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Volatile Organic Compounds Emitted from Air Fresheners: Plug-Ins at Home and Little Trees in Cars

Adriana Pino-Delgado[1], Danielle Vado[1], Dajon McLeese[1], Shizuka Hsieh[1]

[1]Trinity Washington University, Washington, D.C., USA

ABSTRACT

Air fresheners contain volatile organic compounds (VOCs), some of which are possible hazardous air pollutants, carcinogens, or chemicals associated with adverse health effects such as asthma. This screening study identifies VOCs emitted from two types of air fresheners. Plug-in air fresheners are household products that release scents from a heated liquid. Little Tree air fresheners are commonly used solid devices designed to hang in from the rear view mirror in cars. VOCs that were released from heated samples of air fresheners were identified by gas chromatography mass spectroscopy (GCMS). Amongst VOCs commonly known to have scent properties, this screening also revealed the presence of compounds that are not associated with fragrances and that may have adverse health effects. In a pilot study to characterize how air fresheners affect air quality in real settings, air samples were collected on solid-phase microextraction (SPME) field samplers in a home setting and inside a vehicle, before and during air-freshener use. This pilot demonstrated some limited potential for SPME field samplers to track changes in VOC levels in real-life settings.

Introduction

Air fresheners work by releasing scented compounds into the air that overpower odors. There are concerns as to whether air fresheners increase the indoor air pollution due to the fact that they release volatile organic compounds (VOCs) into the air (Steinemann, 2017; Kim, 2015; Nazaroff, 2004). VOCs are chemicals that can turn into a gas or vapor at room temperature. These VOCs may be responsible for health effects associated with fragranced product use, which include respiratory problems, migraine headaches, asthma attacks (Steinemann, 2019), and asthma (Weinberg, 2017). Companies are not required to list all fragrance ingredients on their labels; therefore, many people are not aware of what they are inhaling. Indeed, chemical analyses have confirmed the presence of compounds not listed in the ingredients (Udhe, 2015; Steinemann, 2011). The NRDC (National Resources Defense Council) tested 14 common air fresheners and found that 86% of the products tested contained hazardous chemicals, including products labeled to be “all-natural” or “unscented” (Cohen, 2007; Solomon, 2007). The hazardous chemicals they identified have been associated with changes in hormone levels, asthma or other adverse health effects. A survey of 134 fragranced household products, including 12 air fresheners, revealed the presence of potentially hazardous and carcinogenic compounds in their emissions (Nematollahi, 2019). The combination of chemicals that comes from air fresheners is concerning because the cocktail may have a more toxic effect than each chemical would on its own. VOCs can also react with ozone, a common air pollutant that can come from indoor sources, such as photocopy machines or printers, or smog outdoors (Nazaroff, 2004). Reactions with ozone can form new VOCs or ultrafine particulate matter, which can also have adverse health effects (Kim, 2015), and formaldehyde (Lamorena, 2008; Destaillats, 2006; Singer, 2006), which is a probable carcinogen. Thus, air fresheners may mask unpleasant smells but in reality be exposing people to hazardous chemicals in a freshly scented environment.

The chemical analysis in this work screened for VOCs emitted from a small collection of two different types of air fresheners. Plug-in air fresheners are household products that release scents from a heated liquid. Little Tree air fresheners (LTAFs) are commonly used solid devices designed to hang in from the rear view mirror in cars. Plug-in air fresheners have been amongst the products tested in studies that tracked VOCs such as d-limonene, known for its citrus scent, and other scented compounds in the class of terpenoids (Destailles, 2006; Singer 2006; Singer 2006b; Udhe, 2014). These studies determined concentrations in air and also how quickly the VOCs reacted with ozone (Destailles 2006) and in polluted areas during the warm-weather season (Singer 2006b). Car air fresheners have been the target of studies that recognize the high concentrations of VOCs that can build up in the small confined space of a vehicle, in addition to the possibility of reactions with air pollutants from traffic (Lamorena 2006). Jo *et al.* monitored concentrations of VOCs emitted from gel-type car air fresheners in a chamber and in a vehicle, to determine the extent to which VOCs accumulate (2008). Both of these studies (Lamorena, 2006 and Jo, 2008) used air fresheners that were available in Korea: gel, liquid, and vaporizer. Thus the only study to our knowledge that specifically examines emissions from LTAF-type air fresheners is a recent work from Steinemann (2020) that identifies VOCs emitted from 12 different car air fresheners, three of which were the type that hang inside the car.

