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Unintended VOC emissions from cotton and viscose pads complicate olfactory experiments

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The release of volatile organic compounds (VOCs) from sweat pads is modulated by both material composition and conditions of use, particularly moisture content and thermal treatment. In this study, we investigated the impact of water addition and heat exposure on VOC emissions from cotton and viscose–polyester unsampled sweat collection pads using dynamic headspace sampling coupled with two-dimensional gas chromatography–high-resolution time-of-flight mass spectrometry (DHS-GC× GC-Q-TOF-MS). Water addition led to a pronounced increase in VOC release – including key VOCs with low ppbv odor thresholds like acetic acid, 2,4-nonadienal and 2,3-butanedione – with at least a five-fold increase observed for 110 compounds extracted from cotton pads at ambient temperature. Extraction at elevated temperature (60 °C) resulted in even higher fold changes in VOC release. Heat treatment at 150 °C prior to water dosing further amplified VOC emissions. These observations suggest that cellulose–water interactions play a fundamental role in mediating VOC emission dynamics. Our findings highlight the material- and condition-dependent variability in VOC release from sweat collection pads and underscore the need for rigorous control of hydration and thermal effects in chemical and psychophysical studies involving olfactory stimuli. Given the widespread use of cotton pads in psychological and chemosensory research, these methodological considerations are critical for ensuring reliable chemical analysis and reproducible perceptual outcomes. However, these findings do not invalidate previous work, but rather suggest that it should be interpreted with greater caution. The pad technique may still be useful–provided that large sample sizes and appropriate controls are employed–but it is associated with increased noise.

Human chemosensory cues have been central to numerous psychological studies investigating the effects of olfactory stimuli on perception, emotion, and physiological responses. A large fraction of these studies frequently follow the protocol introduced by Doty and Laing (2003) in the *Handbook of Olfaction and Gustation*[1]. This method involves collecting axillary sweat using cotton pads, which are then cut and pooled according to emotional conditions before presentation to participants. This standardized approach has been employed across multiple studies investigating chemosensory anxiety signals[2–8], fear- and happiness-related chemosignals elicited via film clips[9–13], and other stress-inducing paradigms[14–17], as well as male aggression-related sweat exposure[18]. Despite the widespread use of cotton pads, relatively few studies have investigated the detailed volatile organic compound (VOC) profiles captured with this method.

A seminal study[19] demonstrated that humans can detect emotional stress through chemosensory cues–even unconsciously–by comparing sweat collected during skydiving with sweat obtained during exercise. However, the study did not quantify the amount of sweat collected, and analysis was limited to semi-volatile compounds extracted from the cotton pads. Another study collected 144 sweat samples from 24 nonsmoking Caucasian males under three emotional conditions–fear, happiness, and neutrality–and identified a total of 23 VOCs using GC×GC-Q-TOF-MS[20]. These results suggest that chemosensory signatures are chemically complex, and intra-emotional variability may reflect distinct physiological or metabolic processes. In a recent study, key anxiety-related chemosignals–dodecanoic acid and 3-hydroxy-3-methylhexanoic acid–were identified using GC and GC-olfactometry in condition-specific pooled samples collected via cotton pads[21]. Similarly, sitosterol was identified as an individual stress marker in a paired stress study with scalable chemical interrogation[22].

Additionally, a growing body of research also investigates interspecies chemical communication involving human olfaction. Exploratory studies have shown that humans may be able to detect emotional states–such as

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fear–in animals like horses and dogs through body odor[23,24]. Conversely, dogs have been reported to differentiate between various human emotional states[25,26], and even predict epileptic seizures[27]. Many of these studies used cotton swabs or pads to collect sweat samples, highlighting the widespread use of this method.

Emotion induction has also been shown to significantly increase sweat production. For example, one study found that exposure to fearful stimuli increased sweat production by approximately fourfold in both males and females, with absolute amounts rising from about 50–100 mg in the neutral condition to 150–450 mg in the fear condition[28]. Another study using emotional film clips reported a six-fold increase in sweat production, with averages rising from 10 mg (neutral) to 60 mg (fearful)[11]. Another similar study reported mean sweat production of 30 mg in neutral state, 200 mg in happy state and 250 mg in fearful state[13]. These findings highlight how environmental conditions, sex of the donor, stimulus intensity, and sampling duration all influence sweat quantity.

