What smell can tell: using Magic Chemisorbers and GC/MS to capture and analyse volatiles off-gassing from plastic coatings

O que o odor pode dizer: utilização de *Magic Chemisorbers* e GC/MS para capturar e analisar os gases voláteis libertados pelos revestimentos plásticos

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Abstract

Odour that may be released when opening packaging of plastic objects is often a concern for collection managers. Volatile compounds emitted by three separate plastic-coated textile objects from Centraal Museum Utrecht were identified. Samples are analysed with Fourier Transform Infrared Spectroscopy (FTIR) and (pyrolysis) Gas Chromatography Mass Spectrometry (GC/MS). Polar and apolar Magic Chemisorbers are used to trap volatile organic compounds (VOCs) that the plastic coatings are off-gassing. Two of the objects have a plasticised polyvinylchloride (PVC-P) coating giving off a faint chemically sweet smell, where the DEHP plasticiser is detected as the main off-gassing compound. The third object has a polyurethane (PUR) ester coating which has a very rancid smell, most likely a result of the off-gassing of butanoic acid and naphthalene derivatives. This pilot study shows that polar (PEG) and apolar (PDMS) Magic Chemisorbers are useful sorbents to trap compounds that are off-gassing from plastic coatings.

KEYWORDS
Smell
Volatile organic compounds (VOCs)
Plastic coatings
Magic Chemisorbers
Off-gassing

Resumo

Muitas vezes, o odor libertado durante a abertura de embalagens com objetos de plástico é uma preocupação para os gestores de coleções. Foram identificados compostos voláteis emitidos por três objetos têxteis com revestimentos plásticos do Centraal Museum Utrecht. As amostras foram analisadas por FTIR e (py-) GC/MS. *Magic Chemisorbers*, quimiossorventes polares e apolares, foram utilizados para capturar os compostos orgânicos voláteis (VOCs) libertados pelos revestimentos. Dois dos objetos têm um revestimento de poli(cloreto de vinilo) plastificado (PVC-P) que liberta um odor ligeiramente adocicado e o plastificante DEHP foi detetado como o principal composto gasoso a ser libertado. O terceiro objeto tem um revestimento de poliuretano (PUR) à base de éster e liberta um odor rançoso, provavelmente resultante da libertação de ácido butanóico e derivados de naftaleno. Este estudo piloto mostra que os quimiossorventes *Magic Chemisorbers* polares (PEG) e apolares (PDMS) são sorventes úteis para capturar os compostos gasosos libertados por estes revestimentos plásticos.

PALAVRAS-CHAVE
Odor
Compostos orgânicos voláteis (VOCs)
Revestimentos plásticos
Magic Chemisorbers
Libertação de gases

Introduction

The odour that may be released when opening packaging of plastic objects is a concern for collection managers. What is that smell? Does the smell say something about the type of plastic, its condition, the possible effect on materials in the immediate environment or the effect on people working in the museum? There is a growing number of plastic objects in museum collections, subsequently the notion that certain plastics can off-gas particular smells increases. This results in more inquiries at the Cultural Heritage Agency of the Netherlands (RCE) about the nature of smell released from plastics.

Throughout *Project Plastics* a collection survey was carried out in 2019 at the Centraal Museum Utrecht, one of the partners in the project. During the survey days, fashion objects ranging from shoes, hats, dresses, accessories, including plastic-coated textiles releasing strong odours were examined. The odorous objects partly inspired the idea for the *Smelly Plastics* project, as follow-up on *Project Plastics* [1-3]. Project *Smelly Plastics* aims to develop a method for sampling and analysis of a wide range of volatile organic compounds (VOCs) off-gassing from plastic museum objects.

When a plastic degrades, the polymer chain is oxidised or hydrolysed and disintegrates into small, often polar, molecules. It is to be expected that degrading plastics will emit acids, aldehydes, ketones, and alcohols [4]. The assumption is made that additives to a plastic, like plasticisers, antioxidants, and/or UV-stabilisers, can evaporate together with volatiles originating from the degradation of the plastic. The smell of a plastic is defined as a mixture of volatile and less volatile, polar and apolar compounds.