This work also contributes a preliminary use of solid-phase microextraction (SPME) field samplers for the use of tracking changes in VOC concentrations in real-life applications of air fresheners. SPME field samplers provide an easy method for capturing and, when coupled with GCMS, identifying VOCs in air. The method has proved useful in a variety of environments to identify compounds present. Examples include identifying VOCs emitted by pandas during the breeding season (Wilson, 2018) and VOCs accumulated in sealed museum display cabinets (Alvarez-Martin, 2020). While it is possible to quantify VOCs in air using samples captured on SPME (Hippelein, 2006; Larroque, 2005), the procedure was beyond the capabilities and equipment limitations in our laboratory. Validation experiments have illustrated how 10-minute SPME samples captured changes in traffic-related VOCs with time (Cebellos 2007). The same work indicated the limited reproducibility for SPME, with standard error for replicates ranging from 3 to 17%, and variability due to humidity and temperature (Ceballos 2007). Yet because SPME is easy to use for sample collection, we hypothesized that instead of absolute measurements, it could still be useful for tracking relative changes in VOCs concentrations specifically associated with air freshener use. We used SPME to track VOCs from air fresheners over several days, following the installation of a new air freshener. The real-life environments were a home for the plug-in and a car for the LTAF.

Methods

Chemical analysis

Chemical mixtures were analyzed on a Agilent 7890B/5977BMSD gas chromatograph mass spectrometer (GCMS). Briefly, the GC separated the mixture into its constituent chemicals so that each peak in a chromatogram corresponded to a different compound. The MS ionized the molecules and detected the fragment ions, generating a corresponding mass spectrum for each peak. GCMS parameters are described in the Supplementary Information. Compound identification was based on the best available match between the experimental mass spectrum and those in the NIST (National Institute of Standards) library database.

VOCs emitted by air fresheners

To detect the VOCs emitted by air fresheners, samples were placed in vials, which were then heated. A heated needle extracted air above the sample and injected it into the GCMS. For the plug-ins, 4.0 mL of each liquid was placed in a 20-mL headspace vial. Three vials were prepared for each brand. Vials were incubated for 15 minutes at 56.5°C. The temperature for the plug-ins was chosen because that was the temperature reached by the liquid in an actual plug-in apparatus. LTAFs were cut with a hole puncher, and 0.8 g of the pieces were placed in 20-mL headspace vials. Three vials were prepared for each scent. Vials were incubated at 60°C for 15 minutes. This temperature was selected since higher temperatures were expected to generate stronger emissions, but it was still within the range of hotter temperatures that cars are known to reach in the summer. The incubation time of 15 minutes was selected because additional incubation time did not increase the instrumental signal.

Collecting VOCs in real settings

VOCs from real settings were collected on solid-phase microextraction (SPME) field samplers (Supelco 504831), by exposing the adsorbent fiber material to the environment for one hour. After sample collection, each fiber was retracted to stop sample collection. Each field sampler was sealed and stored on ice after sample collection. GCMS analysis took place within 24 hours. The SPME fiber was inserted into the port, and the fiber was exposed during the entire run. For each test, one fiber was left unexposed, to verify that the fibers were only picking up VOCs during the intended exposure period and from the intended location. In cases where more than one fiber was exposed at the same time, results were averaged.

A Glade brand plug-in was used for in-home testing. The home setting had been exposed to a plug-in air freshener previously, but the previously-used plug-in was removed three days prior to testing. For testing, three SPME field samplers were positioned two feet away from the electrical outlet and at the same height as the plug-in. Two were exposed while one was left unexposed, all for one hour. There were three initial days of data collection without any plug-in. The test plug-in was installed for a week, during which there were four days of data collection. Samples were also collected for five days after the plug-in was removed.