Given the chemical complexity and high variability reported across studies, establishing reliable control conditions is critical. In the present study, we address key methodological challenges in chemosignal research and VOC analysis using sweat collection pads. These pads, characterized by their heterogeneous composition and lack of specific design for chemical sampling, tend to release VOCs unpredictably. This uncontrolled release introduces variability and batch effects, rendering experimental outcomes susceptible to environmental factors such as humidity and temperature–conditions that can compromise the reliability of both analytical results and olfactory exposure experiments. A promising advancement in sweat sampling may lie in transitioning from pad-based methods to flow-through sampling systems, as demonstrated by Vautz et al.[29]. Another potential alternative is the use of sorbent-based sampling materials, as also suggested in recent literature[21,30].

Results

Comprehensive compound identification with nontarget analysis by GC×GC-Q-TOF

The identification workflow resulted in a total of 462 detected features. Of these, 364 were tentatively identified by meeting the combined criteria for spectral library matching, linear retention index (LRI) agreement, and molecular or characteristic ion formula matching. An additional 79 peaks were classified as class unknowns, indicating that although definitive identification was not possible, certain chemical characteristics could be inferred–primarily based on fragmentation patterns and retention time behavior. For example, one class unknown at LRI 1889 was presumed to be a lactone, as it followed the homologous series of lower-boiling lactones that were tentatively identified and exhibited a shared characteristic fragment ion. Furthermore, 17 class unknowns displayed high spectral similarity scores but deviated substantially in LRI values, preventing conclusive identification. Finally, 20 features were categorized as true unknowns, lacking sufficient spectral or chromatographic evidence for classification. A comprehensive list of identifications is provided in (Supplementary Table; Tab1). The feature table was prepared based on the results of this nontargeted analysis by the application of the extracted ion image (EII) - peak template pairs (Supplementary Table; Tab2).

Effect of water addition to cotton pads using ambient extraction temperature (25 °C)

This set of results is particularly relevant as it provides an important control experiment for studies employing sweat collection pads to assess the impact of emotional and other stimuli on sweat production and its VOC composition. To address this question, we performed an extraction under inert gas flow at ambient temperature. Despite the large variability in the data (Figure S1, full dataset in Supplementary Table; Tab4), we observed a substantial increase in VOC release from the pads as a result of higher water dosing. We detected a set of 145 compounds showing statistical significance ($p_{adj} < 0.05, n = 4$), and a minimum threefold increase in peak volumes, while 110 were highly significant ($p_{adj} < 0.01, n = 4$), with at least a five-fold increase.

With cotton pads, VOC release appears exponential (appearing as linear on the logarithmic plot), with the most pronounced differences observed above 200 μL of water added. A selection of representative compounds is presented in Table 1, while the complete dataset is available in Supplementary Table; Tab5. This supplementary table includes the fold change values and statistical significance for all detected compounds across the experimental conditions discussed below. Readers are encouraged to refer to this material to examine the behavior of any compound or compound class of potential interest.

In Figure 1, we also included a rough estimate of the amounts of VOCs extracted. This estimation is based on the known quantity of d_5-chlorobenzene that was added to the sorbent tubes prior to analysis. It should be noted that these values are indicative; they represent estimated extracted amounts and do not directly correspond to perceived odour intensity, which depends on various factors such as volatilization from aqueous solutions–the context in which odour thresholds are typically defined[31]. Furthermore, we are not presenting precise quantitative results in this section, but rather aiming to provide context and perspective for the reader.

Effect of water addition to cotton and viscose/polyester pads using higher extraction temperature (60 °C)

Addition of water to the cotton pads resulted in at least a 10-fold increase in VOC release (Supplementary Table; Tab5), with highly significant results ($p.adj < 0.00001$) for a total of 265 compounds. When using pristine blank pads as the control condition, similar results were obtained when using 50 μL or 100 μL as the control condition. Figure 2A presents the curve describing the effect of added water on VOC release from cotton pads. Although internal standard normalization was applied, data variability remains high, generally ranging between 20% and 100% RSD ($n = 5$). Higher variability was also observed, though only for features near the detection limit. Nonetheless, the incremental trend in VOC emissions is evident. A set of VOC compound classes is presented in Figure 2A, demonstrating a release pattern similar to that observed during DHS extraction at 25 °C. This suggests that the interaction between cellulose and water occurs independently of extraction temperature, implying that water contact itself is a confounding factor in chemical analysis when using sweat pads.

