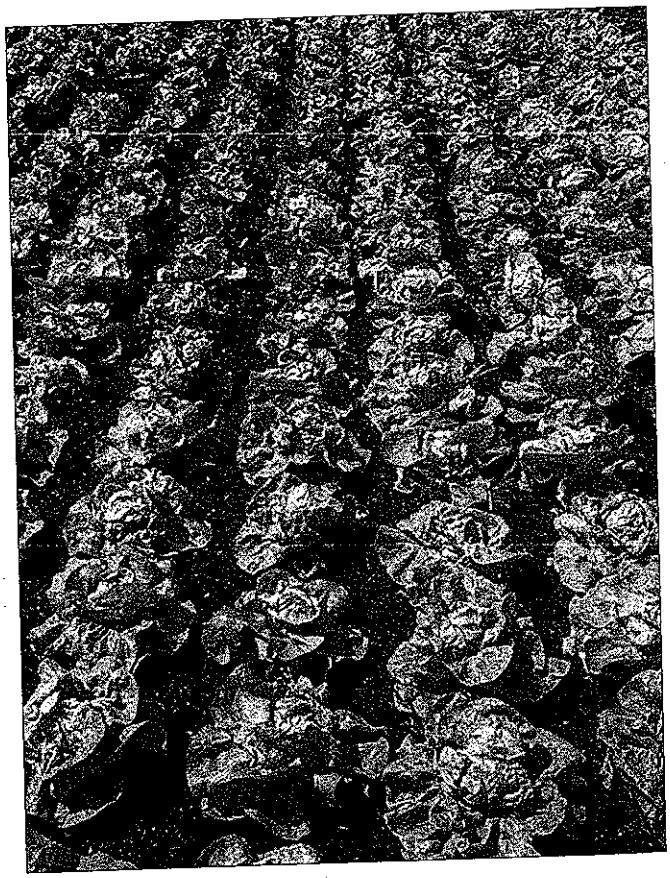
Carbon Cycle:
Measuring CO_2 Flux from Soil

Measuring soil respiration familiarizes students with an important component of the carbon cycle and makes the invisible mechanisms of soil biology and climate change more tangible



by Robert Lessard, L. Dennis Gignac and Philippe Rochette

Subject areas: biology, chemistry, science, mathematics

Key concepts: carbon cycle, greenhouse effect, soil respiration, soil organic matter, acid-base titration

Skills: using the scientific method, observation, taking scientific measurements, data synthesis and analysis, presentation skills

Location: outdoors or indoors

Time: 1 day for preparation and set up, 4–24 hours for incubation and sample collection, 1–2 hours of lab work for titration and calculations

Materials: air chambers, small yogurt containers, stoppered vials, titration equipment and chemicals (see "Materials" section below)

Teaching concepts that relate to environmental problems is not always easy since many of the underlying components may be abstract, complex or invisible. Such is the case with climate change and global warming. Greenhouse gases are virtually undetectable without the use of sophisticated gas-analyzing equipment that is unaffordable to most elementary and secondary schools. It is therefore difficult for students to realize the large amounts of gases emitted into the atmosphere. Students are told that greenhouse gas concentrations are increasing and that these changes are globally important since they will result in climate change. However, for many students, Earth's atmosphere remains a mysterious black box. One way to demystify this concept is to have students measure carbon dioxide (CO_2) flux, or rate of emission, from soil to the atmosphere.

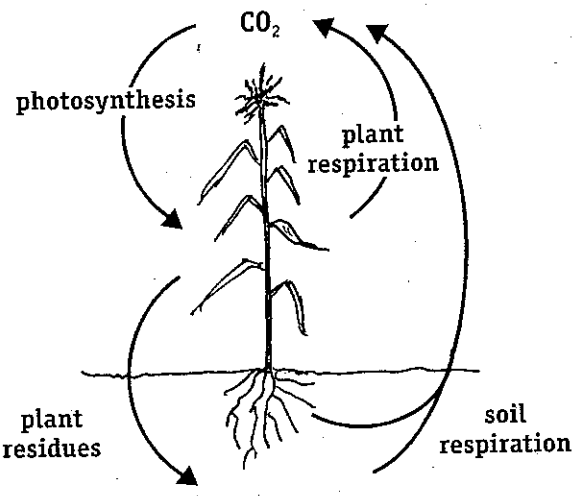
The activity that we describe here is designed for secondary school students and attempts to familiarize them with one aspect of the global carbon cycle: the production of CO_2 through soil respiration. It can also be used to demonstrate how the soil can become a sink for carbon, thus reducing concentrations in the atmosphere and alleviating the trend toward global warming. Furthermore, it presents a practical and inexpensive method for measuring CO_2 fluxes from soils.

