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TURBIDOMETRIC TECHNIQUE FOR THE MEASUREMENT OF VARIOUS SUBDIVISIONS OF CLAY IN SOIL

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A turbidometric technique has been developed to quantify the coarse and fine clay in soil. This helps to elucidate the variations of clay sizes and amounts in the soil samples. The technique will be useful for pedologists in order to evaluate the coarse to fine clay ratio (C/F) of various soil series, which sometimes clearly demonstrate the illuviation (clay movement, translocation and deposition) process to be operative in the soil profile, which otherwise could only be concluded after studying the soil thin-sections with very specialized equipment. These equipment are not easily available in laboratories. The turbidometric technique also gives close agreement for the total clay values (<2 μm) to those as measured by the routine hydrometer method.

INTRODUCTION

Soil texture is important in the sense that it is a major factor in water holding capacity, permeability, infiltration rate, consistence and porosity etc.. The analysis for soil texture can be made in the field and more accurately, in the laboratory. However, the quantification of the finer clay fractions poses problems in the routine analysis of soil texture. Possible alternative technique to quantify the various size ranges within clay fraction, the use of Coulter Counter (Coulter Electronics, Inc., 1975) has also been investigated. Samples were prepared according to Pennington and Lewis, (1979), but this technique was found to be inadequate to quantify particles below 0.4 μm. The lower limit for the Pipette method is (cumulatively) less than 0.2 μm, further quantification of finer clay is impossible by the existing routine techniques. It was therefore a need to develop a technique which measures all size ranges within the clay fraction. It was for this reason that a turbidometric technique was developed, which allows the quantity of the various sizes of clay to be measured through the measurement of the optical density of the clay suspensions in spectrophotometric glass tubes. This measurement of coarse/fine clay is helpful to elucidate the results of clay migration in the soil profile, a pedogenic process of world importance.

MATERIALS AND METHODS

Two disturbed soil samples were collected at 65-70 cm depths of the Lyallpur (typic camborthid) and Khurrianwala (natric camborthid) soil series. These were air dried and sieved (less than 2 mm) for routine analysis. Routine physical and chemical measurements were carried out according to the methods as prescribed by Richards (1954) except otherwise mentioned. All measurements were made on duplicate air dried soil samples and results are presented on an oven dried basis (105°C). Soil texture was determined according to Day's (1965) method.

For the development of turbidometric technique, the integrated form of Stoke's equation as quoted by Hathaway (1956); Irani and Collis (1963) and Livesey (1964) was used which allows the calculation of sedimentation time of a particle of known equivalent spherical diameter (e.s.d.) in a centrifugal field. The equation as quoted by Hathaway (1956) is described below, alongwith the method of clay separation from the soil.

Separation of less than 2 μm clay from soil: The fine earth fraction of soil (less than 2 mm) was subjected to the removal of organic matter, sodium hypochlorite being used for its oxidation (Anderson, 1963) to avoid physical disruption of clay particles by H_2O_2 (Barshad & Kishk, 1968; Douglas & Fiessinger, 1971). The soil was then dispersed in 100 ml of 5% Calgon solution ultrasonically and soil texture was determined according to Day (1950, 1965). After the soil texture measurements, the suspension of clay (less than 2 μm) was separated from the cylinder through siphoning and used for further investigations.

Subdivision of clay for calibration curves: A Beckman-14 refrigerated centrifuge with fixed angle rotor (Fig. 1) was used for clay fractionations. The temperature was maintained at 10°C. Dispersed clay (less than 2 μm) was used to separate four size ranges of clay, based on the Massachusetts Institute of Technology (MIT) log scale $2 \times 10^{n/2}$ i.e., <2 μm, <0.63, <0.2 and <0.063 μm. These were separated from the dispersed clay by a progressive elimination procedure. These were then totally sedimented individually at high speed of 11×10^3 rpm and prolonged settling time of 75 minutes, and then dried at 30°C.