For LTAF testing, data collection was done inside a 2002 Mitsubishi Eclipse with a Vanillorama air freshener. For testing, two SPME fibers were exposed for one hour and one was left unexposed. Three days of data collection without an air freshener was conducted first. After installing the LTAF, testing occurred over a week. On each day, data collection was within the same 4-hour window (10AM-2PM).

Results

VOCs emitted from air fresheners

Figures 1 and 2 show total ion chromatograms, in which each peak corresponds to a different chemical. Differences in patterns reflect how each air freshener emitted a unique mixture of chemicals. A list of chemicals with prominent peaks is in the Supplementary Information. Most chemicals fell into one of two classes of compounds, terpenes/terpenoids and esters, both of which are associated with scents and fragrances. Some chemicals, however, were more commonly used as solvents, to dilute organic compounds. In an analysis of four plug-in air fresheners and three LTAF, the VOCs detected in the highest number of air fresheners were, three terpenoids (D-limonene in 6 of 7, beta-pinene in 6 of 7, and dihydromercenol in 5 of 7 products), and two esters (acetic acid 3-methylbutyl in 5 of 7 and acetic acid 4-tert-butylcyclohexyl also in 5 of 7 products).

The chromatogram for the generic brand contained a unique feature. As shown in the inset at the bottom left of Figure 1, peaks were lying on top of a wide, bumpy, jagged feature with indistinguishable peaks. Mass spectral analysis of the feature indicated that it was due to a mixture of different alkane hydrocarbons. Two of the chemicals with prominent peaks for the generic brand were alkane hydrocarbons (2,2,11,11-tetramethyl-dodecane and 5-methyl-5-propyl-nonane). To see if alkane prevalence was a feature in other generic brands, three years after the initial experiments were conducted, we tested newly-purchased plug-ins (Figure 1, right) from dollar stores. One of these dollar store plug-ins also showed a broad, wide, feature, that was even more prominent that for the generic brand that we had initially tested (Figure 1, bottom right). Mass spectral analysis showed that this feature also corresponded to a mixture of alkane hydrocarbons. A second dollar store plug-in had prominent peaks corresponding to light alkane hydrocarbons, *n*-hexane and 2-methyl pentane (Figure 1, middle right). A third dollar store plug-in had a very prominent peak (Figure 1, top right) attributed to ethanol. While some other plug-ins also emitted ethanol, the peak's intensity indicated it to be a significant ingredient only in the dollar store plug-in.

