Monitoring Food Spoilage Based on a Defect-Induced Multiwall Carbon Nanotube Sensor at Room Temperature: Preventing Food Waste

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ABSTRACT: We have developed an electronic nose based on carbon nanotubes (CNTs) synthesized by using a plasma-enhanced chemical vapor deposition, aiming to be a convenient monitoring device for food spoilage. The prepared CNTs showed a crystalline structure and smooth surface with a diameter of 11.3 nm and a length of ~10 \( \mu \)m. The Raman spectrum showed that the CNTs fabricated were multiwalled carbon nanotubes (MWCNTs). The characteristic graphite peak (G) observed at 1595 cm\(^{-1}\) in the Raman spectrum showed low intensity as compared to the defect peak (D) observed at 1330 cm\(^{-1}\), which referred to defect-induced points in CNTs. The CNTs were used to fabricate a sensor for ethylene gas produced by banana fruits for in situ measurements at room temperature. The sensor demonstrated good performance toward detecting the produced gas. The gas sensing signal was used as early indicators of the spoilage to help prevent food waste. The calibration curve was shown for the sensor responses evaluated at ripening days over 5 days. The sensor showed a response of 3.2% on the first day and increased to ~7.0% by the third day and then gradually decreased. This sensor is appropriate for detecting the spoilage of food because it shows a good sensing response to a low level of produced gas from a single banana. Insight into food spoilage status of a specific level of gas shows its potential to be applied for quality assurance of food. The sensor sensitivity toward ethylene produced by a banana was confirmed based on the sensor response toward chemical ethylene gas.

1. INTRODUCTION

Ethylene (C\(_2\)H\(_4\)) is an unsaturated hydrocarbon molecule, which is a plant hormone. During the blooming of flowers and ripening of fruits, ethylene gas is emitted, which is colorless and has a sweet smell.\(^1\) Ethylene is produced by most plants that use it as a hormone to stimulate growth, maturity, and other key stages of their life cycle. It affects the growth and development process, involving the ripeness and aging of fruits.\(^2,3\) The production of ethylene significantly affects the quality of the collected fruit. Its effect might be useful or harmful, depending on the ripeness stage.\(^4,5\) For example, bananas produce increasing amounts of ethylene when they are ripe and change color.\(^1\)

The commercial market strategies for products are to avoid exposure to ethylene and to reduce and control ethylene issues at the ripeness, collection, storage, transportation, and supply. The people moving the fruit around want to know how it goes and whether they need to take actions to keep ethylene low while it is being transported. To manage any type of long-term stored product, the community wants to be able to measure ethylene to determine if it is stagnant or whether it is ripening.

Fruit ripening in the distribution chains is generally controlled by observing the levels of ethylene and evaluating the quality of fruits at each stage of the journey to the market.\(^6,7\) Consequently, there is a need for better food management and less food waste. A sensor for detecting such stages is required, which can be used to monitor fruits and vegetables while they are being shipped and stored, helping reduce food waste.

Some recent literature has been focused on the ethylene sensor to detect the ripening of agriculture products to avoid food waste.\(^8,9,10,11\) At present, micro gas chromatographs with a preconcentrator,\(^9\) optical-based sensors,\(^10,11\) surface acoustic wave (SAW) sensors,\(^12\) electrochemical sensors,\(^13\) and chemoresistive sensors\(^14,15\) are applied to detect ethylene. Nevertheless, the target to reach a method of cost-effectiveness, simplicity, and high performance is still challenging. Fong et al.\(^8\) designed a new type of ethylene sensor based on carbon...
nanotubes (CNTs) but is operated with the Wacker oxidation mechanism. Instead of incorporating a metallic-like copper that binds directly to ethylene, they used a metallic catalyst of palladium that adds oxygen to ethylene during an oxidation process. The conductometric/chemiresistor sensor has the advantage of configuring a miniature sensing system, and CNTs or metallic nanoparticles have been used as sensors for chemiresistor-based sensors, which have good conductivity and intrinsic gas sensing property.16–18