For the analysis and identification of volatiles in air, Gas Chromatography Mass Spectrometry (GC/MS) is the method of choice [4-7]. In contrast to previous studies regarding the off-gassing of VOCs from plastics, the starting point of this study is the concern of museum employees about the smell of plastic objects. Research has been conducted into the source and cause of these odours.

Materials and methods

The focus of project *Smelly Plastics* is on plastic artworks and objects present in modern and contemporary art collections. For this research the smell of three plastic-coated textiles from the collection of Centraal Museum Utrecht have been examined (Figure 1).

Figure 1. Three modern, plastic-coated textiles from the collection of Centraal Museum Utrecht: *a)* Girl's raincoat (1972) by Pennel et Flipo; *b)* Children's raincoat (1974) by Barbara Farber; *c)* Woman's ensemble *L'apparence du Vide* (1995) by Viktor & Rolf.

Of the investigated garments the first two are raincoats: a girl's raincoat with a cherry design from Pennel et Flipo dating from 1972 (Figure 1a) and a green children's raincoat from Barbara Farber dating from 1974 (Figure 1b). Both coats have a plastic coating which makes them water repellent. They are stored on a clothes hanger protected with tissue paper.

The third garment is a gold-coloured woman's ensemble named *L'apparence du vide* (1995) by the Dutch fashion house Viktor & Rolf (Figure 1c). It is one of the five garments from Viktor & Rolfs Spring/Summer 1996 collection, which was first shown at a contemporary art gallery in Paris in October 1995. They were presented in the form of an installation, without the use of any models, titled *The appearance of emptiness*. Golden dresses were suspended in the gallery space. On the floor lay black garments made of organza, like shadows. The names of supermodels were written across one wall, while voices on the soundtrack playing in the gallery whispered the names of these supermodels. The supermodel boom had become something of a social phenomenon at the time, causing a huge rise in the fees paid to such models [8]. More attention was often paid to the supermodels themselves than to the designer's collection. Viktor & Rolf were irritated by this situation so they devised a plan to restore the rights of clothes in fashion. Although the golden dresses are three-dimensional, there are no heads or limbs, they are clothes with a shiny surface but no substance, and suspended in the air, they symbolize the emptiness of the fashion world that was in a frenzy over supermodels. The suit is wrapped in acid free tissue paper and stored in an acid free box.

Both sides of the fabric of the three garments were examined. Samples taken were analysed by Fourier Transform Infrared Spectroscopy (FTIR) and pyrolysis Gas Chromatography Mass Spectrometry (py-GC/MS) to determine the material composition of the garments.

FTIR spectral data are collected on a Perkin Elmer Spectrum 100 FTIR spectrometer combined with a Specac Golden Gate Single Reflection Diamond ATR. Taken samples of both sides of the fabric were scanned with 16 scans from 4000 to 450 cm^{-1}. Obtained spectra were compared to a compiled library with plastic reference spectra.

The GC/MS instrument available for both material identification and analysing volatiles emitted by plastics (VOCs) is a Thermo Scientific GC/ISQ-7000 mass spectrometer combination equipped with the Frontier Laboratories 3030D Pyrolyzer and the MicroJet Cryo-Trap 1030Ex. The pyrolyzer is septum-less connected to the GC-column. A split exit is constructed at the base of the pyrolyzer, the carrier and split flows are regulated by the GC [9].

The Frontier Pyrolyzer is used for the material identification of the plastic coating by pyrolysis GC/MS (py-GC/MS), for the analysis of volatiles emitted by the plastics, with material thermal desorption (TD-GC/MS), and for the desorption of VOCs trapped on the Magic Chemisorbers.

For the material identification, type of column and GC/MS settings used are according [9]. The pyrolyzer temperature, for this research, was set at 550 °C.