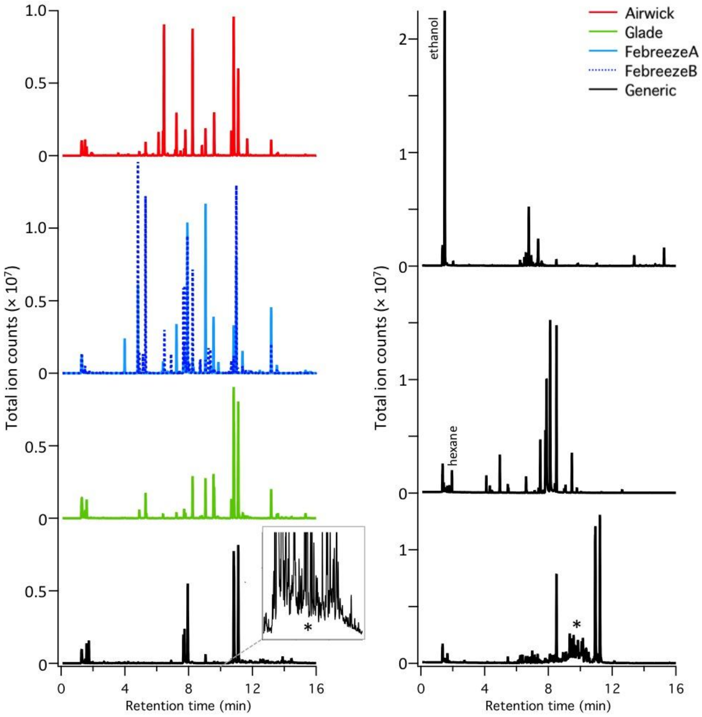

Figure 1. Total ion chromatograms for plug-ins, where each peak represents a VOC. On the left are data from the original experiment. Chromatograms for the two Febreeze vials that came in the same package are overlaid. The inset at the bottom is a detail of the generic brand chromatogram. On the right are data from new experiments conducted to investigate the presence of alkane hydrocarbons in dollar store brands. The asterisk marks a broad jagged feature due to a mixture of alkane hydrocarbons.

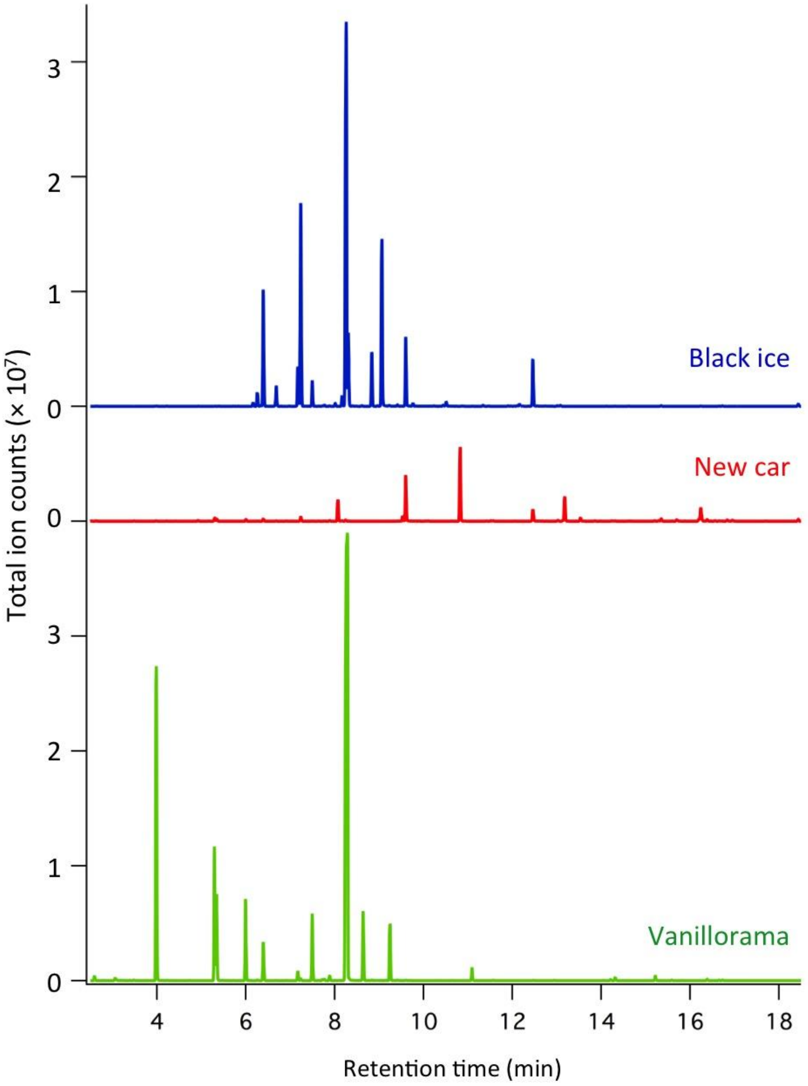

Figure 2. Total ion chromatograms for emissions from Little Tree air fresheners.