The VOCs exhibiting the highest fold changes during room-temperature extraction were largely the same as those showing the most pronounced increases at 60 °C. Their fold changes were also very similar. However, some exceptions were observed: certain compounds, including naphthalene, 2-vinylfuran, 2-*Z*-heptenal, and 2-methylbenzofuran, were not significantly elevated at room temperature but became highly significant at 60 °C. Notably, these compounds had normalized peak volumes near the detection limit. As expected, increasing the extraction temperature from 25 °C to 60 °C during dynamic headspace extraction leads to a significant increase in extracted VOC total amounts, particularly for less volatile organic compounds.

As observed with the cotton pads, the extracted peak volumes from viscose-polyester pads exhibited high variability at each added water volume, generally ranging between 20% and 100% RSD (n=5). Higher variability was observed for features near the detection limit in this case as well. We identified a total of 212 chemicals with a fold change of at least 10 that were highly significant ($p.adj < 0.0001$). This number was notably lower than what was observed for cotton pads. The water addition–VOC release curve for viscose-polyester pads is presented in Figure 2B with all compounds in Supplementary Table; Tab5. The fold changes in VOC peak volumes were comparable to those observed for cotton pads, although the specific set of detected chemicals differed. The shape of the release curve also differed: in viscose-polyester pads, VOC release increased exponentially at lower water volumes (0 - 150 μL) but transitioned to a more linear trend at water volumes higher than 200 μL.

Effect of thermal pretreatment of the pads at 150 °C

Another aspect investigated was the effect of heat treatment on VOC release from the pads. This procedure was previously applied in our study[32] to reduce VOC background originating from the pads prior to exposure. The decision to include heat treatment was based on method validation, where no significant VOC background was detected when spiking 100 μL of a water/methanol mixture. However, substantial batch-to-batch variation was observed.

Heat treatment generally increased the number of highly significant ($p_{adj} < 0.002$) chemicals exhibiting more than a tenfold increase relative to the control condition, reaching 328 and 275 for cotton and viscose-polyester pads, respectively. Additionally, fold changes were generally higher. This provides scientific evidence that while thermal treatment reduces VOC background in dry, pristine sweat pads, upon water addition, it introduces another set of confounding variables. Representative examples are shown in Fig. 2C and D for cotton and viscose-polyester pads, respectively. In cotton pads, more than two-thirds of detected chemicals increased following heat treatment. In contrast, results for viscose-polyester pads were more variable, with some chemicals exhibiting decreasing trends.

Discussion

In Table 1, we present the same representative selection of compounds that exhibited substantial increases in abundance following the addition of water to cellulose-based sweat pads. For this subset, we conducted a literature survey to compile their known sensory descriptors and odor threshold values. These thresholds refer to the concentration of a given compound in aqueous solution at room temperature required for olfactory detection. Some compounds with highly characteristic or pungent odors have thresholds in the ppb to ppt range, indicating that only picogram to nanogram quantities are sufficient for detection. Notably, several compounds extracted from the pads in our study were present in the high nanogram range[32], underscoring the relevance of these findings. This highlights a critical consideration: variations in individual sweat production alone may lead to differences in pad odor profiles, which could in turn affect odor perception in experimental designs where such pads are used to present stimuli to sensory evaluation panels.

Surprisingly, there are only a handful of studies in which the authors report the tentative identities of the detected sweat VOCs sampled by the collection pads. In a study by de Groot[20], a set of 23 compounds were identified as significant chemosignals associated with different emotional states, using movie clips as stimuli. Of these, we found 9 compounds among the highly significant chemicals released from the same type of cotton sweat pads upon the addition of water. Additionally, two other compounds in our dataset showed increased fold-change but were not statistically significant in the dose-response analysis; however, they reached high significance when the extraction was performed at 60 °C (Supplementary Table; Tab5). The remaining compounds reported in other publications were not detected in our VOC dataset, likely due to methodological differences[20–22]. The full list of VOCs is provided in Supplementary Table (Tab5). No statistically significant decreasing tendencies were observed.

