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Quantitative and sensory evaluation of malodour retention of fibre types by use of artificial skin, sweat and radiolabelled isovaleric acid†

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ABSTRACT: The retention of malodours, especially sweat on fabrics, is a widely discussed problem in the textile industry and science. Since a quantitative analysis of the retention of sweat odour molecules on fibres has not been addressed so far, we used liquid scintillation counting to measure the adhesion/dehesion of ^{14}C-labelled isovaleric acid, a lead substance of sweat odour, to knitted fabrics of comparable textile structure made of pure cotton, wool or polyester. Significant retention differences were detected, with polyester showing the highest release of isovaleric acid after 3 h and 20 h. Fabric finishes with β-cyclodextrin enhanced odour retention on cotton and polyester considerably. To study local influences of the clothing materials on odour release, we simulated in diffusion chamber cells a fabric/human skin wear situation using an artificial skin with skin-like composition, topology and mechanical characteristics. Unidirectional transepidermal water vapour release of the skin model modulated the odour retention. Finally, all samples were subjected to a sensory evaluation test with trained panellists using olfactometry and a non-labelled artificial sweat containing isovaleric acid as lead substance. Again, polyester showed little retention capacities, whereas the cyclodextrin finish enhanced binding of the artificial sweat and thus reduced its perception. The phase II approach revealed that perceived malodour intensity clearly depended on the finish and fibre type. The presented data and methods provide a basis for future optimization of clothes in terms of sweat odour management in their respective end uses, e.g. sport or business activities. Copyright © 2013 John Wiley & Sons, Ltd.

Keywords: olfactometry; scintillation counting; sports wear; adhesion; textile

Introduction

Sweating is not only essential for thermo-regulation of the human body, e.g. in sports, but also shows up in emotional, stressful situations, e.g. in daily business. While eccrine sweat is mainly responsible for thermo-regulation, apocrine sweat indicates stress conditions and, consequently, apocrine glands respond to more emotional stimuli.[1] Although fresh sweat secreted by apocrine and eccrine glands is odourless, it is known that the action of skin bacteria is needed to generate the odoriferous substances from non-smelling precursors.[2] The unpleasant body odour is not only embarrassing for the odour-emitting person, but can also have economic consequences such as the replacement of clothes, socks or shoes[3,4] or the need to wash clothes more often. Therefore, the market for functional textiles has extended to apparel textiles for individual consumers who demand odour-free, cleaner or fresher clothing, particularly sportswear, underwear and footwear.[5,6] Hence the textile industry has developed new fibres and finishing techniques to prevent odour build-up within apparel. Absorber finishes use cyclodextrin, a cavity-forming molecule which is able to complex small odour molecules, for instance citronellol, and retain them for up to a year.[7,8] In contrast to that, antibacterial-treated textiles aim to reduce the bacterial number on the fibre surface and, consequently, their metabolism in order to prevent the turn-over of precursors into the odoriferous components.[9] Although sweat odorants and their respective precursors are soaked up together with skin bacteria by the first layer of textiles, i.e. the clothes worn directly on the skin,[10] little is known how odour builds up in a fabric, and if there is an influence of the fibre type, the fabric construction, or the fabric finish on malodour.

While interactions of fabrics with skin bacteria have been studied,[11–14] textile literature relating specifically to the interaction of odour molecules with fabrics is rather limited. This is mainly due to the huge variety of fabric constructions, finishes, fibre types and blends. For example, Munk *et al.*[11] found that polyester retains a detectable odour intensity after it has been worn once, in contrast to cotton, which had to be worn twice. In addition, the same group showed that body odour remained on polyester and cotton fabrics even after laundering.[12] Field studies (wear trials) also revealed polyester being more odorous than cotton or wool after wearing.[11,14]

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Many studies so far have mainly focussed on the characterization of bacterial strains and sweat odorants, especially in the axilla, since the axilla is known to be a major contributor to human body odour and is equipped with a high density of apocrine and eccrine sweat glands.[2] The axillary microflora tends to be dominated by *Staphylococcus* or *Corynebacterium* spp.,[15,16] with *Proprionibacterium* and *Micrococcus* species also present,[17,18] but it is the aerobic corynebacteria that have been implicated as the main cause of axillary malodour.[17] The foot, another high-odour body site, is predominately populated with staphylococci and coryneform bacteria.[19] Furthermore, *Bacillus licheniformis* and *S. haemolyticus* have been found to produce intense odour when incubated on sterile axillary sweat, with *S. haemolyticus* producing the most sulfur-like odour.[20] A variety of odour molecules of the axilla has been identified so far. Volatile carboxylic acids are a key class of human body odorants, with main components such as (*E*)-3-methyl-2-hexenoic acid (3M2H)[21] and 3-hydroxy-3-methyl hexanoic acid (HMHA).[22] A second class includes volatile sulfur compounds, especially (*S*)-3-methyl-3-sulfanylhexan-1-ol.[20] The third class comprises odoriferous steroids, mainly 5a-androst-16-en-3-one and 5a-androst-16-en-3a-ol, which have been found in early studies by Brooksbank *et al.*[23] Although further steroids with low perception threshold[24] as well as sulfurous compounds[25] contribute to malodorous axillary sweat, it must be considered that those compounds can only be sensed by 50% of the population.[26] A main lead substance of sweat odour is isovaleric acid, a short fatty acid of acidic odour[27,28] and low odour threshold of 120–700 ppm.[24,29–31] Although the relative amount of other acids such as acetic acid is higher in foot sweat, there is a major contribution of isovaleric acid to foot and axillary odour because of its low odour threshold.[15,21,28,32–34]