In this work, for detecting ethylene with an efficient approach, we proposed a novel ethylene gas sensor based on multiwalled carbon nanotubes (MWCNTs) by using it in fruit ripeness monitoring. The proposed sensor enables us to monitor the fruits’ natural ripeness at different stages and with high accuracy. In the proposed sensor, we have used a process-ripening banana as a source of ethylene for a systematic investigation of the sensor performance toward the gas. Ethylene was released from the fruit sample and then introduced to a CNT gas sensor fabricated on Au electrodes. The output of the sensor was performed at a temperature of 30 °C and was estimated at various ripening days of the banana as well as at various chemical ethylene gas concentrations. The mechanism of gas sensing was explained in terms of the reaction of the gas with the CNT surface.

2. RESULTS AND DISCUSSIONS

2.1. Morphology and Structural Characterizations.

The as-prepared CNTs were characterized by field emission scanning electron microscopy (FESEM) with 15.0 kV and different magnifications, as shown in Figure 1. The FESEM image confirms the fabrication of CNTs on the SiO2 substrate. Curved and lengthy CNTs contacting with each other and forming a porous network were observed. The inset figure shows a higher magnification image of CNTs with a 100 nm scale, which can show that the diameter of CNTs is about 11.3 nm. This wall thickness of the CNTs might confirm the formation of MWCNTs. The morphology and microstructure of the materials affect the gas adsorption onto the material surface. Thus, the sensor performance including the sensitivity and response and recovery times is related to the adsorption behavior, which is controlled by the porosity of the nanostructure.

The Raman spectrum of the prepared CNTs is shown in Figure 2. The most known vibrations in the Raman spectrum of CNTs are G (graphitic) and D (disordered induced) bands.19 The bands are ascribed to stretching vibrations of C–C bonds of the pure carbon materials. The G band appears due to the in-plane bond stretching motion of pairs of sp2-hybridized carbon atoms and has E2g symmetry as well. However, the D band is a breathing mode of A1g symmetry, which appears due to the presence of disorder and does not exist in the perfect graphite.20 Thus, the Raman spectrum shown in Figure 2 confirms the formation of CNTs via the observation of its corresponding phonon modes. The Raman spectrum of CNTs shows G and G’ (2D) bands at 1595 and 2645 cm⁻¹, respectively. Also, a band at 1330 cm⁻¹ for the D band is seen. The G’ band is due to the various interactions of interlayers occurring at different depths within the CNTs. The intensity of the D band relative to the intensity of the G band is often used to measure the quality of CNTs and the defects present in carbon-based materials. For the current CNTs, the intensity ratio I_D/I_G ratio is ~2.13, which explains a high disorder and defects present in these CNTs. The intensity ratio of I_D/I_G is about 0.17, which indicates multilayers that build up the CNTs. It is known that MWCNTs have a very similar spectrum of single-walled carbon nanotubes (SWCNTs) but with the absence of radial breathing modes (RBMs) in the MWCNT and a significant dominance of the D band. The D band is expected to be more prominent in the MWCNT to a certain extent due to the multilayers and more chaos in the structure. The RBM peak completely disappears when we move to the double-wall tube and that the D and G’ bands become relatively larger when we add layers to the tube walls. Thus, the observed Raman spectrum shown in Figure 2 confirms the formation of multiwalled and defected CNTs.22

The measured surface area of the as-prepared MWCNTs is in the range of 10–500 m²/g.23 However, the theoretical external surface area of CNTs has been reported in the range of 50–1315 m²/g.24 The theoretical specific surface area (SSA) of MWCNTs was calculated in terms of the external diameter and the number of layers of CNTs by eq 1:

\[
\text{SSA(MWNT)} = \frac{1315 \, d_e}{n_d \times 0.68 \times \sum_{i=1}^{n_l} \overline{i}}
\]

Figure 1. FESEM image of the prepared CNTs.

