ORIGINAL INVESTIGATION

Detection of Secondhand Cigarette Smoke via Nicotine Using Conductive Polymer Films

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ABSTRACT

Introduction: The 2006 U.S. Surgeon General's Report found that there is no safe level of exposure to secondhand smoke (SHS). Many smokers attempt to protect others from exposure to SHS; however, it is difficult to assess effectiveness of these behavior changes. There is a need for personal monitoring devices that provide real-time SHS exposure data; at present, there is no device that measures ambient nicotine levels in real time. The development of such a sensor is the objective of this research.

Methods: A nicotine sensing film comprising the conductive polymer polyaniline was linked with a reporting layer, recording changes in chemiresistance due to adsorption of nicotine. Experiments were carried out in a microprocessor-controlled smoking chamber using sidestream smoke from standard reference cigarettes; up to 10 cigarettes were smoked simultaneously. The exposure chamber was calibrated for total suspended particle, carbon monoxide, and nicotine concentrations.

Results: We found significant *real-time* increases in the resistance of films upon exposure to SHS. The sensors were shown to be sensitive to the number of cigarettes consumed and ambient nicotine and demonstrated reasonable recovery between measurements. The sensors have sufficient sensitivity to detect off-gassing of nicotine or "thirdhand smoke."

Conclusions: A sensing element has been developed that can reliably detect secondhand and thirdhand tobacco smoke in real time through the adsorption of ambient nicotine vapor. The device was calibrated to the number of smoked cigarettes and to nicotine concentration. Development of integrated personal sensors to record exposure to SHS using this technology is currently underway.

INTRODUCTION

There is no safe level of secondhand smoke (SHS) exposure, with SHS causing increased risks of cancer, cardiovascular disease, and childhood illness (U.S. Department of Health and Human Services, 2006). While many smokers believe they are taking sufficient steps to eliminate SHS exposure and harm by smoking away from nonsmokers (e.g., in other rooms, other floors, in front of exhaust fans, out of windows, on balconies/porches), they do not realize that they continue to expose their families. Parents, in particular, desire to protect their vulnerable children; however, there is a lack of accurate measurements of children's exposure. Urine, saliva, or serum cotinine levels are specific to nicotine exposure (tobacco, tobacco smoke, or medicinal nicotine), but there is significant variability in cotinine levels across individuals from similar exposure due to differences in individual metabolism (see review in Avila-Tang et al., 2012). Therefore, a detectable level indicates exposure but is difficult to quantify that exposure with precision, and it is impossible to detect *where or when* that exposure occurred

within the preceding several days. In-home room-level testing for tobacco smoke exposure has been conducted using passive or active air sampling procedures; however, the devices involved tend to be costly (e.g., particulate matter pumps with a cost of ~\$3000 per unit) or require sending samples to a chemistry lab for assay of tobacco-specific components via gas chromatography and mass spectrometry (Apelberg et al., 2012). Unfortunately, particulate matter detection may also be affected by other sources including cooking with solid fuels, burning candles or incense, or outdoor air pollution (Fernandez et al., 2009; Klepeis, Ott, & Switzer, 2007; Lopez et al., 2012). Monitoring of a tobacco-specific vapor such as nicotine currently suffers from the lack of a real-time measurement (Apelberg et al., 2012); ambient nicotine measurements are typically averaged over a prolonged sampling period of days to weeks given low effective sampling rates (Hammond & Leaderer, 1987). Thus, methods for detecting "how well" a smoker is protecting his/her family are highly problematic.

Previous research efforts have attempted to use feedback on child's cotinine levels as triggers for parental smoking behavior

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change, with largely disappointing results (Wakefield et al., 2002; Wilson, Farber, Knowles, & Lavori, 2011) excepting modest effects from a study within a population of children with asthma (Wilson et al., 2001). Studies using direct measures of in-home concentrations of ambient nicotine (Emmons et al., 2001) or PM_{2.5} have shown better success (Wilson, Ritchie, et al., 2013; Wilson, Semple, et al., 2012), suggesting that the specificity of the information regarding smoke exposure from within the home may be more persuasive. The ability to provide real-time feedback in response to specific smoking situations may be even more persuasive to change the behaviors of smokers; however, the possibility of such feedback has previously not been possible given technological limitations. A reliable sensing element is the first step in developing a personal monitoring device for SHS, with the ultimate goal of developing a small, affordable, and wearable device that records cigarette smoke exposure in *real time*, allowing the direct connection of SHS exposure to a smoking event.