Background

Soil respiration is defined as the production of CO_2 as a result of two processes: the breakdown, or oxidation, of carbon-rich organic matter by soil microorganisms, and respiration by plant root cells. The rate of CO_2 production is scientifically important because it provides an indication of the rate of breakdown of organic matter and therefore of the amount of soil carbon that is lost. Measurements of soil respiration help to determine the contribution of soil to the atmospheric CO_2 budget.

Carbon, an essential element for plant growth, is obtained from the atmosphere through photosynthesis. However, when plants die, their carbon-rich tissues are returned to the soil and decomposed by living organisms. Soil organic matter is therefore the sum of organic residues (plants and animals) at various stage of decomposition. Organic matter improves soil quality, helps prevent runoff, increases soil humidity and helps to moderate daily temperature fluctuations in the top layers of the soil. Soil organic matter also functions as an enormous storehouse for carbon: it is estimated that living organisms account for about one-quarter of all the carbon in terrestrial ecosystems, while the remaining three-quarters is stored in the organic matter contained in soils.

Soil carbon does not accumulate forever. It is released from the soil when organic matter is broken down by several types of aerobic organisms that use carbon for their own growth. This process liberates plant nutrients, which can then be taken up by living plants, but it also releases CO_2. The rate of microbial activity, and hence of soil respiration, is affected by soil temperature and humidity as well as by the quantity and quality of soil organic matter. Since all aerobic organisms release CO_2 as a result of the breakdown of organic molecules, and since there can be millions of these organisms in as little as a teaspoon of soil, soil respiration is an important source of atmospheric CO_2, contributing up to 100 billion metric tons of carbon to the global carbon cycle each year.



Much of the atmospheric carbon obtained by plants through photosynthesis is eventually stored in the soil as organic matter. Carbon is released back to the atmosphere through cellular respiration and the breakdown of soil organic material.

The increase in atmospheric CO_2 levels since the beginning of the Industrial Revolution is largely due to the burning of fossil fuels and changes in land use. Agricultural practices, often overlooked as a source of greenhouse gases, are responsible for approximately ten percent of greenhouse gases emitted by human activity in most developed countries. The quantity of carbon that is retained in the soil or lost to the atmosphere largely depends on the method of tillage used. When fields are plowed, fresh organic residues are thoroughly mixed into the topsoil. Under these conditions, microbial activity increases, resulting in more of the soil's organic carbon being turned into atmospheric CO_2. There is also a net loss of carbon when fields are left fallow. This is due in part to higher soil temperature and humidity, which accelerate decomposition, and also to the fact that no carbon is added to the soil during a year when there is no crop.

Sound land management practices help to conserve organic matter in soil, thus reversing the tendency of soils to lose their carbon to the atmosphere. One of these practices is no-till agriculture, which consists of seeding a crop in the residues of the previous year's crop. Studies show that the establishment of a permanent vegetation cover also contributes to the sequestration of carbon in the soil. Thus, along with reducing our consumption of fossil fuels, practicing good land management is another method of reducing the concentration of greenhouse gases in the atmosphere, by storing carbon dioxide as organic matter in the soil.

Since soil respiration plays such an important role in the carbon cycle, measuring it is a valuable component of effective teaching about the mechanisms that produce global warming. However, finding an inexpensive but effective method of measuring the CO_2 that is produced through soil respiration is fundamental. The method proposed for this activity is based on the ability of some alkaline compounds, such as sodium hydroxide (NaOH), to react with CO_2 from the air.

