PERFORMANCE OF A RECIPROCAL SHAKER IN MECHANICAL DISPERSION OF SOIL SAMPLES FOR PARTICLE-SIZE ANALYSIS[1]

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SUMMARY

The dispersion of the samples in soil particle-size analysis is a fundamental step, which is commonly achieved with a combination of chemical agents and mechanical agitation. The purpose of this study was to evaluate the efficiency of a low-speed reciprocal shaker for the mechanical dispersion of soil samples of different textural classes. The particle size of 61 soil samples was analyzed in four replications, using the pipette method to determine the clay fraction and sieving to determine coarse, fine and total sand fractions. The silt content was obtained by difference. To evaluate the performance, the results of the reciprocal shaker (RSh) were compared with data of the same soil samples available in reports of the Proficiency testing for Soil Analysis Laboratories of the Agronomic Institute of Campinas (Prolab/IAC). The accuracy was analyzed based on the maximum and minimum values defining the confidence intervals for the particle-size fractions of each soil sample. Graphical indicators were also used for data comparison, based on dispersion and linear adjustment. The descriptive statistics indicated predominantly low variability in more than 90 % of the results for sand, medium-textured and clay samples, and for 68 % of the results for heavy clay samples, indicating satisfactory repeatability of measurements with the RSh. Medium variability was frequently associated with silt, followed by the fine sand fraction. The sensitivity analyses indicated an accuracy of 100 % for the three main separates

(total sand, silt and clay), in all 52 samples of the textural classes heavy clay, clay and medium. For the nine sand soil samples, the average accuracy was 85.2 %; highest deviations were observed for the silt fraction. In relation to the linear adjustments, the correlation coefficients of 0.93 (silt) or > 0.93 (total sand and clay), as well as the differences between the angular coefficients and the unit < 0.16, indicated a high correlation between the reference data (Prolab/IAC) and results obtained with the RSh. In conclusion, the mechanical dispersion by the reciprocal shaker of soil samples of different textural classes was satisfactory. The results allowed recommending the use of the equipment at low agitation for particle size- analysis. The advantages of this Brazilian apparatus are its low cost, the possibility to simultaneously analyze a great number of samples using ordinary, easily replaceable glass or plastic bottles.

Index terms: soil texture, pipette method, mechanical dispersion, accuracy analysis.

RESUMO: *AVALIAÇÃO DO DESEMPENHO DE MESA AGITADORA RECIPROCANTE NA DISPERSÃO DE AMOSTRAS DE SOLO PARA FINS DE ANÁLISE GRANULOMÉTRICA*

A dispersão da amostra de solo é uma etapa fundamental da análise granulométrica, sendo realizada mediante o uso de dispersantes químicos e agitação mecânica. O objetivo deste trabalho foi avaliar a eficiência de mesa agitadora reciprocante de baixa rotação na dispersão mecânica de amostras de solos de diferentes classes texturais. Foram realizadas análises granulométricas em 61 amostras com quatro repetições, empregando o método da pipeta para determinação da fração argila e tamisagem para determinação das frações areia grossa, areia fina e areia total, sendo o silte determinado por diferença. Na avaliação de desempenho, os resultados obtidos com uso da mesa agitadora reciprocante (MAR) foram comparados com dados disponíveis para as mesmas amostras oriundos de relatórios do Ensaio de Proficiência IAC para Laboratórios de Análises de Solos - Prolab/IAC. Análises de acurácia foram realizadas com base nos valores dos intervalos de confiança definidos para cada fração granulométrica componente de cada amostra ensaiada. Indicadores gráficos também foram utilizados na comparação de dados, por meio de dispersão e ajuste linear. A estatística descritiva indicou preponderância de baixa variabilidade em mais de 90 % dos resultados obtidos para as amostras de texturas arenosa, média e argilosa e em 68 % dos obtidos para as amostras de textura muito argilosa, indicando boa repetibilidade dos resultados obtidos com a MAR. Média variabilidade foi mais frequentemente associada à fração silte, seguida da fração areia fina. Os resultados das análises de sensibilidade indicam acurácia de 100 % nas três frações granulométricas - areia total, silte e argila - para todas as amostras analisadas pertencentes às classes texturais muito argilosa, argilosa e média. Para as nove amostras de textura arenosa, a acurácia média foi de 85,2 %, e os maiores desvios ocorreram em relação à fração silte. Nas aproximações lineares, coeficientes de correlação igual (silte) ou superiores (areia total e argila) a 0,93, bem como diferenças menores do que 0,16 entre os coeficientes angulares das retas e o valor unitário, indicam alta correlação entre os resultados de referência (Prolab/IAC) e os obtidos nos ensaios com a MAR. Conclui-se pelo desempenho satisfatório da mesa agitadora reciprocante de baixa rotação para dispersão mecânica de amostras de solo de diferentes classes texturais para fins de análise granulométrica, permitindo recomendar o uso alternativo do equipamento quando se emprega agitação lenta. As vantagens do uso do equipamento nacional incluem o baixo custo, a possibilidade de análise simultânea de grande número de amostras e o uso de frascos comuns, de vidro ou de plástico, baratos e de fácil reposição.

Termos de indexação: granulometria, método da pipeta, dispersão mecânica, análise de acurácia.

INTRODUCTION

Soil texture is based on different combinations of sand, silt, and clay separates that define the particle-size distribution of a soil sample (Gee & Or, 2002). Particle-size distribution is a natural and permanent soil property and one of the most frequently used for soil characterization (Hillel, 1982). Because of the correlation between specific surface and particle size, the percentage distribution of the various sizes of individual particles within a soil is an important soil characteristic (Baver et al., 1972). The particle-size distribution of a soil is determined by particle-size analysis. Particle-size analysis is defined as a measurement of the size distribution of the individual (primary) particles in a soil sample, according to texture fractions in a given classification scheme (Baver et al., 1972; Gee & Or, 2002). The analysis of particle sizes is a common and essential physical analysis of the soil, for which conventionally "fine earth" is used, or the soil fraction that can be sieved through 2 mm mesh. The size limits of the three main fractions of soil particles sand, silt and clay are given by diameter ranges, according to different scales. The sand fraction contains the largest particles, with diameters between 2.0 and 0.02 mm (ISSS) or 2.0 and 0.05 mm (USDA). Silt consists of medium-sized particles, with diameters from 0.02 to 0.002 mm (ISSS) or from 0.05 to 0.002 mm (USDA), and the clay fraction contains the smallest soil particles, with diameters below 0,002 mm or 0,2µ on both scales (Gee & Or, 2002).

