comparable silt and sand fractions.

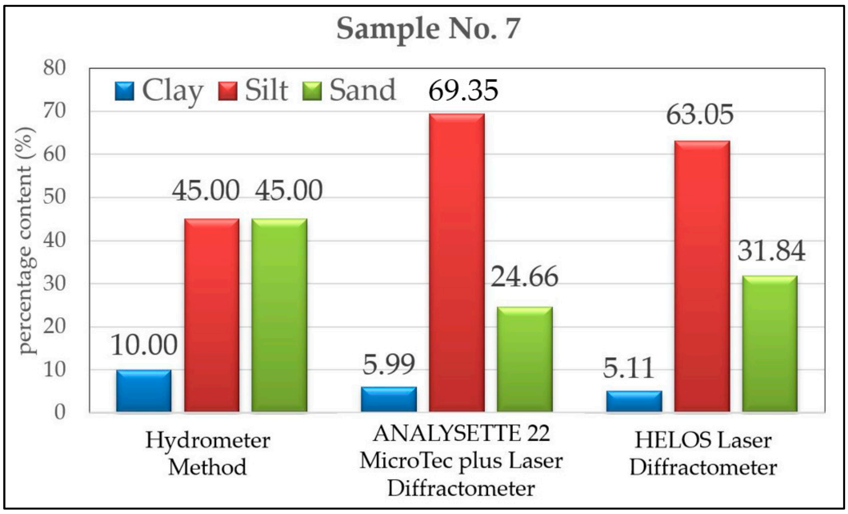

Figure 18. A summary of the results of sample No. 7.

Considering all the examined samples of medium-organic soils, it can be generally concluded that in most cases, certain, occasionally even high, agreement was observed between the test results performed using the laser diffractometer. The results obtained by the hydrometric method were not only incomparable with those obtained by the laser methods, but were basically incomparable among all the samples analyzed.

3.2.3. The High-Organic Soils

Figures 19–23 present and compare the results of tests on high-organic soil for sample Nos. 8–12, carried out using all the selected methods. When analyzing the test results of sample No. 8 it can be seen that the reference method was dominated by the sand fraction, whereas the results of the laser methods were grains corresponding to the silt (Figure 19). In the case of the laser methods (Analysette 22 and Helos laser diffractometer), a high mutual similarity of values and trends was observed, which (except for the clay fraction) was not reflected in the results of the hydrometric method, which was considered to be the basic method.

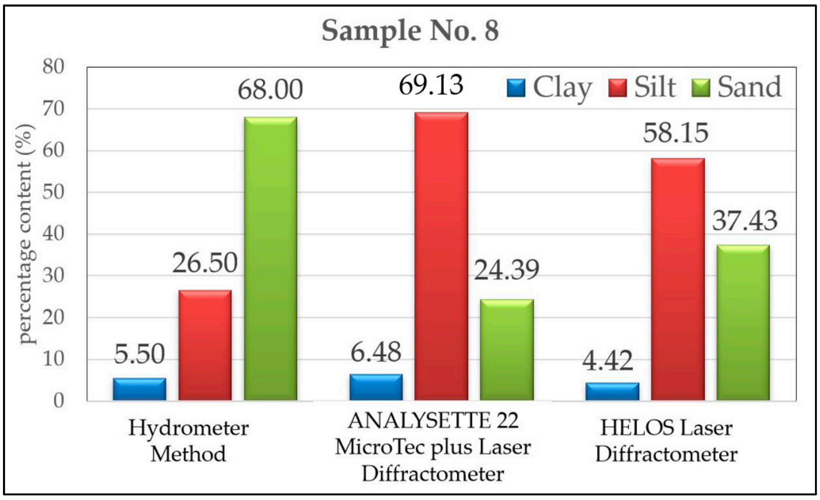

Figure 19. A summary of the results of sample No. 8.