Changes in VOCs concentrations with use

Figure 3 tracks the integrated peak areas for selected compounds over three periods, before installing a new plug-in, while the plug-in remained installed, and after removing the plug-in. All were detected in the home before installing the plug-in. No dramatic changes in concentrations occurred in the home environment, even after the plug-in installation and removal. There were, however, differences in patterns over time.. The top trace shows integrated peak areas for five compounds, all of which were prominent VOCs emitted from the Glade brand plug-in liquid. While limonene was also a prominent component of Glade emissions, its peak areas did not follow the same pattern. As shown in the bottom trace, its pattern followed that of pinene, a VOC that did not appear as prominently in the emissions from the Glade plug-in as it did for other products.

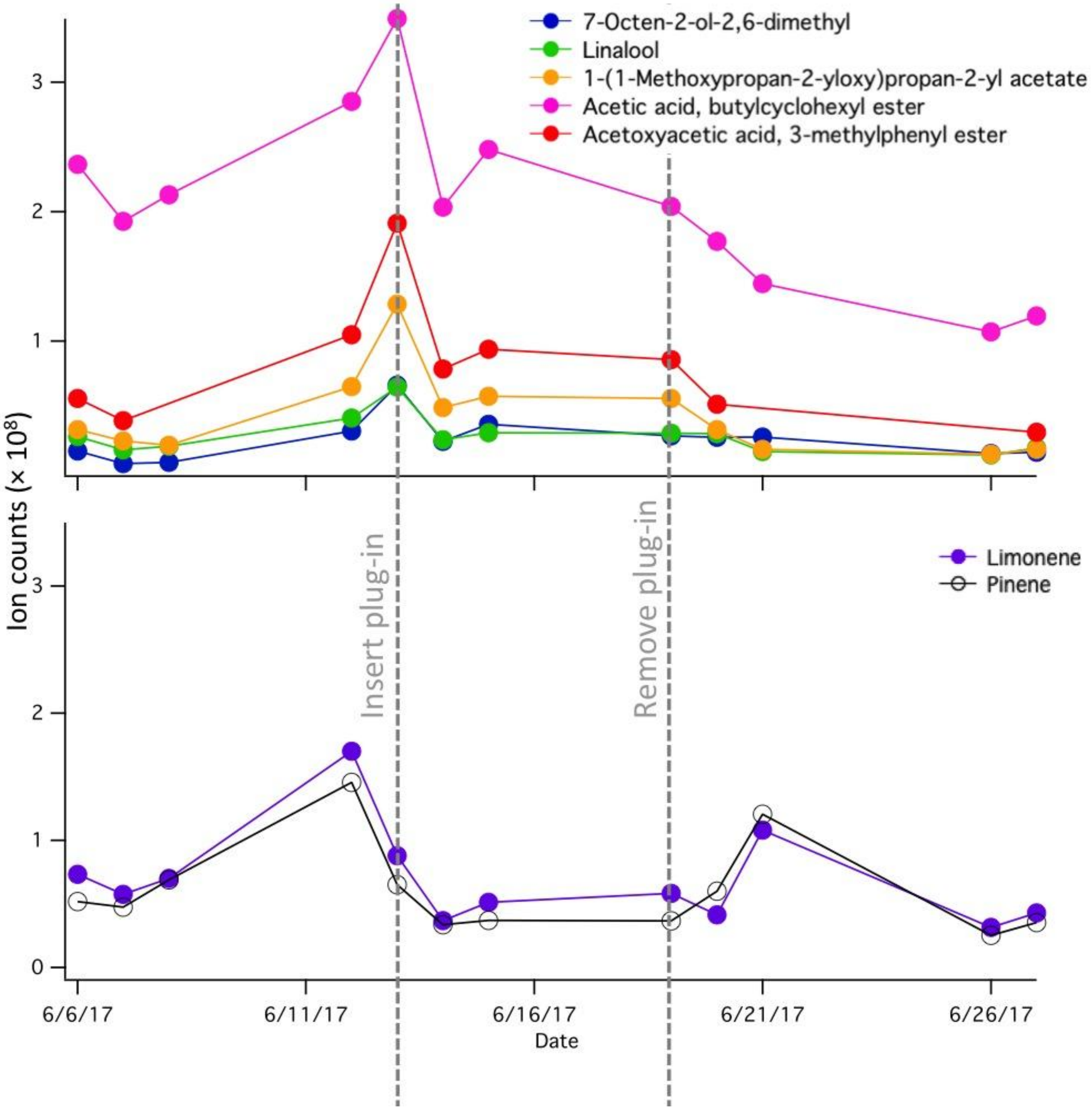

Figure 3. Relative changes in concentrations of compounds emitted from a Glade plug-in, in an in-home setting.