Soil texture is widely recognized as being fundamental for soil identification and classification. More recently, particle-size distribution has been widely used as an independent variable in pedotransfer functions for the estimation of more complex soil physical properties (Tomasella et al., 2000, Silva et al., 2008). In the soil, organic matter, iron oxides and carbonates act as cementing agents, keeping the particles together and forming aggregates. The success of particle-size analysis depends firstly on the sample preparation to ensure a complete dispersion of all aggregates into their individual primary particles without breaking up the particles themselves and secondly, on the accurate fractionation of a sample into its different separates (Baver et al., 1972). Thus, the dispersion phase consists of the individualization of the soil primary particles in aqueous suspension, by using chemical agents and physical methods (Gee & Or, 2002). The chemical agents are used to eliminate the flocculating ions, such as Al and Ca, to increase the repulsion between the primary particles, and to stabilize the individual particles in the suspension throughout the analysis (Gee & Or, 2002; Ruiz, 2005). In Brazil, the Brazilian Agricultural Research Corporation - Embrapa (1997) recommends the chemical agents Na-hydroxide (NaOH) or Na-hexametaphosphate buffered with Na-carbonate for normal soils, the use of hydrogen chloride (HCl) at

10 % for calcareous soils, and Na-hexametaphosphate for saline soils. In São Paulo State, the Agronomic Institute of Campinas (IAC) recommends the use of a mixture of Na-hydroxide and Na-hexametaphosphate (Camargo et al., 1986). Sodium can adsorb high quantities of water and when the Na ion is adsorbed on the surface of soil particles it induces repulsion between them, which in turn facilitates the stabilization of the individual particles in suspension. In recent research, Neto et al. (2009) evaluated the effectiveness of different chemical agents for the dispersion of a Rhodic Hapludox irrigated with calcium-rich water. They concluded that the combination of hydrogen chloride (HCl) with Na-hydroxide (NaOH) was the most efficient way of recovering the clay fraction. Classical physical methods of soil dispersion include fast or slow shaking or rolling of the soil suspension. In the past 20 years, electronic dispersion, primarily by the use of sonication, has become increasingly popular (Gee & Or, 2002). In Brazil, Embrapa (1997) recommends the use of electric stirrers at high speed (10,000 to 12,000 rpm) for a short stirring time, varying from 5 min (for sandy soils) to 15 min (for clayey soils). In São Paulo, Grohmann & Raij (1977) demonstrated the superiority of slow shaking for a more efficient soil dispersion and clay determination for soil particle-size analysis performed in the IAC laboratories. Later, Camargo et al. (1986) recommended the use of low-speed stirrers (30 rpm), for example the Wiegner shaker, with longer stirring time, of about 16 h. According to Gee & Or (2002), not only a standardization of the treatments but also the testing of specific methodologies are needed, since the mechanical techniques can result in the fragmentation of the primary particles. The reciprocal shaker evaluated here belongs to the category of slow-speed shakers, for which there are no comparative performance studies based on the results obtained with methodologies and agitators commonly used in the laboratories. The advantages of this reciprocal shaker include its relatively low cost, the capacity to shake a large number (40) of samples simultaneously, and the possibility of using cheap and easy replaceable glass or plastic pots. Given the above, our objective was to evaluate the efficiency of a slow reciprocal shaker for the dispersion of soil samples of different textural classes for particle-size analysis.

MATERIAL AND METHODS

Experimental location and characterization of the soil samples

The experiment was conducted at the Soil Laboratory (Labsol) of the College of Agricultural Engineering, State University of Campinas, Campinas, SP.

The tests were conducted with 61 soil samples of different textural classes. According to the textural groups defined by Embrapa (2006), the texture of nine of the soil samples was sandy (containing <150 g kg⁻¹ clay and > 700 g kg⁻¹ sand), of 22 medium (with clay contents < 350 g kg⁻¹ and > 150 g kg⁻¹), of 20 samples clayey (with clay contents > 350 g kg⁻¹ and < 600 g kg⁻¹), and of the last 10 samples the texture was Heavy Clay (with clay contents > 600 g kg⁻¹).

The samples were selected from a soil bank of the Labsol, due to its participation in the IAC Proficiency Testing for Soil Analyses Laboratories - Prolab/IAC. The choice of this soil sample bank was due to the availability of results of particle-size analyses performed by more than 80 laboratories participating in the program. The data were used as references in the performance evaluation of the reciprocating shaker. The statistical procedures used in the Prolab/IAC define the average value and the acceptable range of results obtained for each particle-size fraction (coarse sand, fine sand, total sand, silt and clay) for each soil sample (Quaggio et al., 1994). The acceptable range depends on the coefficient of variation (CV) of the results for each particle-size fraction for each soil sample, according to the following criteria: a) for CV > 40 %, the acceptable range is the average ± 1.0 standard deviation *(s)* calculated from the results obtained by all laboratories; b) for CV between 20 and 40 %, the acceptable range is the average ± 1.5 *s,* and c) for CV < 20 %, the acceptable range of results is the average ± 2.0 *s.* These data sets of particle-size analyses determined independently and as a part of a soil analysis quality program, were considered appropriate to draw conclusions not only about the accuracy but also on the precision of the results obtained by mechanical dispersion with a reciprocal shaker (RSh).

Analysis methods

The particle-size analyses were carried out by the Pipette Method described by Day (1965) for clay determination, by sieving for separation of the sands and by the difference between the former to obtain silt, according to the procedures described by Embrapa (1997) and Camargo et al. (1986).

a) Principles

The pipette method is a direct sampling procedure based on the particle settling speed in an aqueous suspension according to Stokes' law (Equation 1). The basic assumptions to apply Stokes' law to soil suspensions are: a) the terminal velocity is reached as soon as settling begins; b) resistance to settling is entirely due to the viscosity of the fluid; c) particles are smooth and spherical; d) there is no interaction between individual particles in the suspension (Gee & Or, 2002). Since the soil particles are not smooth and spherical, *d* must be regarded as equivalent rather than actual diameters. The methods of particle-size analysis based on the settling velocity determine the soil particles more precisely according to the *settling*

time, as defined by equation 2. For clay fraction determination, after the dispersion of the soil sample, the time and the distance of vertical displacement of the particles through the aqueous suspension are fixed, so that only the clay particles remain in suspension at that depth. At the time *t*, a small subsample is taken from the suspension at depth *h*, according to equation 2. After oven-drying and subtracting the dispersant weight (blank test), the clay mass of the soil sample is determined. To determine the sand fraction, the soil suspension is passed either through a set of two sieves, to separate the coarse from the fine sand fractions (ISSS), or through a set of five sieves, to separate the sand in very coarse, coarse, medium, fine and very fine (USDA). Then, the sand fractions are oven-dried and weighed for content determinations. After determining the sand and the clay fractions, the silt fraction is calculated by the difference (Camargo et al., 1986; Embrapa, 1997). The pipette method is often used as a standard method and the results of the particle-size analysis are expressed in g kg⁻¹ by the International System.

$$v = \frac{d^2 g\,(\rho_S - \rho_F)}{18\eta} \qquad \text{Eq. 1}$$

$$t = \frac{9\eta h}{2(\rho_S - \rho_F)\,gr^2} \qquad \text{Eq. 2}$$

b) Analytical procedure

For soils with less than 5 % of organic matter (OM) (< 50 g kg⁻¹), 10 g of the < 2mm fraction of air-dried soil was weighed and transferred to a 500 mL glass pot with 50 mL of dispersant solution* (a mixture of 20 g of Na-hydroxide PA and 50 g of Na-hexametaphosphate in 5 L of distilled water, stirred with magnetic stirrer until the reagents were dissolved). After closing, the glass pot was placed on the low-speed reciprocal shaker (RSh) for mechanical stirring at 130 rpm for 14-16 h (Figure 1).