Figure 2. Raman spectrum of the prepared CNTs. The inset is a scheme of MWCNT.
where \( d_e \) is the external diameter of CNT and \( n \) is the number of layers/shells composed by CNT. The specific surface area of the current MWCNTs calculated via the above equation was 154 m\(^2\)/g, where \( d_e \) is 11.3 nm, and \( n \) is \( \sim 14.0 \) layers based on the fact that the inner diameter of the first tube is 2.0 nm (as for SWCNT)\(^{23} \) and the inner shell distance, \( d_s \), is 0.34 nm.

### 2.2. Sensing Response of the MWCNT Sensor
Xiao et al.\(^{1} \) have reported a significant study of important ripeness restrictions, depending on ethylene production and measured firmness in bananas in the case of natural ripeness and 1-MCP-delayed and ethylene-induced ripening. They reported that ethylene production in natural ripening fruits increased drastically after day 15 of storage, reached the highest production on day 18, and then decreased. The hardness started to decrease on day 18 of storage, where the firmness gains the lowest value on the 23rd day as well. Thus, we have used this study as guidance for banana ripening and the behavior of ethylene production.

To test the sensor’s capabilities, we placed carbon nanotubes on gold electrodes deposited on a glass substrate. Figure 3 indicates the flow-on and the flow-off of air and in the air including gas on the fabricated sensor at room temperature (30 °C). To show the effect of airflow on the sensor output, the jar is emptied to make sure that the effect of airflow can be ignored. It is shown that the sensor output is almost stable when the air flows on the CNT sensor, as shown in Figure 3a. When the air is flowing to the chamber, there is a very slight change in the sensor output, which can be ignored compared to the signal coming from the gas produced by the banana, as shown in the sensor signal of Figure 3b. It shows that the sensor resistance increases quickly with gas exposure and then decreases upon the gas exhaust.

We have used this sensor to monitor the ethylene production in the banana. The ethylene production was measured over 5 days, allowing us to track the ethylene levels with banana ripeness. The gas sensor response of the current-synthesized CNTs is investigated in detail during these 5 days for the as-received banana from the market. Figure 4 shows the sensor signal over 5 days. The sensor responds to the produced ethylene as soon as ethylene adsorbs on the surface of CNTs. It reaches the saturation value within tens of seconds of exposure, and once the gas flow is stopped, the sensor quickly responds to the action. However, it takes a long time from this transient state to reach its original state. In this figure, we show the cyclic signal of the sensor on days 1 (as received), 3, 4, and 5 for the banana. To repeat this cycle, we have waited an interval period of 5 min (300 s) before starting the next cycle. This time is consumed in the recovery period, and then the sensor was exposed for 120 s so the flow of air can pass through the jar that contains the banana. After the day measurement was finished, the banana was kept out of the jar at an ambient atmosphere of 20 °C for the next day. The as-received banana has a whitish green color, as shown in Figure 4, which shows a low peak of the sensor signal. With the second and third day of the experiment, the color changes from green to yellow, which gives a higher sensor signal. It was observed that, on the fourth day, a dark color appears on the banana surface and increases by the fifth day, and again a low signal is detected compared to the third day.

The reversibility of the sensor signal upon exposure to the gas produced by the banana is shown over the experimental days in Figure 4. It is seen that the sensor is very sensitive for the reversible signal for each measurement when the gas is switched on/off. The signal is repeated several times, and there is no observed drift, although the experiment is carried out at a low temperature of 30 °C. Each cycle consists of 420 s, including the response and recovery periods.

It was found that there is a rapid increase in the concentration of ethylene on the third day of the experiment, and thereafter spots of a dark color start to coat the surface of the banana. These all are within 3 days, and then it becomes clear that the ethylene concentration decreases to the levels those observed for the second and the first day. The dependence of the response on ethylene production as a function of measurement days is given in Figure 5. First, it increased to the maximum response on the third day for \( \sim 7.0\% \) and then gradually decreased with a further ripening. On the first and fifth day, the calculated sensor response is about 3.2%. From Figure 5, it is shown that there is a gradual increase in ethylene that started during the first day (as-received sample) and continued until the third day and started to decrease when the dark brown color spread on the surface of the banana.