Conductive polymers such as polyaniline (PANi) are of great interest as components of electrochemical devices (Lai, Tang, et al., 2011; Lai, Zhang, Li, & Gao, 2011; Zhang, Kong, Wang, Luo, & Kang, 2009) and as agents for a variety of sensing applications (Gopalan, Lee, Ragupathy, Lee, & Lee, 2009; Srivastava, Kumar, Singh, Singh, & Vijay, 2011; Tran, Nguyen, Nguyen, Do, & Nguyen, 2011). As a sensor for nicotine, the key element of doped, electrically conductive PANi is the presence of protonated nitrogen atoms that give up a proton to nicotine, a base, which is adsorbed to the film from the nascent vapor. Thin film materials can optimize the density and availability of protonated receptor sites, minimize the diffusion distance necessary for the nicotine to travel during binding events, and increase the responsivity of the sensor when the reporting electrode lies beneath the film, as in this research. The various techniques for creating these thin films include electropolymerization (Waltman & Bargon, 1986), spin casting (Psuja, 2007; Yokota, Kitaoka, & Wariishi, 2007), and laser deposition (Chrissey et al., 2003; Frycek et al., 2006). Measurement using conductive polymer films can be performed either by coating the surface of an electrode with the doped polymer and measuring electrical changes with reference to a redox electrode or by making a true planar chemiresistive structure. The latter has advantages, in that it can be used with a variety of conductive polymers, may be designed to create a higher value of resistance, and has the potential for rapid measurements with small, personal-sized devices. In this article, we describe the development of a planar chemiresistive sensor that reports on exposure to SHS from ambient nicotine concentrations in *real time*. This is an important distinction from active or passive time averaging of ambient nicotine or nonspecific PM_{2.5} measurements, in that it allows for demonstration of changes in air nicotine from a specific smoking event.

METHODS

Materials

Polyaniline (purchased from Polysciences, Inc.) was used as the undoped emeraldine base, with a molecular weight of 15,000 and a conductivity of 10^{-10} S/cm. Formic acid (>98%; purchased from EMD Chemicals) was used to dissolve the polyaniline prior to spin casting and acts as a primary dopant.

Secondary doping using 1.0M aqueous HCl (purchased from Fisher Scientific) increased the sensitivity of the films. For bench laboratory studies, 99% nicotine was used (purchased from Alfa-Aesar). All reagents were used as received without any further treatment. The cigarettes used in the smoking chamber were filtered University of Kentucky 3RF4 reference cigarettes, containing ~0.8 mg of nicotine (Tobacco & Health Research Institute).

Fabrication of Conductive Sensors

The sensing assembly consists of the PANi nicotine-sensing film and an electronic reporting layer. The reporting layer is an electrically conductive electrode patterned into an interdigitated grid with 40 μ m fingers and 20 μ m spacing, produced on prime grade silicon wafers with a 5000-Å oxide layer. The chromium layer was patterned by photolithography and subsequently wet etched to produce the final electrodes with a total area of 376 mm², following vapor deposition of 200 Å of chromium and a 1000 Å overlay of nickel.

Preparation of PANi Solutions and Films

The sensing concept is based on the production of a conductive film that is sensitive to nicotine adsorption, using spin casting of PANi from solution for preparing films. The spin casting solution was produced from formic acid as a 1% (by weight) polymer solution.

An aliquot of 0.5 ml of the PANi polymer solution was dropped onto the silicon reporting layer (described above) and allowed to spread for 20 s. The spin coater was then brought up to 4000 rpm for 30 s. This resulted in deposition of films with a typical thickness of approximately 100 nm. Because the pK_a of formic acid is 3.77 (the amine and imine nitrogen atoms have different pK_a values), PANi in this solution is 50% protonated. To complete the protonation process and increase the sensitivity of the film, secondary protonation in 1.0M HCl was employed by dip coating for 30 s. After this treatment, background (washed) resistance values were measured, and morphology and roughness were investigated by atomic force microscopy using a Pacific Nanotechnology Nano-1 microscope in close contact mode.