We emphasize the critical importance of appropriate control samples in experimental settings of this nature. Unintended or parallel processes may occur during odor collection, presentation or chemical analysis–particularly when employing materials that are not specifically designed for chemical sampling–which can confound results. To mitigate such effects, we recommend adopting more accurate and targeted sampling techniques. These include the use of low-volume sampling media such as cotton swabs[33], direct headspace analysis using sorbent tubes[21,30], and flow-through sampling strategies[29]. Additionally, well-established solid-phase extraction techniques from analytical chemistry, such as solid-phase microextraction (SPME)[34,35], and its variants including thin-film microextraction (TFME)[36,37] and stir-bar sorptive extraction (SBSE)[38], should be considered for their sensitivity and reliability.

Compound name	Class	Fold change	p_{adj}	Odor threshold	Sensory descriptor	Source
Acetic acid	Acids	9	<0.01	6 ppbv	Sour, acidic, pungent	[46]
Propionic acid	Acids	9	<0.01	26 - 170 ppbv	Sour, pungent, rancid	[31,47,48]
Benzyl alcohol	Alcohols	10	<0.01	10 ppm	–	[31]
1-Butanol	Alcohols	28	<0.01	500 ppb	Alcoholic, sweet, banana	[49,50]
1-Pentanol	Alcohols	11	<0.01	–	Alcoholic, sweet, oily	[49,51,52]
1-Hydroxycumene	Alcohols	15	<0.01	–	green, sweet, earthy	[53]
2,4-Nonadienal	Aldehydes	12	<0.01	0.09 ppb	–	[53]
Heptanal	Aldehydes	18	<0.01	3 ppb	Fruity, grass, green	[49,54,55]
Hexanal	Aldehydes	18	<0.01	4.5–5 ppb	Grass, green	[49,55,56]
Benzene, propyl-	Aromatic hydrocarbons	40	<0.01	–	–	–
Ethyl acetate	Esters	34	<0.01	5–5000 ppb	–	[54,56]
Benzoic acid, methyl ester	Esters	26	<0.01	–	–	–
Butanoic acid, methyl ester	Esters	16	<0.01	60 - 76 ppb	Fruity, apple	[57]
Decane	Hydrocarbons	14	<0.01	11 ppb	Gasoline-like	[58]
Octane	Hydrocarbons	32	<0.01	725 ppm	Gasoline-like	[53,59]
2,3-Butanedione	Ketones	10	<0.01	2.3–6.5 ppb	Buttery	[60–64]
2-Cyclopenten-1-one	Ketones	20	<0.01	–	–	–
4-Cyclopentene-1,3-dione	Ketones	17	<0.01	–	–	–
3-Pentanone, 2-methyl-	Ketones	18	<0.01	–	Acetone-like	[65]
2-Pentanone	Ketones	20	<0.01	70 ppm	Acetone-like	[54]
2-Propanone, 1-hydroxy-	Ketones	17	<0.01	–	sweet, caramellic, ethereal	[66]
1-Butanamine, N-butyl-N-methyl-	Nitrogen compounds	14	<0.01	120 ppb	Fishy	[67]
4-Pyridinecarboxaldehyde	Nitrogen compounds	14	<0.01	–	Fruity	[53]
Formamide, N,N-dimethyl-	Nitrogen compounds	15	<0.01	2.2 ppm	Fishy, unpleasant	[68]
2(3H)-Furanone, 5-methyl-	Oxygen heterocycles	11	<0.01	–	Coconut	[53]
2(5H)-Furanone	Oxygen heterocycles	36	<0.01	10 ppb	Buttery	[69]
3-Furaldehyde	Oxygen heterocycles	16	<0.01	–	–	–
Furan, 2-hexyl-	Oxygen heterocycles	15	<0.01	–	–	–
Furfural	Oxygen heterocycles	30	<0.01	3–23 ppm	Almond, caramel	[31,49,54,61]
Unknown 02 (LRI = 756)	Unknowns	27	<0.01	–	–	–
Aromatic class unknown (LRI = 1004)	Unknowns	44	<0.01	–	–	–

Table 1. Example compounds significantly increased in cotton pads following DHS extraction at 25 °C (paired t-tests with FC >5.0, p adj. < 0.01). The table reports fold changes (FC), adjusted p-values (p_{adj}, n = 4), odor thresholds, sensory descriptors, and known sources. Statistical results refer specifically to the extraction performed at ambient temperature.