Experimental design

In this experiment, plastic containers are placed upside down on several sampling sites to act as air chambers in which CO_2 from the soil collects. A smaller container of NaOH is placed inside each air chamber to trap the CO_2. Alkali trapping uses the weakly acidic properties of CO_2 in an acid-base neutralization that produces carbonate:

$$CO_2 + 2Na^+ + 2OH^- \rightarrow CO_3^{2-} + 2Na^+ + H_2O$$

As long as there is an excess of OH^- ions (a result of NaOH dissociation), the equilibrium shifts towards the right, producing carbonate. Following an incubation period, the NaOH is collected and the carbonate is precipitated using barium chloride ($BaCl_2$) The quantity of CO_2 that is absorbed in the alkaline solution can then be measured using a simple titration.[1]

Materials

- air chambers, one per sample: 5-liter plastic pails, approximately 18 cm in diameter and 18.5 cm deep will work well
- NaOH containers, one per sample: small plastic yogurt containers, approximately 9 cm in diameter, cut to a height of approximately 4 cm. Any small plastic container can be used; however, to maximize the absorption of CO_2, the area of the opening of the NaOH container should be greater than 26% of the area of the opening of air chamber.
- one stoppered vial for each sample
- one or two flat boards (for control sites) measuring 30 cm x 30 cm, or larger
- electrical tape
- 250 ml Erlenmeyer flasks
- 10 ml and 25 ml graduated cylinders
- 5 ml pipettes
- 400 ml beakers
- 50 ml burettes mounted on retort stands
- 1 liter of 0.25 M NaOH
- 500 ml of 3 N $BaCl_2$ (1.5 M)
- 2 liters of 0.1 M HCl
- phenolphthalein indicator

Safety precautions

The solutions used in this activity are very corrosive. The sample collection sites should be fenced and have warning signs. Gloves, eye protection and lab coats or aprons should be worn whenever the chemicals are handled. At the end of the experiment, ensure that all solutions are properly disposed of in chemical waste containers.

Preparing solutions

NaOH: To prepare the 0.25 M NaOH solution, add 10 grams of NaOH pellets to 500 ml of distilled water. Swirl and then complete the volume to 1 L by adding more distilled water. Secure the stopper tightly to protect the solution from exposure to air.

HCl: To prepare the 0.1 M HCl solution, add 16 ml of concentrated acid to 1 L of distilled water. Swirl the solution and then add 1 L of distilled water to make a total volume of 2 L. It's a good idea to standardize the dilute acid using a 0.5 M NaOH standard solution. HCl and NaOH concentrations can be adjusted according to the anticipated CO_2 soil emissions. Lower concentrations of acid and alkali solutions are used if CO_2 emissions are expected to be low. To ensure best results, the HCl concentration is kept between one-half and one-quarter of the concentration of the NaOH.

$BaCl_2$: $BaCl_2$ is used in this experiment to precipitate the carbonate produced as insoluble $BaCO_3$. To prepare the 1.5 M solution, add 156.2 grams of $BaCl_2$ to 400 ml of distilled water and swirl. Then complete the volume to 500 ml. If $BaCl_2H_2O$ is used instead, 183.2 grams are needed to produce the 1.5 M solution.

Phenolphthalein indicator: The phenolphthalein solution serves as a pH indicator dye. Prepare a 1% solution by adding 1 gram of phenolphthalein to 100 ml of 95% (vol/vol) ethyl alcohol. Only two drops of this solution will be needed for each titration.

Selecting sites

Since the sample collection sites will have open containers of NaOH, they should be in an area where public access is limited. Select sites of approximately 50 cm by 50 cm in an area with little or no vegetation. Any soil may be used for this experiment as long as the surface of the ground is relatively flat. For best results, sample several locations that have different types of soil. The important variable that distinguishes the type of soil is the quantity of organic material it contains. Generally, if the soil temperature is high enough (> 10°C) and there is enough moisture, soils that contain the most organic matter will have the highest rate of soil respiration. After selecting suitable sampling locations, remove any vegetation from the sites at least 24 hours in advance in order to eliminate CO_2 that

may be produced as the result of soil disturbance.

