SIMPLE, SIMULTANEOUS GRAVIMETRIC DETERMINATION OF CALCITE AND DOLOMITE IN CALCAREOUS SOILS

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ABSTRACT

Literature for measuring calcite and dolomite, describes slow methods that require expensive apparatus. The objective of this study was to modified the carbonate meter (CM) method, to quantify calcite and dolomite in calcareous soils without using specialized equipment. The separation was done placing between 0.1800 – 0.2200 g of fine calcareous soil, 6 mL glass vial with 4.0 mL 4M HCl-3% $FeCL_2.4H_2O$ in a 50 mL Erlenmeyer flask. The Erlenmeyer was capped and the carbonate meter (CM) weight recorded with four decimals as initial weight. Three more weights were taken at 30 s, 30 min and 22-24h after reaction. Linear regressions and correlations coefficients for calcite and dolomite in calcite-dolomite-soil-mixtures with the modified carbonate meter (MCM) calcite and dolomite results were excellent. The MCM method separate calcite and dolomite in calcareous soils with good precision and accuracy.

INTRODUCTION

Total inorganic carbon (TIC) in calcareous soils has two main components: Calcite and dolomite. Performing the separation of calcite and dolomite in calcareous soils, allows three new soil categories: Calcitic, dolomitic and calcitic-dolomitic, very useful tool for characterizing calcareous soils.

OBJECTIVE

Modified the carbonate meter (CM) method to quantify calcite and dolomite in calcareous soils.

MATERIALS AND METHODS

The CM method was modified in regard to the weight loss of CO_2 release to the atmosphere. Two more weights are needed occurring between the initial and final weight. The second and third weights occur 30 s and 30 min, respectively, after initial reaction. Partial net IC (%) at 30 s and partial net IC (%) at 30 min and total net IC (%) at 22-24h, were used to calculate % calcite and % dolomite. With careful timing, multiple samples can be analyzed with a maximum of 15 samples per batch. A technician should be able to analyze 144-192 samples per week.

RESULTS AND DISCUSSION

Linear regressions and correlations coefficients for calcite and dolomite in the standards mixtures are presented in Fig. 1. Both correlations are highly significant, the intercepts and slopes for calcite and dolomite approach zero and one, respectively, indicating that the MCM method can be used for analysis of calcite and dolomite in calcareous soils up to 450 g kg^{-1}. Precision for calcite and dolomite was mainly less than 10%, while recovery for calcite and dolomite was in general more than 90%. Rodriguez et al. (2016) working with pure calcite and pure dolomite, found similar results for total inorganic carbon (TIC).

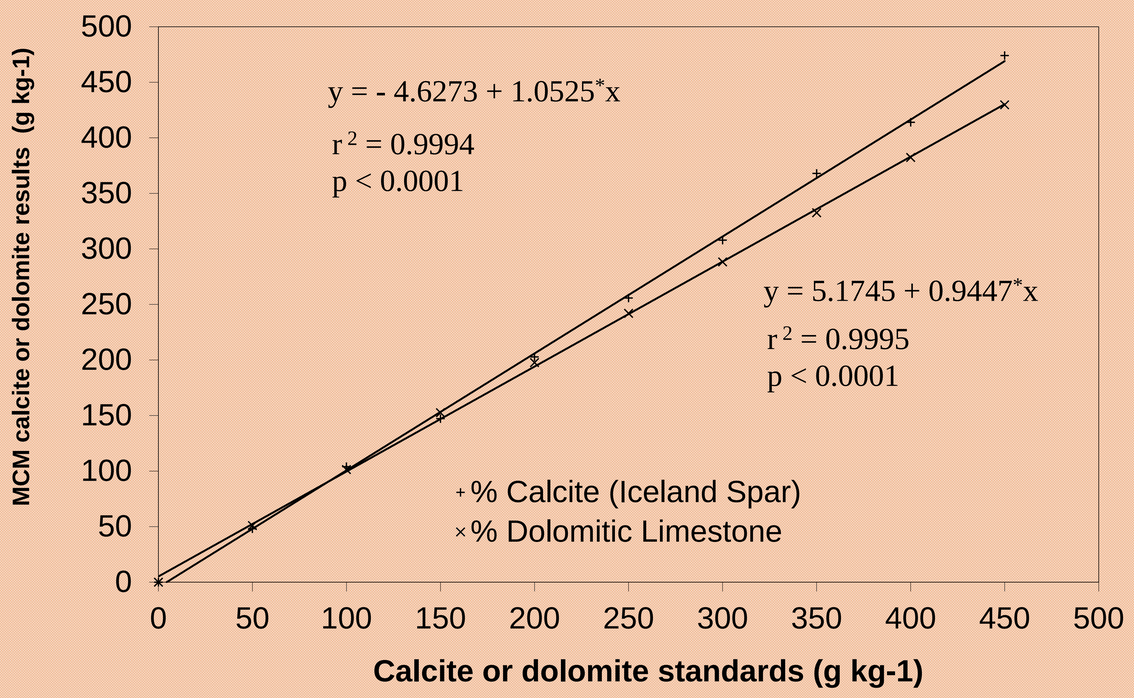

Fig. 1. Calcite or dolomite standards and MCM calcite or dolomite gravimetric results.

