

Detection of Isoprene in the Environment Using Microcantilever Sensor

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Abstract

Isoprene is a colorless liquid produced in abundance by plant life throughout the environment. It is highly volatile, though, and is normally in a gaseous state when found in nature. By measuring the amounts of gaseous isoprene in soils we may be able to gain insight into relative levels of biological activity that is occurring within the soils. The levels of biological activity in various soils are directly correlated with the soil respiration of greenhouse gases such as methane or carbon dioxide. In this study, we have designed a small, portable piezoresistive microcantilever sensor for the detection of isoprene. Measurements of isoprene gas are taken in forest and farm soils using the microcantilever sensor and compared with similar measurements taken with a quadrupole mass spectrometer.

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Introduction

Piezoresistive microcantilever sensors are small, portable and robust sensors that have been used in a wide variety of applications in the detection of various gases and microbes in gaseous and liquid environments [1-7]. In a typical application, a sensing material is used that is designed to undergo a tiny volumetric change when exposed to the desired analyte [6, 8, 9]. This volumetric change in the sensing material is measured by a tiny piezoresistive microcantilever that is partially embedded in the sensing material itself. Volumetric changes in the sensing material result in a tiny strain in the microcantilever, which results in a measurable change in the electrical resistance of the cantilever that is proportional to the amount of the strain. Piezoresistive microcantilever sensors are tiny, portable, resistant to external shock, suitable for applications in gaseous or liquid environments, and require only simple support electronics to operate.

In soils found throughout nature, including farmland soils and forest soils, various types of microbes are involved in the emission or absorption of a number of various gases. These gases may include greenhouse gases such as carbon dioxide (CO₂), methane (CH₄), isoprene (C₅H₈), and others. The absorption or emission of these gases is often referred to as soil respiration. Soil respiration may depend upon several environmental factors such as soil water level or humidity, temperature, amount and type of decaying plant material, soil mineral composition, and microbial presence and activity within the soil [10-19]. Microbial processes in soils are important and varied, including the production of isoprene. Isoprene itself has been shown to serve as a marker for the overall level of microbial activity in the soil [20-22]. *Bacillus*, and relatives, produce large amounts of isoprene along with the forest plants themselves, while other bacteria in the soil may act to consume isoprene. Measuring the relative levels of isoprene in forest soils in conjunction with simultaneous measurements of methane and carbon dioxide may allow us to more exactly correlate the shifts in soil greenhouse gas respiration with the levels of microbial activity in the soils themselves.

In this study, we have constructed a piezoresistive microcantilever sensor suitable for the detection of isoprene. Measurements taken with this device in forest soils were also directly compared with isoprene concentrations as measured in parallel with a portable quadrupole mass spectrometer. Levels of isoprene in various soils, considered along with levels of methane and carbon dioxide may allow us to correlate the amounts of soil greenhouse gas respiration with the levels of microbial activity within the soils. The overall design of the

isoprene sensor is suitable for long-term in-situ monitoring. The microcantilever sensor itself may be buried in the soil and is resistant to water and other environmental changes that may occur over time. The support electronics for the sensor are simple [23], requiring only the measurement of the sensor resistance. We may power these devices with only two AA batteries, and they may be interfaced with sensor mesh transmitters for longer-term, remote applications.

Materials and Method

The sensors utilized in this study are based on a flexible semiconductor piezoresistive microcantilever. These particular microcantilevers were designed by Cantimer, Inc., Menlo Park, CA. A single microcantilever is approximately 200 microns in length and 40 microns wide. The cantilevers are integrated onto a silicon chip and extend into a small circular area of diameter 1 mm to contain the sensing material and also to protect the cantilever from shock, vibration and other physical disturbances such as insertion into soil of liquid. Each microcantilever also contains an integrated thermistor for temperature measurement and correction. The final assembly is mounted on a small plastic circuit board for interfacing external electronics (Figure 1).