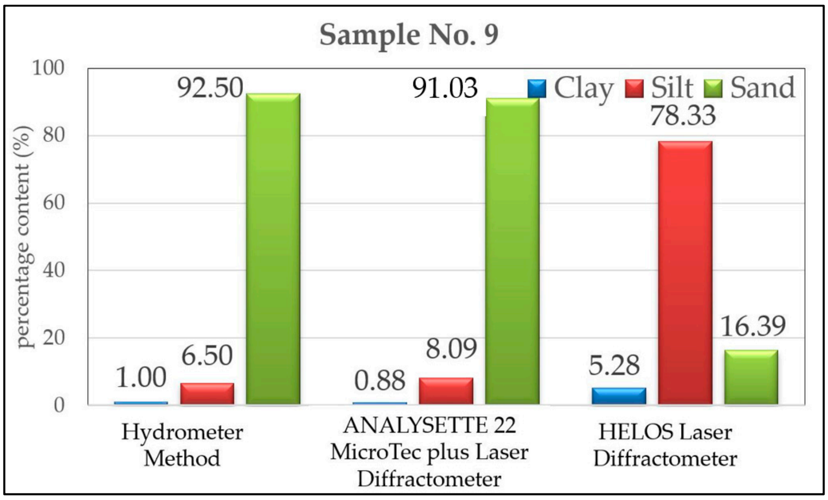

Figure 20. A summary of the results of sample No. 9.

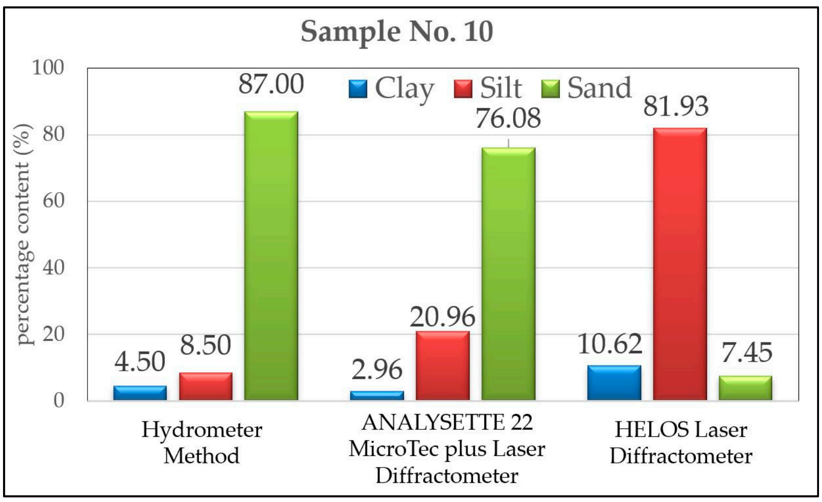

Figure 21. A summary of the results of sample No. 10.

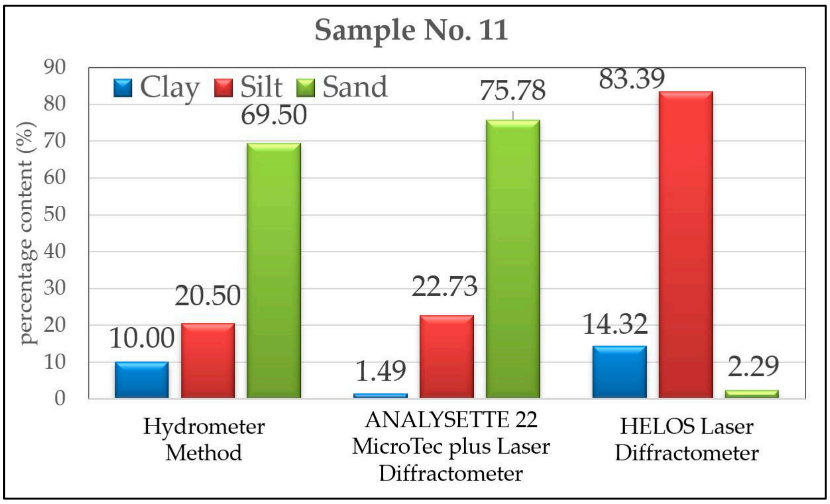

Figure 22. A summary of the results of sample No. 11.

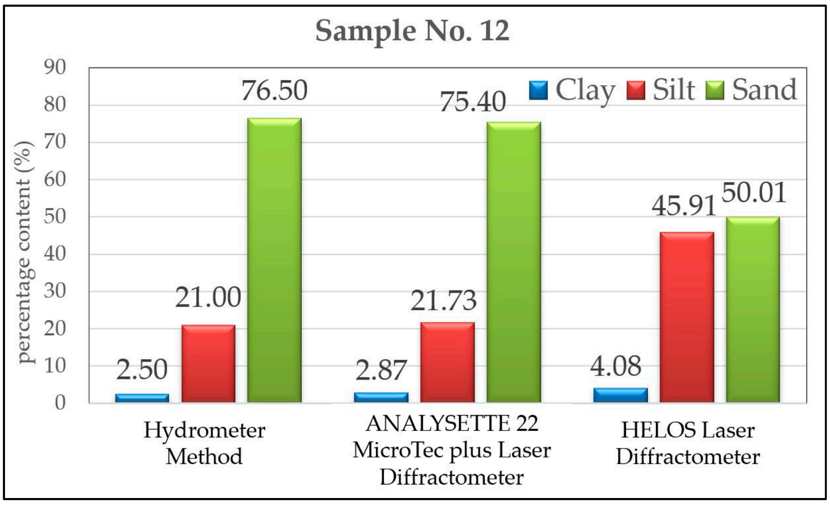

Figure 23. A summary of the results of sample No. 12.