The prevention of sweat odour build-up on fabrics is a widely discussed issue in the textile industry. Therefore, the purpose of our approach was to study the possible retention of sweat odour molecules on fabrics with respect to the common clothing materials cotton, wool and polyester and their surface properties. To examine adhesion/dehesion of odour molecules and fibre types in detail, we used liquid scintillation counting to quantify the retention of ^{14}C-labelled isovaleric acid on these fibre substrates. We evaluated the effect of a hydrophilic finish for polyester and a fabric finish with β-cyclodextrin on sweat odour retention of our samples. To study local influences of the three fabric materials on sweat evaporation, we simulated a fabric/human skin wear situation in diffusion chamber cells using an artificial skin with skin-like composition, topology and mechanical characteristics. In addition, to correlate the *in vitro* results to a sensory evaluation, all samples were also soaked with a non-labelled artificial sweat containing isovaleric acid as lead substance, subjected to olfactometry and evaluated by trained panellists.

Experimental

Fabric Characterization

White fabrics of cotton (CO) and polyester (polyethylene terephthalate, PES) were obtained from Trigema, Burladingen, Germany. The wool fabric (WO) was supplied by WFK Testgewebe GmbH, Brüggen, Germany (wool shrinkage test fabric, reference material). It was not further bleached or finished and thus the colour turned a yellowish white. All textile materials were of comparable structure (1-by-1 rib construction, jersey knit). The natural fibres, cotton and wool, were not further processed after manufacturing. Since polyester is a hydrophobic polymer, it was hydrophilised by a chemical modification with a mixture of cationic and anionic starches.[35] All fabrics were laundered five times with a perfume-free detergent prior to evaluation and finally UV sterilized. Final baseline weights and thicknesses of fabrics were 200 g/m^2 and 870 μm for WO, 140 g/m^2 and 350 μm for CO and 140 g/m^2 and 400 μm for PES. Furthermore, all fabrics were characterized with regard to their moisture management. Water vapour permeability (WVP) was measured according to DIN 53122 (cup method) during 24 h at 25°C and 90% humidity and calculated as WVP (in (g/m^2/24 h) = $\Delta m/A \times 10^4$, where m is mass of absorption medium, and A is area of textile. Water sorption (WS) was examined according to DIN 53923 (Testing of textiles; determination of water absorption of textile fabrics by gravimetric analysis) with artificial sweat instead of water and calculated in % of original dry weight: WS = $(m_{wet} - m_{dry})/m_{dry}$, where m_{wet} and m_{dry} are mass of textile after wetting and mass of dry textile prior to wetting, respectively. All samples were pre-conditioned at 65% relative humidity and 20°C.

β-Cyclodextrin Fabric Finish

All chemicals used were purchased from Sigma-Aldrich (Munich, Germany). For the preparation of β-cyclodextrin finishes, 8 mmol of cyanuric acid were added to 24 ml of pre-cooled deionized H_2O at 5°C under continuous agitation. A solution of 5 mol/l NaOH was added dropwise and the suspension was stirred vigorously for 10 min. β-Cyclodextrin (4.5 g) together with another 5 ml of NaOH solution (5 mM) were added. The suspension was stirred at 10°C until it became slightly yellow due to the formation of monochlortriazinyl-β-cyclodextrin. Deionized water was added to a final volume of 100 ml and 40% (w/w) β-cyclodextrin concentration with a pH of 12. Fabrics were passed twice through a two-roller laboratory padder foulard with a running rate of 450 cm/min and roller pressure of 5 bar and subsequently dried for 60 s at 100°C (PES) or 150°C (CO), respectively, and then weighed. Wool fabric was padded under acidic conditions (pretreated with 10% acetic acid solution) before drying at room temperature without fixation. All samples were rinsed with water and dried again. The final amount of β-cyclodextrin was 1% on CO and PES fabrics and 0.3% for WO. The phenolphthalein method was used to verify a successful β-cyclodextrin finish on the fabrics. Water sorption and water vapour permeability were measured as described above.

Binding Experiments of Fabrics with Radiolabelled ^{14}C-Isovaleric Acid

^{14}C-Radiolabelled isovaleric acid (American Radiolabeled Chemicals, St. Louis, MO, USA) was diluted in an artificial sweat solution according to DIN EN ISO 105-E04 to a final molarity of 9 μM with a resulting radioactive activity of 18 500 Bq/ml and a pH of 6.3. Textile sample swatches, with or without a cyclodextrin finish, stamped into pieces of 4 cm^2, were pre-conditioned at 21°C in an open Petri dish. Subsequently, 50 μl of radiolabelled isovaleric acid was placed onto each sample swatch. After an incubation time of 1, 3 or 20 h at 21°C and 60% relative humidity, swatches were placed into a scintillation vial with 6 ml of deionized water. Rotiszint® eco plus scintillation cocktail (Carl Roth, Karlsruhe, Germany) was added to each vial 8 ml, mixed by gentle shaking and radioactivity was detected in a Hidex 300 SL liquid scintillation counter (Hidex, Turku, Finland).