Integrated Stoke's equation of Hathaway: The final form of the integrated Stoke's equation according to Hathaway (1956) is as follows:

$$t = \frac{n \log_{10} (R2/R1)}{3.81\, r^2 N^2 (e-e')} + 2/3\,(ta + td)$$

This formula is quoted by Hathaway (1956) where:

t = the time (seconds) taken for the larger particles in the size fraction to travel the distance from the surface of the liquid to the bottom of the centrifuge bottle.

ta = the time of acceleration (seconds) of the centrifuge to reach at the required speed.

td = time of deceleration (seconds) of a centrifuge to stop.

n = viscosity of suspension in poises.

R1 = initial distance (cm) of particles from the axis of rotation.

R2 = final distance (cm) of particles from the axis of rotation.

r = equivalent spherical radius (cm) of particles to sediment.

e = density of the particles in g cm^{-3}.

e' = density of medium in g cm^{-3}.

N = rotory speed of the centrifuge machine in revolution per second.

The position of the bottle in rotor at a fixed angle of 25° is shown in Fig. 1, where R1 is the distance from the axis of the rotor to the surface of the liquid. During operation, the surface of the liquid should remain parallel to the axis of rotation, R2 is the distance from axis of rotation to the top of the clay pellet, but may vary according to the size of the pellet. During calculations, it was assumed that the size of the pellet was 0.5 cm for >0.063 µm sediment, 0.4 cm for >0.2 µm and 0.3 cm for >0.63 µm. During calculations the following additional points should be taken into account:

1. The centrifugation speed in Table 1 is given in rpm but it was corrected to rps for the calculation.
2. (e-e') was taken as 1.65 g cm^{-3}.
3. ta and td are measured by timing the acceleration of the centrifuge machine upto various rpm, with the polythene bottles filled up to 6 cm depth in the rotor. For td the centrifuge machine was set at maximum break, and similarly timed.

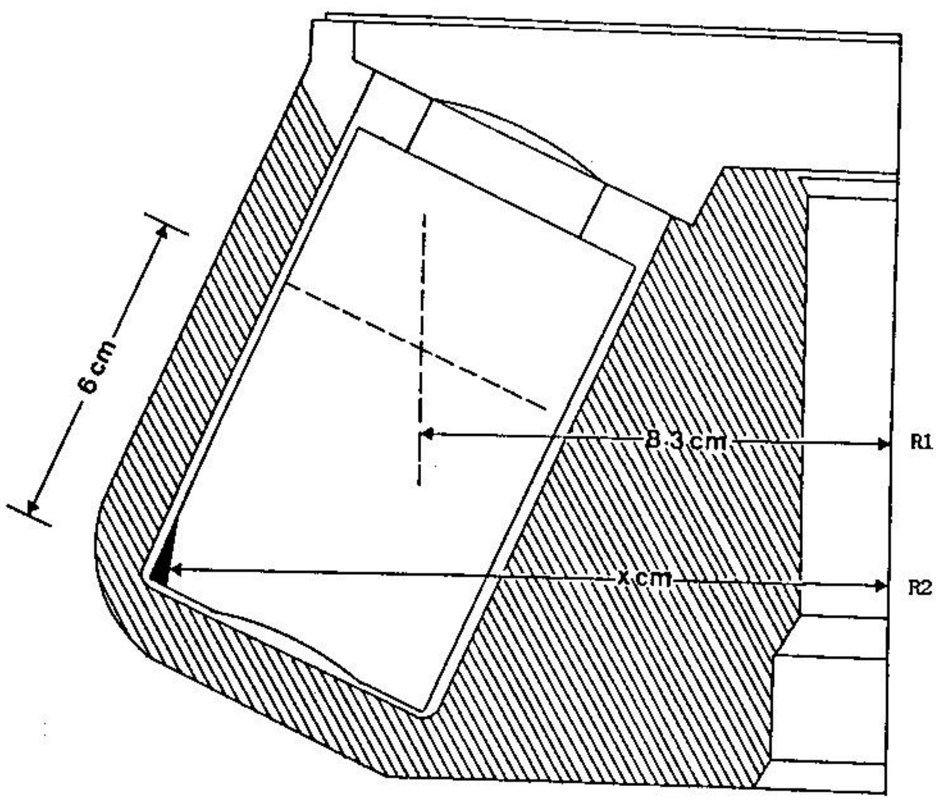

Fig. 1. Centrifuge bottle in a fixed angle rotor of Beckman JA-14

Table 1. Centrifugation timings for the separation of various subdivisions of clay for calibration curves