The sensor responds to the produced gas within a few seconds. However, the response and recovery time constants are characterized by the time required for the response to reach 90% of the equilibrium value in gas and air, respectively. They are important parameters to assess the performance of the sensing materials, depending on the time. Figure 6 demonstrates the definition of the response and recovery time constants for the first cycle of the as-received banana. The time constants depend on how the gas diffuses into the layer and how fast the gas concentration fills in the chamber.
current MWCNT sensor has short response−recovery times. As shown in the figure, the response time constant is \( \sim 75 \text{ s} \), and the recovery time constant is \( \sim 140 \text{ s} \). It is reasonable that the recovery time is longer than the response time due to the slow desorption of the gas at this low operating temperature. However, the response and recovery time constants are still reliable for fast monitoring.

To confirm that the sensor is sensitive to ethylene gas, the sensing signal of the sensor toward chemical ethylene gas was investigated. For carrying out this experiment, we have used diluted 100% ethylene gas with a purity of 99.999%. A 1000 mL solution of ethylene gas was transferred to a balloon (shown in the inset of Figure 7), which was washed/purged by zero air three times in advance. To dilute the gas to a low concentration, specific amounts of 0.2, 0.3, 0.5, 1.0, and 2.0 mL of ethylene gas were injected into the measurement jar at 1500 mL in volume under stirring. Thus, the new gas concentration inside the jar was diluted by a factor of \( \sim 1/1500 \). Gas amounts of 0.2, 0.3, 0.5, 1.0, and 2.0 mL of 100% ethylene gas are corresponding to 0.13, 0.2, 0.33, 0.66, and 1.3 \( \mu \text{L} \), respectively. The sensor signal was recorded at these gas concentrations, as shown in Figure 7. The sensor response increased from \( \sim 1.9 \) to 9.0 when the gas concentration was increased from \( \sim 0.13 \) up to \( \sim 1.3 \mu \text{L} \), respectively. This result obtained here for ethylene gas confirmed that the present sensor is very sensitive toward ethylene gas, which was produced during banana ripeness.

2.3. Sensing Mechanism of the MWCNT Sensor. Here, we propose a sensing mechanism that shows how the defect-
induced MWCNTs respond to ethylene gas. In the chemiresistor gas sensor of carbon nanotubes, the main gas sensing mechanism is the change of the charge carrier density induced in the charge transfer in the carbon nanotubes. This transfer causes a change in the resistance of the carbon nanotubes. Few mechanisms can cause charge transfer, such as physisorption of gas molecules, the dipole–dipole interaction between gas molecules and CNTs, and the contact resistance modulation between the CNT-CNT and the CNT-electrode. In physisorption-induced charge transport, ethylene molecules are trapped in the interstitial defects, in contact between CNTs, or the surface defects of MWCNTs, causing charge capture by ethylene molecules during absorption on the surface of CNTs. When the gas was turned off and the air was flowed, ethylene desorbs and leaves the charge back into the carbon nanotubes.

In the present experiment, the as-prepared MWCNT sensor is used at room temperature. As it is known, if the metal and semiconductor are connected to each other, the Fermi levels will equilibrate. Thus, the Fermi level of Au goes near the lowest unoccupied molecular orbital (LUMO) of the CNT.25,26 When ethylene flows to the CNTs, it is bound with CNTs through the interstitial defects, contacts between CNTs, or the surface defects of MWCNTs, causing the electron transport to decrease. Thus, the resistance of the CNT sensor increased, as shown in Figure 3b. As a result, the defect-induced MWCNT-based sensor responds to ethylene once it flows on its surface. It is stated that graphene interacted with CO, NO, and NO2 when it was in a defective phase.27 The study showed that the response of the graphene sensor was enhanced for this defective graphene layer. Hence, it is practical that the current defect-induced MWCNT-based sensor shows a reasonable response toward ethylene gas.