Teague Smoking System/Laboratory Exposure System

Sensors were tested using two different controlled environments with two different goals: a bench laboratory test measured sensitivity to nicotine vapor directly and a smoking machine measurement tested the sensitivity to nicotine from SHS as a function of the number of cigarettes smoked in the machine. In both experimental environments, the change in the resistance of the sensor was measured using a multimeter connected to a laboratory computer.

The laboratory sample system (lab chamber) consisted of a small nylon box, containing spring-mounted electrodes and a small (~3 cm³) well filled via a syringe through a septum. The sensor assembly was placed on the electrodes above the well and a nylon cap attached using a torque wrench to ensure reproducible pressure of the sensor against the spring-mounted electrodes. Nicotine (1 mL) was injected into the well and the response of the sensor recorded. To follow the recovery of the sensor after exposure to nicotine, dry nitrogen was passed

through the well to evaporate the nicotine. Nicotine was injected at different concentrations and resistance recorded as a function of time at 22 °C. The vapor-phase concentration of nicotine was varied by making liquid mixtures of nicotine in chloroform. Rault's Law provides that the vapor pressure of each component is determined by the mole fraction in the liquid phase. Our polymer sensor is insensitive to chloroform vapor.

The smoking machine experiments were carried out in a Teague Enterprises TE-10 smoking system equipped with two exposure chambers (Teague Enterprises). The TE-10 smoking system incorporates a microprocessor-controlled smoking machine that produces both mainstream and sidestream smoke, or sidestream smoke alone, in conjunction with mixing, aging, and exposure chambers. Smoke is drawn from the combustion chamber into a mixing chamber and then into exposure chambers. Cigarettes are loaded into a wheel, lit, puffed, and ejected; the Federal Trade Commission method of puffing is used, with a 2 s puff, once per minute, smoked for 8 min each. One to 10 cigarettes may be smoked simultaneously. Expended cigarettes are automatically ejected and extinguished in a water bath. Valves control airflow volume during smoke generation and exposure conditions. Valves also permit purging of smoke from within the chambers into dedicated ducting that conducts all smoke outside the building. All measurements using the Teague Enterprises system were made with polymer sensors in an exposure chamber, using calibrated operational parameters for total suspended particle, carbon monoxide, and nicotine concentrations by selected mixing valve settings.

Sensor Response

Measurement of the sensor response to nicotine is accomplished through changes in resistance as a function of time. The resistance, R , of the polymer sensor was measured using a Keithley Model 2100 6 ½ Digit Multimeter. During the measurement, constant current of 1 mA was applied and the voltage through the film recorded, providing a resistance value via Ohm's law. Total dissipated power within the sensor was less than 0.5 mW. Sensitivity analysis found that four point measurements were unnecessary, thus all of the reported data were obtained using two contacts. Data were taken at a rate of 1 Hz over as long as 9 hr but typically over considerably shorter times. The background resistance background was measured prior to each exposure (typically 600 Ω); data are reported as normalized resistance, referenced to the initial, out-of-chamber background value.

The films were exposed to a range of analyte concentrations that ensured a challenge to the adsorption process. The results indicate that both the change in the resistance value and the rate of change in the resistance are proportional to the quantity and identity of the analyte adsorbed.

RESULTS

Sensor Morphology

The morphology of the film surface was investigated by atomic force microscopy of films produced on both silicon oxide and glass under the coating conditions described above. The undoped film (prior to treatment with HCl) is rougher than the

doped material and more irregular with surface defects. The doped film is smoother, and the minimal occurrence of surface defects provides an ideal material for adsorption of the target nicotine molecule from the vapor phase (data not shown).

Changes in Resistance in Response to Adsorbed Nicotine

Sensor functionality depends upon detecting differences in electrical resistance as a function of the adsorption of nicotine onto the sensor chip. We tested numerous films using pure nicotine in the small lab-built chamber and cigarette exposure as measured in the Teague smoking system. Data and figures presented here are typical of these observations.

