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Analogy of Soil Parameters in Particle Size Analysis through Laser Diffraction Techniques

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ABSTRACT

A study was undertaken to optimize the parameters for particle size analysis through laser diffraction techniques. Fifty soil samples with varying soil texture, organic matter, sesquioxide content and calcareousness were collected and analyzed for soil texture by conventional (International Pipette Method-IPm) and Instrumental (Particle Size Analyser-PSA) methods. The study reveals that PSA is more accurate and preferable compared to IPm in determining the soil particle sizes. The clay content of the different samples estimated by International Pipette method and by Particle size analyzer varied from 0.9 to 48.4% and 0.35 to 41.2 %, respectively. PSA showed a good agreement (72% samples) for silt size fractions, and a slight shift in the upper limit of clay from conventional size of 2 µm could help in analysis of soil texture by PSA.

Keywords: Soil texture, international pipette method, particle size analyser

INTRODUCTION

Soil texture is a qualitative classification tool used in both field and laboratory to determine classes for agricultural soils. The classes are distinguished in the field by the 'textural feel' which can be further clarified by separating the relative proportions of sand, silt and clay using grading sieves. The class is then used to determine crop suitability and to approximate the soil's responses to irrigation and management practices. Traditional particle size determination techniques include sieves for the larger size ranges, usually above 63µm (230 mesh size). Sieves are limited in resolution (number of sieves = number of data channels), they are slow and operator intensive, and has limitation for determining the smaller size classes. Pipette or sedimentation method is generally used for the finer fractions; however, this technique is slow and is affected by particle shape.

Modern automated analytical techniques are used for sizing sediments which includes laser diffraction and digital image processing. They are fast, easy, operator independent, have a much broader range and higher resolution with many more data channels. In these techniques, particles are to be independently suspended in the flow-cell and the desirable condition is achieved by agitation and ultrasonification whereas the chemical means of removing cementation is not followed. Laser Diffraction techniques are occasionally applied to soil material (Cooper et al. 1984). Laser Diffraction Technique measures light scattered from the particles suspended in the measurement cell. The angle of scatter is related to the size of the particles. The measurement is essentially instantaneous, although total analysis times are in the order of 30 seconds for most samples. The instrument is popular for this application because of its wide size range (0.02-2000 µm), speed, stability and ease of use. With this background, the present investigation was attempted to study the variability and relationship between the international pipette method and laser diffraction technique for varying soil properties.

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MATERIALS AND METHODS

Fifty numbers of surface soil samples were collected from different parts of Western Agro-climatic Zone of Tamil Nadu representing various soil textures and having a wide range of organic matter content, sesquioxide content and calcareousness. The samples were analyzed for texture both by International pipette method (Piper 1966) as well as by Laser diffraction technique in Microtrac S3500 particle size analyzer. The samples were also analyzed for organic carbon content (Walkley and Black 1934), sesquioxide content from HCl extract (Hesse 1973) and free calcium carbonate by Rapid titration method (Piper 1966).

The methodologies followed for particle size determination are detailed below.

International Pipette Method

Twenty grams of soil sample was treated with 60 ml of 6% hydrogen peroxide and kept over water bath for 30 minutes to oxidize the organic matter. To this 200ml of N/5 hydrochloric acid was added and kept overnight to destroy all the carbonates present in the samples. The contents were filtered through Whatman No.50 filter paper and washed with water till the filtrate ran free of chloride. The contents were transferred from filter paper to another beaker and 400 ml water was added. To this 8 ml of 1*N* sodium hydroxide was added to deflocculate the finer particles present in the samples. The entire sample was stirred through mechanical stirrer for 10 minutes to disperse all the soil separates. The volume was made up to 1.0 litre using distilled water in a measuring cylinder without spout. The cylinder was tightly closed with a rubber stopper and the content was mixed thoroughly by repeated inversions holding the rubber stopper tightly so as to avoid spilling of the soil water suspension. The clay fraction, silt fraction and the sand fraction were determined using the pipette method as described by Piper (1966).

Laser Diffraction method

Microtrac S3500 particle size analyzer with a 780 nm wavelength laser beam was used for studying particle size distribution (PSD) of soil samples. Microtrac FLEX software was used for calculation of the particle size distribution. The analysis was carried out in the laboratory of Metrohm India limited, Chennai. The instrument measures particle size over the range of 21 nm-2800 µm in wide angle range of nearly 160 degree. Microtrac employs three lasers to emit laser light into particles from the best angle. While many other particle size analyzers are designed to detect particles at a point, the Microtrac detection mechanism is designed to detect all the scattered light on an entire surface.