Option: If finding suitable sites is a challenge, a schoolyard or lab experiment may be easier to manage. Growing beds packed with soil that is amended with different concentrations of organic matter can be used to simulate soils having different levels of carbon content.

Setting up collection chambers

Have students work in pairs, each pair being responsible for measuring the soil respiration at one sampling site. Assign one or two pairs of students to control sites. Controls are necessary in order to obtain a measure of ambient CO_2 in the atmosphere. This value will be subtracted from the quantity of CO_2 measured from soil samples in order to determine how much of the trapped CO_2 is contributed by soil respiration. Set up each sampling site as follows:

1. Measure and record the diameter of the opening of the air chamber in order to calculate the surface area that it will cover when placed upside down over the NaOH trap.

2. Position the NaOH trap approximately 4 cm above the soil. This is done by taping three small sticks approximately 15 cm in length to the sides of the container to act as a tripod (see Figure 1).

3. Place 25 ml of NaOH solution in the bottom of the NaOH trap (see Figure 2). The soil should not be disturbed during this step. In addition, students should not breathe directly on the surface of the liquid since this will contaminate it with CO_2. Due to the corrosive nature of NaOH, the 25 ml of solution for each trap should be transported in a stoppered vial to the study site.

4. Quickly place the air chamber over the trap and set its edges approximately 2 cm into the soil (see Figure 3). Some of the surrounding soil may be gently pushed along the edge to ensure a complete seal and prevent loss of CO_2 to the atmosphere.

5. Leave the chamber undisturbed for 24 hours, depending on the organic content of the soil. Soils that contain a large proportion of organic matter usually require less time than those that do not. The time must be accurately recorded in order to calculate fluxes.

Controls: Set up the control chambers in the same manner as the others, with the following exceptions. Lay a flat board on the surface of the soil to act as a barrier between the soil and the NaOH trap. Place the NaOH trap directly onto the board (do not elevate the trap on a tripod). Mount the air chamber over the trap and seal the edges to the board using electrical tape (see Figure 4). Controls are left for the same time period as the treatments.

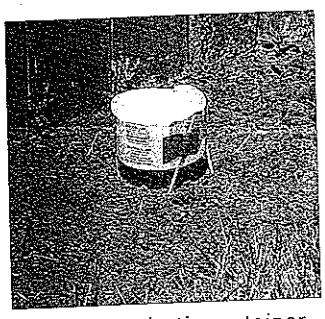
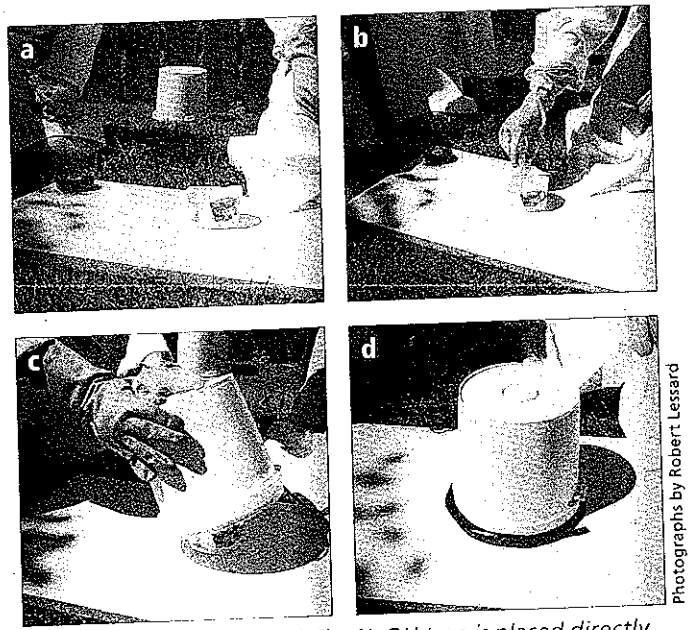