The stirring velocity was determined based on preliminary tests at different speeds, which demonstrated that 130 rpm was the ideal rotation speed to promote an effective movement of the suspension within the glass pot. For soils containing more than 5 % of organic matter (> 50 g kg⁻¹), a pre-treatment was required to eliminate OM as follows: fill 10 g of the < 2mm fraction of air-dried fine earth into a 800 mL beaker, add 200 mL of Na-pyrophosphate 0.1 mol L⁻¹ and 50 mL of hydrogen peroxide (H_2O_2 ~30 %), and let it stand overnight. The next day, maintain at 40 °C in water bath for 8 h, and stir with a glass rod every 2 h. To remove the excess of H_2O_2, raise the temperature to 80 °C, until almost dry. Wash the sample, centrifuge it twice with distilled water, and remove the supernatant. Air-dry the sample, grind and weigh the quantity required for the particle-size analysis.

After the stirring period, the suspension was sieved through a 0.053 mm (270 mesh) into a 0.5 L sedimentation cylinder for sand separation. The material retained on the sieve (sand) was transferred to a 0.4 L beaker, and oven-dried at 105 °C. Then the dried sands were transferred to a set of two sieves: 0.21 mm (coarse sand) and 0.05 mm (fine sand), and shaken for 30 min on a sieve vibrator. The masses of the coarse sand and of the fine sand were weighed (precision 0.01 g).

The volume of the suspended material was completed to 0.5 L and the test tube placed in a water bath. The suspension was stirred for 30 s with a glass rod with a plunger slightly smaller than the cylinder diameter attached to its lower end. The settling time was defined according to the suspension temperature.

After the sedimentation period required for clay recovery only, 10 mL of the suspension were pipetted from a depth of 5 cm, transferred to a tared beaker (precision 0.0001 g), and dried at 105 ^{0}C for 24 h. After drying, the beaker was placed in a desiccator until reaching room temperature, then weighed (precision 0.0001 g) and the weight of the clay + dispersing agents determined.

A blank test was performed to determine the weight of the dispersing agents by preparing a solution with the same concentration used in the analysis (50 mL of dispersing solution* in a sedimentation cylinder + water to complete 0.5 L). A 10 mL aliquot of the solution was transferred to a beaker and oven-dried at 105 ^{0}C for 24 h. After drying, the beaker was placed in a desiccator until reaching room temperature; then it was weighed (precision 0.0001 g) to determine the mass of the dispersing agents contained in the 10 mL aliquot. This value was subtracted from the weight of clay + dispersing agents to determine the clay content only.

Figure 1. A view of the reciprocal shaker (RSh) as used for the mechanical dispersion of the soil samples using common glass pots.

The silt fraction was calculated by the difference between the sum of the sand and the clay fractions in relation to 1000g, since the results should be expressed in g kg^{-1} (Camargo et al., 1986; Embrapa, 1997). For each soil sample, particle-size analysis was performed in four replications.

c) Statistical analysis of the particle-size analysis results and performance evaluation of the reciprocal low speed shaker (RSh)

The results were subjected to descriptive analysis to determine the following values: mean, standard deviation, and the maximum, minimum and coefficient of variation, using the SAS statistical program. The data variability, expressed by the coefficient of variation (CV %), was evaluated according to the Warrick & Nielsen (1980) criteria, by which coefficients below 12 % indicate low variability; the coefficients varying between 12 and 60 % medium variability; and the coefficients > 60 % high variability.

For a performance analysis and to validate the results obtained with the RSh, the data accuracy was analyzed (Fletcher et al., 1986), based on confidence intervals (IC) as the criteria of acceptance, as defined by the particle-size data of the analysis performed with the same samples by the laboratories of the Prolab/IAC program (Tables 1 and 2). Graphic indicators were also used to compare the results, using the ORIGINPRO^R 7.5 software, which visualized data dispersion and the linear fit between the average content of a given particle-size fraction (sand, silt or clay) for each soil sample (reference data obtained from the Prolab/IAC program) and the value obtained using the RSh.

RESULTS AND DISCUSSION

The results of the descriptive analysis for coarse sand, fine sand, total sand, clay and silt fractions for nine sandy soil samples (containing < 150 g kg^{-1} clay and > 700 g kg^{-1} sand) are shown in Table 3. For all sandy samples (9), the coefficients of variation (CV) of the results for fine sand, total sand and clay were lower than 12 %, indicating low variability. The variability in the results for coarse sand was also low, except for sample 2, for which the CV of 13.95 % indicated medium variability, according to the criteria of Warrick & Nielsen (1980). For silt, medium variability was observed for three samples (2, 4 and 6); however, for the remaining six samples, variability was also low. In general, low variability was characterized for 91 % of the results obtained for the sandy soil samples.

The results shown in table 4 were obtained from 21 medium-textured soil samples. For almost 92 % of the data, including coarse sand, total sand and clay fractions of all tested samples, variability was low

including the clay fraction (all samples), coarse sand (samples 54, 55, 56, 57), fine sand (samples 52, 54, 55, 56, 57, 58, 59, 61), total sand (samples 52, 54, 55, 56, 57, 58) and silt (samples 53, 54, 56, 57, 58). Medium variability was inferred for coarse sand (samples 52, 53, 58, 59, 60, 61), fine sand (sample 53), total sand (samples 53, 59, 60, 61), and silt (samples 52, 55, 59, 60, 61).

The prevalence of low variability for various data sets indicates a good repeatability of the results of the analysis using the low speed reciprocal shaker (RSh). Medium variability was characterized

Table 2. Confidence intervals for coarse sand, fine sand, total sand, silt and clay contents for 20 Clayey soil samples and for 10 Heavy Clay soil samples

Sample	Sand			Clay	Silt
	Coarse	Fine	Total		
			g kg^{-1}		
			Clayey soil samples		
32	183 to 264	105 to 159	307 to 417	401 to 568	94 to 203
33	271 to 361	79 to 141	379 to 479	322 to 466	109 to 242
34	122 to 180	92 to 148	216 to 329	457 to 622	116 to 261
35	118 to 188	89 to 150	215 to 333	459 to 603	133 to 248
36	196 to 268	104 to 163	327 to 403	404 to 570	102 to 195
37	215 to 336	71 to 147	308 to 483	310 to 509	126 to 248
38	138 to 171	99 to 143	229 to 322	472 to 638	101 to 215
39	188 to 264	103 to 173	325 to 400	434 to 542	95 to 200
40	126 to 178	96 to 143	242 to 304	480 to 604	116 to 251
41	239 to 341	145 to 235	454 to 506	365 to 478	62 to 128
42	30 to 62	69 to 134	103 to 194	406 to 603	240 to 456
43	27 to 60	52 to 103	76 to 160	421 to 623	251 to 448
44	252 to 347	147 to 223	461 to 506	373 to 460	71 to 131
45	24 to 53	46 to 100	76 to 148	436 to 694	194 to 423
46	237 to 340	154 to 227	455 to 507	380 to 452	72 to 135
47	227 to 314	151 to 225	431 to 485	391 to 492	63 to 136
48	242 to 322	92 to 168	374 to 451	353 to 518	100 to 205
49	231 to 340	74 to 134	361 to 426	411 to 502	105 to 200
50	124 to 287	71 to 136	205 to 408	332 to 574	176 to 308
51	85 to 128	145 to 226	250 to 338	482 to 610	101 to 219
			Heavy Clay Soil Samples		
52	40 to 77	60 to 110	100 to 214	526 to 737	122 to 283
53	11 to 32	7 to 27	16 to 75	578 to 735	220 to 381
54	40 to 76	65 to 108	113 to 181	520 to 741	142 to 280
55	40 to 77	69 to 102	106 to 186	560 to 720	133 to 283
56	48 to 83	97 to 139	147 to 214	508 to 698	136 to 282
57	43 to 89	96 to 142	153 to 215	541 to 683	124 to 279
58	42 to 85	99 to 139	147 to 218	542 to 677	135 to 272
59	44 to 91	103 to 142	156 to 226	543 to 677	125 to 282
60	52 to 86	101 to 146	159 to 229	554 to 677	131 to 253
61	49 to 88	104 to 146	167 to 223	559 to 673	128 to 241

Source: Values extracted from the Prolab/IAC reports.

