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Title: Monitoring Pedogenic Inorganic Carbon Accumulation Due to Weathering of Amended Silicate Minerals in Agricultural Soils.

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Author Questionnaire

1. **Microscopy**: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. **Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **No**

3. **Interview statements:** Considering the COVID-19-imposed mask-wearing and social distancing recommendations, which interview statement filming option is the most appropriate for your group? **Please select one**.

   ☒ Interviewees wear masks until videographer steps away (≥6 ft/2 m) and begins filming, then the interviewee removes the mask for line delivery only. When take is captured, the interviewee puts the mask back on. Statements can be filmed outside if weather permits.

4. **Filming location:** Will the filming need to take place in multiple locations? **No**

Current Protocol Length

Number of Steps: 11
Number of Shots: 15

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Rafael M. Santos:** The verification method described here is adaptable for monitoring pedogenic inorganic carbon sequestration in various agricultural soils amended with rocks containing alkaline earth metal silicates, such as wollastonite, basalt, and olivine.

1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.2. **Fatima Haque:** This method can be readily exploited by private or governmental entities for verifying soil inorganic carbon content, in view of qualifying farmers for negative emission carbon credits.

1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

OPTIONAL:

1.3. **Reza Khalidy:** Enhanced weathering of minerals spread onto lands can also lead to carbon sequestration in scenarios beyond agriculture, such as pasture, forestry or rehabilitated lands and urban soils.

1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.4. **Fatima Haque:** The heterogeneity of agricultural soils, both areal and depth-wise, makes it challenging to determine inorganic carbon content accurately, and sub-sampling of samples also contributes to reduced precision.

1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

1.5. **Reza Khalidy:** The use of standard additions of calcium carbonate, sample dividers, extensive analysis replication and statistical analysis can help first-time researchers gain confidence with the proposed methodology.

    1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

Introduction of Demonstrator on Camera

1.6. **Rafael M. Santos:** Demonstrating part of the field procedures will be **Stephen Vanderburgt**, a Master's Student from our laboratory.

    1.6.1. INTERVIEW: Author saying the above.

    1.6.2. The named demonstrator(s) looks up from workbench or desk or microscope and acknowledges the camera.

Protocol

2. Soil sampling and core collection

2.1. Begin by determining leveling of each plot **[1]** using a GPS receiver **[2]**, then place flags at the boundaries of each plot **[3]** to ease subsequent sampling **[4]**.

2.1.1. LAB MEDIA: Corn field.mp4.

2.1.2. LAB MEDIA: 00003.MTS, 00004.MTS, or 00005.MTS.

2.1.3. LAB MEDIA: 00007.MTS, 00008.MTS, or 00009.MTS.

2.1.4. LAB MEDIA: 00010.MTS or 00011.MTS.

2.2. Collect core samples from random points within each sub-plot, one per sub-plot. Use a soil probe or a soil core sampler to collect the soil core down to three depth zones of 0 to 15 centimeters, 15 to 30 centimeters, and 30 to 60 centimeters **[1]**.

2.2.1. LAB MEDIA: 00014.MTS, 00015.MTS, 00017.MTS, 00018.MTS, 00019.MTS.

2.3. Use an extendable auger to collect deep soil samples from additional locations, down to three depth zones of 60 to 100 centimeters, 100 to 175 centimeters, and 175 to 250 centimeters **[1]**. Transfer the soil samples into buckets, one for each sampled depth at each plot **[2]**.

2.3.1. LAB MEDIA: 00035.MTS or 00036.MTS.

2.3.2. LAB MEDIA: 00038.MTS.

2.4. Hand-blend the soils in each bucket thoroughly **[1]**, then place the portable moisture tester into the mixed soil sample and wait until the moisture content fixes at a stable point on the gauge of the device. Press the holder button and record the value as the real-time moisture content of blended soils **[2]**.

2.4.1. LAB MEDIA: 00042.MTS.

2.4.2. LAB MEDIA: 00039.MTS or 00041.MTS.

2.5. Label sample bags properly with information about the plots, the soil depth, and date of sampling, then store the composite samples in the bags **[1]**.

2.5.1. LAB MEDIA: 00044.MTS, 00045.MTS, or 00047.MTS.

3. Soil fractionation prior to chemical analysis

3.1. Air-dry the soil samples as soon as possible after sampling to minimize the oxidation of soil carbon. Place the soil samples in cardboard boxes **[1]** and place the boxes in a drying cabinet at 50 degrees Celsius for 24 to 48 hours, until the soil is dry **[2]**. Store the air-dried samples in sample bags until further analysis **[3]**.

    3.1.1. Talent placing a sample in a carboard box.
    3.1.2. Talent placing the box in a drying cabinet.
    3.1.3. Talent placing the dried sample in a bag.

3.2. Prior to soil fractionation, run the soil samples through a 2-millimeter sieve to remove large fragments of rocks and plant remains **[1]**. Oven-dry the sieved soils by placing them in a muffle furnace maintained at 105 degrees Celsius for at least 15 hours **[2]**.

    3.2.1. Talent running a sample through the sieve.
    3.2.2. Talent placing the sample in a muffle furnace.

3.3. For soil fractionation, place 1 kilogram of the oven-dried sample onto the top mesh of the sieve shaker consisting of different mesh sizes **[1-TXT]**. Shake the sieves at 60 rpm for 15 minutes **[2]**. Use pan fractions of less than 50 micrometers for analyses, as this is the pedogenic carbonate-enriched soil fraction **[3]**. *Videographer: This step is important!*

    3.3.1. Talent placing the sample on the sieve shaker. **TEXT: 710 to 50 µm**
    3.3.2. Sieve shaking.
    3.3.3. Soil fraction prepared for analysis.