Artificial Skin

All chemicals used were purchased from Sigma-Aldrich. To investigate sweat odour behaviour on fabrics in a simulated wear situation with human skin *in vitro*, an artificial skin model was used. The skin model, which mimics the physical and chemical properties of human skin, used in the *in vitro* tests, was prepared in accordance with patent US4877454A with minor modifications. In brief, 10 g of porcine skin gelatin, were dissolved in 77 g of water while stirring on a hot plate. To prevent bacterial contamination, propylparabene was dissolved in the resulting warm gelatin, followed by 5 ml NaOH (1 M) and 3 g of 2-(alkoyloxy)-1-[(alkoyloxy) methyl]-ethyl-7-(4-heptyl-5,6-dicarboxy-2-cyclohexane-1-yl)heptanoate.

The skin was stabilized with formaldehyde and allowed to set and dry under ambient conditions. The average thickness of the skin model was ~300 ± 10 µm.

Adhesion Experiments in Diffusion Chambers

Adhesion experiments in a simulated wear situation were conducted in diffusion chamber cells (Franz type). Unidirectional vapour pressure was produced by fixing a 9 cm^2 piece of rehydrated artificial skin onto the lower compartment of the chambers which contained 0.9% NaCl solution kept at 35°C. Subsequently, a 9 cm^2 fabric sample swatch of PES or CO was placed on top of the artificial skin. After closing the diffusion cells with the upper compartment, fabric samples were completely covered by adding 112.5 µl of radiolabelled isovaleric acid. The diffusion cells were incubated for 3 h at 35°C in a water bath. After incubation, the fabrics were each mixed separately with 6 ml deionized water and 8 ml scintillation cocktail (Rotiszint® eco plus). Radioactivity was measured as described above. As a control the undiluted artificial sweat solution was measured. All tests were carried out in duplicates.

Olfactometry

Olfactometric sensory analysis was performed with four trained panellists (trained according to Maxeiner *et al.*[36]) with a four-port olfactometer device Ecoma TO 8 (Odournet, Kiel, Germany). Written informed consent was obtained from all panellists. For the olfactometric experiments a concentrated artificial sweat solution was mixed. In brief, we followed the composition based on Ara *et al.*[32] with modifications (45 µM of isovaleric acid were added as major odour compound for olfactometry). Isovaleric acid (45 µM) was diluted in an acidic buffer solution according to DIN EN ISO 105-E04 and adjusted to pH 6.3. The odour of the resulting composition was rated 'sweat-like' by 10 test persons. After pre-conditioning the fabrics for 24 h at 21°C and 60% humidity, 312.5 µl of artificial sweat was applied onto each 25 cm^2 fabric swatch. Samples were incubated in gas-tight bags at 35°C for 1 h, 3 h and 20 h, respectively, to simulate sweat odour build-up in a permanent wear situation. In this set-up, a closed system without evaporation was used to avoid loss of odorants below the perception threshold. The odour bags were then transferred to the olfactometer device as described above. The determination of odour intensity with dynamic olfactometry, declared as odour units per m^3 (OU/m^3), was carried out following DIN EN 13725. This technique is a standardized method to determine odour concentrations for odour assessment, with an estimated 300 laboratories worldwide using this particular method.[36]

Results and Discussion

Water transfer and absorption properties are crucial parameters for both the microclimate of the skin and sweat odour management of fabrics.[37] Since sweat odour molecules are transferred into fabrics via liquid sweat and water vapour, the wicking properties of all samples towards artificial sweat were determined by measuring water sorption and WVP (Table 1). Although all fabric samples were of similar construction and showed similar WVP, water sorption and wettability differed significantly depending on the fibre type and finish. Untreated wool is known for its low water sorption and bad wettability;[38] as expected, the wool sample displayed the lowest water sorption value. In general, untreated PES is also hydrophobic with a wettability of >600 s.[39] In contrast to that, for our studies we chose a finished, hydrophilised PES material, typically used as first-layer clothing, which displayed a wettability of 118 s. The β-cyclodextrin finish increased WVP of all fabrics, but water sorption was considerably increased in WO, slightly increased in PES, and slightly decreased for CO. Wettability (i.e. the time for absorbing the artificial sweat) of WO was not modulated by the finish, whereas this value increased for CO and decreased for PES. Figure 1 shows representative sweat absorption pictures of the samples.