Each sensor utilizes a "sensing material" that expands or contracts a small amount when exposed to the desired analyte. Here, we have used polyisoprene dissolved in an organic solvent to construct our sensors. Polyisoprene is an exact match for our analyte, gaseous isoprene, based on its solubility parameter of 16.5 mPa^{1/2} [3]. In the actual construction of a sensor, a small amount of sensing material in liquid form is deposited onto separate Si substrate. Using a micromanipulator under a microscope, the microcantilever tip is positioned to partially insert or contact the sensing material. The Si substrate is then bonded to the cantilever chip while held in position using epoxy, forming a rigid sensor assembly [5, 24]. The baseline electrical resistance of a microcantilever sensor is approximately 2.2 kΩ.

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Figure 1: Piezoresistive microcantilever isoprene sensor mounted on circuit board prior to insertion into the soil. The microcantilever sensor extends into a small circular area at the end of the circuit board used to protect the sensor. The microcantilever is nearly invisible to the human eye owing to its very small size.

Each microcantilever sensor is operated by using a resistance measuring electronic chip. Here, we use a small AD7793 24-bit A/D converter interfaced to a laptop computer through a USB serial converter to make measurements. The AD7793 provides two programmable current sources which are each set to 100 μ A. The controlled constant current is passed through the piezoresistive microcantilever and the temperature sensor on each sensor chip. Finally, the voltage drops across each of the two elements is measured by the A/D converter and recorded by the laptop computer. Commands to the AD7793 are facilitated by a custom assembly language program on the laptop computer and transmitted to the AD7793 by using a U421 USB serial converter.

In Figure 2 below, we show an isoprene microcantilever sensor interfaced to a battery-powered mote transmitter using Zigbee radio frequency protocol. Sensor mote construction and sensor interfacing electronics have been described in an earlier publication [3]. Here, the small sensors may be deployed for long periods of time in a mesh network with many such mote-sensor combinations that communicate with each other and to a single base station. In the current study, we used a laptop computer for interfacing to a single sensor for our testing purposes.



Figure 2: Microcantilever interfaced to battery-powered mote. Here, only the microcantilever sensor is active, the integrated temperature sensor is not connected.

Results and Discussion

For controlled exposure to isoprene in the laboratory, we placed the sensors in a 22.5 liter plexiglass chamber. The chamber is equipped with sealed electrical feedthroughs for sensor interfacing without opening the chamber to the environment. The chamber also contains an insertion port for the introduction of analytes. In the case of injecting liquid isoprene into the chamber, an amount equivalent to 200 ppm gaseous isoprene was used. When the liquid is injected, it is fully evaporated within 60 sec owing to its high volatility. Figure 3 shows the sensor response to 200 ppm isoprene exposure.

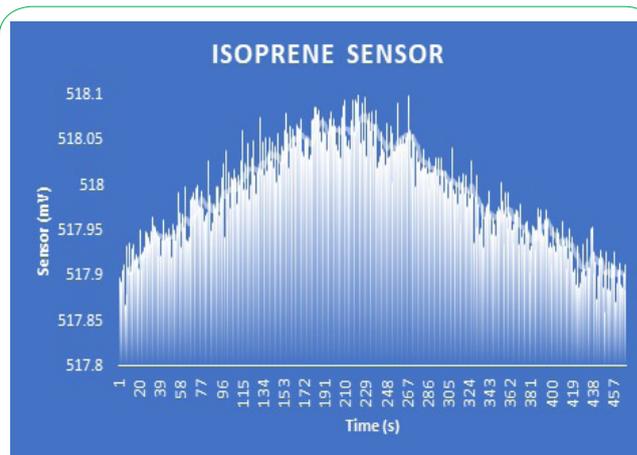


Figure 3: Isoprene sensor response to gaseous isoprene at 200 ppm. The isoprene was injected into the chamber at time 0 sec, and at time 250 sec the chamber was vented to test the sensor return to equilibrium.

The microcantilever sensor response to isoprene introduction into the test chamber is rapid, however it takes approximately 250 sec of exposure to for the sensor to reach maximum sensor response. We expect this is due primarily to a relatively slow rate of diffusion or partitioning of the gaseous isoprene molecules into the polymeric sensing material. In addition, the liquid isoprene itself takes approximately 60 sec to fully evaporate after it is introduced into the chamber. When the chamber is vented to ambient air after 250 sec, the sensor slowly recovers to its original state. The temperature remained constant at 21.5°C during this time period.