Lab Chamber Results

Injection of nicotine into the sample well evoked an immediate rise in the measured resistance. The theoretical values of nicotine concentration were confirmed experimentally by use of a photoionization detector that was sensitive to nicotine but not chloroform. (RAE, 2012) The results of these preliminary studies are shown in Figure 1, where the initial slope upon injection of the sample into the chamber is plotted against nicotine concentration. Figure 1 also includes a typical plot of the signal for pure nicotine vapor as a function of time, postinjection.

The data in Figure 1 provide a calibration of the rate of relative resistance change as a function of parts per billion (ppb) of nicotine in air. The measurements shown were reproducible to within 5%. The data are fit via linear regression with a slope of $1.90 \times 10^{-6} \Omega_{\text{rel}} \text{ s}^{-1} \text{ ppb}^{-1}$ and a correlation coefficient of 0.99. The slopes are a preferred measure of the concentration; use of absolute resistance changes is problematic since the sensor resistance will continually increase if nicotine remains present in the ambient atmosphere. This calibration is intended for use in reporting sensor data in terms of ppb of nicotine rather than as a function of cigarette exposure.

Teague Smoking Machine Results

Figure 2 presents the entire response curve over time in response to smoking a single cigarette in the Teague system. During the smoking process, sidestream smoke was fed into the exposure chamber and the resistance increased as long as the smoking continued, indicating continued adsorption of nicotine into the film. The signal stopped increasing as the cigarette was extinguished and decreased slowly because air entering into the exposure chamber from the smoking system contained no additional nicotine. After approximately 6 min, the chamber was purged with 100% fresh (room) air and the sensor resistance dropped to a level approximately 20% above the chamber background. The initial slope of the signal was determined to be $8.73 \times 10^{-4} \Omega_{\text{rel}} \text{ s}^{-1}$, indicating, from our calibration curve in Figure 1, a nicotine level of 450 ppb for this exposure. The system calibration at the inflow/outflow settings of the exposure chamber provides that the dynamic nicotine concentration in this situation from the cigarette smoke alone is 0.5 ppb. Note that the background reading of the sensor (the resistance at the zero time point in the smoking apparatus) immediately increases as the film is placed into the exposure chamber, indicating a background level of nicotine before engaging the smoking apparatus and hence a higher total concentration.

Detection of secondhand cigarette smoke

Figure 1. Response of the sensor to vapor-phase nicotine from liquid nicotine at 22 °C. Plot of signal as a function of time for pure nicotine vapor (inset).

Figure 2. Response of the sensor, in terms of relative resistance, generated from a single cigarette in the Teague system. Note that the sensor detects nicotine adsorbed on the walls in the chamber from previous experiments, so-called thirdhand smoke.

Figure 3 demonstrates a set of sequential exposures in the Teague system, using varied numbers of simultaneously smoked cigarettes, each followed by a brief fresh air blowout. Cigarettes were smoked over a period of 8 min (in a manual lighting mode), during which sidestream smoke was mixed with

an equal volume of fresh air and fed into the exposure chamber. Following the extinguishing of the cigarette, fresh air was blown into the exposure chamber for a period of 6 min (purge). The decrease in resistance during the fresh-air phase shows that nicotine is desorbed from the sensor, restoring the resistance to a

Figure 3. Response of the sensor, in terms of relative resistance, generated from cigarette smoking in the Teague system. The plot shows the response to different numbers of cigarettes smoked simultaneously. Clean, smoke-free air was passed through the chamber between successive experiments.

smaller value. The slopes of the rising signals are also related to the number of cigarettes simultaneously consumed. The system provides nominal dynamic concentrations of 0.75 ppb and 1.11 ppb of nicotine from sidestream smoke generated by two and three cigarettes, respectively. The initial slopes for the three different smoking levels were $9.62 \times 10^{-4} \Omega_{\text{rel}} \text{ s}^{-1}$ (510 ppb), $1.63 \times 10^{-3} \Omega_{\text{rel}} \text{ s}^{-1}$ (1030 ppb), and $1.72 \times 10^{-3} \Omega_{\text{rel}} \text{ s}^{-1}$ (1,100 ppb), respectively, during consumption of one, two, and three cigarettes. Note the agreement between the slopes in Figures 2 and 3 for a single cigarette and the measured increases in nicotine exposure for multiple cigarette exposure. Sensor fatigue caused by the insufficient 'off time' to remove nicotine from the sensor was observed for the final cycle shown in the figure.