Methods

Sweat pad materials

We investigated two types of commercially available absorbent pads that are widely used in the literature: the overwhelming majority of studies used cotton pads[3,5,6,8,9,12–17,17–22,25,26,28,39–41], while the remaining studies used a combination of polyester and some type of treated cellulose, such as rayon or viscose[10,11,32,42–44]. We used these pad types to evaluate volatile emissions under varying experimental conditions: Dermatess (10 x 10 cm, 67% viscose/33% polyester; Pietrasanta Pharma, Italy) and Cutisorb (10 x 10 cm, 100% cotton; BSN Medical, Germany). Dermatess pads were sourced from Pietrasanta Pharma, while Cutisorb pads were purchased via Homoempatia. Pads were used as received, with no chemical or physical pre-treatment unless specified.

Water dosing experiments

Different volumes of ion-exchanged water (Arium Pro, Sartorius AG, Germany, conductivity > 18.2 MΩ*cm, pH ≈ 5.5) were evenly spread over each pad to simulate sampling of varying sweat volumes. We applied the water volumes listed in Table 2 with 4–5 replicates for each condition. The extractions were performed in a randomized manner. The pads were analysed immediately after water dosing. Water amounts were decided according to the typical sweat production values[11,13,28].

Extractions at ambient temperatures (25 °C) and at 60 °C

To evaluate the impact of extraction temperature on volatile emission profiles, DHS experiments were performed at ambient laboratory temperature (25 °C). This temperature was chosen because the studies applied pad extraction methods performed at ambient temperature. Pads were placed in individual DHS stainless steel chambers and water was added in volumes of 0, 100, 200, 300, and 500 μL, with four replicates per condition. No thermal pretreatment was applied in this set (Table 2).

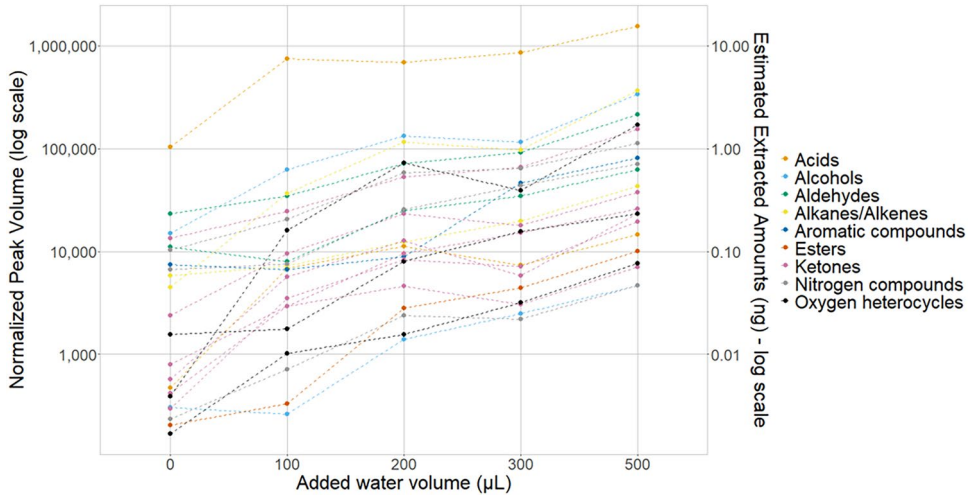

Fig. 1. Normalized responses of a representative set of 22 chemicals across the dataset, illustrating the impact of water addition on VOC release from cotton sweat pads during DHS extraction at room temperature (25 °C). The x-axis shows the volume of water added, while the left y-axis displays the log_{10}-transformed normalized peak volumes obtained via DHS-GC×GC-Q-TOF analysis. The right y-axis indicates the estimated VOC amounts, expressed relative to the peak volume of d5-chlorobenzene used as the internal standard.

For the extraction at 60 °C the procedure was similar, water was added in volumes of 0, 100, 150, 200, 300, and 500 μL. No prior cleaning or conditioning of the pads was performed. Extractions were conducted at 60 °C, with five replicates per water volume per pad type (Table 2).

