Smart Gardening IoT Soil Sheets for Real-Time Nutrient Analysis

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Agriculture sector has been greatly influenced by the recent advances in electrical engineering. Nitrogen (N), from fertilizer, remains one of the largest inputs to surface and groundwater contamination, resulting in environmental and human health degradation. This paper explores the use of wireless potentiometry in field settings for in situ N monitoring. We report a disposable IoT gardening soil sheet capable of analyzing real-time soil nitrate concentration during leaching and irrigation events. The nitrate doped polypyrrole ion selective electrode (N-doped PPy ISE) sensor array sheet features a fault tolerant circuit design multiplexed to an oxidation and reduction potentiometer that can rapidly detect nitrate levels in soil leachates. Measurement data are transmitted via Waspnote ZB Pro SMA 5dBi radio, 6600mAh rechargeable battery, 7.4-volt solar panel, and a Meshlium ZigBee PRO access point to cloud server and mobile device. This paper investigates the gardening IoT sheets as a viable tool for in situ nitrate mapping, and to potentially help everyday home and commercial gardeners reduce excessive fertilizer application.

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Materials and Methods

Sensor sheets, prepared in-house, were printed onto a photopaper substrate with aqueous ink, which was formed using a combination of solvents. The ink is composed of well-dispersed silver nanoparticles.\(^*\)

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nanoparticles (10 nm) with a silver concentration of 20 wt%, viscosity of $\sim 9.5$ cP and a surface tension of $\sim 36$ mN m$^{-1}$ which met inkjet printer (MFC-J680DW) requirements. The nanoparticles were protected by a capping layer of poly (N-vinylpyrrolidone) (PVP). Pyrrole(98%), sodium nitrate (NaNO$_3$-), and PBS buffer solution were purchased from Sigma and were all of analytical grade. Pyrrole, being light sensitive, was refrigerated in the dark. Sensors were calibrated immediately prior to polymerization. Pyrrole is sensitive to ambient light with 1 M, 0.4 M and 0.1 M of NaNO$_3$. All solutions were prepared immediately prior to polymerization. Pyrrole is sensitive to ambient light with three separate pyrrole concentrations (1 M, 0.5 M, 0.1 M) mixed with various nitrate concentrations. Electropolymerization was conducted for duration of 5–20 minutes on the working electrodes. After each cycle, 100 $\mu$A were applied for duration of 5–20 minutes on the working electrodes. Each cycle, 100 $\mu$L were added to cell solution to measure sensor performance against nitrate on screen printed electrodes was performed electrochemically using a Princeton Applied Research Potentiostat-Galvanostat (Model 263A). A constant current of 100 $\mu$A were applied for duration of 5–20 minutes on the working electrodes. After each cycle, 100 $\mu$L were added to cell solution to measure sensor performance against nitrate concentrations. Electropolymerization was conducted with three separate pyrrole concentrations (1 M, 0.5 M, 0.1 M) mixed with 1 M, 0.4 M and 0.1 M of NaNO$_3$. All solutions were prepared immediately prior to polymerization. Pyrrole is sensitive to ambient light and atmospheric oxygen. Therefore PPY solution was refrigerated and not exposed to light.

Three soil samples (L1, L2, and L3) were collected from three 12m$^2$ garden beds at Florida International University’s Modesto-Ma启迪 Campus in Miami, Florida. Soils were identified via the USDA – NRCS’s Web Soil Survey as limestone-derived udorthents.

**Results and Discussion**

Electrodes were inkjet printed on a paper substrate. Working electrodes were modified for nitrate sensitivity by electropolymerization of pyrrole. As a result a thin film polymer membrane of N-doped pyrrole was formed capable of soil nitrate measurements. A prototype sheet that comprises of 8 electrodes in a 5 x 3 inch area was developed (Figs. 1a, 1b).