Table 3. Descriptive statistics of the results from particle-size analyses of nine Sandy soil samples (n= 4 replications)

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			g kg^{-1}			%
1	Coarse Sand	269	9.12	283	242	7.12
	Fine Sand	475	18.92	500	458	3.98
	Total Sand	744	3.00	746	740	0.4
	Clay	117	3.37	122	115	2.88
	Silt	140	2.87	144	138	2.06
2	Coarse Sand	315	43.96	352	256	13.95
	Fine Sand	507	29.85	548	485	5.81
	Total Sand	822	14.32	837	804	1.74
	Clay	66	2.63	68	62	3.99
	Silt	112	14.08	130	96	12.63
3	Coarse Sand	303	23.64	336	284	7.80
	Fine Sand	532	18.23	546	505	3.43
	Total Sand	835	7.59	841	824	0.91
	Clay	76	2.75	79	73	3.61
	Silt	89	7.54	98	82	8.45
4	Coarse Sand	419	19.41	447	402	4.63
	Fine Sand	510	17.26	524	485	3.38
	Total Sand	929	2.50	932	926	0.27
	Clay	43	3.37	47	39	7.83
	Silt	28	4.92	32	23	17.75
5	Coarse Sand	372	30.72	402	341	8.25
	Fine Sand	470	25.72	494	442	5.47
	Total Sand	842	6.24	850	835	0.74
	Clay	45	1.16	46	44	2.57
	Silt	113	5.32	119	106	4.73
6	Coarse Sand	355	23.42	388	336	6.60
	Fine Sand	570	18.08	585	544	3.18
	Total Sand	925	5.45	932	920	0.59
	Clay	44	3.32	48	40	7.45
	Silt	31	5.48	35	23	17.67
7	Coarse Sand	330	20.63	357	307	6.26
	Fine Sand	508	29.87	545	472	5.87
	Total Sand	838	9.90	852	829	1.18
	Clay	69	5.60	74	62	8.11
	Silt	93	4.97	97	86	5.34
8	Coarse Sand	326	20.05	353	308	6.15
	Fine Sand	499	20.04	514	471	4.01
	Total Sand	825	3.40	828	821	0.41
	Clay	84	2.06	87	82	0.45
	Silt	91	1.92	92	88	2.12
9	Coarse Sand	320	30.93	349	293	9.66
	Fine Sand	508	28.04	533	482	5.53
	Total Sand	828	3.11	831	824	0.38
	Clay	84	3.50	88	80	4.15
	Silt	88	1.71	90	86	1.94

Table 4. Descriptive statistics of the results from particle-size analyses of 21 Medium textured soil samples (n = 4 replications)

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			———————————— g kg⁻¹ ————————————			%
10	Coarse Sand	379	38.06	416	340	10.05
	Fine Sand	264	28.47	288	235	10.80
	Total Sand	642	10.48	651	628	1.63
	Clay	275	13.40	287	256	4.86
	Silt	83	8.42	93	73	10.17
11	Coarse Sand	432	13.24	446	415	3.06
	Fine Sand	113	9.63	123	104	8.51
	Total Sand	545	6.60	550	536	1.21
	Clay	328	9.39	339	320	2.86
	Silt	127	10.86	135	111	8.55
12	Coarse Sand	334	13.52	346	317	4.05
	Fine Sand	259	10.34	272	249	4.00
	Total Sand	592	4.24	598	589	0.71
	Clay	331	22.80	365	316	6.88
	Silt	77	21.26	95	46	27.70
13	Coarse Sand	424	2.38	427	422	0.56
	Fine Sand	121	2.62	125	119	2.16
	Total Sand	545	2.06	547	543	0.37
	Clay	325	9.35	334	312	2.87
	Silt	130	7.74	141	123	5.95
14	Coarse Sand	420	25.10	447	396	5.98
	Fine Sand	120	12.44	132	109	10.35
	Total Sand	540	13.54	557	526	2.50
	Clay	328	11.81	342	316	3.60
	Silt	133	10.21	147	124	7.70
15	Coarse Sand	307	15.77	324	287	5.13
	Fine Sand	266	10.07	277	253	3.77
	Total Sand	574	7.25	581	564	1.26
	Clay	325	8.22	336	316	2.52
	Silt	101	6.50	110	95	6.45
16	Coarse Sand	476	51.59	521	406	10.83
	Fine Sand	84	9.60	98	76	11.40
	Total Sand	561	49.18	597	489	8.77
	Clay	290	8.00	302	286	2.75
	Silt	149	50.62	225	117	33.86
17	Coarse Sand	336	13.96	347	316	4.14
	Fine Sand	247	7.78	258	241	3.15
	Total Sand	584	6.45	588	574	1.10
	Clay	332	7.27	337	321	2.19
	Silt	85	13.67	105	76	16.13
18	Coarse Sand	283	6.97	293	277	2.46
	Fine Sand	285	6.48	293	280	2.26
	Total Sand	569	5.03	574	562	0.88
	Clay	332	6.83	341	325	2.05
	Silt	99	4.00	105	97	4.04
19	Coarse Sand	347	34.59	380	304	9.97
	Fine Sand	238	21.30	263	217	8.95
	Total Sand	585	13.42	597	567	2.29
	Clay	331	7.22	338	325	2.18
	Silt	84	7.52	95	78	8.96

Table 4. Cont.

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			g kg⁻¹			%
20	Coarse Sand	336	13.24	346	317	3.94
	Fine Sand	245	10.47	255	231	4.28
	Total Sand	581	7.23	587	572	1.24
	Clay	336	13.42	354	324	3.99
	Silt	83	6.50	89	74	7.80
21	Coarse Sand	340	16.87	363	326	4.96
	Fine Sand	240	15.34	257	220	6.39
	Total Sand	580	4.85	583	573	0.83
	Clay	327	10.23	341	318	3.12
	Silt	93	5.85	99	86	6.30
22	Coarse Sand	329	31.03	363	295	9.43
	Fine Sand	248	36.76	289	210	14.82
	Total Sand	577	6.05	584	571	1.04
	Clay	326	10.47	336	312	3.21
	Silt	98	4.65	104	93	4.77
23	Coarse Sand	489	26.21	508	450	5.36
	Fine Sand	180	22.36	213	167	12.46
	Total Sand	668	6.68	676	662	1.00
	Clay	217	5.12	224	212	2.36
	Silt	115	3.94	121	112	3.42
24	Coarse Sand	411	4.03	416	407	0.98
	Fine Sand	107	4.57	114	104	4.26
	Total Sand	519	4.04	522	513	0.77
	Clay	346	3.77	349	342	1.09
	Silt	136	5.88	144	130	4.32
25	Coarse Sand	292	23.36	305	257	8.00
	Fine Sand	301	16.17	325	292	5.37
	Total Sand	593	7.22	597	582	1.21
	Clay	299	7.43	309	292	2.48
	Silt	109	3.10	111	104	2.86
26	Coarse Sand	415	23.18	449	400	5.59
	Fine Sand	111	13.22	120	91	11.97
	Total Sand	525	11.16	540	516	2.12
	Clay	337	12.01	349	325	3.56
	Silt	138	4.71	145	135	3.41
27	Coarse Sand	428	10.62	436	413	2.48
	Fine Sand	75	44.37	106	10	58.58
	Total Sand	504	44.76	534	437	8.89
	Clay	338	6.84	348	334	2.02
	Silt	159	47.57	229	128	29.96
28	Coarse Sand	345	9.69	359	338	2.81
	Fine Sand	262	11.72	273	245	4.47
	Total Sand	607	2.62	611	605	0.43
	Clay	297	3.41	301	293	1.15
	Silt	96	2.06	99	94	2.14
29	Coarse Sand	353	14.27	368	336	4.04
	Fine Sand	250	13.88	265	234	5.56
	Total Sand	602	0.95	603	601	0.15
	Clay	297	6.68	304	288	2.25
	Silt	101	7.50	111	93	7.44
30	Coarse Sand	586	10.87	599	575	1.85
	Fine Sand	83	4.69	88	79	5.65
	Total Sand	669	6.65	678	663	0.99
	Clay	229	22.54	262	213	9.85
	Silt	103	18.44	114	75	17.99