Thermal pretreatment of pads at 150 °C before the experiments

Similar to a study investigating the effects of different cleaning regimens of cotton swabs[33], we assessed the impact of thermal cleaning on background volatiles. Additional experiments were conducted using pads that had undergone a prior thermal treatment step. Pads were placed in a GC oven at 150 °C for 20 minutes before water dosing (Table 2). Extraction temperature was 60 °C.

Chemical analysis with DHS-TD-GC×GC-Q-TOF

Dynamic headspace extraction

The methodology for the chemical analysis is described in detail in our earlier work[32], but a brief summary is provided here. Dynamic headspace extraction was performed using a Markes μ-Chamber (Llantrisant, UK). The stainless steel chambers were thermally cleaned at 150 °C for 20 minutes under a nitrogen flow of 150 mL min^{-1}. Nitrogen (purity >4.0) was supplied by an LNI Swissgas NG EOLO 10 L generator (Versoix, Switzerland). After thermal cleaning, the temperature of the μ-Chamber was allowed to return to ambient, the nitrogen flow was stopped, and packed sorbent tubes (TenaxGR) were inserted into the connectors of each chamber. The chambers were then heated to the desired extraction temperature (25°C or 60 °C), and the headspace purging was initiated with nitrogen at an extraction flow rate of 75 mL min^{-1} for 20 minutes.

Thermal desorption

Before analysis, an internal standard mixture was added to all tubes prior to tube desorption. We acquired a 50 L (148 bar) gas cylinder from Apel-Riemer Environmental Inc. (Miami, Florida) containing three certified, nonnaturally occurring VOCs (1,4-difluorobenzene, d_5-chlorobenzene, and 1-bromo-4-fluorobenzene) at a concentration of 1 ppmv. The reduction valve on the gas mixture cylinder was set to 1 bar. The cylinder was connected to the internal standard addition loop (1 mL loop volume) available on the Centri. A loop purge time of 1 minute was used before each injection.

The detailed description of the thermal desorption methodology was also presented in our previous work[32], but we briefly summarize it here. To eliminate excess humidity, a pre-purge of 500 mL of helium (purity 5.0) was performed at a flow rate of 250 mL min^{-1}. Tube desorption was carried out starting at 35 °C and ramped up to 300 °C over 5 minutes, during which the cold trap was maintained at 2 °C. The desorption flow rate was 35 mL min^{-1}. Subsequently, the cold trap was flash-heated to 300 °C and held at this temperature for 7 minutes. The total trap desorption flow rate was 4 mL min^{-1}, with a column flow rate of 0.5 mL min^{-1} and a split flow of 3.5 mL min^{-1}. To minimize carryover, the cold trap was thermally cleaned for 5 minutes at 300 °C at a flow rate of 50 mL min^{-1}.

GC×GC analysis with Q-TOF-HRMS detection

Comprehensive two-dimensional gas chromatography (GC×GC) was performed using an Agilent 7890B system equipped with a capillary flow technology modulator (Agilent Technologies, Santa Clara, CA, USA). The first-dimension column was a J &W DB-5MS (30 m x 0.25 mm x 0.25 μm), and the second-dimension column was a