Figure 4 similarly shows integrated peak areas for some of the prominent compounds associated with the Vanillaroma LTAF, following its installation in a car. Over the eight days after installing a new Vanillaroma LTAF in the vehicle, concentrations of these compounds decreased dramatically. Fitting points to exponential decays yielded a half-life of one day for limonene, indicating that the LTAF loses its potency within days. Pinene, 2(3H)-dihydro-5-pentyl furanone and acetic acid, *p*-methoxybenzyl ester took longer to dissipate than the rest of the compounds, perhaps due to a slower release from the LTAF. While it was hypothesized that reactions might generate new volatile organic compounds, none were detected.

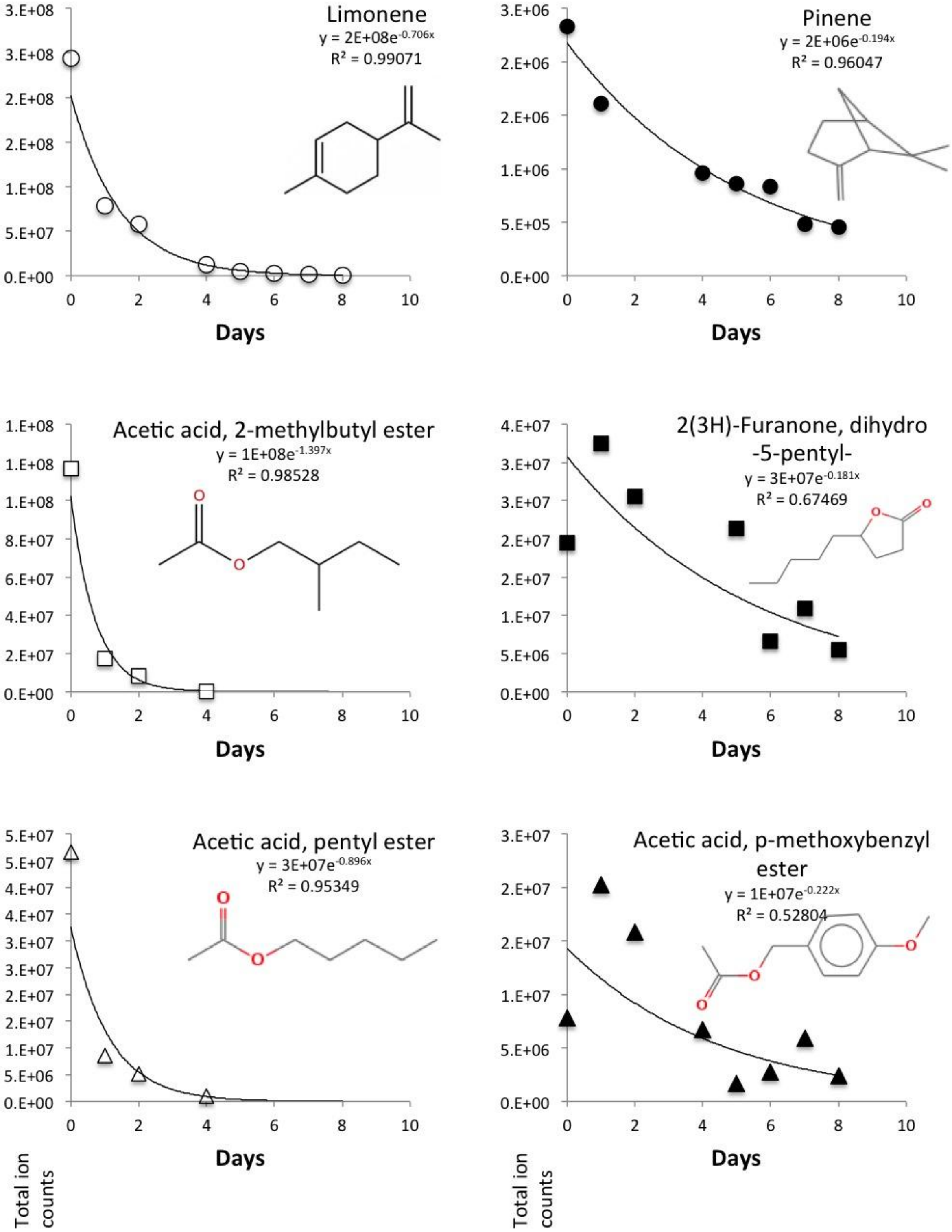

Figure 4. Relative changes in concentrations in a closed automobile on the day of and 8 days after hanging a new Little Tree air freshener (Vanillorama) in a car.

Discussion

VOCs found in air fresheners

This work is similar to others in the literature (Steinemann, 2020; Nematollahi, 2019; Steinemann 2011) that list VOCs detected in air freshener emissions and identify the ones with prominent peaks in GCMS analysis. Without quantitative analysis of concentrations in air, however, human exposure levels are left unknown. Since health effects depend on exposure and dose, this and similar works therefore cannot rate the toxicity of using air fresheners. However, this kind of work can raise potential concerns due to VOC associations with adverse health effects.

Terpenes and terpenoids are a large class of VOCs that are found in nature, in plants and fruits. Because of their fragrance properties, they are prevalent in air fresheners and cleaning products. In a survey of 25 products, Steinemann *et al.