Table 5. Descriptive statistics of the results from particle-size analysis of 20 Clay soil samples (n = 4 replications)

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			———————————— g kg^{-1} ————————————			%
32	Coarse Sand	236	5.16	242	230	2.18
	Fine Sand	133	4.65	138	127	3.51
	Total Sand	369	0.57	369	368	0.15
	Clay	493	7.14	498	482	1.45
	Silt	139	6.78	149	134	4.87
33	Coarse Sand	296	11.23	309	283	3.79
	Fine Sand	127	8.96	135	115	7.08
	Total Sand	422	2.87	424	418	0.68
	Clay	442	9.53	450	428	2.15
	Silt	136	12.35	154	126	9.08
34	Coarse Sand	161	7.67	167	150	4.76
	Fine Sand	115	8.50	128	111	7.37
	Total Sand	277	1.91	278	274	0.69
	Clay	566	9.67	574	554	1.70
	Silt	157	8.13	168	150	5.17
35	Coarse Sand	156	9.57	168	148	6.15
	Fine Sand	118	4.35	120	111	3.70
	Total Sand	273	12.67	288	259	4.64
	Clay	572	3.59	577	569	0.62
	Silt	155	13.93	172	142	9.00
36	Coarse Sand	237	9.39	248	225	3.96
	Fine Sand	132	5.56	138	125	4.22
	Total Sand	369	7.18	375	359	1.95
	Clay	499	12.50	508	481	2.50
	Silt	133	7.88	144	126	5.92
37	Coarse Sand	253	27.42	291	226	10.86
	Fine Sand	105	23.6	140	91	22.44
	Total Sand	358	24.93	389	337	6.97
	Clay	470	35.77	507	425	7.61
	Silt	173	26.97	204	146	15.61
38	Coarse Sand	151	7.87	158	140	5.21
	Fine Sand	116	9.60	125	104	8.24
	Total Sand	267	5.19	274	262	1.94
	Clay	578	8.13	585	567	1.40
	Silt	155	9.27	168	147	5.98
39	Coarse Sand	244	9.83	256	233	4.02
	Fine Sand	120	18.51	130	92	15.46
	Total Sand	364	12.36	377	348	3.39
	Clay	506	7.54	512	498	1.49
	Silt	131	15.59	154	120	11.90
40	Coarse Sand	163	4.78	167	156	2.94
	Fine Sand	113	2.58	116	110	2.28
	Total Sand	276	4.11	281	272	1.49
	Clay	565	12.60	575	547	2.23
	Silt	159	14.94	181	148	9.39
41	Coarse Sand	299	20.96	324	276	7.01
	Fine Sand	190	10.32	202	178	5.43
	Total Sand	489	10.67	502	478	2.18
	Clay	410	9.03	418	398	2.20
	Silt	101	16.50	124	89	16.37

Table 5. Cont.

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			g kg^{-1}			%
42	Coarse Sand	40	4.35	42	33	11.03
	Fine Sand	93	3.26	97	89	3.51
	Total Sand	132	5.68	139	126	4.29
	Clay	544	5.88	550	536	1.08
	Silt	323	10.96	338	315	3.39
43	Coarse Sand	37	5.56	42	29	15.25
	Fine Sand	76	5.56	83	70	7.37
	Total Sand	112	4.89	118	106	4.37
	Clay	554	9.97	563	542	1.08
	Silt	334	8.22	346	327	2.45
44	Coarse Sand	265	20.00	291	243	7.54
	Fine Sand	207	14.75	221	187	7.14
	Total Sand	472	5.80	478	464	1.23
	Clay	423	14.66	438	407	3.46
	Silt	106	9.32	115	97	8.84
45	Coarse Sand	33	1.15	34	32	3.49
	Fine Sand	82	15.26	104	72	18.72
	Total Sand	115	14.73	136	104	12.86
	Clay	614	50.09	688	580	8.15
	Silt	272	44.23	301	206	16.29
46	Coarse Sand	264	4.39	269	259	1.66
	Fine Sand	210	2.94	214	207	1.40
	Total Sand	474	1.82	476	472	0.38
	Clay	417	9.10	423	408	2.17
	Silt	109	11.09	120	99	10.22
47	Coarse Sand	280	19.36	295	252	6.90
	Fine Sand	187	13.44	206	175	7.18
	Total Sand	467	6.18	471	458	1.32
	Clay	434	5.90	442	429	1.36
	Silt	99	10.04	112	88	10.12
48	Coarse Sand	295	10.72	303	279	3.64
	Fine Sand	140	1.25	141	138	0.90
	Total Sand	434	10.96	444	419	2.52
	Clay	460	5.35	466	453	1.16
	Silt	106	12.76	120	94	12.06
49	Coarse Sand	295	16.34	312	273	5.54
	Fine Sand	108	9.10	120	98	8.41
	Total Sand	403	7.27	410	393	1.08
	Clay	452	17.46	468	427	3.86
	Silt	146	11.70	163	139	8.04
50	Coarse Sand	189	11.02	202	177	5.84
	Fine Sand	96	6.07	102	91	6.31
	Total Sand	285	16.67	303	268	5.85
	Clay	476	12.70	493	463	2.66
	Silt	239	7.07	249	234	2.95
51	Coarse Sand	103	2.98	106	99	2.90
	Fine Sand	195	4.57	201	190	2.34
	Total Sand	298	7.34	307	289	2.46
	Clay	548	9.27	561	540	1.69
	Silt	154	3.74	159	150	2.42

Table 6. Descriptive statistics of the results from particle-size analyses of 10 Heavy clay soil samples (n = 4 replications)