Diameter of particle to sediment (μm)	Temp. (°C)	Sampling depth (cm)	R1 (cm)	R2 (cm)	Rotor speed (rpm) (10^3)	ta sec	td sec	Time of settling Min	Sec
> 0.063	10	6	10	8.3	12.9	125	213	27	50
> 0.200	10	6	4	8.3	13.0	42	114	16	56
> 0.630	10	6	1.5	8.3	13.1	17	67	12	05

1. Number of repetitions to spin all particles of desired diameter from the suspension was 5 in all separations.
2. Different concentrations of the <2 μm clay were prepared by using <2 μm clay straight away, without using the centrifuge machine.

Turbidometric technique: Soil material (less than 2 mm) already treated with sodium hypochlorite was used. It was dispersed ultrasonically with 5 ml of 0.2% Calgon solution for 3 minutes and the volume was raised to 50 ml (2 g L^{-1}). The soil suspension was redispersed ultrasonically for one minute.

The treated soil suspensions were centrifuged in a Beckman model L8 55M refrigerated centrifuge with swing-out rotor for the required time for a specific particle size. The temperature was maintained at 10°C in the centrifuge machine. The optical density was measured on a Cecil-202 spectrophotometer. The time of settling of

Table 2. Centrifugation timings for microquantitative estimation of clay (Beckman Model L8 55M refrigerated centrifuge)

Diameter of particle to sediment (μm)	Temp. (°C)	Sampling depth (cm)	R1 (cm)	R2 (cm)	Rotor speed (rpm) (10^3)	ta sec	td sec	Time of settling Min	Sec
>2.000	10	6.5	9.5	16	1	15	21	3	47
>0.630	10	6.5	9.5	16	2	25	27	9	7
>0.200	10	6.5	9.5	16	3	32	34	38	20
>0.063	10	6.5	9.5	16	10	187	98	37	15

ta, td, rpm, R1 and R2 are already described in the text.

various soil particles is given in Table 2. Quantities of various subdivisions were calculated by using the optical density values on respective curves. The amount of 2-0.63, 0.63-0.2, 0.2-0.063 and <0.063 μm clays were calculated by taking the difference between the upper and lower limits of the cummulative values. The results are expressed on the basis of less than 2 mm and <2 μm oven dried soil and clay (Table 3).

Turbidometric technique: The technique was developed from studies by Sarkar (1976) by using the centrifuge machine and various fractions of clay. Various steps involved are described as under:

Calibration: Linear calibration curves of four subdivisions of clay were obtained upto 1000 mg L^{-1}. These curves are shown in Fig. 2. An optical density value of 0.5 at 680 nm was found adequate for such calibration for less than 2 μm and less for other fractions. The value at 680 nm was chosen as it gives maximum sensitivity for this technique.

Table 3. Microquantitative estimation of clay subdivisions in the samples of the Lyallpur and Khurrianwala soil series as measured by the turbidometric technique

Subdivision sizes (μm)	Lyallpur soil series	Khurrianwala soil series
<2 μm (Hydrometer method)	34%	25%
<2 μm (Turbidometric technique)	33%	24%
2-0.63 μm (Turbidometric technique)	43% of the total <2 μm clay*	46% of the total <2 μm clay*
0.63-0.2 μm (turbidometric technique)	15% of the total <2 μm clay	23% of the total <2 μm clay
0.2-0.063 μm (Turbidometric technique)	34% of the total <2 μm clay	7% of the total <2 μm clay
<0.063 μm (Turbidometric technique)	8% of the total <2 μm clay	24% of the total <2 μm clay
Total coarse clay (2-0.2 μm)	58% of the total <2 μm clay	69% of the total <2 μm clay
Total fine clay (<0.2 μm)	42% of the total <2 μm clay	31% of the total <2 μm clay

*clay as measured by the turbidometric technique.

RESULTS AND DISCUSSION

Physical and chemical characteristics of the soil samples used in the development of turbidometric technique are shown in Table 4. The values of EC_e, pH_s and ESP classified the sample from Lyallpur soil series as normal and Khurrianwala soil series as saline-sodic (Richards, 1954). Soil texture is silty clay loam for both the soil samples.

