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# ATMOSPHERIC ENVIRONMENT

## Tobacco smoke aging in the presence of ozone: A room-sized chamber study

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#### ABSTRACT

Exposure to tobacco pollutants that linger indoors after smoking has taken place (thirdhand smoke, THS) can occur over extended periods and is modulated by chemical processes involving atmospheric reactive species. This study investigates the role of ozone and indoor surfaces in chemical transformations of tobacco smoke residues. Gas and particle constituents of secondhand smoke (SHS) as well as sorbed SHS on chamber internal walls and model materials (cotton, paper, and gypsum wallboard) were characterized during aging. After smoldering 10 cigarettes in a 24-m<sup>3</sup> room size chamber, gas-phase nicotine was rapidly removed by sorption to chamber surfaces, and subsequently re-emitted during ventilation with clean air to a level of  $\sim 10\%$  that during the smoking phase. During chamber ventilation in the presence of ozone (180 ppb), ozone decayed at a rate of 5.6  $h^{-1}$  and coincided with a factor of 5 less nicotine sorbed to wallboard. In the presence of ozone, no gas phase nicotine was detected as a result of re-emission, and higher concentrations of nicotine oxidation products were observed than when ventilation was performed with ozone-free air. Analysis of the model surfaces showed that heterogeneous nicotine-ozone reaction was faster on paper than cotton, and both were faster than on wallboard. However, wallboard played a dominant role in ozone-initiated reaction in the chamber due to its large total geometric surface area and sink potential compared to the other substrates. This study is the first to show in a room-sized environmental chamber that the heterogeneous ozone chemistry of sorbed nicotine generates THS constituents of concern, as observed previously in bench-top studies. In addition to the main oxidation products (cotinine, myosmine and N-methyl formamide), nicotine-1-oxide was detected for the first time.

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#### 1. Introduction

Over the past decade significant progress has been made around the world in controlling involuntary exposures of non smokers to secondhand tobacco smoke (SHS). In some countries, expanding workplace restrictions now protect a majority of working adults, but homes remain the most important exposure setting for children (US DHHS, 2006). SHS includes thousands of substances, of which more than 60 are known or suspected carcinogens and several are strong irritants (Jenkins et al., 2000; Hoffmann et al., 2001). Recently, the term *thirdhand smoke* (THS) has been adopted to describe the residues of tobacco smoke that remain adsorbed to indoor surfaces after the smoke clears, react to produce secondary pollutants and/or are re-emitted back to indoor air over extended periods of time (Matt et al., 2004; Winickoff et al., 2009; Fortmann et al., 2010; Sleiman et al., 2010a). While inhalation of SHS is a major pathway of exposure indoors, long after smoking has ceased, contact with toxicants in THS can also take place by other routes such as dermal uptake, hand-to-mouth transfer and inhalation of sorbed species that are re-emitted to the gas phase. These pathways of exposure are especially relevant in homes with toddlers because young children have more contact with contaminated surfaces such as carpet and furniture.

Physicochemical transformations of SHS and THS components that occur in indoor environments after smoking takes place – aging – impact both short and long term exposure patterns of nonsmokers because aging can generate secondary pollutants over periods ranging from a few hours to several months. Ozone and other atmospheric oxidants may produce secondary pollutants by reaction with sorbed SHS and THS (Destaillats et al., 2006; Petrick et al., 2010). Moreover, nicotine re-emitted from surfaces during



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aging can react with ozone in the gas phase to produce ultrafine particles that contain oxidized byproducts with high asthma hazard indices (Petrick et al., 2010; Sleiman et al., 2010a). Thus, a significant fraction of the widespread respiratory symptoms associated with passive smoking may be associated with irritating gas-phase oxidation products and secondary aerosol particles produced by the reaction of tobacco smoke components with indoor ozone.

Despite its high reactivity, the atmospheric lifetime of ozone is long enough to allow for its transport to the indoor environment, where it reacts at rates that are often higher than typical ventilation removal rates. Weschler (2000) reviewed available information on simultaneous indoor—outdoor (I/O) ozone measurements, finding that in most cases I/O ratios were between 0.2 and 0.7. Ozone may also be generated indoors in large quantities from devices marketed as "air purifiers" (Boeniger, 1995; Hubbard et al., 2005). Such devices are commonly used to remove odors related to secondhand tobacco smoke, despite the growing body of evidence showing that they generate products that are more toxic and/or irritating than their precursors (Phillips and Jakober, 2006; Waring et al., 2008).

While some homogenous gas phase reactions are important indoors, the available experimental evidence indicates that the most important decay processes for indoor ozone involve surface reactions that are major sources of secondary pollutants (Morrison and Nazaroff, 2002; Aoki and Tanabe, 2007; Zhao et al., 2007) and sorbed chemicals (Fan et al., 2003; Fick et al., 2005; Singer et al., 2006; Wang and Morrison, 2006; Coleman et al., 2008; Petrick and Dubowski, 2009). The characterization of ozone-initiated chemical reactions on indoor surfaces will contribute to understanding their link to health effects and provide important insights into design of effective engineering solutions that eliminate or reduce exposure to toxic chemicals generated by these reactions (Weschler, 2004; Weschler and Wells, 2004; Weschler, 2006; Morrison, 2008).