Finally, Figure 4 contains the results from several successive runs in which 10 cigarettes (nominally providing 3.16 ppb of nicotine) were simultaneously smoked in order to test the response and recovery in a heavy smoking situation. The sensor background resistance was measured in ambient room air prior to insertion in the exposure chamber. After a 6-min delay (following cigarette extinguishment), a new burn was begun, followed by two additional 10-cigarette exposures. The first 10-cigarette burn resulted in a steep increase with a slope of $1.91 \times 10^{-3} \Omega_{\text{rel}} \text{ s}^{-1}$ (1,000 ppb), less than expected based on the previous figures but reflecting a saturation of easily reached binding sites near the surface of the sensor.

DISCUSSION

This series of experiments demonstrates that a novel sensor based on a polyaniline film can detect ambient nicotine levels in real time, within two different experimental environments, across a

wide range of nicotine and sidestream smoke concentrations. This is a leap forward in SHS exposure detection technology as detection of any level of ambient nicotine is specific to tobacco smoke having been present within that space. Previous research has demonstrated that exposure to ambient nicotine is correlated with exposure to the harmful components of tobacco smoke (see [Apelberg, 2012](#) for review). The sensor/chip assembly is particularly innovative, in that it does not require any disassembly of the device or transport to a laboratory for subsequent analysis; the sensor needs only to be linked to a computer to retrieve the data and computationally interpret the normalized relative changes in resistance as nicotine exposure in ppb. This information can then be provided to the end user. Such information could be used to confirm adherence to smoking bans such as in rented motor vehicles, hotel rooms, apartment buildings, restaurants, or bars. Personal use, as noted above, can provide feedback to smokers regarding how well they are achieving true smoke-free environments for their nonsmoking family members.

Notably, as illustrated in each of the smoking system experiments and conspicuously absent in the lab chamber experiments, the background reading of the sensor, or the resistance at the zero time point in the smoking apparatus, immediately increases by 20% as the film is placed into the exposure chamber, indicating a background level of nicotine before engaging the smoking apparatus. Using the experiment illustrated in Figure 2 as an example, prior to our use for the experiment, the smoking chamber had been in constant use for 8 hr and the walls were not cleaned. This suggests that the sensor is capable of measuring nicotine outgassing from the enamel covered metal surface of the chamber's interior walls. This tobacco smoke contamination of rooms and automobiles, or "thirdhand smoke," has been described ([Sleiman et al., 2010](#)) and is currently the topic of a new area of research.

Figure 4. Response of the sensor, in terms of relative resistance, generated from repetitive smoking of 10 cigarettes in the Teague system. The experiment shows that the sensor recovers even in this heavy smoking regime. Note that the sensor again detects nicotine adsorbed on the wall in the chamber from previous experiments.

Limitations

This new technology shows substantial promise for quantification of ambient nicotine, and the same sensor can be repeatedly used; however, the baseline resistance of the sensor does rise with repeated exposures if the sensor is not in a fresh-air environment, allowing nicotine to desorb from the sensor. This can be seen in Figure 3, where the fresh-air phase of the repetitive experiment was not sufficient to bring the sensor back to the original baseline. However, the final exposure cycle shown in Figure 3, with a longer smoke-free period, indicates that a return approximately to the original baseline is possible. Indeed, a resistance measurement made several hours after completing the experiments that are shown in the figure resulted in a value nearly equal to the initial resistance (data not shown). With these repetitive exposures within the smoking chamber, we note simply that the first, single cigarette increases the signal by ~60% and a second consecutive single cigarette furthers the increase by 32%. The next sample, involving two cigarettes, resulted in a 40% signal increase, and the final sample of three cigarettes resulted in a 42% increase in resistance. These differences in resistance measurements with high-intensity exposures are also noted in Figure 4, where the first cigarette burn (of 10 cigarettes) resulted in substantially lower resistance than expected (based on the previous figures), reflecting a saturation of easily reached binding sites near the surface of the sensor. Figure 4 also shows that the absence of significant recovery time following each 10-cigarette exposure decreases the absolute increase in signal (although the third and fourth exposures provide similar increases), with measured changes in resistance of 110%, 25%, 15%, and 9% with these repeated high-intensity exposures. The intention of this particular experiment was to saturate the sensor, exposing it to levels of tobacco smoke that would unlikely be encountered if used as