Retention of ^{14}C-Isovaleric Acid on Fabrics

In this study we followed practical test approaches. Therefore adhesion/dehesion of odour molecules and fibre types was not studied via chemical analysis and solid-phase micro-extraction. Instead, we used liquid scintillation counting to quantify the

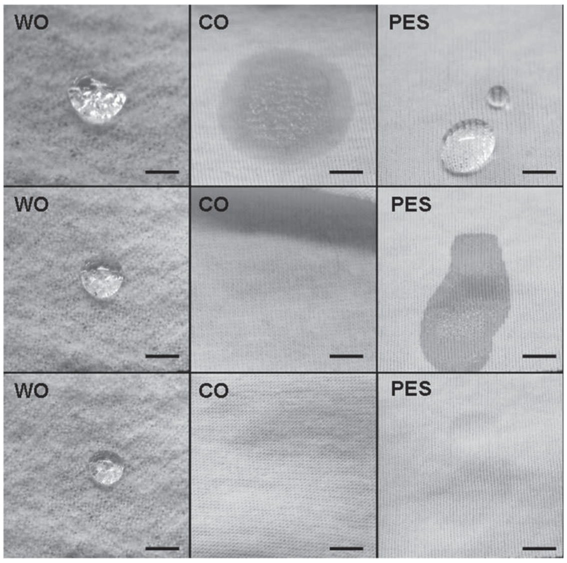

Figure 1. Stereoscopic image of the sorption capacity of wool (WO), cotton (CO) and polyester (PES) loaded with artificial sweat after 1 s (upper row), 2 min (middle row) and 5 h (lower row). After 18 h all fabrics were dry (data not shown). Please note the even distribution of sweat on CO in contrast to the uneven distribution and minor surface area of sweat on PES. Bar = 5 mm

Table 1. Construction, water sorption (WS), water vapour permeability (WVP) and wettability of finished and unfinished fabric samples

Fabric	Construction	WS (%)	WVP (g/m^2/24 h)	Wettability (s)
WO	Jersey knit	1.7	641	>600
CO	Jersey knit (single)	6.3	588	0.1
PES	Jersey knit (interlock)	5.5	611	118
WO + β-CD	Jersey knit	7.1	679	>600
CO + β-CD	Jersey knit (single)	5.2	646	10
PES + β-CD	Jersey knit (interlock)	6.3	654	34

WO, wool fabric; CO, cotton fabric; PES, polyester; CD, cyclodextrin.

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retention of ^{14}C-labelled isovaleric acid with these fibre substrates. Fabrics have long been studied for their thermo-regulatory and skin microclimate properties. If a wearer exceeds the metabolic rate for which the clothing system was designed, they rapidly begin to sweat. The unique water (liquid sweat) and humidity (vaporous sweat) sorption properties are important in thermo-regulation and comfort, but have not yet been addressed in terms of malodour management; hence, sweat odour compounds are transferred and released to a hitherto unknown extent into and out of fabrics via liquid sweat and water vapour. In this study we investigated liquid sweat/fabric/malodour interaction and quantified the amount of isovaleric acid retained within the fabrics by extracting the radioactive labelled compound.

The *in vitro* scintigraphy showed that WO, CO and PES differed in their retention behaviour towards the short fatty acid isovaleric acid at 21°C and 60% relative humidity (Figure 2). One hour after inoculation most of the radiolabelled sweat odour component remained on all swatches, independent of fibre type. After 3 h, 98% of the applied isovaleric acid was retrieved on WO, half of it from CO, and PES was unable to withhold the volatile compound. After 20 h almost all of the odour molecules evaporated from PES.

The results summarized in Figure 2 show that the cyclodextrin finish and fibre types considerably determine the adhesion/dehesion of isovaleric acid on fabrics. Finishing the samples with cyclodextrin enhanced retention of the fatty acid, except for WO, which most likely resulted due to an incomplete and unfixed fabric finish and a resulting minor cyclodextrin concentration (0.3%) on the wool surface (Figure 2B). It can be assumed that the isovaleric acid was almost completely entrapped within the β-cyclodextrin (β-CD) cavities at the fibre surfaces of CO and PES. The differences in the retention of isovaleric acid on the non-finished fabrics are most likely influenced by the water sorption properties of the samples as well as their physico-chemical interactions with isovaleric acid. It is known that CO and PES, for example, differ in swelling of the fibres or in their liquid retention within the pores of a corresponding fabric. In this respect, Liu *et al*. found *cis*-3-hexenyl salicylate[40] and several other aroma compounds[41] distributed throughout the fibre diameter of CO, whereas on PES fibres, with their typical round cross-sectional shape and smooth surface, the compounds were only found sporadically at the fibre surface due to the lower polarity of PES. Cantergiani and Benczedi[42] studied the absorption of fragrance molecules to CO towels by using inverse gas chromatography and dynamic vapour sorption, and attributed the adhesion of fragrance molecules to changes in the physico-chemical properties of the CO fibre, i.e. binding of fragrance molecules to amorphous and microcrystalline phases of CO at elevated humidity. In a comprehensive study, Prada *et al*. carried out headspace analyses of a variety of human hand odour volatiles, focussing on their release from different fabrics, including WO, CO, PES and rayon.[43] The authors found a higher release of

