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Soil Carbonate Analysis Using the Solvita Compost Maturity Gel System

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Core Ideas

- The Solvita compost gel system was well calibrated with calcium carbonate C.
- The Solvita compost gel system was highly correlated to the standard pressure calcimeter method.
- The Solvita compost gel system is an alternative method for inorganic C determination.

Abstract: Determination of inorganic carbon (IC) as calcium carbonate is important for soil fertility and soil health assessments in alkaline soils. Methods have been developed to determine IC, including the widely used pressure calcimeter method. We compared 19 alkaline soils using the pressure calcimeter method and the Solvita gel system, which was recently developed to measure CO_2 respiration. Results from the study indicated calibration curves for the Solvita gel system could be developed within an approximate range of 1 to 30 mg IC. Comparison of the Solvita gel system with the pressure calcimeter resulted in a near 1:1 relationship, where slight overestimation occurred when IC was less than 13 g kg^{-1} and slight underestimation above this level. The results of this study indicate the Solvita gel system can be considered as an alternative method for IC determination.

Alkaline calcareous soils have large quantities of inorganic carbon (IC) as calcium carbonate ($CaCO_3$) (Dari et al., 2019). In these soils, IC affects nutrient availability and is a factor for phosphorus (P) recommendations (Robertson and Stark, 2003; Moore et al., 2009). Understanding the IC content of alkaline soils is necessary to select appropriate soil tests that minimize IC interference. On high pH soils, Olsen bicarbonate is used for P, and ammonium acetate (pH 8.5 buffered) is used for secondary nutrient determination to minimize the effect of IC (Olsen et al., 1954; Knudsen et al., 1982; Miller et al., 2013). Additionally, the Haney, Haney, Hossner, Arnold (H3A) test (Haney et al., 2006, 2010, 2017, 2018), a component of the soil health tool, has P detection limit issues in high pH/IC soils (Harmel et al., 2009; Haney et al., 2017; Dari et al., 2019).

Multiple methods have been developed to analyze soil carbonate (Loeppert and Suarez, 1996; Miller et al., 2013). The pressure calcimeter method is commonly conducted where acid dissolution of $CaCO_3$ is used to produce carbon dioxide (CO_2) gas for measurement via the pressure generated or concentration of the CO_2 captured (Loeppert and Suarez 1996; Sherrod et al., 2002; Fonnesbeck et al., 2013).

Increased interest in soil health assessment has resulted in novel methods to measure soil respiration/CO_2 production. The Solvita gel system (Woods End Laboratories) is one method that has shown to be comparable to chemical titration and infrared gas analysis with the advantage of potentially simplifying the process through the elimination of more hazardous and expensive equipment (Haney et al., 2008). A modified Solvita gel system (Woods End Laboratories) is used for determining compost maturity (Changa et al., 2003; Storino et al., 2016).

No known work to date has investigated the potential of the gel system to measure IC in calcareous soils. Thus, the objective of this study was to determine

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Abbreviations: DCR, Digital Color Reader; IC, inorganic carbon.

if the Solvita gel system could produce comparable results as compared to the traditional pressure calcimeter method and thus be considered as a method for use in soil testing.

Materials and Methods

Calibration Curve for Pressure Calcimeter and Solvita Gel System

A laboratory study was conducted to compare the efficiency of measuring $CaCO_3$ using pressure calcimetry and the compost maturity Solvita gel system. All samples were analyzed in duplicate and averaged. A calibration curve was developed based on the methods of Sherrod et al. (2002) and Fonnesbeck et al. (2013). Standards were prepared using reagent grade $CaCO_3$ at amounts of 1.2, 6, 12, 18, 24, and 30 mg IC.

Calcimeter standards were analyzed using a 60-mL gas-tight vial, where 5 mL of distilled water was added. Vials were then sealed and 2 mL of 6 M HCl containing 3% (w/w) iron (II) chloride was injected through a septum. Measurements were taken after 2 h via a pressure transducer (Setra 280E, Setra Systems Inc.). A linear calibration curve was developed using the data from the pressure transducer.

The Solvita compost maturity gel system was used to develop the calibration using high CO_2 (compost) Solvita paddles. Standards were the same as above where a 473-mL wide-mouth Mason jar with an 86-mm dome lid was used (Rogers et al., 2017). Five milliliters of distilled water was added, and a 4-mL sample cup with 2 mL of 6 M HCl containing 3% (w/w) iron (II) chloride was placed inside each jar with a Solvita high CO_2 (compost) paddle. The paddle was lifted using a 5.7-cm plastic test tube to avoid the paddle touching the acid/water mix. The containers were then capped, the sample cup tipped over, and the acid swirled. In preliminary studies, the Solvita gel system required 6 h to equilibrate (data not shown); thus, this timeframe was used for extractions. After 6 h, the containers were opened and each paddle was analyzed under the high CO_2 (compost/manure) and low CO_2 (CO_2 burst) modes on a Solvita Digital Color Reader (DCR) Multi-Mode Unit (Woods End Laboratories). As IC measurement is separate from developed methods for the DCR, we recorded the color reading for our analyses. The calibration curve was fit to determine the amount of IC in the soil using the high CO_2 and low CO_2 values. Finally, these calibrations were used to determine the IC content of 1-g soil samples.