Figure 1: *A plastic container cut to a height of about 4 cm serves as the NaOH trap. It is suspended above the soil on a tripod made of three sticks taped to the container.*

Figure 2: *The NaOH is transported to the study site in a glass vial and then poured into the trap.*

Figure 3: *The air chamber is a 5-liter plastic pail placed over the trap and pushed into the soil to a depth of 2 cm. Right: Soil surrounding the chamber is pushed around the edges in order to complete the seal.*



Photographs by Robert Lessard

Figure 4: *Control set-up. a) The NaOH trap is placed directly on a flat board. b) 25 ml of NaOH is poured into the trap. c) The air chamber is placed over the trap. d) The chamber is sealed to the board using black electrical tape.*

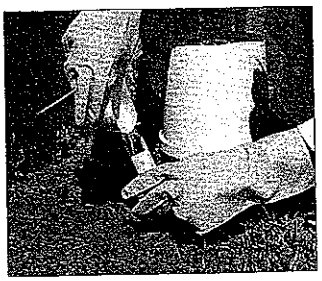


Figure 5: At the end of the experiment, the chamber is gently removed and the NaOH is poured into a glass vial for transport to the lab.

6. After the incubation period, collect the NaOH samples in suitably labeled vials. To do this, simply pour the liquid in the traps into the vials, making sure that none is lost and without breathing directly on it (see Figure 5). Then return the samples to the lab for titration.

Titration

Titrate each of the NaOH samples three times as follows.

1. Place a 400 ml beaker under a 50 ml graduated burette. Open the stopcock and rinse the burette a few times with distilled water. Add 50 ml of 0.1 M HCl to the burette and allow it to run through the tip. Close the stopcock and add 0.1 M HCl to the burette until the bottom of the meniscus is at 0 ml.

2. Place 5 ml of the NaOH solution from the sample into a 250 ml Erlenmeyer flask and add 10 ml of distilled H_2O.

3. Precipitate the carbonate contained in the NaOH solution by adding 10 ml of 3 N $BaCl_2$. The quantity of white crystals formed is proportional to the concentration of carbonate present in the alkali solution. The reaction, including $BaCl_2$, is:

$$CO_3^{2-} + 2Na^+ + Ba^{2+} + 2Cl^- \rightarrow BaCO_3 + 2NaCl$$

4. Add 2 drops of phenolphthalein indicator to the flask. This should turn the solution pink.

5. Place the flask under the burette. Slowly add drops of the HCl to the sample. After each addition, pause and mix the solution by swirling gently or stirring with a glass rod. When the color of the sample changes from pink to transparent (equivalence point), stop the titration and record the volume of HCl that was used.

6. Repeat the titration two more times for each sample. Find the mean volume of HCl required for titration.

Quantification of CO_2 concentrations in the samples

The quantity of CO_2 absorbed by the NaOH trap (QCO_2) for each sample is calculated as follows:

$QCO_2 = (T - C)(N)(E)(Vtr/Vti)$ where:

T = mean volume of HCl used to titrate the CO_2 in the control

C = mean volume of HCl needed to titrate the CO_2 in each of the samples

N = normality of the HCl used = 0.1

E = conversion factor: use 22 to obtain mg of CO_2, or 6 to obtain mg of C absorbed by the trap

Vtr = volume of NaOH in the trap in ml = 25 ml

Vti = volume of NaOH used in titration = 5 ml

Sample Calculations

In this example, NaOH that had incubated for eight hours was collected from a trap above a sampling site and from a control. The mean volume of HCl (0.1 N) needed to titrate the NaOH from the control was 36 ml; the volume needed for the soil sample was 30 ml.