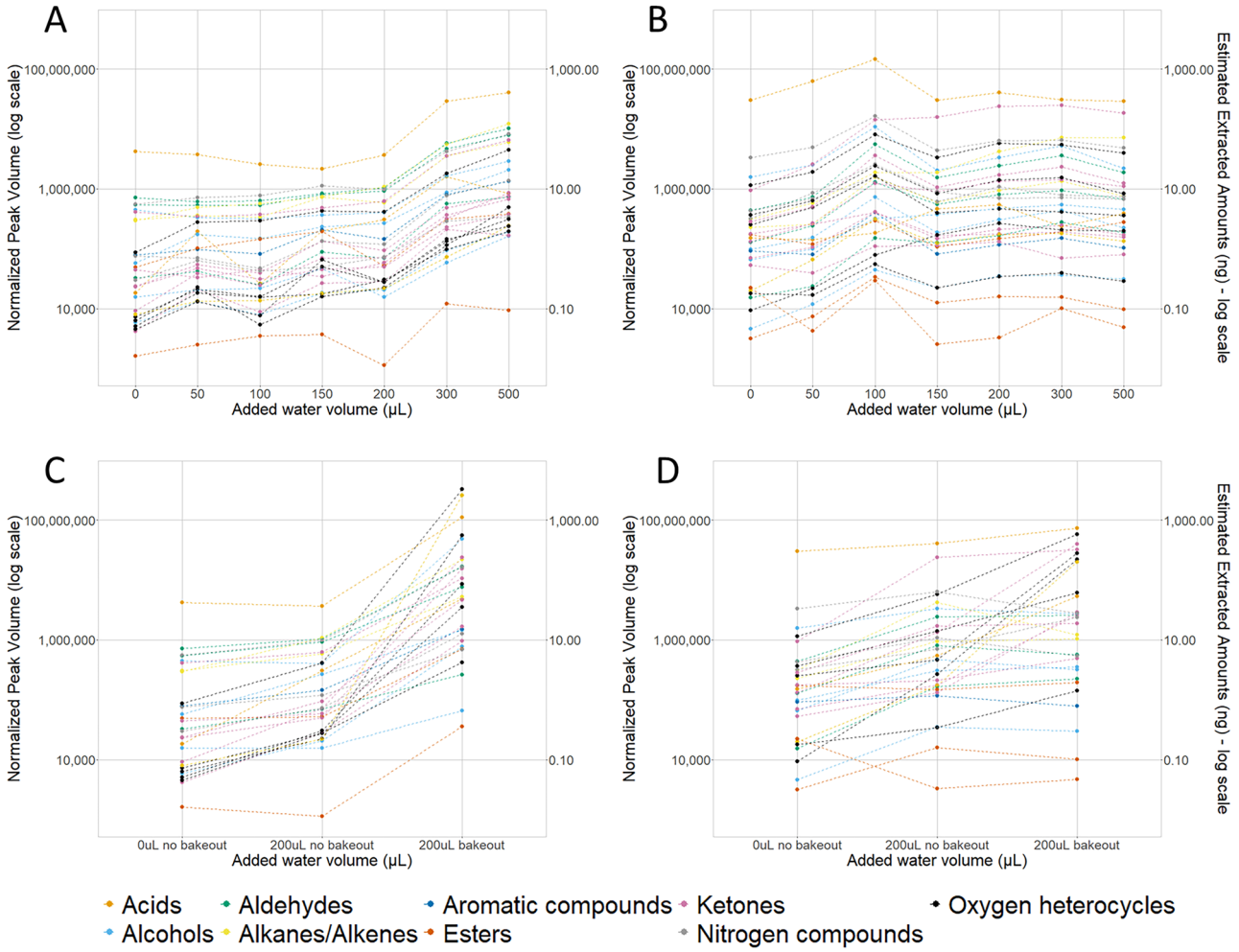

Fig. 2. Normalized responses of four representative compound subsets, illustrating the effect of added water on VOC release from cotton, viscose, and polyester sweat pads under the experimental conditions. The x-axis shows the volume of water added, while the left y-axis displays $\log_{10}$-transformed, normalized peak volumes measured via DHS-GC×GC-Q-TOF. The right y-axis indicates estimated VOC amounts (ng), calculated relative to the peak volume of d_5-chlorobenzene used as an internal standard. (**A**) Cotton pads with DHS extraction at 60 °C. (**B**) Viscose and polyester composite pads with DHS extraction at 60 °C. (**C**) Effect of thermal pretreatment on cotton pads. (**D**) Effect of thermal pretreatment on viscose/polyester pads.

J &W DB-INNOWAX (5 m x 0.25 mm x 0.15 μm). The separation was performed in constant-flow mode, with a volumetric carrier gas (helium, 5.0) flow rate of 0.5 mL min^{-1} for the first dimension and 18 mL min^{-1} for the second dimension, utilizing a modulation period of 2 seconds. Details about the methodology are reported in our previous work[32].

Molecular identification with GC×GC-Q-TOF

Non-targeted data processing was performed using GC Image version 2.8 (Lincoln, NE, USA), following the methodology outlined in our previous work[32]. In brief, the procedure comprised the following steps: (1) Conversion of raw data files into image files, with spectral filtering to exclude air background ions. (2) A random selection of 20–25 images was used to generate composite chromatograms through GC Image's automatic template generation feature. (3) Peak detection was conducted both automatically and with manual curation. (4) Spectra from detected peaks were compared against the 2017 National Institute of Standards and Technology (NIST17) spectral library. (5) First-dimension linear retention indices (LRI) were matched against the LRI database in the NIST17 library (Supplementary Table; Tab3). (6) Accurate mass measurements allowed to assign elemental formulas to molecular ions (when available) or qualifying characteristic ions.