Sample	Textural fraction	Mean	Standard deviation	Maximum	Minimum	CV
			g kg^{-1}			%
52	Coarse Sand	63	7.93	74	56	12.69
	Fine Sand	87	1.82	89	85	2.09
	Total Sand	150	7.04	159	142	4.71
	Clay	677	20.57	699	658	3.03
	Silt	174	24.89	197	151	14.34
53	Coarse Sand	12	4.57	18	7	38.92
	Fine Sand	8	0.95	9	7	12.35
	Total Sand	20	3.87	25	16	19.86
	Clay	639	32.48	660	591	5.07
	Silt	342	28.59	384	322	8.37
54	Coarse Sand	64	6.58	72	56	10.28
	Fine Sand	90	2.87	94	88	3.20
	Total Sand	154	6.94	160	144	4.51
	Clay	685	11.97	699	670	1.74
	Silt	160	8.99	173	152	5.61
55	Coarse Sand	57	5.12	62	50	8.94
	Fine Sand	86	5.25	90	78	6.15
	Total Sand	143	4.57	148	138	3.20
	Clay	677	31.91	704	634	4.71
	Silt	181	29.08	218	157	16.11
56	Coarse Sand	66	5.91	71	59	9.03
	Fine Sand	116	2.06	118	113	1.78
	Total Sand	181	4.85	186	175	2.67
	Clay	643	9.21	648	629	1.43
	Silt	175	7.52	185	168	4.27
57	Coarse Sand	66	5.32	71	60	8.12
	Fine Sand	120	3.55	124	117	2.96
	Total Sand	186	6.02	191	177	3.24
	Clay	641	15.35	652	618	2.39
	Silt	174	11.51	191	166	6.61
	Coarse Sand	61	8.18	68	52	13.52
	Fine Sand	114	8.18	122	103	7.15
58	Total Sand	175	4.34	181	171	2.48
	Clay	645	6.78	650	635	1.05
	Silt	180	10.30	194	169	5.71
	Coarse Sand	84	32.46	131	58	38.87
	Fine Sand	127	4.20	131	122	3.32
59	Total Sand	210	34.91	262	187	16.62
	Clay	632	9.67	641	618	1.53
	Silt	159	37.21	188	104	23.47
	Coarse Sand	85	33.43	135	63	39.21
	Fine Sand	127	6.94	135	120	5.45
60	Total Sand	213	38.33	2700	193	18.04
	Clay	631	9.12	642	620	1.44
	Silt	157	38.10	187	101	24.34
	Coarse Sand	85	32.38	133	65	38.09
	Fine Sand	131	3.51	134	127	2.69
61	Total Sand	216	34.61	166	192	16.06
	Clay	633	14.01	642	612	2.21
	Silt	152	43.19	194	92	28.41

primarily for silt fraction, followed by fine sand, and also for the results for the Heavy Clay soil samples.

The accuracy analysis for nine sandy soil samples showed that the estimations of sand and clay fractions using the RSh were 100 % accurate, once all obtained results were within the confidence intervals defined in table 1 and figure 2. For the silt fraction, the accuracy of the estimations dropped to 55.6 %, because the estimated values of four out of nine samples were not within the predefined confidence interval. However, the overall sensitivity or accuracy of the particle-size analysis using the RSh in the dispersion of the sandy soil samples was high, reaching 85.2 %. For the medium-textured soil samples, all values obtained using the RSh were within the confidence intervals defined in table 2, with 100 % accuracy for the three particle size fractions sand, clay and silt (Figure 3). The estimations of the three main textural fractions, sand, clay, and silt were also found to be 100 % accurate for the clayey soil samples (Figure 4) and for the Heavy Clay soil samples (Figure 5). In summary, considering all 61 soil samples of the different textural classes, the mean accuracy of the estimations using the RSh was approximately 96 %, a high value.

The dispersion charts for mean values of sand, clay and silt extracted from the reports of Prolab/IAC (X axis) and those determined using the RSh (Y axis) are illustrated (Figure 6: Sandy and Medium textured soil samples; Figure 7: Clayey and Heavy Clay soil samples). The linear approximations indicate similarities between the two data sources, as evidenced by the proximity of the angular coefficient values ($0.84 < m < 1.5$) to the unit, which characterizes the straight line of perfect correlation. The values of the correlation coefficients were always greater than 0.93, thus confirming the high correlation between the Prolab/IAC values (reference) and the results obtained with RSh. Of the textural classes, total sand and clay provided the best results, with correlation coefficients greater than 0.98 and differences between the angular coefficients of the straight line and the unit value less than 0.16. Considering all textural classes tested, the largest discrepancies, not only in relation to the correlation coefficient values (> 0.93), but also to the deviations of the angular coefficients from unit (<0.5), were observed for the silt fraction estimations. This can be explained by the fact that the silt fraction was determined by difference, leading to cumulative errors in the estimations of this fraction. Ruiz (2005)
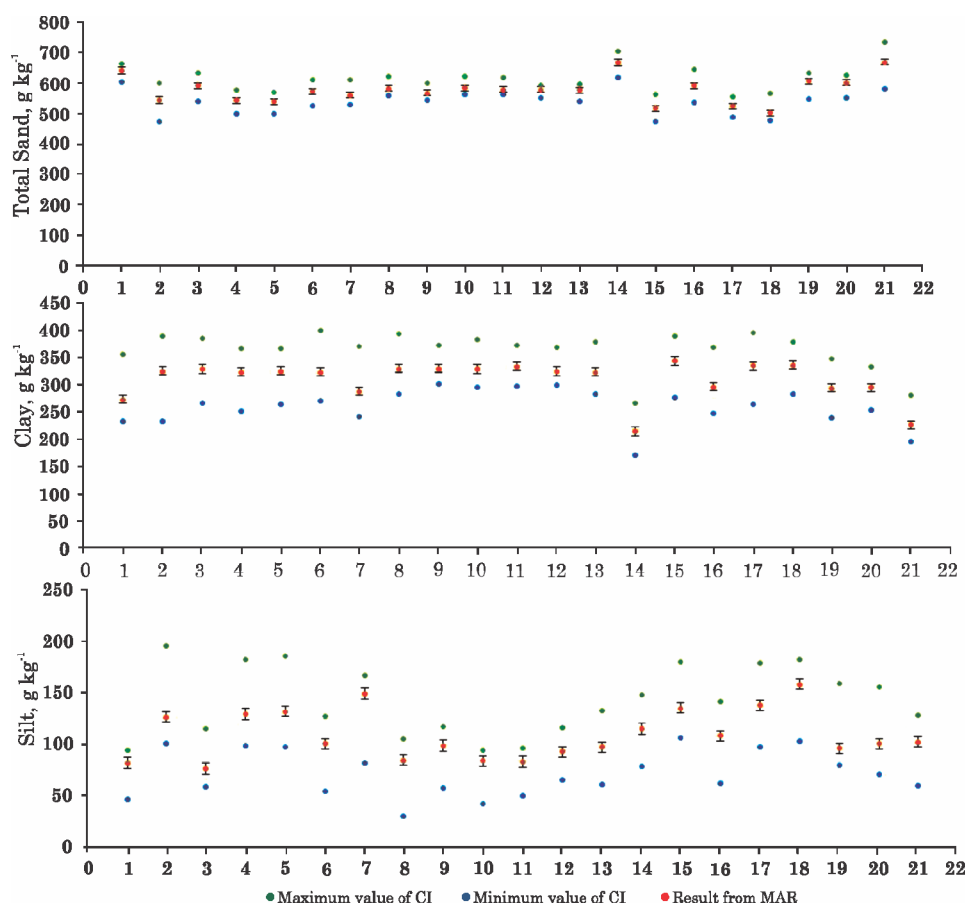