CO_2 concentrations in samples

A conversion factor of 22 is used to calculate the quantity, in milligrams, of CO_2 absorbed by the NaOH trap. The volume of NaOH in the trap was 25 ml and the volume used for each titration was 5 ml. These values are entered in the equation $QCO_2 = (T - C)(N)(E)(Vtr/Vti)$ as follows:

T = 36 ml N = 0.1 N Vtr = 25 ml
C = 30 ml E = 22 Vti = 5 ml

Thus, $QCO_2 = (36 - 30)(0.1)(22)(25/5) = 66$ mg of CO_2

CO_2 flux from the soil

Continuing with the above example, the diameter of the opening of the air chamber was 18.2 cm, or 0.182 m. The surface area of soil thus exposed was $A = (\pi)(0.182/2)^2 = 0.02602$ m². Since the incubation time was 8 hours, the CO_2 flux (FCO_2) = 66/(0.02602)(8) = 317.06 mg CO_2 $m^{-2}h^{-1}$

Quantity and flux of carbon

To find the quantity of carbon absorbed by the trap, QC, use the formula with the conversion factor E = 6. The conversion factor is obtained by multiplying 22 by the portion of carbon in a mole of CO_2 (22 x 12g C / 44 g CO_2). This gives QC = 18 mg of carbon. The flux of carbon (FC) would thus be 18/(0.02602)(8), or 86.5 mg of carbon m^{-2} h^{-1}.

Quantification of soil respiration

This calculation takes into account the surface area of the soil that was exposed and the incubation time. Soil respiration is calculated as the rate of CO_2 production by a surface area during a period of time and is then defined as the CO_2 flux (mg/m²/hr). The flux (FCO_2) is calculated as follows: $FCO_2 = QCO_2/(A)(t)$, where A is the area of exposed soil in square meters and t is the time of incubation in hours.

Extensions

1. Most of the CO_2 emitted at the soil surface is produced by microorganisms, and the warmer the soil, the more intensive the microbial activity. Soil temperature is therefore one of the most important environmental factors controlling the rate of CO_2 production in soils. By using temperature probes, students could measure this factor over several days. Students could then draw a graph of soil respiration as a function of soil temperature.

2. A second variable that affects soil respiration is the moisture content of the soil. The moisture content is easily measured by weighing soil samples, drying them in an oven at 60°C for 24 hours, and then weighing them again. The percent moisture content *(MC)* is calculated as: *MC = [(fresh weight – dry weight)/dry weight] x 100.*

3. Organic content is an important variable that affects soil respiration. The percent of organic content can be estimated by combustion, as follows. Remove a handful of the top 10 to 15 cm of soil from the study location. Heat a sub-sample of this soil at low intensity in a crucible to evaporate the water, and then weigh the soil to obtain the dry weight. Next, heat the soil at high intensity for a few minutes until the color no longer changes. Cool the soil and then reweigh it to obtain the burned weight. The percent organic content *(OC)* of the soil is calculated as: *OC = [(dry weight – burned weight)/dry weight] x 100.*

4. Other factors that change CO_2 fluxes from soils are quantity and quality of organic residues being decomposed. There are several ways in which organic soil amendments may influence CO_2 fluxes. For example, adding readily decomposable materials such as fresh manure, flour or even sugar to soil should generate higher fluxes compared to soils amended with substrates that decompose at a lower rate (wood chips, straw). Designing experiments in which the type and/or the quantity of organic matter added to soil changes will help students understand how organic amendments affect soil respiration.

Evaluation

At the end of the exercise, students should be able to measure CO_2 and understand that it is produced by soils and accumulates in the atmosphere. They should also understand that several factors, such as temperature and humidity of soils, affect their rate of CO_2 production. If extension activities 3 and 4 (see above) are used, they will also grasp the relationship between the amount and type of organic matter added to a soil and the quantities of CO_2 released to the atmosphere. A possible discussion question that students could reflect on is: How does returning more organic material to soils (crop residues, manure, etc.) increase agricultural sustainability *(elements of answer: reduced erosion, better retention of water and nutrients, higher intrinsic fertility, better soil aeration, less compaction)* and act on the total greenhouse gas balance?

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Note

1. P. Rochette and G.L. Hutchinson, "Measurement of soil respiration in situ: Chamber techniques," in J. Hatfield and J. M. Baker, eds., *Micrometeorology in Agricultural Systems*, ASA monograph #47, Madison, WI: American Society of Agronomy, 2005, pp. 247–286.

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