Our previous work showed that nicotine sorption is affected by substrates (Petrick et al., 2010) and that it can be substantially depleted by its reaction with ozone over timescales that are competitive with desorption and re-emission into the gas phase (Destaillats et al., 2006; Petrick et al., 2011). As a consequence, reemitted nicotine gas phase levels decreased by 1-2 orders of magnitude compared with similar conditions in the absence of ozone, and stable oxidation products such as *N*-methylformamide, cotinine, nicotinaldehyde and myosmine were emitted to the gas phase. This study investigates whether similar processes occur when SHS ages in a scaled-up environmental chamber that contains several indoor materials. The present study also tracks a wider range of SHS constituents in the gas phase, on airborne particles, and on the model surfaces gypsum wallboard, cotton cloth, and cellulose paper. The effect of substrate type on nicotine surface oxidative kinetics is also examined.

#### 2. Experimental

Chamber smoke aged in clean air (RH = 44–63%, T = 20-27 °C, no ozone) was compared with smoke that aged in the presence of ozone under otherwise identical conditions. After smoke generation the following types of samples were collected and analyzed: a) gas (and particulate) phase compounds on Tenax TA in sorbent tubes, b) semi-volatile organic compounds (SVOCs) on XAD-coated denuders, c) particles on filters and d) SVOCs sorbed to model surfaces (cotton cloth, wallboard, and paper).

#### 2.1. Chamber parameters

The walls and ceiling of the  $24 \text{ m}^3$  smoking chamber were covered with  $42 \text{ m}^2$  of painted gypsum wallboard, while the floor

(10 m<sup>2</sup>) was covered with vinyl tiles; all surfaces had previously been exposed to tobacco smoke over a period > 10 years. An axial fan (52 cm diameter) was operated inside the chamber to ensure rapid mixing. Filter cigarettes from a leading US brand were purchased from local California retailers and used for SHS generation. At experimental onset 10 cigarettes were smoldered simultaneously. The chamber was housed within a small building and outdoor air ventilation was continuously supplied to the chamber through a dedicated system that had a carbon trap to remove organic gases, and held the chamber at a slightly positive pressure. The ventilation rate was 0.75 h<sup>-1</sup> determined using CO<sub>2</sub> as a tracer gas (EGM-4, PP systems). Temperature and relative humidity in the chamber were logged continuously (HOBO<sup>®</sup> U10-003, Onset Corp., USA).

Experiments were performed in 2 phases: a smoking phase and a ventilation phase, as shown in Fig. 1 and Table S1 in Supplementary Information (SI). The smoking phase started at the point of cigarette lighting, and lasted for  $\sim$  2.5 h, corresponding to roughly 3 air exchanges. Clean air was introduced continuously during both phases. However, oxidation studies were performed by adding ozone during the ventilation phase ('ozone'), for comparison with ventilation by clean air ('clean'). Ozone was generated by flowing Ultra High Purity O<sub>2</sub> (Airgas) through a corona discharge ozone generator (Yanco Industries M/N GE30/FM100) at a rate of 100 mL min<sup>-1</sup>. The ozone flow was introduced at the center of the chamber. Gas phase ozone concentrations in the chamber in the absence of tobacco smoke were <200 ppb (steady state of  $[O_3] =$ 180 ppb was reached after 24 h), and were monitored with a continuous ozone monitor (2BTechnologies Model 202.). These ozone levels are higher than typical concentrations associated with infiltration from outdoor air, but consistent with those observed during operation of ozone-generating indoor "air cleaners" (Phillips and Jakober, 2006; Waring et al., 2008).

#### 2.2. Model surfaces and wall wipes

Wallboard specimens were cut and the edges sealed with Teflon tape to limit direct exposure to the gypsum core. Strips of chromatography paper of  $17.5 \times 13.5$  cm (Whatman 3MM, No. 3030-153), cotton cloth samples of  $10 \times 3$  cm, and wallboard samples of  $9 \times 1 \times 1.3$  cm were placed in the center of the chamber for exposure, at a distance of  $\sim 1$  m from the smoldering cigarettes. Multiple equivalent samples of each model surface were exposed simultaneously to the same chamber air, and were retrieved at different times to follow their SHS/THS loading as a function of time. The effective surface areas of the substrates were determined by N<sub>2</sub>-BET measurements. Samples were conditioned at 50 °C for 20 h under a dry N<sub>2</sub> flow (FlowPrep 060, Micromeretics) followed by analysis of N<sub>2</sub> surface gas adsorption at 77 K (TriStar 3000 Micromeritics).

#### 2.2.1. Wall wipes

During experiments the chamber walls were wiped using laboratory lint-free tissues (KimWipes<sup>®</sup>) at selected times during chamber studies (sampling conditions described in Table S2). The extracts were analyzed following procedures that were similar to those used for the surface materials (described below).

#### 2.3. Sample collection and analysis

#### 2.3.1. Extraction

Model samples exposed to SHS were extracted twice at 100 °C at 1500 psi, in methylene chloride (OmniSolv, spectrophotometric grade) in an accelerated solvent extractor (ASE200 Dionex Corp.) using a total volume of up to 30 mL. A methanol aliquot of 0.5 mL

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