Figure 2. Accuracy analysis of nine sandy soil samples: representation of the minimum and maximum values of the confidence intervals (CI), means and average standard errors (bars) for total sand, silt and clay contents as determined by particle-size analysis using the reciprocal shaker (RSh).
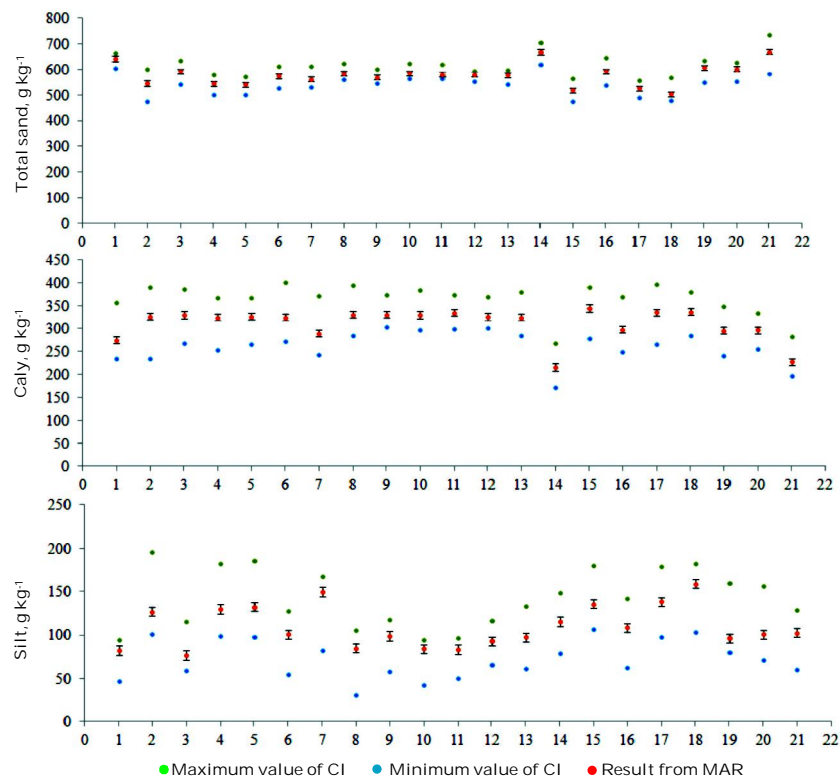
Figure 3. Accuracy analysis of 22 Medium-textured soil samples: representation of the minimum and maximum values of the confidence intervals (CI), means and average standard errors (bars) for total sand, silt and clay contents as determined by particle-size analysis using the reciprocal shaker (RSh).
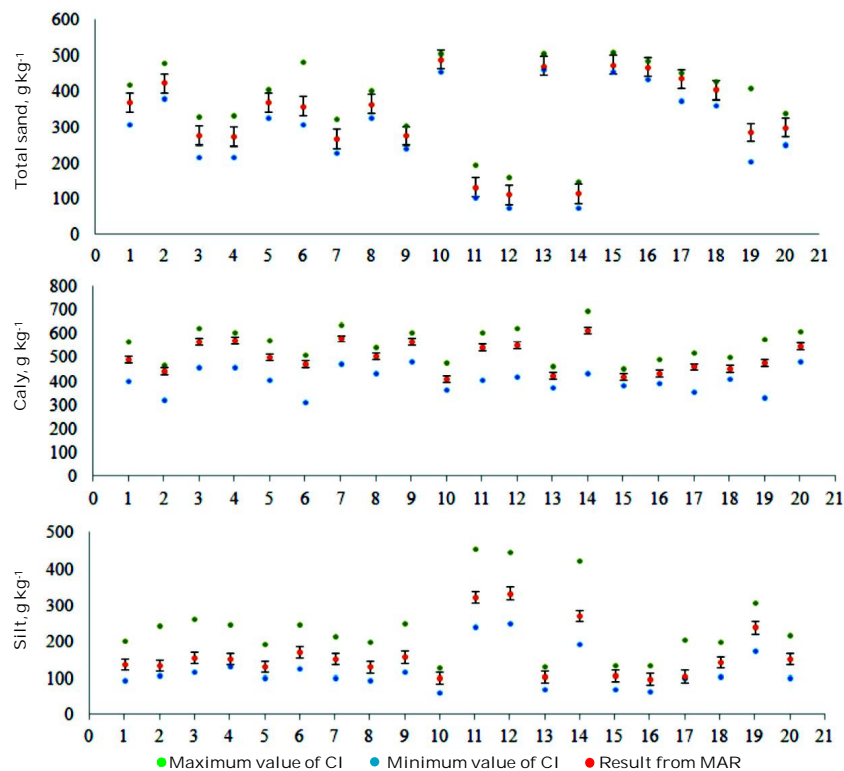