In the plot below, Figure 4, we show the microcantilever sensor output taken in the “Cowboy” fire burned forest region within the Coconino National Forest. The Cowboy fire is a wildfire which burned a small forest region south of Flagstaff, AZ in 2016. In the Cowboy fire region, the fire damage was total, with few surviving trees or smaller vegetation. Today in this region, forest growth is returning, including small bushes and grass. Tree canopy growth is back to approximately 50 percent of its original state prior to the fire. For this isoprene measurement, the sensor is inserted into the soil approximately 90 sec prior to measurement and held in the soil for 190 sec additional time. This time interval was chosen to allow the gases in the soil and the instrument to begin to come to equilibrium. The sensor is then removed from the soil and exposed to ambient air for the remainder of the time. We can see that the isoprene level in the soil is less than the surrounding air, and the sensor reflects that as it slowly comes to equilibrium with the lower relative levels in the soil. After the sensor is removed from the soil and exposed to the ambient forest air, it begins to come to equilibrium with the higher isoprene levels in the surrounding forest air. Here, the sensor response is quicker to come to

equilibrium to ambient air that in the lab test chamber, as no time is required to slowly vent the isoprene from test chamber.

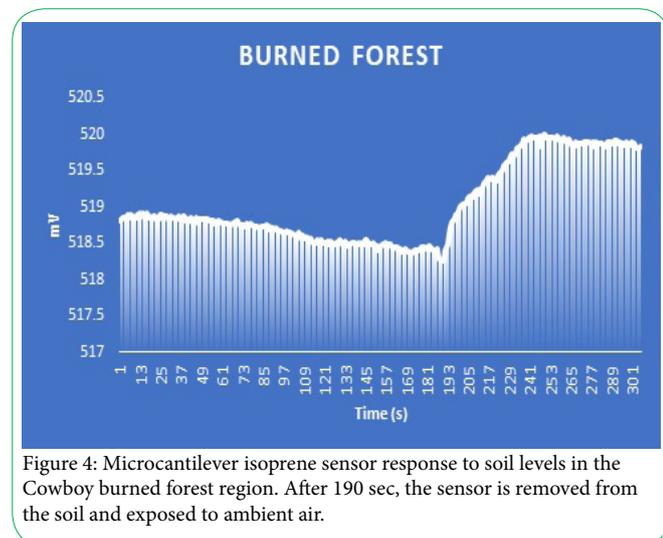


Figure 4: Microcantilever isoprene sensor response to soil levels in the Cowboy burned forest region. After 190 sec, the sensor is removed from the soil and exposed to ambient air.

In previous studies, we have used a quadrupole mass spectrometer to measure forest soil isoprene levels relative to ambient air. Soil microbes such as Actinobacteria and Alphaproteobacteria as well as some fungal species have been shown to consume isoprene in soils [20]. In a laboratory study [20], generic soils were found to eliminate approximately 55% to 80% of the gaseous isoprene provided to the soils. In pristine forest soils, where there have been no wildfires, forest thinning or forest logging operations, the relative isoprene levels are measured by us to be the lowest. In this case isoprene relative levels are typically only about 75% that of the ambient isoprene concentrations. In soils from burned forests, isoprene levels as measured in-situ with quadrupole mass spectrometry, have been observed to be somewhat higher than in pristine forests, but still lower than ambient air. In the "Cowboy" fire forest, the soil isoprene level as measured by the quadrupole was about 80% that of the surrounding air. Thus, from both sensor and quadrupole mass spectrometer measurements, our data indicate that the soil appears to be acting as an isoprene sink.

Conclusions

Isoprene is widely produced in the environment primarily by both plant life and certain types of microbial activity. Within the soil, isoprene may be either produced or consumed, depending on the types of microbes in the soil, soil humidity, temperature, decomposing soil plant litter, and other factors. We have constructed a piezoresistive microcantilever sensor suitable for the detection of the volatile gas phase of isoprene. This sensor is small, portable, requires only simple support electronics, and may be useful in measuring absolute levels of isoprene in forest and other soils over long time periods. Exposure to isoprene levels of 200 ppm under controlled conditions in the lab show the sensor responds readily to gaseous isoprene. The sensor also showed good response to relative isoprene levels in the soils of the Coconino National Forest and the surrounding air.

Competing Interests

The author declares that they have no competing interests.

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