Soil/Standard Description and Characterization

The tested soils in the study include a mix of North American Proficiency Testing (NAPT) program standards and samples taken from across southern Idaho (n = 19) (Table 1). During preliminary experiments, we determined that the DCR was unable to quantify IC below approximately 1 mg and was not reliable beyond 30 to 36 mg IC.

Statistical Analyses

Linear regression models were used to fit the millivolts or Solvita color reading (high and low reading) as a function of IC content of the standards. Pearson correlations and linear regressions were performed to compare the IC

Table 1. Physicochemical properties of soils and North American Proficiency Testing Program (NAPT) samples used in the study. Samples 1–4 were from the NAPT where year-sample numbers are reported.

Sample	Soil texture/series	Sample/taxonomic classification	Soil pH	Inorganic C		
				Calcimeter	Solvita paddle	
					High color	Low color
				——— g kg^{-1} ———		
1	Sandy loam	NAPT-2009-115	7.9	2.2	3.4	3.8
2	Silty clay loam	NAPT-2016-103	8.0	12.4	14.3	14.3
3	Loam	NAPT-2017-104	7.5	3.1	5.5	5.8
4	Clay	NAPT-2018-115	8.1	7.3	8.1	8.4
5	Portneuf SiL	Coarse-silty, mixed, superactive, mesic Durinodic Haplocalcids	8.2	21.8	19.4	18.6
6	Power McCain complex	Power: Fine-silty, mixed, superactive, mesic Xeric Calciargids; McCain: Fine-silty, mixed, superactive, mesic Durinodic Xeric Calciargids	8.1	1.8	3.0	3.3
7	Harston fine sandy loam	Coarse-loamy, mixed, superactive, calcareous, frigid Xeric Torrifluvents	8.2	13.2	13.7	13.8
8	Rad FsL	Coarse-silty, mixed, superactive, mesic Durinodic Xeric Haplocambids	8.3	21.9	21.0	19.9
9	Portneuf SiL	Coarse-silty, mixed, superactive, mesic Durinodic Haplocalcids	8.3	16.6	15.4	15.4
10	Portino SiL	Coarse-silty, mixed, superactive, mesic Xeric Haplocalcids	8.1	15.9	14.3	14.1
11	Pancheri SiL	Coarse-silty, mixed, superactive, frigid Xeric Haplocalcids	8.1	21.8	20.3	19.6
12	Picabo SiL	Coarse-silty, carbonatic, frigid, Oxyaquic Calcixerolls	8.1	21.1	21.3	20.8
13	Potell SiL	Coarse-silty, mixed, superactive, calcareous, frigid Xeric Torriorthents	8.3	9.5	9.8	10.1
14	Declo loam	Coarse-loamy, superactive, mesic Xeric Haplocalcids	8.1	12.9	12.8	12.9
15	Declo loam	Coarse-loamy, superactive, mesic Xeric Haplocalcids	8.2	11.9	14.1	14.3
16	Declo loam	Coarse-loamy, superactive, mesic Xeric Haplocalcids	8.3	12.6	12.8	12.7
17	Declo loam	Coarse-loamy, superactive, mesic Xeric Haplocalcids	8.2	5.3	6.9	7.2
18	Declo loam	Coarse-loamy, superactive, mesic Xeric Haplocalcids	8.4	15.7	14.8	14.8
19	Kucera/Ririe complex	Kucera: Coarse-silty, mixed, superactive, frigid Calcic Pachic Haploxerolls; Ririe: Coarse-silty, mixed, superactive, frigid Calcic Haploxerolls	8.1	6.1	7.5	8.0

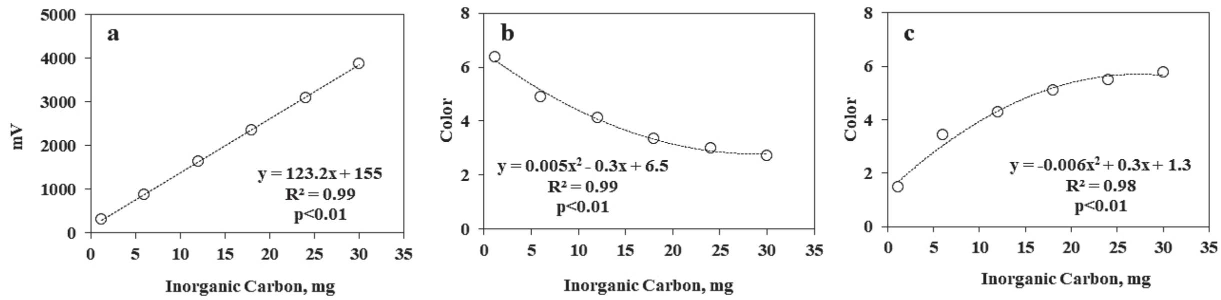

Fig. 1. Calibration curve for calcimeter showing (a) the relationship between inorganic C (IC) and millivolts measured via the pressure calcimeter and (b–c) the relationship between IC and color value measured via the Solvita high color paddles on both the (b) high and (c) low settings.