The comparison of the calibration curves (Fig. 2) reveals that these differed slightly from profile to profile for less than 2 μm and less than 0.63 μm clay fractions, but similar curves are obtained for both the pro-

Table 4. Physical and chemical characteristics of the soils used

Parameter	Unit	Lyallpur soil series	Khurrianwala soil series
pH_s	-	7.5	9.7
EC_e	$dS\ m^{-1}$	1.8	15.9
ESP	-	7.0	83.0
Analysis of the saturation extract			
Na^+	$mg\ kg^{-1}$	46.0	1000.0
K^+	"	4.0	29.0
Ca^{2+}, Mg^{2+}	"	95.0	88.0
CO_3^{2-}	-	Absent	Absent
HCO_3^-	$mg\ kg^{-1}$	138.0	160.0
Cl^-	"	250.0	533.0
SO_4^{2-}	"	102.0	2500.0
Exchangeable cations			
Na^+	me/100 g	1.1	11.4
K^+	"	0.4	0.5
Ca^{2+}, Mg^{2+}	"	15.2	1.8
ESP	-	7.0	83.0
Particle size analysis			
Sand (2000-60 μm)	%	10.8	15.0
Silt (60-2 μm)	"	55.2	59.5
Clay (less than 2 μm)	"	34.0	25.5
Soil texture	-	Silty clay loam	Silty clay loam
Munsell Colour Notation			
(Air-dried)	-	10 YR 6/3 Brown to dark brown	10 YR 6/3 Brown to dark brown

files for less than 0.2 μm and less than 0.063 μm fractions. This suggests that the curves for the finer clays could be generalised to be used for the other similar soil profiles. It should be noted that for higher limits (less than 2 μm and less than 0.63 μm) separate calibration curves should be obtained, for each profile or the value of less than 2 μm could also be obtained from the routine mechanical analysis (Pipette and hydrometer methods) of the soil samples.

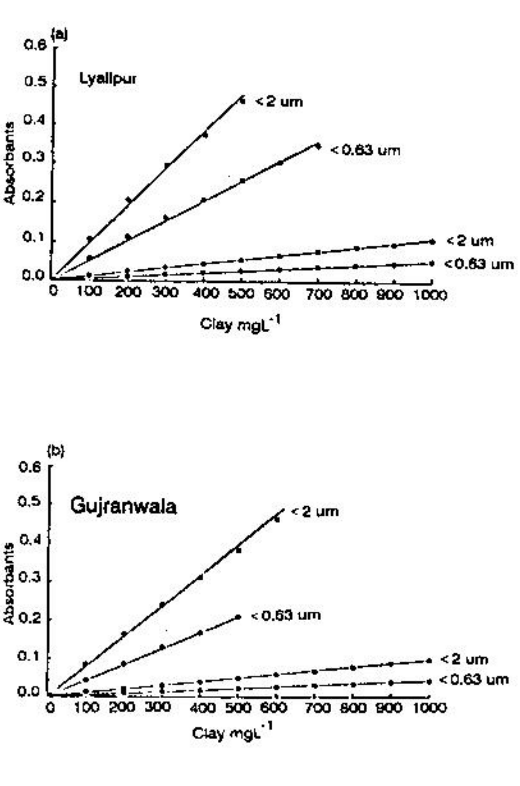

Fig. 2. Calibration curves for microquantitative estimation of clays:
a) Lyallpur soil series
b) Kurrianwala soil series

The comparison of the total clay (<2 μm) values as measured by the routine mechanical analysis and the turbidometric technique revealed similar values by both the techniques (Table 3). It is, therefore, concluded that the turbidometric technique could also be used successfully to quantify less than 2 μm clay in soil.

The present turbidometric technique to measure the fine clay fraction in soil is more reliable and accurate than the existing routine technique of Bascomb (1982), where after centrifugation for a specific period of time for a particular particle size, the suspension is taken out by pipette and transferred into a tared polypropylene dish. This suspension is evaporated and the percentage of clay calculated. In this procedure, the presence of electrolytes in the soil suspension may also evaporate and increase the total weight and hence overestimate the clay, which may mislead the results. In the turbidometric technique, such an error of overestimation is avoided.

The significance of these coarse/fine clay ratios in terms of genesis and properties of these two soils will be discussed in a later paper.

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