3. CONCLUSIONS

An e-nose based on MWCNTs was designed and fabricated to monitor ethylene production during the banana ripening. The banana ripening was the source for ethylene production, where all measurements were carried out for in situ conditions. The sensor was tested at a temperature of 30 °C. The sensor parameters such as sensor response, response time, recovery time, and reversibility were carefully analyzed over 5 days for the as-received banana. The sensor response was 3.2% on the first day of the as-received banana, then gradually increased to reach ~7.0% on the third day of ripening, and then decreased to 3.2% when the banana surface has dark spots on the fifth day. The sensor has shown a trusted response and has the advantages of an easy fabrication process, operating at room temperature, and low power consumption. This sensor has short response–recovery times and has reliability for fast monitoring for food ripening or spoilage. With this small size, simple fabrication, and ease of electrical measurement, the integration of this sensor into sensor array is not complicated and it can be used as a portable e-nose device.

4. MATERIALS AND METHODS

Figure 8 shows the procedures for the synthesis of CNTs and the gas sensor device. A mirror-polished SiO2/Si substrate was used in the current experiment. Before the growth, the substrate was washed with deionized water and then ethanol followed by drying in air. Afterward, ~24 nm of a Ni catalyst thin film was deposited on the substrate surface by DC-sputtering. Also, a Cr layer of ~5 nm was deposited between the Si substrate and the Ni catalyst. After these steps, the plasma-enhanced chemical vapor deposition (PECVD) process was achieved for growing CNTs. The PECVD process was performed for the growth of CNTs using a FirstNano (model 3000 EasyTube) system. The carbon source was ethylene gas, which was delivered through a flow meter system and a furnace that consisted of the substrate. The furnace was heated up to 850 °C, and as the furnace reached the temperature set point, the sample was ready for the growth of CNTs. The CNT growth was performed at 750 °C by passing the mixture of 2 SLM of ethylene gas and 1 SLM of N2 gas through a tubular reactor (in the furnace) in which the Ar gas was flowed to remove the air inside the chamber. Then, ethylene decomposes into carbon and hydrogen species. After 30 min of the reaction time, samples were slowly cooled inside the furnace. Finally, the furnace was cooled to room temperature in the presence of Ar gas, and the CNTs were grown on the substrate.

The morphology of CNTs was examined by a field emission scanning electron microscopy (FESEM) model JEOL JMS-7000 with high magnification. The Raman spectrum was conducted using a confocal Raman microscope (LabRAM HR800) connected to a multichannel charge-coupled detector (CCD). A laser (He–Ne) with a wavelength of 633 nm and a 20 mW output power was used as a source of excitation. The Raman spectrum was measured at ambient temperature in a backscattering configuration.

The gas sensor was fabricated using two gold electrodes deposited in advance by DC-sputtering on a glass substrate.
with a gap of ∼200 μm. The CNT powder was lined in the gap by using a solution mixture in ethanol. The gas sensor was dried at 60 °C for 30 min in ambient air before inserting it into the testing chamber. All the sensing characterizations were then tested at an operating temperature of 30 °C. The sensing measurements were carried out in a gas sensor characterization instrument (GSM-6000A model) system attached to a temperature-controlled Linkam chamber (Linkam HFS600EPB4 Probe Thermal Stage), as shown in Figure 9.

For investigating the sensor performance, the resistance was attained for the baseline in air. The sensor then was exposed to the gas for 120 s and then was recovered for 300 s. It was examined upon exposure to varying cycles of gas and dry air. The system (including jar) shown in the figure is designed to have this investigation under natural conditions. A banana was received from the market and kept in a closed jar before measuring for 300 s. The produced ethylene was generated by passing the dry air via a jar containing the banana placed in a 20 °C ambient atmosphere and then passed to the sensor chamber at a rate of 200 mL/min. The gas sensing setup and the testing platform are demonstrated in Figure 9.

The electrical measurement was conducted using data acquisition (Keithley 2010, Kickstart 2 software). The response of the sensor is calculated using eq 2:

$$S\% = \frac{R_g - R_a}{R_a} \times 100$$

where $R_a$ and $R_g$ are the resistance in air and air containing gas, respectively. The experiments were carried out at room temperature and under in situ conditions.

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**Notes**

The authors declare no competing financial interest.

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