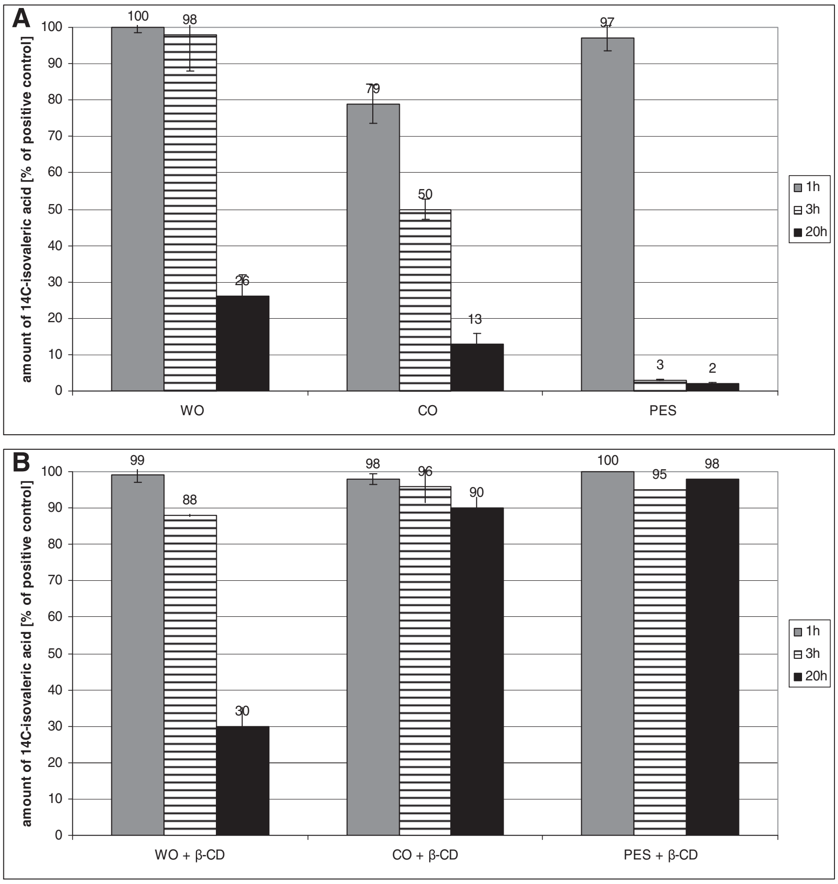

Figure 2. Scintigraphy of the recovery rate of WO, CO and PES samples after 1 h, 3 h and 20 h of incubation with ^{14}C-isovaleric acid. (A) Untreated fabrics; (B) fabrics finished with β-cyclodextrin (β-CD). While the radioactive signal was almost completely present on WO samples after 3 h, it decreased over time on CO and rapidly on PES. The cyclodextrin finish significantly enhanced the retention of isovaleric acid on CO and PES

carboxylic acids from PES than from CO. Although we followed a different methodological approach, studying the odour retention capacity of common fibres towards isovaleric acid, this is in line with our findings of less retention of this lead sweat substance by PES and a greater retention by CO (Figure 2).

Cotton fibres are known to be hydrophilic and are therefore able to absorb vapour, which makes CO an ideal moisture buffer system for the skin microclimate.[44,45] Cantergiani and Benczedi[42] also characterized the interaction with fragrance molecules at different relative humidities with CO fabrics and found that CO absorbed 16% of water at 100% relative humidity, whereas McQueen *et al*. compared WO, CO and PES at 65% relative humidity and found values of 7.5% for CO, 0.6% for PES and 14% for WO of moisture regain.[46] Given the correlation of a high moisture regain and the retention capacity for sweat odour and isovaleric acid as shown in this study, CO seems to be beneficial for odour management of first-layer fabrics. Taking into account that sport garments are in close contact with the skin and that exercise-induced sweating occurs (perspiratio sensibilis), it seems obvious that for first-layer fabrics the odour interaction of molecules with fibres seems to be dominated by wicking properties, i.e. direct uptake of liquid sweat, rather than vapour absorption. However, one should be aware that, in our study, the retention of sweat odour via water vapour uptake was not investigated. Since WO, especially, is known to lead to greater amounts of water vapour inside of the fibre, it might be favourable for non-liquid vaporous sweat (perspiratio insensibilis), e.g. in business activities. To clarify this, it will be necessary to run the retention experiments with all samples with radiolabelled odoriferous molecules, including isovaleric acid, in a future experimental set-up that focusses on vapour uptake. Obendorf *et al*., for example, has already shown the existence of an inverse relationship between vapour pressure of odour substances and chemical retention on fabrics.[47]

In contrast to the unfinished wool test fabric used here with its intact hydrophobic cuticle, commonly available wool garments worn next to the skin are generally less hydrophobic, since they run through a couple of finishing and colouring treatments, hence any non-test fabrics are hard to compare. That implies that, in practice, the retention of isovaleric acid by wool garments and, consequently, its odour emission may greatly differ from our sample. The high recovery value for isovaleric acid on WO after 3 h of incubation can be traced back to the bad wettability of this material, since the drop-like remaining of the inoculum on the wool surface still showed the lowest surface area for evaporation of the fatty acid (Figure 1). Obviously, any diffusion of isovaleric acid from artificial sweat solution into the knitwear is hindered by the hydrophobic lipid layers on the wool fibre cuticle. Although from their physicochemical structure, isovaleric acid as well as fatty acids could possibly bind through ionic interactions to basic amino acids and disulfide bridges on wool fibres,[48] the hydrophobic cuticle effect apparently had a stronger impact on sweat odour evaporation.

