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Characterization of incense smoke by solid phase microextraction—Comprehensive two-dimensional gas chromatography (GC × GC)

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Abstract

Comprehensive two-dimensional gas chromatography in tandem with flame ionization detection (GC × GC-FID) was used for the qualitative fingerprint characterisation of four different types of powdered incense headspace (H/S), and incense smoke. Volatile organic compounds (VOCs) in the incense powder and smoke were extracted by using solid phase microextraction (SPME) with a polydimethylsiloxane/divinylbenzene (PDMS/DVB) 65 μm fiber. Low-polarity/polar, and polar/non-polar phase combinations were tested to contrast the GC × GC separation of components in these two column sets.

A total of 324 compounds were tentatively identified, with more than 100 compounds in incense powders and more than 200 compounds in the incense smoke, by using GC coupled to quadrupole mass spectrometric detection. Identification required at least 90% match with the NIST library; otherwise they were considered as unidentified. The smoke stream comprised compounds originating from the incense powder, and combustion products such as PAH, N-heterocyclics, and furans. However, GC × GC was able to separate many more volatile compounds (possibly hundreds more) present in the complex smoke samples, many of which cannot be separated by conventional 1D-GC; this is a direct consequence of the high-resolution power of GC × GC. GC × GC fingerprint comparison of powder H/S with smoke allows facile subtraction of the former from the latter to assist identification of compounds generated from burning incense.

Keywords: Incense; Smoke; Headspace; Solid phase microextraction; Comprehensive two-dimensional gas chromatography

1. Introduction

Incense is used to mask odour, for aromatherapy, and plays an important role in many religions around the world, especially in eastern religions. The Hindu, Buddhist, Taoist and Shinto religions all burn incense in festivals, processions and many daily rituals, such as paying respect to ancestors and various gods.

There are two broad types of incense. Western incenses are normally burned to produce pleasant fragrance inside home, shopping centres, shops and public places. These incenses are largely made from the gum resins in tree bark (such as frankincense), aromatic woods, flowers, essential oils and synthetic substitute perfume chemicals. Eastern incenses are

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processed from plants such as sandalwood, patchouli, agarwood and vetiver, and are used for ceremonial practices. Incenses are available in various forms including joss stick, cones, coils and rocks.

Incense burning is a long, slow, incomplete combustion process, resulting in the generation of a continuous smoke stream, which is an important source of indoor air pollution due the emission of PM_{10} and $PM_{2.5}$ (particulate matter less than 10 and 2.5 μm in aerodynamic diameter), carbon monoxide (CO) and volatile organic compounds (VOCs) (Lee and Wang, 2004). These researchers measured the emission rates of 10 different incense types varying in combustible volume from 0.29 to 2.8 cm^3 and found that emission rates for $PM_{2.5}$ and PM_{10} ranged between 9.8–2160.3 and 10.8–2536.6 mg h^{-1}, respectively (emission rates did not correlate with incense volume; burn times varied from 25 to 50 min). Lung et al. (2003) showed a significant contribution from incense burning to indoor PM_{10} and particulate polycyclic aromatic hydrocarbon (PAH), which comprises a large group of organic contaminants generated by incomplete combustion of organic material. One incense stick usually lasts about 45–60 min depending on the length and tightness of the powder packing. The greater the mass of material burned, the greater the particulate mass that is generated (Mannix et al., 1996). Other studies indicate that the particulate burden from burning one incense stick is 1.2–2.9 times of that of burning one cigarette (Chen and Lee, 1996; Mannix et al., 1996). Lung and Hu (2003) showed that incense burning produced about 28.3–30.5 μg PAHs per stick.

The aerosol particulate matter emitted from incense burning was found to be mutagenic in the Ames Salmonella test (Lofroth et al., 1991). Incense sticks were found to contain compounds causing allergic contact dermatitis and photosensitization, such as musk ambrette, musk ketone and musk xylene (Roveri et al., 1998).

Solid phase microextraction (SPME) is a solvent-free sampling technique, introduced by Pawliszyn in the 1990s, and is a popular alternative sample preparation and extraction method to classical extraction methods. The transport of analytes from the matrix into the fiber-supported polymer coating commences when the fiber is placed in contact with the sample. In the headspace (H/S) mode, the analytes are transported through the air before they are sorbed into or onto the coating. SPME has been successfully applied in many applications, including wound-induced VOCs from plants (Perera et al., 2002), analysis of PAHs in atmospheric particulates (Vaz, 2003), volatile terpenes in olibanum (Hamm et al., 2005), aroma compounds in palm sugar (Ho et al., 2006) and methoxypyrazines in wine (Ryan et al., 2005).

The hyphenated GC technique of comprehensive two-dimensional gas chromatography (GC × GC) was reported in 1991 by Phillips (Liu and Phillips, 1991; Phillips and Beens, 1999), comprising two serially-coupled GC columns containing different stationary phases with a modulator near their junction. The modulator serves to periodically and rapidly sample effluent from the first column, as a series of concentrated zones, which are then reinjected as narrow peaks into the second column. The second column generates a chromatogram of a few seconds (s) duration. Therefore, the entire sample is submitted to two ‘‘orthogonal’’ separations. The process results in significantly higher peak capacity with substances that co-elute on the primary column ideally resolved on the secondary column. GC × GC has been successfully applied to a wide range of samples including sedimentary petroleum hydrocarbons (Reddy et al., 2002), volatile compounds in strawberry (Williams et al., 2005), natural fats and oils (Mondello et al., 2003), and urban aerosols (Kallio et al., 2003).

Much previous work on incense smokes employed conventional extraction and GC methods; no reports of analysis of incense smoke have used SPME with GC × GC. Previous studies mainly focused on the emission rates of the smoke from incense burning, however, the compounds that were released from the smoke were not well characterized (and chromatograms often not shown in published literature). Therefore, it is important to develop an analytical method that can be used for the chemical characterization of compounds present in the particulate phase especially where the complexity is significant. Notwithstanding the quantitation limitations of SPME, the present study seeks to examine the feasibility of using SPME-GC × GC for sampling and identifying volatile compounds emitted by incense powder and in the smoke stream, and to generate a fingerprint chromatogram for different types of incense. Two different SPME extraction setups were used for the analysis of incense smoke—direct from the smoke stream, and the side stream. Different GC × GC column configurations were studied to contrast the separation of compounds.