* (2011), d-limonene, alpha-pinene and beta-pinene were the most prevalent. Concentrations of d-limonene were highest amongst biological VOCs emitted from air fresheners and cleaning products (Huang, 2011). The three chemicals falling in the category of terpenes and terpenoids that appeared in the most (at least 5 out of 7) tested products in this study were d-limonene, beta-pinene, and dihydromercenol. In their review of potential health effects associated with air freshener emissions, Kim *et al.* (2015) address terpenes, specifically d-limonene, pinene and linalool, all three of which were also found in products tested in this study. The review associates these terpenes with respiratory diseases and skin allergies or dermatitis. When linalool reacts with oxygen over time, the products may cause allergic responses (Kim, 2015).

The three most prevalent esters were acetic acid, 3-methylbutyl ester; acetic acid, phenylmethyl ester; and acetic acid, (4-tert-butylcyclohexyl) ester. While only one of the VOCs identified as most prominent in emissions from 12 air fresheners was an ester, ethyl acetate (Nematollahi, 2019), a survey of car air fresheners revealed (Steinemann 2020) 6 esters out of the list of 17 most prominent VOCs. Esters are expected to be in air freshener emissions, as they have characteristic sweet or fruity smells. Acetic acid, 3-methylbutyl ester (also known as isoamyl acetate or 3-methyl butyl acetate) has a banana or pear smell. Acetic acid, phenylmethyl ester (also known as benzyl acetate) has a floral scent. Acetic acid, (4-tert-butylcyclohexyl) ester (also known as 4-tert-butyl cyclohexyl acetate) smells woody. None of these esters appear to have significant known health effects. For example, the Environmental Working Group's Skin Deep website lists no known cancer, developmental or allergenic effects (n.d.). A toxicity study of 4-tert-butyl cyclohexyl acetate showed no irritation in two studies where the chemical was applied to human skin (Bhatia, 2008).