Before analyzing samples in the instrument, the sample parameters *viz.,* size and refractive index were set up. Since the soil separates are irregular in shape, the particle parameters were set accordingly. Another important particle parameter was Refractive Index (RI), which is a complex number comprised of (i) a real part (n_r) which represents the change in the velocity of light in vacuum; and (ii) an imaginary term (n_i) which represents the transparency and absorptivity of that material. The values of the minerals commonly found in soil falls between 1.48 and 1.71, but for minerals like hematite, the RI may reach values from 2.9 to 3.2. Yet, for most minerals an n_r value of approximately 1.52 was suitable. Thus, the RI value input was set at 1.52 for the soil samples.

Two grams of soil sample was taken in a 100ml beaker. To this 50 ml of water and 1-2 drops of Triton X 100 dispersing agent was added. The sample was subjected to ultrasonification and fed into particle size analyzer. Before feeding the sample to the instrument, samples were drained and filled twice followed by the flow of water. Samples were analyzed by setting the instrument parameter *viz.,* the rate of ultra-sonification at 0.5 cycles with 40% frequency for 5 minutes. A subset of randomly selected samples, ten in number, was subjected to different durations of sample cycling in the flow cell. The soil textural classes for the above samples were identified using soil textural triangle of International Society of Soil Science (ISSS scheme 1929).

Graphical examination of the data was performed using Microsoft Excel spreadsheet program. Calculating the percent deviation or relative difference between IPm and PSA method for clay, silt and sand were done as per the following equation:

$$\text{Per cent Deviation} = \frac{(a - b)}{b} \times 100$$

Where,

a = per cent of clay / silt / sand determined through International Pipette method.

b = per cent of clay / silt / sand determined through Particle Size Analyzer.

RESULTS AND DISCUSSION

The clay content of different soil samples analyzed by IPm varied from 0.9 to 48.4 per cent. The higher percentage of clay (48.4%) was observed in two samples (Sample No. 3 and 6) out of the 50 samples subjected for particle size analysis. The lowest percent of clay was observed (0.9%) in the Sample No.16. The silt content of the samples varied from 2.5 to 20 per cent and the sand content varied from 32.2 to 90.7 per cent. The higher percent of silt (20%) was found in Sample No. 14 and 24. The lowest silt content (2.5%) was observed in the Sample Nos.11, 12, 33, 37, 38, 40 and 49. The higher percent of sand (coarse and fine fractions) was observed in Sample No.13. The lower percent of sand fraction was observed in Sample No.3. Eshel et al. (2004) obtained a good agreement between measured and calculated laser diffraction values for one size class, accompanied by poor agreement between measured and calculated values for the other class.

The clay content of different soil samples analyzed through Laser diffraction technique (PSA) varied from 0.4 to 41.2 per cent. The highest percentage of clay (41.2%) was observed in Sample No 21. The lowest per cent of clay (0.4%) was observed in the Sample No.19. The silt content of the soil samples varied from 5.8 to 33.5 per cent and the sand content varied from 31.2 to 92.6 per cent. The highest percent of silt (33.5%) was found in Sample No.3. The lowest per cent of silt (5.8%) was observed in the Sample No.10. The higher per cent of sand (92.6%) was observed in Sample No.10. The lowest per cent of sand (31.2%) was observed in the Sample No.3. (Table 1)

The per se performance of the soil samples on pH showed good agreement (50%) between IPm and PSA methods of textural analysis. The chi-squared test showed that these groups are significantly different (at 1% level) from each other in terms of showing agreement between the two methods of textural analysis (Table 2). Soil samples based on natural breaks in soil EC value was done to check if this soil property has any bearing on the choice of the method for textural analysis. When compared to the IPm, PSA produced agreeing results (Fig. 1) in terms of soil textural class (38%) for soils with low EC (<0.1 dSm^{-1}) in group I followed by 0.5-1.0 dSm^{-1} in group III and 0.1-0.5 dSm^{-1} in group II. Loizeau et al. (1994) found that laser grain size analysis underestimates the 0-2 micrometer fraction proportional to the clay content as determined by the pipette method.