In the case of sample No. 9 of highly organic soils, the test results of which are presented in Figure 20, high similarity of the test results obtained using the Analysette 22 laser diffractometer with the reference hydrometric method was observed. These results were very similar in the range of all the fractions and indicated clear dominance of the sand fraction, in contrast to the results obtained with the Helos laser diffractometer, with which they did not agree, where the silt fraction was dominant.

Figure 21 shows the test result of sample No. 10, where high similarity was also observed between the results obtained using the Analysette 22 laser diffractometer and the hydrometric method, with a lack of agreement with the test results obtained using the Helos laser diffractometer. A high degree of agreement was also observed between these results and those of sample No. 9 (Figure 20).

The tests of sample No. 11 again showed high similarity of the test results obtained using the Analysette 22 laser diffractometer and the reference hydrometric method, especially in the sand and silt fractions, while the results were lower in the case of the clay fraction. These results are similar to those obtained for sample Nos. 9 and 10. Unfortunately, these tests were again not in agreement with the results of the Helos laser diffractometer tests, although some similarity was observed in terms of the clay fraction (Figure 22).

The results of sample No. 12 of high-organic soil showed absolute dominance of the sand fraction, which was exceptionally observed for all methods used (Figure 23). The exceptional agreement between the test results obtained using the hydrometric method and the Analysette 22 laser diffractometer was repeated. The results of the Helos laser diffractometer tests were not so consistent, but a common tendency was maintained: the highest results were obtained for the sand fraction, then the silt fraction and the lowest for the clay fraction.

Summarizing the review of the test results of all samples of high-organic soils, it should be noted that when comparing the results obtained using the hydrometric method and the Analysette 22 laser diffractometer, there was exceptional agreement in the results obtained, which was not the case when testing low- and medium-organic soils. Unfortunately, the agreement between all research methods used was only observed incidentally.

3.3. The Content of Individual Fractions

The verification of the suitability of the laser diffraction method for the determination the granulometric composition of local organic soils required the compilation of the test results not only according to the classification group (low-, medium- and high-organic), but also in a broader context, taking into account the degree of the assignment of the research material to a specific fraction (sand, silt, clay), and carrying out analyses not only in relation to the test results of a single sample, but also as a whole, i.e., for all samples in the series, and even their origin [1,2].

3.3.1. The Low-Organic Soils

In the case of low-organic soils (Figure 24), the comparison of the test results in the field of the clay fraction showed that there is a certain correlation between the results obtained using the reference method and the Analysette 22 laser diffractometer. In the case of sample No. 2, the test results were very similar (2.96% and 4.5%), but for sample No. 1 the differences were significant (18.48% and 36.60%). When analyzing the test results for all the low-organic soil samples tested, very large differences were observed between individual samples, which most likely resulted from different local conditions of deposition, i.e., genesis. No significant similarities were found between the results obtained using the Helos laser diffractometer and alternative methods, i.e., the Analysette 22 laser diffractometer and the hydrometric method (reference).