4. Pedogenic inorganic carbon sequestration determination

4.1. To determine the inorganic carbon content of soil samples using calcimetry analysis, place 5 grams of a sieved soil sample in an appropriate Erlenmeyer flask **[1]**. Suspend the sample in 20 milliliters of ultrapure water **[2]**.

    4.1.1. Talent placing sample in a flask.
    4.1.2. Talent adding water to the sample.

4.2. Add 7 milliliters of 4 molar hydrochloric acid to a small flat-bottomed glass test tube **[1]**, then place this tube upright inside the flask using a pair of tweezers **[2]**.

    4.2.1. Talent adding HCl to the test tube.
    4.2.2. Talent placing the test tube in the flask.

4.3. Carefully attach the flask to the calcimeter by affixing the rubber stopper **[1]**. Adjust and read the initial water level in the burette and seal its headspace by turning the top valve to the measuring position **[1a]**. Shake the flask, thereby knocking over the acid tube **[2]**, until the water level in the burette reaches a constant value, and no bubbling is observed in the solution **[3-TXT]**. *Videographer: This step is important!*

VO talent this is a long step, and you can split it into 2 sound clips

    4.3.1. Talent attaching the flask to the calcimeter.

4.3.1a. Added shot: Talent adjusting and reading the initial water level in the calcimeter's burette, then turning the top valve to the measuring position.

4.3.2. Talent shaking the flask.

4.3.3. Water level reaching a constant value with no bubbling. **TEXT: Approximately 5 min**

Results

5. Results: SEM-EDS analysis of wollastonite-amended soil

5.1. A typical set of data for a wollastonite-amended soil compared to a control untreated soil is shown here **[1]**. The pH of the amended soil is higher by 1.15 units compared to the control **[2]** and the calcium carbonate content is nearly five times greater **[3]**.

5.1.1. LAB MEDIA: Table 1.

5.1.2. LAB MEDIA: Table 1. *Video Editor: Emphasize the pH for the wollastonite-amended soil.*

5.1.3. LAB MEDIA: Table 1. *Video Editor: Emphasize the calcimeter measurement for the wollastonite-amended soil.*

5.2. The calcium carbonate content was measured over a vertical profile **[1]**. The highest content was 10.13 grams per kilogram, detected in the 0 to 15-centimeter depth zone **[2]**. The two deep profile samples also had a high content of carbonates **[3]**.

5.2.1. LAB MEDIA: Table 2.

5.2.2. LAB MEDIA: Table 2. *Video Editor: Emphasize the 0 – 15 centimeters rows.*

5.2.3. LAB MEDIA: Table 2. *Video Editor: Emphasize the 60 – 100 centimeters row.*

5.3. The various oxides present in the soil were determined by WDXRF **[1]**. Silica was the main oxide present **[2]**.

5.3.1. LAB MEDIA: Table 3.

5.3.2. LAB MEDIA: Table 3. *Video Editor: Emphasize the* SiO_2 *row.*

5.4. The XRD pattern of a wollastonite-amended soil is shown here **[1]**. The main peaks present are quartz **[2]** and albite, which are predominant minerals in sandy loamy soils **[3]**.

5.4.1. LAB MEDIA: Figure 3.

5.4.2. LAB MEDIA: Figure 3. *Video Editor: Emphasize the Q peak at 26.65°.*

5.4.3. LAB MEDIA: Figure 3. *Video Editor: Emphasize the A peak at 28.07°.*

5.5. Wollastonite-amended soil was imaged using SEM after several weeks of weathering **[1]**. A closer look at the wollastonite particles shows the morphological changes occurring at the surface **[2]**. Microanalysis of the wollastonite surface was performed by obtaining an elemental mapping of the sample **[3]**.

5.5.1. LAB MEDIA: Figure 4 A.

5.5.2. LAB MEDIA: Figure 4 B.

5.5.3. LAB MEDIA: Figure 4 C – F.

5.6. The EDS spectrum of the mapped area revealed its semi-quantitative chemical profile. The elemental maps clearly show silicon and calcium as the main components of wollastonite **[1]**.

  5.6.1. LAB MEDIA: Figure 4 G.

5.7. Spot EDS analysis was performed on the smaller fragments scattered in the soil sample **[1]**. The fragments were rich in carbon and oxygen, suggesting that they are made up primarily of organic matter **[2]**.

  5.7.1. LAB MEDIA: Figure 5.

  5.7.2. LAB MEDIA: Figure 5. *Video Editor: Emphasize the C and O peaks in the bottom 2 graphs.*

Conclusion

6. Conclusion Interview Statements

6.1. **Rafael M. Santos:** When attempting this protocol, keep in mind that the sampling depth may vary in different areas depending on the ease of sampling over the vertical profile, the thickness of the surface soil horizon, the depth of the water table, and soil structure.

6.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.2, 2.3.*

6.2. **Fatima Haque:** Measuring stable isotopic and radiogenic carbon signatures over the vertical profile of soil and subsoil could be incorporated in this procedure to further verify the sequestration of atmospheric CO_2 in mineral-amended fields.

6.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.