On the basis of soil organic matter, about 35 per cent samples had good agreement between IPm and PSA methods of textural analysis when the organic carbon content was below 1 per cent (Fig. 2). When the individual soil separates were considered, silt content was not comparing well between the two methods of analysis (Fig. 3). Grouping of soil samples based on calcium carbonate content was done to check whether this soil property has any bearing on the choice of the method for textural analysis. This was in agreement with the findings of Zobeck (2004), who obtained a better co-efficient of determination between the two methods for non-

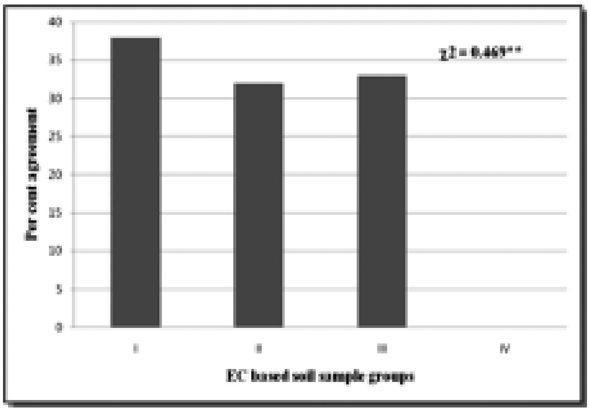

Fig 1: Effect of soil EC on textural analysis

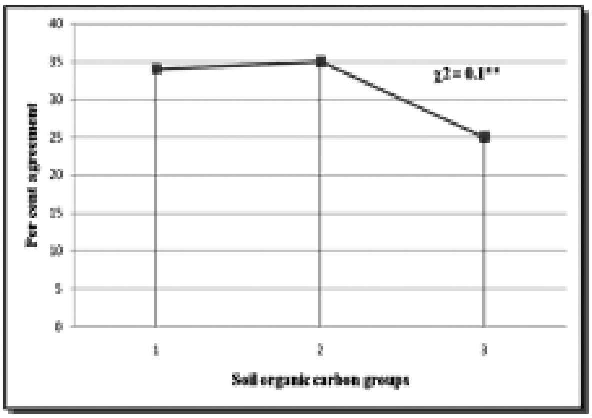

Fig. 2: Percent agreement of soil organic carbon content on textural analysis

Table 1: Comparison of soil particle sizes estimated by IPm and PSA

Sl No	International Pipette Method (IPm)			LASER Diffraction Technique through PSA		
	% Clay	% Silt	% Sand	% Clay	% Silt	% Sand
1	18.4	7.5	70.6	10.5	23.5	65.9
2	23.4	17.5	57.6	25.9	21.9	52.2
3	48.4	17.5	32.2	35.3	33.5	31.2
4	5.9	17.5	76.5	6.7	15.9	77.4
5	35.9	15.0	44.9	25.8	25.5	48.7
6	48.4	10.0	37.3	37.7	30.1	32.2
7	23.4	7.5	65.1	10.4	15.6	74.0
8	20.9	17.5	59.5	11.5	18.2	70.3
9	20.9	10.0	63.9	9.9	14.3	75.8
10	3.4	7.5	85.3	1.6	5.8	92.6
11	25.9	2.5	70.6	11.1	23.3	65.6
12	5.9	2.5	90.7	2.5	11.0	86.5
13	5.9	5.0	85.8	1.8	9.8	88.5
14	18.4	20.0	54.2	2.0	22.8	75.2
15	23.4	10.0	62.0	10.0	27.1	62.9
16	0.9	10.0	87.7	6.9	16.6	76.4
17	10.9	5.0	82.6	6.8	16.6	76.6
18	20.9	10.0	64.4	11.4	22.8	65.8
19	5.9	5.0	88.2	0.4	8.3	91.3
20	10.9	10.0	76.6	6.1	14.5	79.4
21	35.9	15.0	43.3	41.2	21.7	37.1
22	20.9	5.0	71.2	11.8	26.2	62.0
23	20.9	5.0	70.4	15.7	25.3	59.0
24	25.9	20.0	53.1	10.0	20.3	69.7
25	43.4	10.0	46.0	9.2	29.0	61.8
26	45.9	10.0	44.0	23.2	32.5	44.3
27	10.9	10.0	72.7	8.7	22.3	69.0
28	20.9	7.5	65.5	13.2	20.8	66.1
29	15.9	5.0	78.6	6.6	16.3	77.1
30	3.4	4.0	89.0	2.5	10.1	87.5
31	28.4	10.0	57.1	20.5	26.6	53.0
32	8.4	5.0	84.6	3.8	11.0	85.2
33	10.9	2.5	82.4	6.3	13.3	80.5
34	15.9	17.5	59.2	11.8	26.6	61.6
35	5.9	2.5	89.0	6.5	14.8	78.7
36	10.9	5.0	77.4	5.5	18.3	76.2
37	20.9	2.5	73.2	9.4	16.3	74.3
38	15.9	2.5	78.3	9.3	22.1	68.7
39	10.9	7.5	78.3	7.7	20.3	72.0
40	18.4	2.5	78.4	7.9	11.6	80.6
41	15.9	15.0	65.6	21.1	23.5	55.5
42	10.9	7.5	77.4	5.8	23.7	70.5
43	23.4	10.0	64.6	14.3	20.2	65.5
44	5.9	10.0	79.4	4.9	13.1	82.0
45	20.9	15.0	63.5	8.3	16.7	75.0
46	8.4	10.0	73.1	10.7	17.2	72.1
47	13.4	17.5	64.7	13.7	23.5	62.8
48	18.4	17.5	59.5	5.2	14.0	80.8
49	13.4	2.5	83.6	2.2	9.7	88.1
50	15.9	15.0	68.7	5.3	14.7	80.0
Mean	18.3	9.5	68.9	11.1	19.2	69.7
S.D.	11.5	5.4	14.4	9.1	6.5	14.3
C.V.	130.5	28.6	202.3	80.6	41.0	200.1