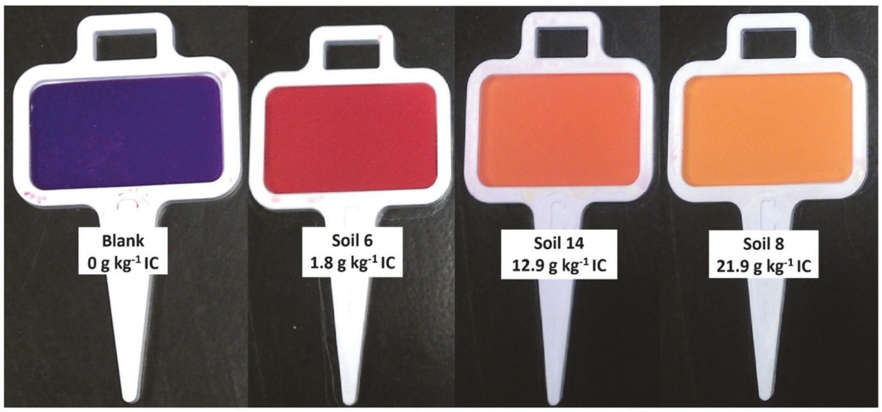

Fig. 2. Demonstration of Solvita paddle colors for various intensities of a range of soil samples where soil and calcimeter measured g kg^{-1} of inorganic C (IC) are reported.

values determined on the soils via the pressure calcimeter and Solvita gel system. All analyses were performed in Sigma Plot 13.0 (Systat Software, 2014).

Results and Discussion

The pressure calcimeter calibration regression produced a linear fit with R^2 = 0.99 (Fig. 1a). High (Fig. 1b) and low (Fig. 1c) color calibrations generated positive and negative polynomial fit with an R^2 of 0.99 and 0.98, respectively.

Soil sample values were calculated based on the calibration of IC to millivolts, high color readings, and low color readings, respectively (Table 1). Visual examples of the range of paddle results are shown in Fig. 2. Correlations indicated that pressure calcimeter IC was strongly related to both the high and low paddle readings where r = 0.99 for both (data not shown). Regression analysis further confirmed the goodness-of-fit as noted by an R^2 = 0.98 and 0.97 for the IC content determined by the high and low color paddles as compared to the IC content determined via the pressure calcimeter, respectively (Fig. 3). Compared with the calcimeter method, the gel system tended to slightly overestimate IC content when IC was less than 13 g kg^{-1} and underestimated above this IC level. However, within the range of the tested samples the difference from the 1:1 line was less than 1 to 2 g kg^{-1} at all points.

Conclusions

The Solvita gel system was strongly correlated with the pressure calcimeter IC in the soil samples used in this experiment. The Solvita gel system could be utilized with little change to existing DCR devices; however, a specifically designed method in the DCR would allow less effort by users and more accurate readings, and could potentially

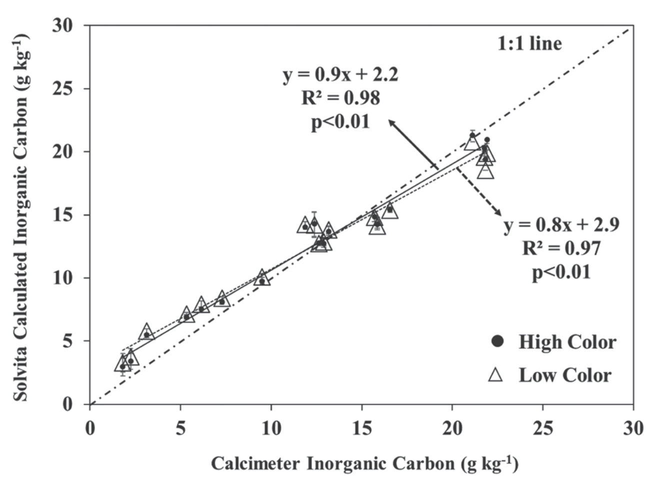

Fig. 3. Regression fit between inorganic carbon (IC; g kg^{-1}) measured using the pressure calcimeter and calculated using Solvita calibration. The solid and dotted lines are the linear regression fits for high and low color Solvita settings, respectively.

be adjusted to provide a wider range of analysis. Use of the Solvita paddles could reduce hazards involved in IC analysis and provide a simple and convenient way to analyze IC in soils.

Conflict of Interest

The authors declare no conflict of interest.

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