Impact of Skin Vapour Pressure on the Retention of Isovaleric Acid on Cotton Fabric and Polyester

Local influences of clothing on sweat odour release were studied by Otomasu *et al*.,[49] who used the hygrometer-based ventilated sweat capsule method to analyse sweat evaporation from clothing materials. Although they found that clothing that was in full contact with skin underwent more relative humidity due to transepidermal water loss (TEWL) than clothing that had partial or no contact, it is unknown, so far, how skin parameters, e.g. the unidirectional vapour pressure of the TEWL, or the temperature gradient between skin surface and the ambient temperature, modulate the release of sweat odour from fabrics. We therefore addressed this question in a simulated wear situation and ran the isovaleric acid retention experiments with a standardised artificial skin model. The artificial skin model showed human skin-like composition, topology and mechanical characteristics and allowed control of the parameter skin, i.e. its temperature (34–35°C), texture and the unidirectional TEWL. Previous experiments in our laboratory revealed the artificial skin to have a mean TEWL of 20 g/h m^2 at 35°C, which is comparable to the data of Mayrovitz *et al*. for human hand or underarm skin.[50] As expected, skin temperature and increased vapour pressure enhanced the sweat evaporation in the simulated wear situation. After 3 h of incubation, the recovery rates for the radiolabelled compound in this open system was cut to 9% for CO and 5% for the hydrophilic PES fabric, thus indicating the enhanced dehesion of isovaleric acid under local skin influence. Thus, unidirectional transepidermal water vapour release of the skin model modulated the odour retention.

Sensory Analysis Using Olfactometry

In a further set of experiments we decided to investigate whether sweat perception might also be affected by increased temperature and water vapour pressure. To confirm this, we ran a sensory analysis with panellists and a four-port olfactometer. Fabric samples were inoculated with an extended artificial sweat containing at least one component of each class of sweat odour and isovaleric acid as lead malodour substance.

In these experiments the panellists evaluated little sweat odour evaporation for unfinished WO (most likely due to its hydrophobicity and small surface area of the sweat drop), medium odour intensity for CO and a burst in odour perception for the hydrophilic PES after 3 h emitting from the sealed bags. During the following 17 h odour release from CO nearly doubled, whereas sweat evaporation of WO did not change considerably. In this respect, the sweat evaporation data of the sensory evaluation test are in record with the recovery experiments using radiolabelled isovaleric acid (Figure 2). Apparently, less retention of isovaleric acid on unfinished fabrics correlates with increased sweat odour perception. In contrast to this, CO and PES samples finished with β-cyclodextrin retained higher amounts of isovaleric acid (Figure 2) and consequently emitted less odour (Figure 3). Unfortunately, it was impossible to evaluate the cyclodextrin effect of the finished WO, due to an insufficient treatment (as already described).

A few studies so far have addressed the question of odour intensity of fabrics. In field tests where cotton was compared to PES for sweat odour intensity, McQueen *et al*.[13,14] and Munk *et al*.[11,12] both also found PES to be more odorous than cotton. Our results should be interpreted carefully on the basis of the sweat sorption capacities data of our samples (Table 1 and Figure 2), where CO displayed an even distribution of sweat which finally led to a high surface area for sweat evaporation, due to the hygroscopic nature of this fibre type. Although the hydrophilic finished PES is less hygroscopic than CO (Table 1), we observed that the release of sweat odour from PES was much quicker. One explanation for this finding could be that, because of their high crystallinity,[51] polyester fabrics lack moisture retention capacities compared to CO. In our hands the artificial

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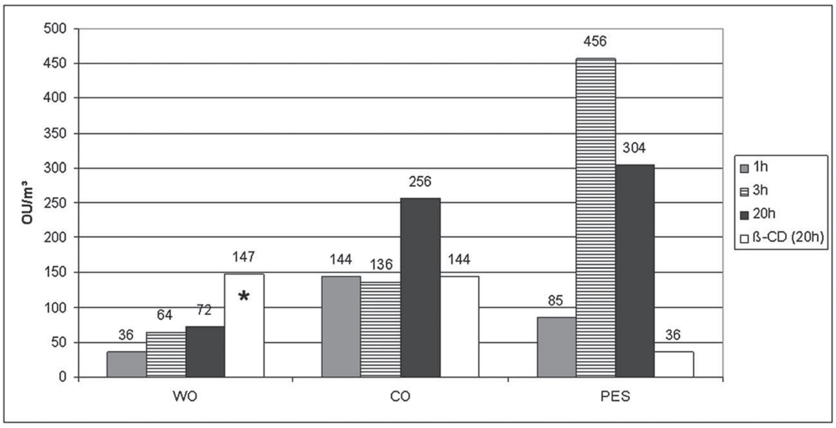