To further validate the technique, the content of select calcareous soils from USDA (Table 2) with a total-CCE range of 0.88 to 71.0% were used to test the method. If the MCM procedure quantitatively separated the calcite and dolomite in each calcareous soil, then the calcite-CCE plus the dolomite-CCE should be equal to the total-CCE done manometrically by the Soil Survey Laboratory Staff (2004). Results indicated that the MCM method, identified the same amount of total-CCE (calcite-CCE + dolomite-CCE) with a sample range of 0.87 to 69.28% (Table 2).

Table 2. Validated results of $CaCO_3$ equivalent (CCE) obtained with the modified carbonate meter (MCM) gravimetric method from calcite-CCE plus dolomite-CCE in USDA calcareous soils.

		Modified Carbonate Meter (MCM)							CCE	
USDA Soil	Series	Calcite	SD	Precision	Dolomite	SD	Precision	Replicates	USDA	CM
		%			%				%	
Arkansas	Dudley	0.87	0.09	9.82	0.00	0.00	0.00	3	0.88	0.87
Montana	Reedwest	1.05	0.12	11.43	0.00	0.00	0.00	3	1.41	1.05
Nevada-1	Patter	2.01	0.10	4.80	0.18	0.02	8.65	3	1.99	2.21
Washington	Warden	2.23	0.08	3.66	0.50	0.05	10.58	3	3.25	2.77
California	McKeohills	3.95	0.12	3.08	1.11	0.13	11.72	3	5.88	5.16
Utah	Begay	7.62	0.13	1.71	1.39	0.08	5.60	3	9.09	9.13
Oklahoma	unknown†	14.09	0.18	1.30	1.20	0.09	7.11	3	15.82	15.39
South Dakota	Still Lake	9.88	0.12	1.26	11.14	0.21	1.89	3	22.86	21.98
Indiana	Blount	10.52	0.20	1.88	15.14	0.17	1.15	3	25.90	26.96
Nevada-2	Urmafot	34.62	0.05	0.15	8.32	0.43	5.21	3	45.65	43.66
Wyoming	Gragnot	7.56	0.33	4.36	46.77	0.27	0.57	3	57.59	58.35
West Virginia	Huntington	66.25	0.35	0.52	2.79	0.44	15.77	3	71.00	69.28

† Soil collected in Beaver County, Oklahoma.

Additional validation was studiied analyzing soils from different states for calcite and dolomite, to verify that the results for calcite-CCE + dolomite-CCE match the total-CCE obtained with the MCM method. Results of calcite-CCE + dolomite-CCE for the soils ranged from 6.85 to 67.36%, and the total-CCE obtained with the MCM method ranged from 6.92 to 66.83% (Table 3). The MCM new method identified calcareous soils from Texas, Kansas, Minnesota and Alaska as calcitic; calcareous soils from Iowa, as calcitic-dolomitic; and calcareous soils from Wisconsin, as dolomitic (Table 3).

Table 3. Determination of calcite and dolomite in several USA calcareous soils with the MCM method.

		Modified Carbonate Meter (MCM)							CCE	
State	Series	Calcite	SD†	Precision	Dolomite	SD†	Precision	Replicates	State	CM
		%			%				%	
Texas	Hidalgo	5.79	0.27	4.69	0.98	0.19	19.71	3	6.85	6.92
Texas	Houston Black	7.44	0.56	7.53	1.17	0.21	17.51	3	8.71	8.25
Kansas St.	Harney	8.87	0.53	6.02	1.25	0.32	25.64	3	10.23	10.42
Iowa	Harps	8.25	0.11	1.33	5.07	0.25	4.88	3	13.76	14.25
Wisconsin	Kewaunee	1.83	0.23	12.75	13.11	0.08	0.61	3	16.07	14.92
Kansas St.	Harney	14.01	0.37	2.64	1.83	0.19	10.12	3	16.00	16.25
Minnesota	unknown	12.74	0.25	1.93	3.96	0.50	12.65	3	17.04	17.25
Alaska	unknown	17.36	0.23	1.31	4.86	0.11	2.18	3	22.64	23.08
Texas	Austin	29.53	0.57	1.93	1.02	0.23	22.35	3	30.64	30.84
Wisconsin	Hortonville	2.77	0.42	15.08	31.70	0.56	1.75	3	37.20	35.25
Texas	Dustin	41.14	0.32	0.79	1.56	0.26	16.81	3	42.83	42.83
Texas	Stephen	48.32	0.07	0.13	1.59	0.17	10.58	3	50.05	51.17
Texas	Austin	59.47	0.48	0.80	1.70	0.12	7.32	3	61.32	62.44
Texas	Pun	65.33	0.22	0.33	1.87	0.16	8.75	3	67.36	66.83

†Standard deviation.

CONCLUSIONS

The modified carbonate meter (MCM) technique, quantify calcite and dolomite and can be used for routine analysis of calcareous soils. The modified carbonate meter(MCM)method, identified calcareous soil from Texas, Kansas, Minnesota and Alaska as calcitic; calcareous soils from Iowa as calcitic-dolomitic and calcareous soils from Wisconsin as dolomitic (Table 3).

REFERENCES

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