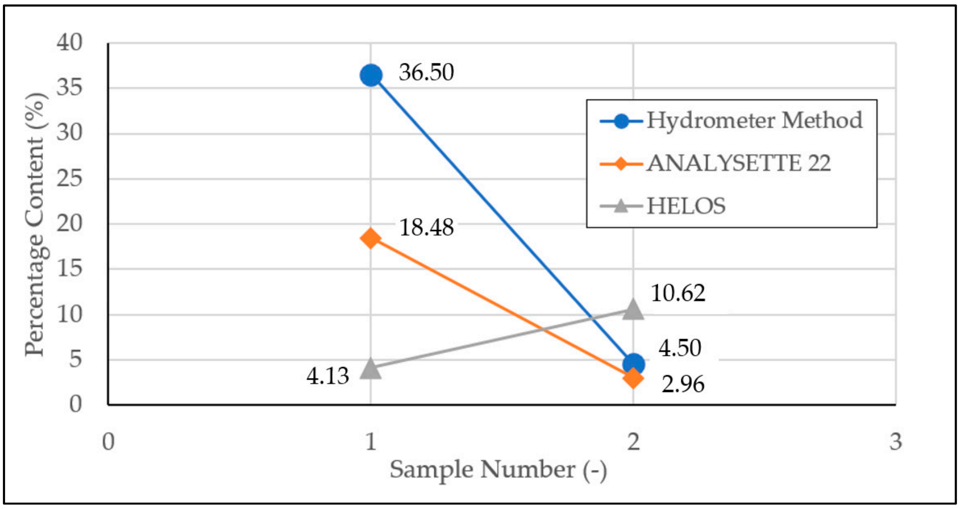

Figure 24. The clay fraction in low-organic soils.

Figure 25 summarizes the test results for the silt fraction, where similar correlations were observed to the clay fraction in Figure 21, but the results were more divergent, as sample No. 2 had silt contents of 8.50% and 20.96%, whereas sample No. 1's silt contents were recorded as 53.50% and 81.44%. Considering the test results of both samples, large discrepancies were also observed. The values obtained with the Helos laser diffractometer were not comparable with the other methods.

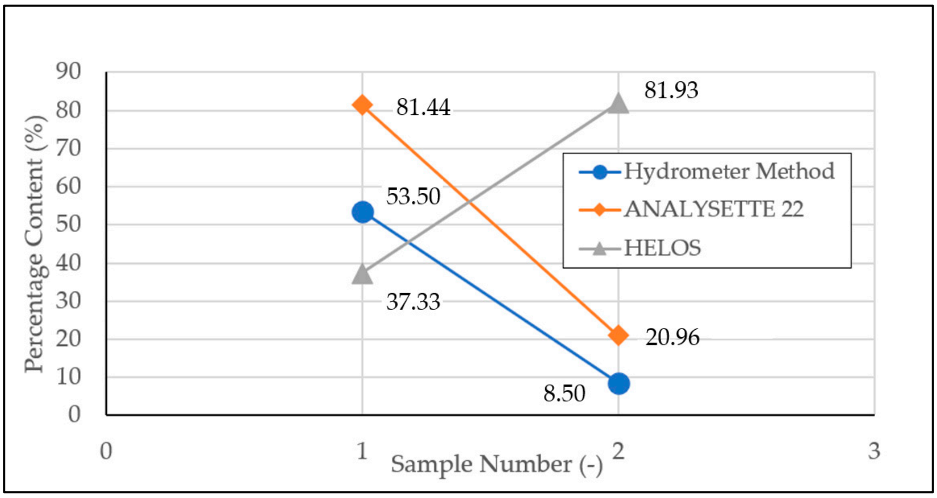

Figure 25. The silt fraction in low-organic soils.

The analysis of the results for the number of grains attributed to the sand fraction presented in the Figure 26 showed a high level of agreement for both samples, as the difference between the reference method and the Analysette 22 laser diffractometer was approximately 10%. Unfortunately, in this case too, the results from the Helos laser diffractometer were not comparable.

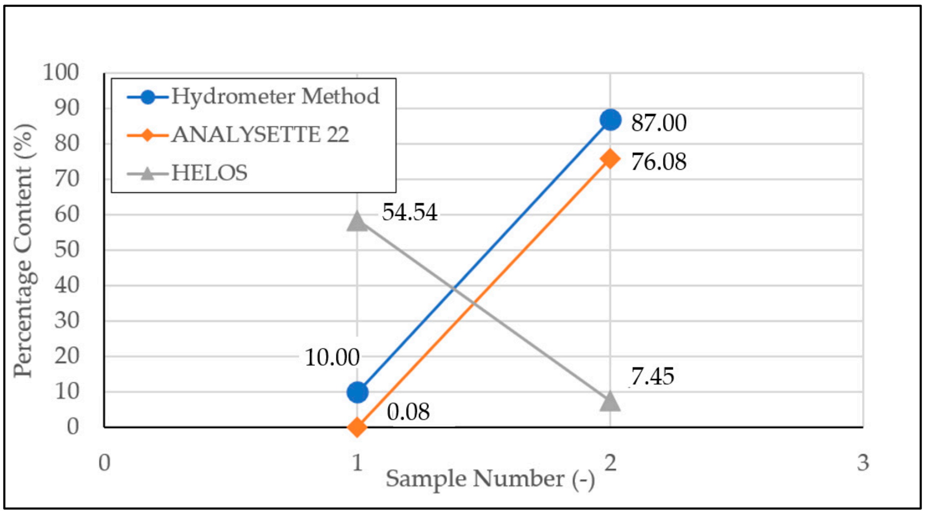

Figure 26. The sand fraction in low-organic soils.

