

A Note on Evaluation of Methods for Routine Analysis of Carbonates in Some Vertisols of Karnataka

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A number of methods for quantitative determination of soil carbonates are in vogue. Each of these has specific advantages and disadvantages (El Mahi *et al.* 1987). The precise quantitative determination of carbonates in soil is important as it is being used as a differentiating criterion for some classes at the family level of Soil Taxonomy (Soil Survey Staff 1975). Hence, the study was undertaken to evaluate three methods for routine analysis of carbonates in the fine earth fraction in Vertisols of Karnataka.

Five Vertisols occurring in the Upper Krishna Project area of north Karnataka, differing in carbonates were sampled from each horizon, air dried and processed (passed through 2 mm sieve) for analysis. Three methods namely, rapid titration (Piper 1966), volumetric using 'SVV' apparatus (Subramanian *et al.* 1975) and acetic acid (Loeppert *et al.* 1984) were used for determination of carbonates as calcium carbonate equivalent (CCE) in the fine earth fraction. Rapid titration method is based on the reaction of HCl with carbonates. The CCE was estimated from the acid consumed to dissolve carbonates. Volumetric method works on the principle of the volume of CO₂ evolved as a result of carbonate

dissolution on treating the soil with HCl. The CCE was estimated from the CO₂ evolved using a standard curve. Acetic acid method measures CCE based on acid consumption. It entails the reaction of soil with an aliquot of acetic acid for measuring the pH of suspension after completion of the reaction. The CCE was estimated based on pH using the formula given by the authors. Least significant difference (LSD) test was used for comparison of the values obtained by these three methods.

The results indicated that Piper's rapid titration method gives significantly higher estimates of CCE than the other two (Table 1). The potential sources of error with this method are masking the titrimetric end point by clays or Fe and Al oxides and/or consumption of H⁺ either through dissolution of noncarbonate material or ion exchange reactions (Loeppert *et al.* 1984 and El Mahi *et al.* 1987). When the latter error is corrected as suggested by El Mahi *et al.* (1987), the corrected CCE using rapid titration method does not differ significantly from the other two methods, but the mean per cent of corrected CCE for all the soils tends to be slightly higher than those of the others, suggesting the reaction of HCl with

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TABLE 1. Calcium carbonate equivalent (CCE) in the fine earth fraction by three methods

Depth (cm)	CEC (NaOAc) ₁ cmol kg ⁻¹	Rapid titration	Rapid* titration (Corrected)	Volumetric	Acetic acid
Pedon 1					
0-18	69.5	17.6	14.1	12.8	13.2
18-40	73.8	20.9	17.2	15.2	16.1
40-62	76.1	25.2	21.4	19.6	22.0
62-86	76.1	28.0	24.2	22.0	22.6
86-110	60.8	32.6	29.6	27.9	28.5
Pedon 2					
0-18	65.2	14.7	11.4	9.7	10.2
18-55	69.5	15.5	12.0	10.5	12.5
55-90	74.9	15.0	11.3	10.7	10.4
90-126	73.8	16.3	12.6	10.2	10.5
Pedon 3					
0-20	76.1	23.2	19.4	17.6	18.1
20-48	79.3	26.1	22.1	20.7	21.2
48-88	78.2	29.1	25.2	23.2	24.2
Pedon 4					
0-22	59.7	16.3	13.3	11.5	12.0
22-58	58.6	13.9	11.0	10.7	10.2
58-93	60.8	15.2	12.2	10.4	12.5
93-138	57.6	15.3	12.4	9.7	10.3
Pedon 5					
0-10	65.2	20.9	17.6	15.7	15.6
10-32	69.5	26.0	22.5	22.0	21.8
32-60	69.5	26.9	23.4	21.6	21.3
60-80	64.1	27.0	23.8	21.9	24.0
Mean	21.3	17.8	16.2	16.9	

*Corrected CCE = % CCE by rapid titration method - 0.05 CEC of Soils (E1 Mahi *et al.* 1987); L.S.D. at 5% = 3.2

non-carbonate minerals.

The results observed with the other two methods, however, do not differ significantly, but the mean of CCE in all the soils using volumetric method is slightly lower than the acetic acid method. The volumetric method using 'SVV' apparatus is relatively time consuming, requires assembling of special apparatus and may lead to underestimation of total carbonates in case when dolomite is present. The acetic acid method is suitable when large samples are taken at a time and when the CCE range is known. It is time consuming if small number of samples are to be handled. Further, the precautions such as to avoid volatilization of acetic acid and loss in any other way, need to be meticulously followed. Moreover, small sample weight employed in this procedure will magnify the errors.

Piper's rapid titration method requires less time and no specialized equipment and ensures complete dissolution of dolomite, if present. Considering the aforesaid points, Piper's rapid titration method after correction as suggested by El Mahi *et al.* (1987) is suitable for routine analysis of carbonates

in these soils

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