Determination of Carbonate Concentrations in Calcareous Soils with Common Vinegar Test

Qiang Zhu, Monica Ozores-Hampton, and Yuncong Li

Introduction
Calcereous soils are those that have free calcium carbonate \((\text{CaCO}_3)\) and have pH values in the range of 7.0 to 8.3. If they are managed properly, calcereous soils can be used to grow any crop. Before employing any management practices, it is important to know how much carbonate exists in the soil. Soil carbonate is usually quantified by acid dissolution followed by the volumetric analysis of the released carbon dioxide \((\text{CO}_2)\). In geological sciences, a simple acid test consists of placing a drop of dilute hydrochloric acid on a rock or mineral and observing if there are \(\text{CO}_2\) bubbles released; the bubbles indicate the presence of carbonate minerals. The household test below uses vinegar and other simple instruments to estimate soil carbonate concentration.

Background and Set-up

Principle
The determination of carbonate is based on the following chemical reaction:

\[
\text{CaCO}_3 + 2\text{H}^+ \rightarrow \text{Ca}^{2+} + \text{CO}_2 \uparrow + \text{H}_2\text{O}
\]

Carbon dioxide will be released during the reaction and expressed as bubbles. Whether the bubbling is weak or vigorous depends on the quantity and particle size of soil tested. Vinegar is a dilute acetic acid solution that produces a weak effervescent reaction with calcite, and it is easy to obtain and safer to use than hydrochloric acid, a strong acid.

Reagent
White vinegar with 5% acidity (supermarket grade)

Instruments
1. Eyedropper
2. Cap (0.8 tsp volume) from bottled spring water
3. Bowl (3.5 fl oz volume)
4. Stirring rod
5. Pen and paper

Procedure
1. Collect a composite soil sample that represents typical soil in the area, spread out, and dry it at room temperature for one week.
2. Remove gravel and large pieces of organic residue from the soil sample.

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3. Put one full cap of soil (Figure 1) into a bowl and add ten drops of spring water to wet the soil.

![Figure 1. One full cap of soil. The volume of the cap is 0.8 tsp, and one cap dry soil is approximately 0.13 to 0.19 oz. Credits: Q. Zhu](image)

4. Use the eyedropper to add white vinegar to the soil sample in increments of ten drops at a time, stir the mixture thoroughly, and record the number of drops added.

5. When the vigorous bubbles become weak after stirring (Figure 2), stop adding another ten drops of vinegar and let the mixture stand for five minutes.

![Figure 2. Vigorous bubbles become weak in the mixture of soil and vinegar. Credits: Q. Zhu](image)

6. Add ten more drops, stir the mixture, and wait for one minute.

7. If there are only a few small bubbles released, stop and sum the number of drops used; if there are still a lot of bubbles, perform steps 4, 5, and 6 again.

### Interpretation of the Vinegar Test

The vinegar test was compared to the volumetric calcimeter method, an accurate method to calculate calcium carbonate concentration (Chaney, Slonim, and Slonim 1982). The comparison showed a significant positive linear correlation ($y = 10.63x + 1.12, y$ is carbonate concentration (%) determined by volumetric calcimeter method, $x$ is the ratio of vinegar volume to soil mass, $R^2 = 0.94$) between the two methods. Therefore, soil carbonate concentration can be estimated using the vinegar test as indicated in Table 1. The table shows how carbonate concentration can be estimated by the total drops of vinegar added to one cap of soil. For example, if 50 drops of vinegar are recorded, the carbonate concentration will be 5%; if 80 drops are used, then the concentration value will be in the range of 5 to 10%.

<table>
<thead>
<tr>
<th>Total drops for one cap of soil</th>
<th>Carbonate concentration (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 10</td>
<td>&lt; 2</td>
</tr>
<tr>
<td>40–70</td>
<td>5</td>
</tr>
<tr>
<td>100–150</td>
<td>10</td>
</tr>
<tr>
<td>200–320</td>
<td>20</td>
</tr>
<tr>
<td>410–650</td>
<td>40</td>
</tr>
</tbody>
</table>

*One cap of dry soil is approximately 0.13 to 0.19 oz.

### Acidifying Calcareous Soils

Soil acidulents such as elemental sulfur, trisulfate salts, or fertigation acids such as nitric acid, phosphoric acid, or sulfuric acid have been successfully used to acidify calcareous soils with less than 3% CaCO$_3$. However, soil acidulents will not acidify soil with high concentrations of CaCO$_3$ (>10%) such as Krome, very gravelly loam, or Marl soils, which are found in south Florida. See Shober, Wiese, and Denny (2011) for recommendations on lowering soil pH by elemental sulfur in soils with less than 3% CaCO$_3$.

### References