The tests of the particle size distribution of low-organic soil samples showed some agreement in the results obtained between the hydrometric method and the Analysette 22 laser diffractometer, while no comparable results were obtained with the Helos laser diffractometer in either case. The differences between laser diffractometer methods may be due to the design of the instruments and the different methods of dispersing the research material in the chambers. The results obtained should be treated with caution due to the very small number of samples tested.

3.3.2. The Medium-Organic Soils

Looking at the grains size content assigned to the clay fraction in the medium-organic soils samples (Figure 27), there was high similarity of the results between all the methods used, except for sample Nos. 3 and 4, where the results of the hydrometric method were significantly higher (32.0% and 42.5%) than the rest, ranging from 2.5% to 12.68%.

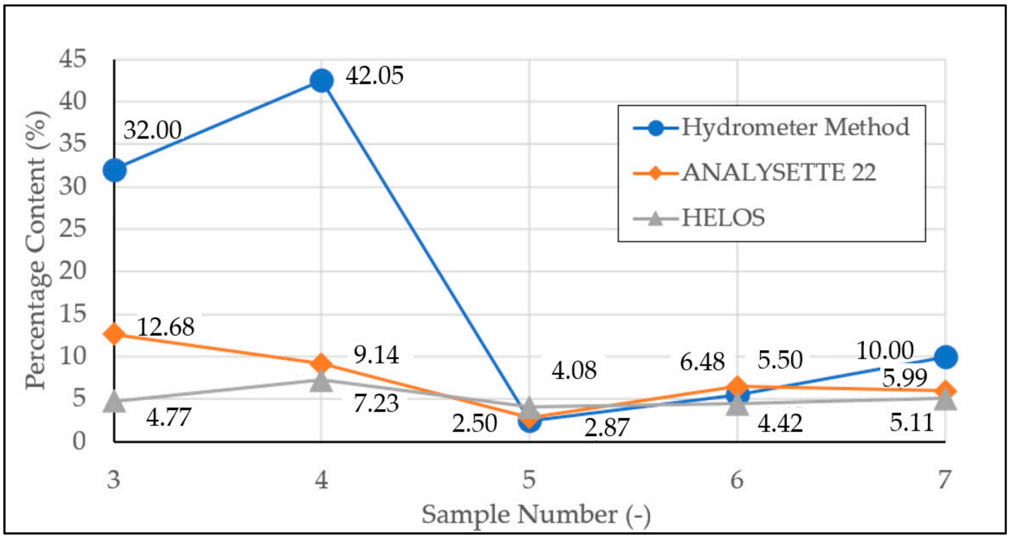

Figure 27. The clay fraction in medium-organic soils.

The analysis of the graphs in Figure 28, showing the test values obtained in the clay fraction, showed that in most cases (sample No. 4, 6 and 7), the laser diffractometer tests were similar. In the case of the hydrometric method, a high level of agreement was observed, but only in one case for the Analysette 22 laser diffractometer (sample No. 5) and Helos (sample No. 3). In general, in this case, values obtained with the reference method were the lowest, with differences with the laser methods of up to 40%, but within a relatively narrow range of values.