Three of the four generic brands emitted alkane hydrocarbons and one had a strong signature of ethanol. Neither alkane hydrocarbons nor ethanol have scent properties to contribute towards fragrance; rather they act as solvents. Such solvents have been detected in other air freshener studies, for example butane and pentane were found in the emissions from an pressurized air freshener spray (Udhe, 2015). The finding of strong solvent signatures from the generic brands tested indicates that some may contain liquids that increase the plug-in's liquid volume but do not add to desirable scent properties. The prevalence of solvents may be of concern, as some are potentially hazardous substances. While ethanol inhalation is not considered hazardous, it may adversely affect individuals with alcohol addiction issues (MacLean, 2017). Inhalation of hydrocarbon mixtures that include both alkane and aromatic compounds, however, can lead to asthma like complications including chemical pneumonitis, acute or chronic inflammation of the lungs due to inhaling irritants. Those that have been exposed to hydrocarbon mixtures typically experience frequent coughing and shortness of breath (Curtis, 2020). A specific alkane hydrocarbon whose health effects have been documented is hexane. It can cause numbness in hands and feet or even paralysis if directly inhaled, as can happen in workplaces or when individuals "sniff" solvents in an attempt to get high (ATSDR, 2011), but exposures to vapors from plug-ins are unlikely to be high enough to cause such severe health effects.

Changes in VOCs concentrations with use

The pilot study to track VOCs from air fresheners over time was more successful for the car than the home setting. While VOCs from LTAF in a car were at their highest concentrations within the first few days of use, the in-home

VOC concentrations showed little impact on indoor air due to the plug-in air freshener. One reason is that the home presents a larger volume in which the VOCs disperse. Another reason is that in the in-home setting, other household products may be contributing to VOCs with similar effects as plug-in air fresheners. Limonene and pinene concentrations followed a different pattern than other VOCs prominently found in plug-in emissions. Since limonene and pinene are found in common household products, it is likely that the SPME fiber collected emissions from other product use. The presence of VOCs found in plug-ins a week after removing the plug-in may reflect a combination of VOCs from other household products and the lingering effects of the plug-in. The slow decrease in VOCs from the plug-in are consistent with a recent study that showed that it took two weeks for limonene from air fresheners to decrease to background level (Goodman, 2020).

Conclusion

This chemical analysis of air emitted from four plug-in and three car air fresheners illustrates the complexity and uniqueness of mixtures used in different brands and for different scents. As found in previous studies, the most commonly found compounds were those associated with scents and fragrances, terpenoids and esters.

Yet air freshener companies are not required to disclose all the ingredients in their products, and several studies have documented the discovery of unlisted chemicals (Steinemann, 2011; Steinemann, 2017; Nematollahi, 2019; Steinemann, 2020). Chemical analyses are therefore necessary to determine what chemicals people are inhaling when they use air fresheners. This work corroborates studies that revealed the presence of unscented components that are used as solvents (Udhe 2015). We found evidence of alkane hydrocarbons, commonly used as solvents, in three of four tested generic plug-in air fresheners, but not in four tested brand-name products. For one of the four generics, the most prominent chemical signature was from ethanol, also an unscented solvent. Our findings encourage the testing of generic brand products alongside commonly-found brand names.

The scented and unscented compounds detected in this work include compounds associated with adverse health effects, yet it is impossible to determine health risks without concentrations in air that determine human exposures. While many studies (Udhe, 2015; Rahman, 2014; Huang, 2011; Singer, 2006) have quantified compounds emitted from air fresheners, this study takes a first step in that direction by applying an easy-to-use method for sample collection in real environments. This pilot study illustrated the relative impact of air freshener use over time, and revealed a potential use of the method for determining when air freshener components have been cleared from a home or car space.

Limitations

This work is limited to identifying volatile organic compounds emitted from air fresheners and how their relative concentrations change during product use. Identification was based on the best match with mass spectra in a library; compound identities were not verified with standards. Experiments also did not quantify concentrations of the chemicals in the air. Relative integrated peak areas or peak heights for different compounds cannot be used to compare concentrations, because the detection sensitivity for GCMS and the collection efficiency for SPME are different for each VOC and were not characterized. SPME collection efficiencies also depend on temperature and humidity, and individual SPME fibers can vary in their ability to collect and deliver VOCs to the GCMS. Limited data from this pilot experiment (23 trials) showed an average of 40% difference when two separate SPME fibers were used to collect data at the same time.

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