Table 2: Comparative study of soil samples on pH basis analyzed by IPm and PSA

pH based Groups	No. of samples	No. of samples showing agreement between IPm and PSA	Per cent agreement
I	12	6	50
II	12	2	17
III	19	7	37
IV	7	2	29

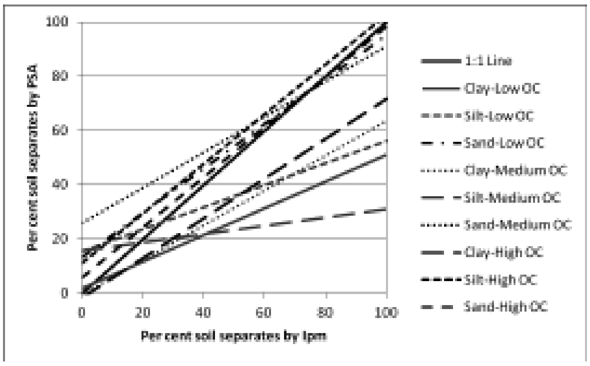

Fig. 3: Agreement between IPm and PSA for Soil separate estimation under different groups of soil based on Organic Carbon Content

calcareous soils. Compared to the IPm, PSA produced agreeing results (50%) in terms of textural class for soils (Fig. 4) with high calcium carbonate (15-20%) in group IV, followed by group II (5-10%), I (0-5%) and III (10-15%). Grouping based on sesquioxide content, about 37 per cent samples had good agreement between IPm and PSA methods of textural analysis when the sesquioxide content was more than 10%. When the individual soil separates were considered, silt content was not comparing well between the methods of analysis (Fig. 5), as also reported by Pieri et al. (2006).

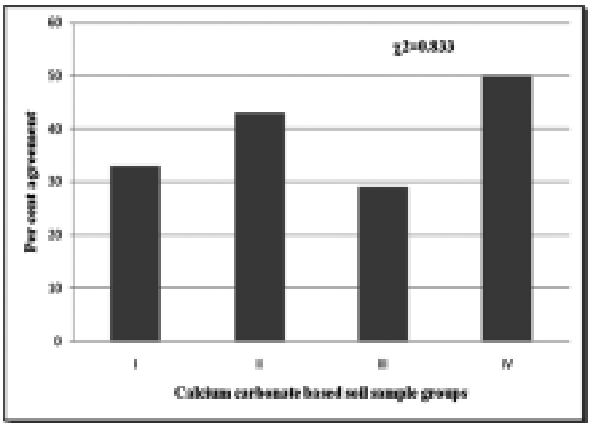

Fig. 4: Effect of Soil free Calcium Carbonate on Textural

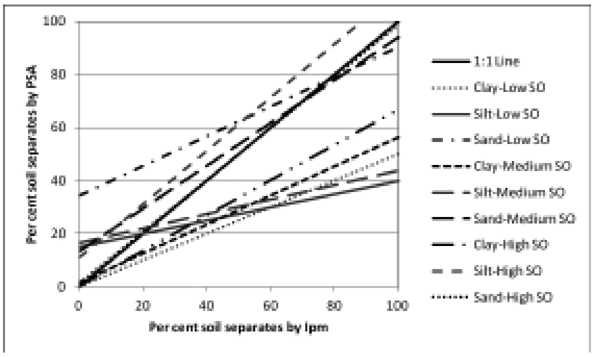

Fig 5: Agreement between IPm and PSA of soil based on Sesquioxide Content

CONCLUSIONS

Even though it is not explicitly established from the present study that what causes the difference between the two methods of analysis of soil separates, it is found that a relook into the definition of size of the soil separates could favour the use of laser diffraction-based soil particle size analysis. The present findings can be discussed in scientific forums and if agreed, the PSA can be recommended as one of the important equipment in the soil laboratories.

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