Figure 3. Sensory evaluation by olfactometry with four panellists. WO, CO and PES were inoculated with artificial sweat after 1 h, 3 h and 20 h of incubation in gas-tight bags. Note the increase in odour intensity of PES between 1 h and 3 h, indicated as odour units (OU) per m^3. *Evaluation not valid due to insufficient cyclodextrin finish on wool

sweat evaporated from PES almost completely during the first 3 h, which is reflected in the outcome of the olfactometry analysis, where we found a stronger odour on PES after 3 h and 20 h compared to the odour intensity after 1 h. Since for the sensory evaluation tests the sample swatches were inoculated with artificial sweat and were then kept in gas-tight bags, we found that the odour molecules evaporated during the first 3 h and remained in the bags until sensory evaluation after 3 h or after 20 h, respectively. We assume a slight adhesion of evaporated isovaleric acid to the nalophan (polyethylene terephthalate) material of the bags during 20 h of incubation which might explain the lower odour intensity compared to the sensory evaluation after 3 h. Apart from isovaleric acid we did not include other important contributors to sweat malodour, such as volatile sulfur compounds or odoriferous steroids. Based on our data on isovaleric acid, it can be assumed that the retention of this fatty acid resembles in its fibre interactions other odoriferous carboxylic acids, e.g. butyric acid, isobutyric acid, (*E*)-3-methyl-2-hexenoic acid (3M2H)[21] or 3-hydroxy-3-methyl hexanoic acid (HMHA). Unfortunately, in their study, Prada *et al.*[43] did not investigate the functional groups of sulfur compounds or steroids, but they included alcohols. Since the sulfur odour compound (*S*)-3-methyl-3-sulfanylhexan-1-ol contains at least a functional hydroxyl and thiol group,[20] and may thus behave similarly in its adhesion to fibres as the alcohols used by Prada, it is tempting to extrapolate a minor retention of (*S*)-3-methyl-3-sulfanylhexan-1-ol by PES fibres, and an increased retention by WO and CO. Munk *et al.* found lipophilic compounds such as steroids to have greater adhesion to PES than to CO.[12] Nevertheless, from our point of view, it is difficult to predict retention or adhesion of odoriferous steroids by PES, CO or WO, due to the variety of functional groups (ketones, hydroxyl groups) of these molecules, their cyclic structure and molecular mass. It is conceded that further investigations are required to generate relevant information on the physico-chemical affinity of odour substances other than isovaleric acid to fabric fibres, and to study them in more detail with respect to fibre composition, surface and fabric construction.

Conclusion

Our findings support the view that the adhesion/dehesion of sweat odour from fabrics is a complex topic due to a variety of influencing parameters. Sweat odour retention and evaporation depend on factors such as chemical structure of the odorants and fibre type, fibre finish, moisture sorption of the fabrics, construction and local influences of the skin, e.g. temperature and TEWL. Nevertheless, the data and methods presented here provide a good basis for future optimization of clothes in terms of sweat odour management in their respective end uses, e.g. sport or business activities. It can be concluded that a smart fibre choice and odour modulating finishes will have the greatest impact on moisture and malodour management.

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References

1. K. Sato, R. Leidal, F. Sato. *Am. J. Physiol.* **1987**, *252*, R166.
2. W. B. Shelley, H. J. Hurley Jr, A. C. Nichols. *AMA Arch. Derm. Syphilol.* **1953**, *68*, 430.
3. A. Benohanian, *Clin. Dermatol.* **2001**, *19*, 398.
4. S. Freeman, *Contact Dermatitis* **1997**, *36*, 247.
5. Y. Gao, R. Cranston. *Textile Res. J.* **2008**, *78*, 60.
6. U. Girrbach. *Int. Textile Bull.* **2003**, *2*, 34.
7. B. Martel, M. Morcellet, D. Ruffin, F. Vinet, L. Weltrowski. *J. Inclusion Phenom. Macrocyclic Chem.* **2002**, *44*, 439.
8. U. R. Bhaskara-Amrit, P. B. Argrawal, M. M. C. G. Warmoeskerke. *Autex Res. J.* **2011**, 11.
9. W. W. Volz. *Textilveredlung* **2003**, *11/12*, 17.
10. D. Höfer. *Curr. Probl. Dermatol.* **2006**, *33*, 67.
11. S. Munk, P. Munch, L. Stahnke, J. Adler-Nissen, P. Schieberle. *J. Surfactants Detergents* **2000**, *3*, 505.
12. S. Munk, C. Johansen, L. H. Stahnke, J. Adler-Nissen. *J. Surfactants Detergents* **2001**, *4*, 385.
13. R. H. McQueen, R. M. Laing, C. M. Delahunty, H. J. L. Brooks, B. E. Niven. *J. Textile Inst.* **2008**, *99*, 515.
14. R. McQueen, R. M. Laing, H. J. L. Brooks, B. E. Niven. *Textile Res. J.* **2007**, *77*, 449.
15. J. J. Leyden, K. J. McGinley, E. Holzle, J. N. Labows, A. M. Kligman. *J. Invest. Dermatol.* **1981**, *77*, 413.
16. N. Shehadeh, A. M. Kligman. *J. Invest. Dermatol.* **1963**, *41*, 39.
17. D. Taylor, A. Daulby, S. Grimshaw, G. James, J. Mercer, S. Vaziri. *Int. J. Cosmet. Sci.* **2003**, *25*, 137.
18. W. E. Kloos, M. S. Musselwhite. *Appl. Microbiol.* **1975**, *30*, 381.
19. J. Marshall, K. T. Holland, E. M. Gribbon. *J. Appl. Bacteriol.* **1988**, *65*, 61.
20. M. Troccaz, C. Starkenmann, Y. Niclass, M. van de Waal, A. J. Clark. *Chem. Biodivers.* **2004**, *1*, 1022.