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**Electropolymerization.**—Polymerization of pyrrole doped with nitrate on screen printed electrodes was performed electrochemically using a Princeton Applied Research Potentiostat-Galvanostat (Model 263A). A constant current of 100 $\mu$A were applied for duration of 5–20 minutes on the working electrodes. After each cycle, 100 $\mu$L were added to cell solution to measure sensor performance against nitrate concentrations. Electropolymerization was conducted with three separate pyrrole concentrations (1 M, 0.5 M, 0.1 M) mixed with 1 M, 0.4 M and 0.1 M of NaNO$_3$. All solutions were prepared immediately prior to polymerization. Pyrrole is sensitive to ambient light and atmospheric oxygen. Therefore PPY solution was refrigerated and not exposed to light.

Three soil samples (L1, L2, and L3) were collected from three 12m$^2$ garden beds at Florida International University’s Modesto-Maidique Campus in Miami, Florida. Soils were identified via the USDA – NRCS’s Web Soil Survey as limestone-derived udorthents. They have a gravelly-loam texture, shallow to bedrock and poorly drained. Experimental plants were grown from seeds, twelve inches apart directly into garden bed soil, while controlled group were grown in 10 L pots of bare loamy soil with no added fertilizers or microbes. All groups were grown under 100% sun with irrigation, weed and pest control done as needed. The Shurflow water pump transferred fluids from the storage tank to the sprayer with an inlet pressure of 2.06 bar (30 psi) and an output flow rate of 4 gallons per minute, providing test beds with water through DIG drip irrigation.

**Potentiostat circuit.**—The input range for the potentiostat circuit (Fig. 2) depend on the values of R1, C1, R2, R3 and the source of input (BATT or USB), there by having a direct control over the input voltage of the potentiostat. For instance, a 5 volt input will yield a linear input range in the $-1$ to $+1$ V range. The circuit supports a power supply range of 3V to 32V. Range of output current sensing is depended on the negative voltage to resistor R5. An applied voltage of $-3.7$ V yields a range of $-150$ to $120$ $\mu$A (at $-5$ V range is $-210$ to 65 $\mu$A). Alternatively, current sensing range can also be tailored by introducing variable resistors at positions R5 and R6. Power dissipation of the circuit was calculated as the products of voltage and current of all the power sources. Total power dissipation at ideal state is 50 mW. The current drawn internally by op amp (X3) is $-18.9$ nA and the current drawn internally by op amp (X5) were found to be $-23.34$ nA.

**Figure 1.** a) Layout of the IoT enabled soil sensor system. b) Seedling pellets on the in-house fabricated nitrate sensor sheet.
layer. Electrodeposited electrode sheets were incubated at 70°C for 15 min for enhanced stability.

Figs. 3a, 3b shows circular grains around 50–200 nm in diameter for 5 and 10 minute (E5 and E10) long electrodeposition. At longer electrodeposition times, larger clusters were observed (Figs. 3c–3d). The non-uniformity and nonconductive nature of the larger clusters were found to hinder the ionic exchange during electrochemical measurements. Linearity and sensing range was found to be affected by the duration of electrodeposition. Electrodes deposited for 15 min (E15) were found to have a linear response in the 5 ppm to 90 ppm nitrate levels. Fig. 4a shows the differential pulse voltammetry (DPV) current response of sensors to varying nitrate levels. Nitrate sensitivity of sensors that were electrodeposited at varying times is depicted in Fig. 4b. As the films grew thicker, the slope of the nitrate response curve was found to be higher nearing a Nernstian response. Apparently from the sensor response, the electrodes that were electrodeposited for a duration of 15 minutes was found to have the best linear sensitivity as compared to the other electrodes. Apparently from the sensor response slopes, electrode E15 was found to be more than 110% sensitive to nitrate levels as compared to E5 and E10. However, electrode E20 was found to be the least sensitive among all the electrodes, despite having a larger area. We assume that the non-uniformity and nonconductive nature of the larger clusters are hindering the ionic exchange during electrochemical measurements.