For identification, we applied a spectral similarity threshold of $\geq$ 750, an LRI proximity threshold of ± 50 units, and a spectral accuracy of $\leq$ 10 ppm for elemental formula assignment (in some cases we accepted a value $\leq$ 20 ppm for low-intensity qualifying peaks that are expected from our instrument). The rationale for these criteria was discussed in detail in our prior method publication[32]. While spectral and LRI matching typically provides a robust foundation for tentative identification, the inclusion of elemental formula assignment based on fragment (or molecular ion) accurate masses further strengthens the confidence in compound identification.

Condition	Description	Cotton pads	Viscose/polyester pads
1	Pads "pristine" (extractions at 25 °C)	0, 100, 200, 300, 500 μL water (4 replicates each)	–
2	Pads "pristine" (extractions at 60 °C)	0, 100, 150, 200, 300, 500 μL water (5 replicates each)	0, 100, 150, 200, 300, 500 μL water (5 replicates each)
3	Pretreated pads at 150 °C (extractions at 60 °C)	200 μL water (5 replicates each)	200 μL water (5 replicates each)

Table 2. Summary of pad control experiments. Each condition describes the experimental treatment of cotton and viscose/polyester pads, along with the volumes of water used and number of replicates. Pristine pads were used as received, pretreated pads were heated to 150 °C for 20 minutes.

Data processing

The full data extraction and suspect screening workflow is described in our earlier work[32] but is summarized in this paragraph. Following the identification of suspected chemicals, characteristic ions were assigned based on their selectivity and spectral prominence ($\geq 30\%$ of the base peak). A narrow, asymmetrical spectral window (m/z - 0.003, + 0.01) was applied to account for mass shifts due to ion saturation, enabling the extraction of ion images (EIIs) from raw data files. These EIIs were generated for specific chemical classes (e.g., m/z 74.0368 and 87.0446 for methyl esters) or individual compounds.

To pair up with each EII, a peak template was developed, defining retention time windows for each compound as polygons or rectangles (Supplementary Table; Tab2). This template was systematically applied across all samples, yielding a final feature matrix with chemicals as variables and samples as observations (Supplementary Table; Tab4).

A total of 88 peak templates were created for both tentatively identified and unknown compounds, incorporating the monitoring of noncoeluting ions per template. Ultimately, 88 EIIs were extracted per sample, ensuring precise and selective monitoring of compounds based on retention time and characteristic ions.

After data extraction, feature tables were generated for all three experimental conditions, containing selective responses for 462 chemicals across all samples. These matrices underwent a data pretreatment workflow. First, negative and missing values were replaced with one-fifth of the smallest detected positive area for each compound. Subsequently, normalization was performed using the most reliable internal standard. In all cases, d_5-chlorobenzene served as the reference internal standard. For comparative analysis, normalization was carried out by dividing each compound's peak volume by the internal standard's peak volume within the same sample, followed by multiplication by the median peak volume of d_5-chlorobenzene across all samples. This approach preserved the original data distribution, facilitating subsequent statistical analyses.

Statistical analysis

Dose-response analysis was used to investigate the effect of added water on VOC release. This analysis quantifies the fold change in the response of each VOC following the addition of a specific volume of water to the pads. Additionally, it provides statistical significance alongside the fold change, based on replicate samples analyzed at each water volume. A detailed description of the method is available elsewhere[45]. During statistical analysis, a logarithmic (log_{10}) transformation was applied to the data; however, the reported fold change values remain untransformed.

Data availability

The directly extracted peak data from the processed data files for all samples is presented in "SI_TALBE.xlsx" on "Tab4_feature_template_ &_stats" tab. Also, please read the file "SI_Description_Figure_S1.docx" for details. Due to restrictions regarding data storage on archive servers and the nature of GCxGC-HRMS data structure (high mass resolution data binning together with a full scan spectrum stored at a data acquisition rate of 50 Hz), one raw data file is approximately 5 GB in size (even after initial processing that compresses data files to between 0.5 - 1 GB which is used to eliminate electronic noise) we can only share the raw data files on request. Please contact the corresponding authors for details.

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Competing interests

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