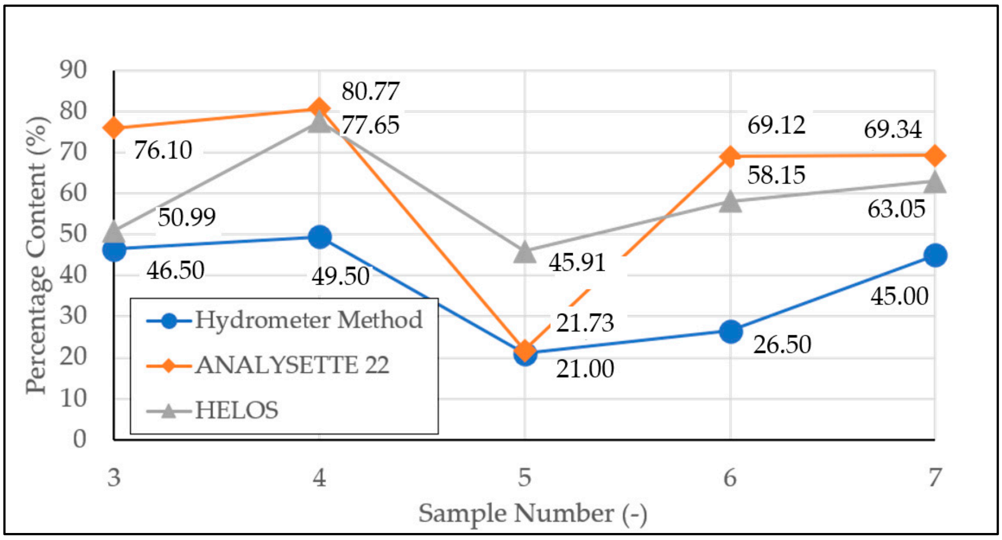

Figure 28. The silt fraction in medium-organic soils.

In the case of identifying the sand fraction (Figure 29), acceptable agreement of the results with all the methods used was only obtained in the case of sample No. 4 (8.0–15.12%). Exceptional agreement of the Analysette 22 laser diffractometer test results with the reference method was recorded in only one case (sample No. 5). Apart from the exceptions described, it is difficult to find significant relationships between the results of these tests.

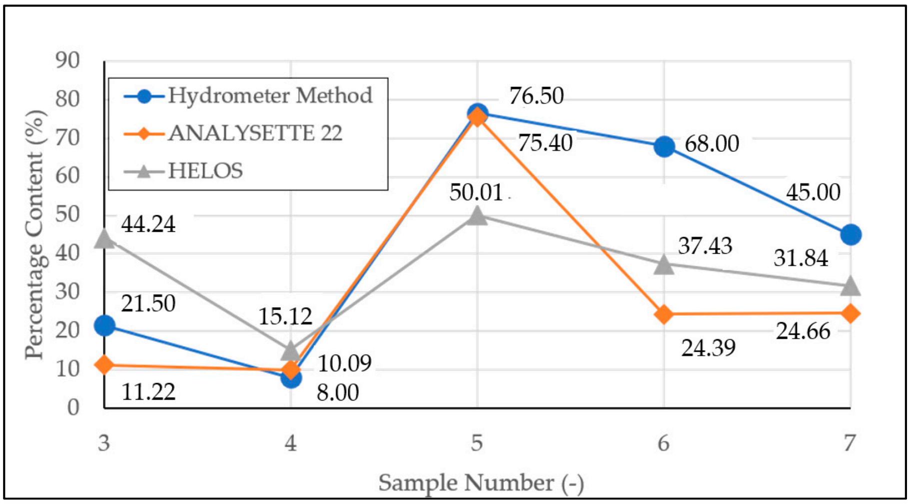

Figure 29. The sand fraction in medium-organic soils.

Paradoxically, in the case of medium-organic soils, the method whose results varied most was the hydrometric method used as a reference. Depending on the type of fraction or sample, the degree of agreement between the results and laser methods ranged from very high similarity to total disagreement.

3.3.3. The High-Organic Soils

When analyzing the clay fraction content of in highly organic soils, it was found that the laser diffractometer results were similar for all samples (No. 8–12), and in the case of sample No. 10–12, they were also in agreement with the results of the reference method. The largest deviations in the results compared to the hydrometric method were observed only in the case of sample Nos. 8 and 9 (Figure 30).

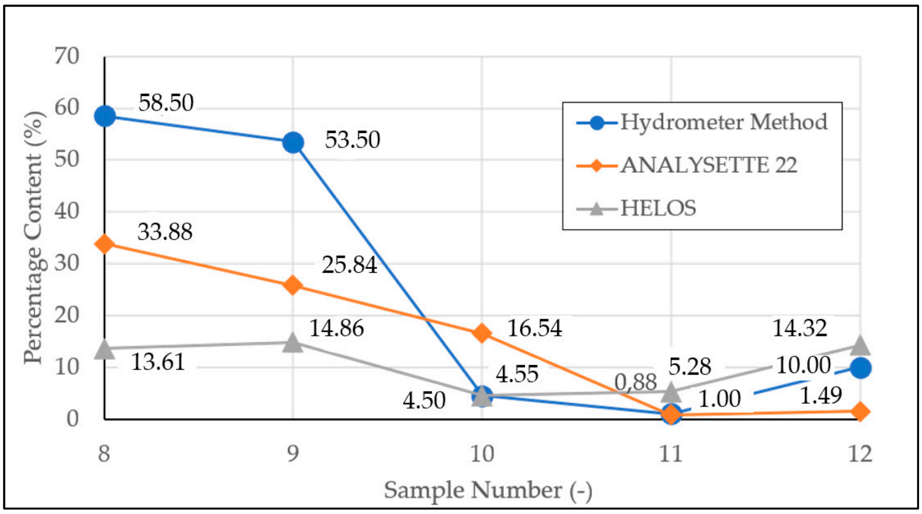

Figure 30. The clay fraction in high-organic soils.