Fig. 5 depicts the amount of leached nitrate as detected by the sensor. Three different soil types of limestone-derived udorthents with known carbon and nitrogen contents (Table I) were used for this

Table I. Nitrate leachate levels of three lime stone-derived udorthents soil samples during irrigation cycle (LOW: 5–40 ppm, MED: 40–100 ppm, HIGH: 100–300 ppm) as predicted by the IoT soil sensor network.

| Irrigation Cycle | Soil Sample | 1 | 2 | 3 | 4 | 5 | 6 |
|------------------|-------------|---|---|---|---|---|---|
| 1                | HIGH        | HIGH | MED | LOW | LOW | LOW |
| 2                | HIGH        | HIGH | MED | LOW | LOW | LOW |
| 3                | HIGH        | HIGH | MED | LOW | LOW | LOW |

Figure 2. Linear sweep potentiostat circuitry.

Figure 3. SEM micrographs of (a) cross section of the electropolymerized working electrodes. Electrodeposition of pyrrole for varying time durations (b) 5 (c) 10 (d) 15 (e) 20 minutes, at a constant current of 100 μA.
study. Sensors were installed one inch beneath the top of the soil beds. Readings were taken 600 seconds after irrigation allowing time for leachate to come in contact with the sensor. Soil samples, spiked with 100 mM nitrate solution, were used for leaching studies. Irrigation was applied at a fixed daily rate and the leachate samples from the bottom drain were analyzed for six consecutive irrigation cycles. The response of the sensor to varying irrigation cycles reflected the leaching rates. Leaching trends were found to be very similar for all three soil samples. Leaching was found to be highest for the first 2 irrigation cycles. This can be due to loosely bound nitrate. Depending
on the nitrate leach rate, the sensor response has been categorized into three; LOW: 5-40 ppm, MED: 40–100 ppm, HIGH: 100–300 ppm (Table 1). When the response of the sensor is HIGH for two consecutive times, an alert signal is sent out by an external micro-controller to the IoT network. However, stability of the sensor was found to be degrade after 3 consecutive tests. This can be due to the deterioration of the pyrrole surface from the mud particulates in the leachate.

Waspmote Agriculture Sensor Board Pro serves as the microcontroller and the IoT network. Data packets are transmitted via a Waspmote ZB Pro SMA 5dBi radio, 6600mAh rechargeable battery, 7.4-volt solar panel, and a Meshlium ZigBee PRO access point. Data obtained from individual sensors are directed to Meshlium access point and stored directly to the hard drive or sent to a cloud service. Meshlium is a Linux router which works as the gateway to the waspmote sensor network. Inserting a sim card onto the waspmote sim slot allows for data and commands to be transmitted to cellular devices. A ZigBee radio transmit data frames to the meshlium, which operates at 2.54 Ghz, using a transmission power of 50 mW and a line of sight 5dBi dipole antenna to cover a range of 7000 meters. Out of three soil samples tested, all of them showed HIGH leachate levels consecutively for two irrigation cycles. A leaching rate > 100 ppm of leaching for two consecutive cycles (Cycle 1 and 2). After Cycle 3, the leachate level was found to drop consistently. Similar trend was observed with samples 2 and 3. Hence, re-fertilization of the soil will be based on the crucial information from our soil sheet sensor.

Conclusions

We developed a IoT enabled soil sensor sheet sensor capable of electrochemically detecting nitrate leachates. The sensor sheet was inkjet printed on a paper substrate and modified via electrodeposition. Initial studies indicate that the analytical current response is linearly proportional to soil leachate nitrate content, however, the stability of the sensor was found to be degrade after 3 consecutive tests. This can be due to the deterioration of the pyrrole surface from the mud particulates in the leachate. Sensor deterioration can be due to various reasons, such as concentration of soil nutrients, pH, ionic composition, non-specific adsorption on the surface of the electrode, weather conditions, and soil microbial interaction. Currently, we are addressing the sensor abnormalities as observed from our experiments. The potential of our wireless sensing platform was demonstrated by detecting the soil leachate levels in three samples of limestone-derived udorthents with respect to irrigation cycles. Measurement data are transmitted via Waspmote ZB Pro SMA 5dBi radio and a Meshlium ZigBee PRO access point to cloud server and mobile device.

Figure 5. Nitrate leaching in limestone-derived udorthent soil samples.

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