Figure 4. Accuracy analysis for 21 Clayey soil samples: representation of the minimum and maximum values of the confidence intervals (CI), means and average standard errors (bars) for total sand, silt and clay contents as determined by particle-size analysis using the reciprocal shaker (RSh).
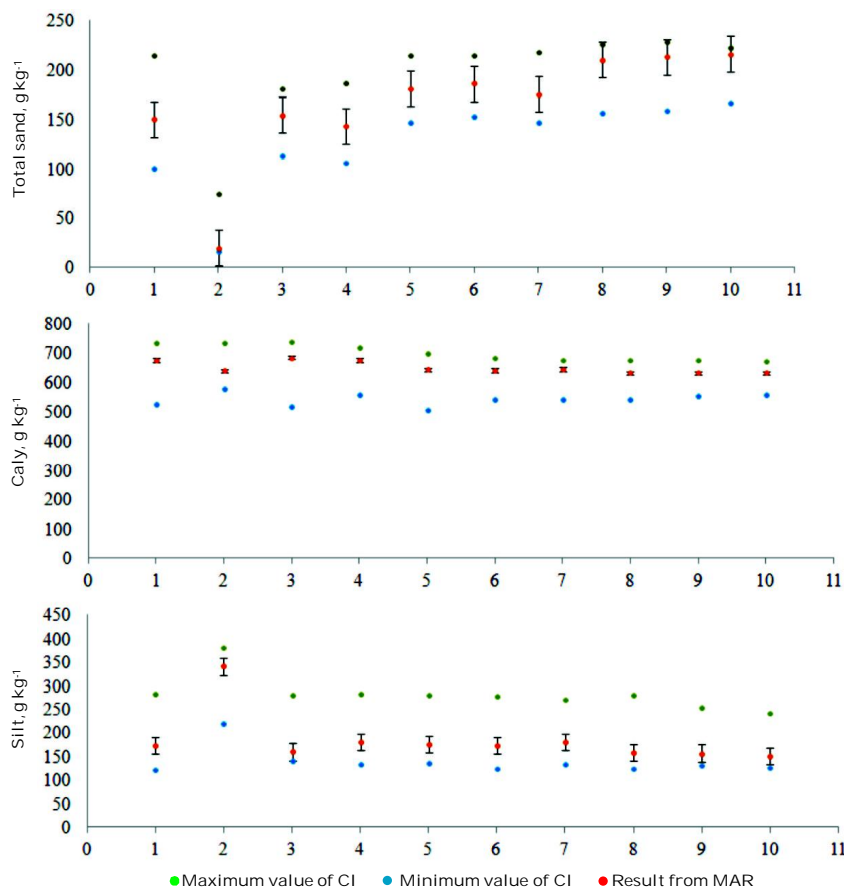
Figure 5. Accuracy analysis for 11 soil samples of the Heavy clay soil textural class: representation of the minimum and maximum values of the confidence intervals (CI), means and average standard errors (bars) for total sand, silt and clay contents as determined by particle-size analysis using the reciprocal shaker (RSh).
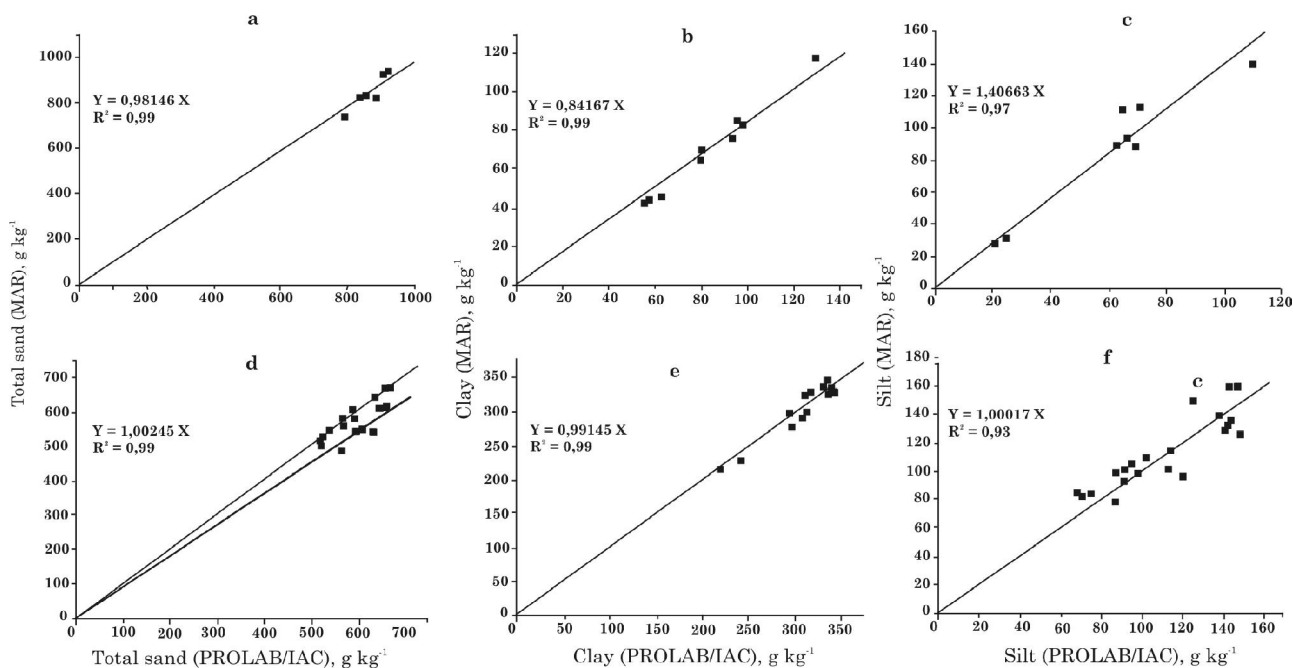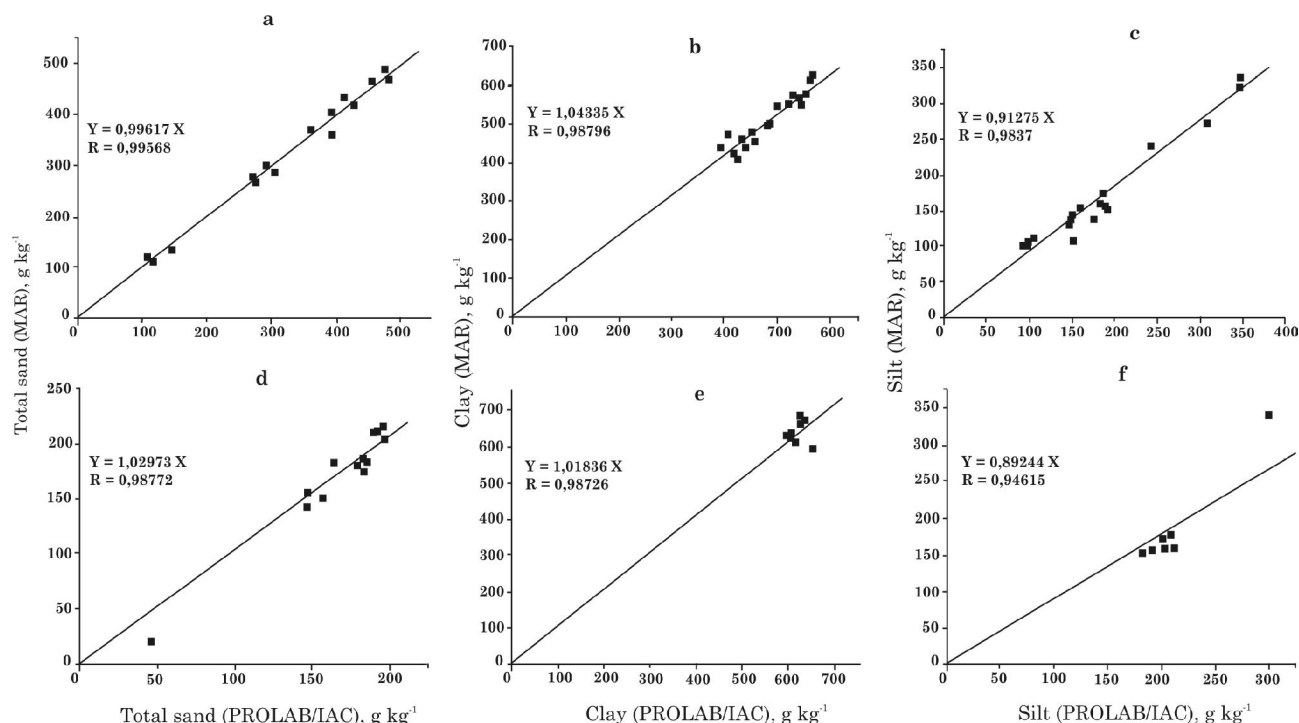

Figure 6. Dispersion charts for mean values of total sand, clay and silt extracted from the data of the Prolab/IAC and results determined using the RSh for 9 Sandy soil samples (*a*, *b* and *c*) and for 21 Medium-textured soil samples (*d*, *e* and *f*).

Figure 7. Dispersion charts for mean values of total sand, clay and silt extracted from the data of Prolab/IAC and results determined using the RSh for 20 clayey soil samples (*a*, *b* and *c*) and for 10 heavy clay soil samples (*d*, *e* and *f*).

demonstrated that the value of the silt fraction calculated by subtracting the other fractions is overestimated. To minimize this problem and possibly increase the accuracy of the determination, the author suggested that an additional volume of the silt and clay suspension should be sampled to estimate silt. However, the effect of this procedure on the accuracy of determinations was not discussed here.

In conclusion, the performance of the reciprocal shaker (RSh) was satisfactory enough to allow its recommendation as a suitable alternative to the conventional devices used for mechanical soil sample dispersion in particle-size analysis. Additional advantages of the equipment are its low cost, the possibility of simultaneous dispersion of up to 40 soil samples, and the option of using ordinary, cheap and easily replaceable glass pots.

CONCLUSIONS

1. The mechanical dispersion of soil samples from different textural classes, even of the Heavy Clay class by the reciprocal shaker was satisfactory.

2. The tested equipment is a viable alternative for the mechanical dispersion of soil samples for particle-size analysis.

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