The graph in Figure 31 shows the percentage of grains attributed to the silt fraction, clearly showing that the hydrometric method results were significantly lower than the others, but within a relatively narrow range (6.5–25.0%) for almost all samples. The Analysette 22 laser diffractometer results were remarkably close to the results of the reference method, but only for samples 11 and 12, while for samples 8–10, high agreement with the Helos laser diffractometer results was observed. The narrowest range of results was observed for the results obtained with the Helos laser diffractometer, where the maximum difference in results between samples was only 6.36%.

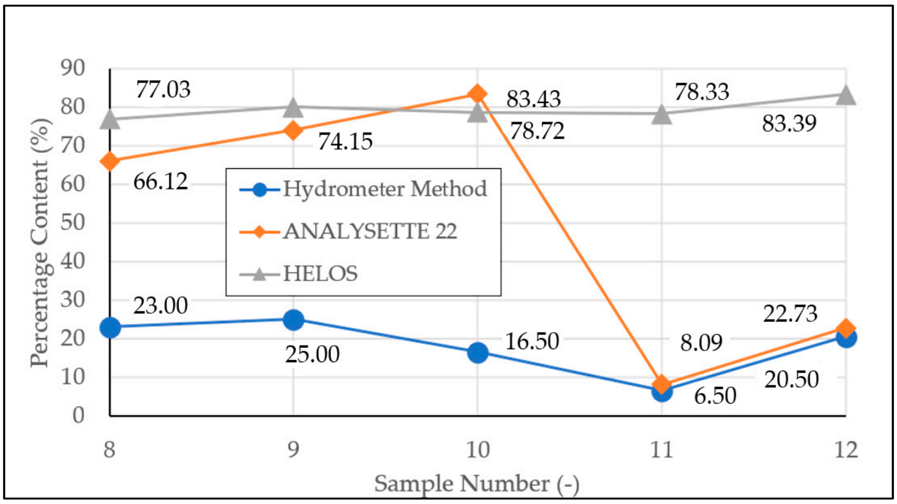

Figure 31. The silt fraction in high-organic soils.

In the case of the sand fraction content determined for highly organic soils, the results of both the reference method and the Analysette 22 laser diffractometer were extremely inconsistent, ranging from 0.0% to 92.5%, with mutual similarities only for the results of sample No. 11 and 12 (Figure 32). In contrast, the analysis of the Helos laser diffractometer results showed that they were relatively close for all samples (2.29%–16.73%), but it is difficult to compare them with the results of the alternative methods, except for sample Nos. 8 and 9, where they were between the results of the hydrometric method and the Analysette 22 laser diffractometer.

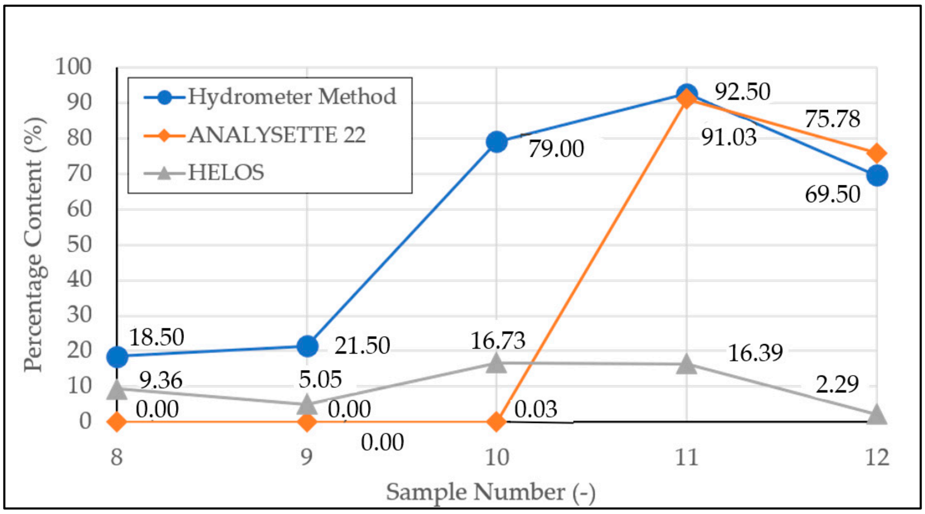

Figure 32. The sand fraction in high-organic soils.