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21. X.-N. Zeng, J. J. Leyden, H. J. Lawley, K. Sawano, I. Nohara, G. Preti. *J. Chem. Ecol.* **1991**, *17*, 1469.
22. A. Natsch, H. Gfeller, P. Gygax, J. Schmid. *Int. J. Cosmet. Sci.* **2005**, *27*, 115.
23. B. W. Brooksbank, R. Brown, J. A. Gustafsson. *Experientia* **1974**, *30*, 864.
24. J. E. Amoore. *Chem. Sens. Flav.* **1977**, *2*, 267.
25. A. Natsch, J. Schmid, F. Flachsmann. *Chem. Biodivers.* **2004**, *1*, 1058.
26. J. Labows, C. Wysocki. *Perfumer Flavorist* **1984**, *9*, 21.
27. J. N. Labows, K. J. McGinley, A. M. Kligman. *J. Soc. Cosmet. Chem.* **1982**, *33*, 193.
28. D. B. Gower, B. A. Ruparelia. *J. Endocrinol.* **1993**, *137*, 167.
29. G. Ohloff. In *Progress in the Chemistry of Organic Natural Products*, vol. 35. Springer: Vienna, **1978**; 431.
30. R. G. Buttery, R. Teranishi, L. C. Ling, J. G. Turnbaugh. *J. Agric. Food Chem.* **1990**, *38*, 336.
31. F. A. Fazzalari. *In ASTM Data Series DS 48A*. American Society for Testing and Materials: Philadelphia, **1978**; 497.
32. K. Ara, M. Hama, S. Akiba, K. Koike, K. Okisaka, T. Hagura, T. Kamiya, F. Tomita. *Can. J. Microbiol.* **2006**, *52*, 357.
33. Y. Hasegawa, M. Yabuki, M. Matsukane. *Chem. Biodivers.* **2004**, *1*, 2042.
34. A. Caroprese, S. Gabbanini, C. Beltramini, E. Lucchi, L. Valgimigli. *Skin Res. Technol.* **2009**, *15*, 503.
35. R. Samu, A. Moulee, V. G. Kumar. *J. Colloid Interface Sci.* **1999**, *220*, 260.
36. B. Maxeiner, J. Ennen, S. Rutzel-Grunberg, B. Traupe, K. P. Wittern, R. Schmucker, R. Keyhani. *Int. J. Cosmet. Sci.* **2009**, *31*, 193.
37. X. Q. Dai, R. Imamura, G. L. Liu, F. P. Zhou. *Eur. J. Appl. Physiol.* **2008**, *104*, 337.
38. C. Wang, Y. Qiu. *J. Appl. Polym. Sci.* **2012**, *123*, 1000.
39. Y. Boguslavsky, T. Fadida, Y. Talyosef, J.-P. Lellouche. *J. Mater. Chem.* **2011**, *21*, 10304.
40. H. Liu, S. K. Obendorf, T. J. Young, M. J. Incorvia. *J. Appl. Polym. Sci.* **2004**, *91*, 3557.
41. H. Liu, S. K. Obendorf, M. J. Leonard, T. J. Young, M. J. Incorvia. *J. Surfactants Detergents* **2005**, *8*, 311.
42. E. Cantergiani, D. Benczedi. *J. Chromatogr. A* **2002**, *969*, 103.
43. P. A. Prada, A. M. Curran, K. G. Furton. *J. Forensic Sci.* **2011**, *56*, 866.
44. T. Yasuda, M. Miyama, H. Yasuda. *Textile Res. J.* **1992**, *62*, 227.
45. L. Benisek, P. R. Harnett, M. J. Palin. *Melliand English* **1987**, *12/1987*, 878.
46. W. E. Morton, J. W. S. Hearle, *Physical Properties of Textile Fibres* 3rd edition. Textile Institute: Manchester, **1993**.
47. S. K. Obendorf, H. Liu, M. J. Leonard, T. J. Young, M. J. Incorvia. *J. Appl. Polym. Sci.* **2006**, *99*, 1720.
48. W. Xu, G. Ke, J. Wu, X. Wang. *Eur. Polym. J.* **2006**, *42*, 2168.
49. K. Otomasu, M. Yamauchi, N. Ohwatari, T. Matsumoto, K. Tsuchiya, M. Kosaka. *Eur. J. Appl. Physiol. Occup. Physiol.* **1997**, *76*, 1.
50. H. N. Mayrovitz, M. Bernal, F. Brlit, R. Desfor. *Skin Res. Technol.* **2012**. epub ahead of print (DOI: 10.1111/srt.12000)
51. H. K. Rouette, M. Peter, *Handbuch Textilveredlung*, Deutscher Fachverlag, **2006**.

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