a personal sensing device (given air flow) or even if used as a room-level monitor, given the unlikelihood of such intense exposure within such a small space. The repeated 10-cigarette exposure within the 1 m³ volume of the smoking chamber would rival that of the smokiest bar with no ventilation at all. Most importantly, this particular experiment indicates that the film is sensitive to its environment, even if the ambient atmosphere has a remarkably heavy concentration of cigarette smoke. The repeated intense exposure/saturation issues will be addressed in subsequent refinement of the device, but may be addressable with computer algorithms to interpret resistance slopes by time.

Impact

The preceding results and discussion primarily focus on changes in the resistance as a function of sensor exposure, but we have ascertained and quantified (Figure 1) the rate of change (slope) of the resistance with both nicotine concentration (in the laboratory chamber) and cigarette exposure (in the smoking system). This dual calibration allows two ways to present exposure data. While in scientific studies one prefers to use nicotine concentrations in ppb, the calibration of detector response to the number of cigarettes consumed in the smoking apparatus provides a useful means of relating SHS exposure to smokers and nonsmokers. Relating SHS exposure of a child (or other nonsmoker) to “the equivalent of xx cigarettes” provides a more striking and readily assimilated message. We hypothesize that such a message would have maximum impact, similar to the ways in which delivering the PM_{2.5} levels worked in the cited REFRESH study (Wilson, Ritchie, et al., 2013, Wilson, Semple, et al., 2012). We should also be able to correlate our electronic measurements to others currently in use, such as the real-time PM_{2.5} studies (Semple, Apsley, & MacCalman, 2012), by a cross calibration.

Future

While the size of the polymer films used in the studies depicted above was approximately 1 in.², the size of the sensing element may be reduced to one-half to one-third without compromising functionality. This allows for smaller and less obtrusive personal sensors. We have explored the application of small, restricted instruction set computer processors to handle data input, storage, and output and are currently refining appropriate candidate electronics. We have developed prototype devices that are powered by a small, long-life (rechargeable) battery that can operate the sensor for several days if data are stored internally and downloaded at the conclusion of a study. The cost of the reusable sensing chip (polymer film plus reporting chip) is currently of the order of USD 30. We found that the polymer may be stripped and recoated for as many as 20 cycles, at which time the chip was still functional and testing halted. Recoating the interdigitated electrodes with fresh polymer for reuse costs pennies for the raw materials. The computer processor is the most expensive aspect of a personal sensor. The current cost of such devices ranges from USD 25–300, depending upon the vendor and required capabilities, which depend on the desired use of the device.

CONCLUSIONS

We have demonstrated that a chemiresistor based on a polyaniline film and interdigitated electrode can provide a *real-time* indication of exposure to secondhand cigarette smoke. The polyaniline film was shown to be sensitive to the number of cigarettes consumed, demonstrated reasonable recovery between exposures, and was functional in the presence of a wide range of exposures to nicotine and tobacco smoke from simulated light-smoking to heavy-smoking environments. The detection of nicotine outgassing or “thirdhand smoke” was also demonstrated to be feasible using the polymer film assembly. These results provide the basis for the development of wearable personal exposure sensors. These exposure sensors can be used to detect personal exposures and to detect nicotine in spaces such as rooms or cars, providing real-time indications of cigarette usage in those environments.

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DECLARATION OF INTERESTS

Dartmouth College has a patent pending for this technology.

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