Analyzing the results of the tests of high-organic soils (Figures 30–32) in terms of belonging to individual fractions, it was found that in most cases, much narrower ranges of values were obtained than in the case of low- and medium-organic soils, especially when tested with the Helos laser diffractometer. The content of organic matter in this group was the highest and in the widest range (33.79%–84.14%) compared to the low-organic (4.35%–5.73%) and medium-organic (6.95%–19.64%) soils, suggesting that the distribution the results obtained would be much higher. Regarding the similarity of the results obtained between the methods used, it is difficult to make a clear or definitive statement. It depends on which samples were compared, because although the research materials were assigned to the same classification group, they came from different locations and could have different origins, and could therefore have different chemical compositions or structures, which would be reflected in the research results, under identical test conditions. Nevertheless, the test results for some samples were comparable, as can be seen from the corresponding charts. The largest discrepancies in relation to alternative methods were attributed to the test results obtained using the Analysette 22 laser diffractometer. Unfortunately, in this case, the test results obtained with the reference method can hardly be qualified as useful for verifying the tests results obtained with both laser methods.

The scientific reliability and confirmation of the credibility of these analyses require that the imperfections and limitations of the methodology used are again recognized and described, and the differences in the results are discussed. When analyzing the results from a general perspective, different degrees of similarity were found between the results obtained using the methods applied, ranging from no similarity at all to a high degree of similarity. According to the authors, the main reason for this is the research material itself, i.e., organic soil, which is a composite of mineral grains and organic substances clumped together in varying proportions. Due to the pilot nature of this research, the organic soils were grouped only by organic matter content, as recommended by the standard. However, this classification does not take into account the genesis of the soil, which has a decisive influence on the shape and chemical composition of the organic substance, which is reflected, among other things, in the strength of the bonds between the components that build mineral–organic aggregates, so that even soils with the same content of organic matter may have completely different geotechnical parameters. It is likely that the strength of these solutions may affect the efficiency of the dispersion process at the stage of preparation for diffractometric analysis, as evidenced by the inconsistency of the results between these methods in some cases. One of the main limitations is the choice and reliability of the method for verifying of the laser diffractometer tests results, which is currently limited to a single reference method (hydrometric), because no appropriate alternative method was found despite an intensive search of the available literature. The method is also limited by the research capabilities of the instruments, both the measurement method itself and the way in which the research material is dispersed, which vary depending on the device and can produce inconsistent test results. At this stage of our work, we are not able to eliminate these imperfections or explain all the unknowns, but we hope that this manuscript will become a reason for increased interest in this type of research, and their continuation, although tedious, expensive and time-consuming from a certain perspective, may bring significant benefits for geoengineering.

4. Conclusions

This manuscript presents detailed results of tests of the usefulness of two types of diffractometers, i.e., Analysette 22 and Helos, for determining the grain composition of the full spectrum of organic soils and draws the following conclusions:

- In the group of low-organic soils (sample No. 1, 2), the conclusions are not clear, because the soil results of sample No. 1 did not show any significant similarity between the laser methods and the reference method, whereas in the case of No. 2, an apparent similarity was observed between the laser diffractometer method results, which also differed significantly from the reference method.
- In the group of medium-organic soils (sample Nos. 3–7), there was, in most cases, some, sometimes even high, agreement between the test results performed using both laser diffractometers, but they were not comparable with the results of the reference method.
- In the group of high-organic soils (sample Nos. 8–12), we found exceptional agreement of the test results between the hydrometric method and the Analysette 22 laser diffractometer in almost all cases.

When analyzing the above conclusions, it should be borne in mind that the results obtained may be influenced not only by the quantity, but also by the type of substances and the type of connection of the materials constituting the mineral–organic aggregate. Unfortunately, this requires a comprehensive physico-chemical study, which is beyond the scope of this work. The tests also showed that the dispersion method can also affect the results obtained, and the capabilities of a specific diffractometer may also have an impact on the obtained results. A fully reliable, alternative reference method is also lacking. However, these limitations do not diminish the research potential of laser diffraction methods, whose main value lies in the speed and repeatability of the test and the minimally simplified procedure for preparing samples for testing, which effectively eliminates errors associated with the human factor.

In conclusion, the authors believe that the laser diffractometer method, if reliably verified, could be an effective tool for determining the grain composition of a material with an extremely complex skeleton structure, such as organic soils, for which classical or standard testing methods are not fully effective.

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