To analyse VOCs in air, passive sampling with solid phase extraction (SPE) and GC/MS identification were chosen, with the advantage of direct transfer of the captured substances to the inlet of the GC without sample loss due to extraction [10]. With solid phase extraction, VOCs are captured in an activated layer, a sorbent applied on a carrier. The sorbent is subsequently desorbed in the heated inlet of the GC. SPE can be selective and competitive in their sorption of analytes, hence, given the polarity of the expected VOC mixture, it was decided to use both a polar and an apolar coated SPE device.

The capture of VOCs is performed by the recently developed Magic Chemisorber Solid Phase Extraction Elements by Frontier Laboratories Ltd, Japan. They are specially designed for use in their pyrolyzer [11]. Magic Chemisorbers are available in two varieties: polar, de-activated steel coated with polyethylene glycol (PEG matrix) and apolar, titanium coated with polydimethylsiloxane (PDMS matrix). For sampling and analysis of VOCs, the small Magic Chemisorbers are mounted on a (stainless steel) Eco-stick (Figure 2), also necessary to desorb the chemisorbers in the pyrolyzer. The Magic Chemisorbers are cleaned in advance before each sampling by heating them in the pyrolyzer, under an inert atmosphere, for ten minutes at 200 °C (polar chemisorber) and 280 °C (apolar chemisorber). After cleaning, the chemisorbers, mounted on the Eco-stick, are stored in separate sealed vials under an inert atmosphere. To the authors knowledge, this is the first study that uses Magic Chemisorbers in the cultural heritage field to capture the off-gassing compounds released from plastic coatings.

The column used for the analyses of the VOCs is the ZB-5ms (Phenomenex, 7HG-G010-22), an apolar column with a length of 30 m, a diameter of 0.25 mm and a film thickness 1.0 µm. The VOCs obtained are cryo-trapped at -160°C for five minutes at the head of the column prior to analysis. The GC/MS settings used are, helium as carrier gas with a constant flow of 1.4 ml/min, the temperature of the GC inlet is set at 290 °C, the split ratio is set to 1:7. The temperature program used is 80 °C, stable for three minutes, subsequently raised to 200 °C with a rate of 10 °C/min, and to 290 °C with a rate of 6 °C/min, kept stable for three minutes. The temperature of the MS interface is 270 °C, and the temperature of the ion source is set at 250 °C. Mass spectra are recorded from 10 amu until 600 amu with a speed of 5 scans per second.

Direct analysis (TD-GC/MS) of samples of the plastic-coatings was performed, to gain insight about the nature of compounds emitted by the plastics and to verify the results gained with the Magic Chemisorbers. For this, a small sample (app. 10 µg) is placed in a stainless-steel Eco-cup and inserted in the pyrolyzer at 110 °C and kept at that temperature for five minutes. Decomposition of plastics is not expected at 110 °C.

For sampling of the VOCs in air, the object was (partially) covered by a glass bell jar to create a headspace for capturing the compounds emitted by the object. The VOCs are sampled in the immediate vicinity of the artwork, without any direct contact with the SPE devices that are inserted into a piece of polystyrene foam [12]. For analysis of the captured VOCs, the capture elements are thermally desorbed in the heated furnace of the Frontier 3030D pyrolyzer; the polar ones were desorbed at 200 °C, the apolar ones at 280 °C. Desorption time was set to five minutes. Figure 2 depicts the steps in this process. Blank measurements are carried out without the presence of an object under the bell jar and with the presence of the SPE devices in the polystyrene foam mount.

Xcalibur (4.2, Thermo Fisher Scientific) software is used to record the mass spectral data. Amdis 4.71 in combination with a Microsoft Office Excel template is used to identify, sort, and report the results of the analyses [13]. AMDIS (Automated Mass Spectral Deconvolution and Identification System) is a program developed by the National Institute of Standards and Technology (NIST) for the purpose of compound identification in GC/MS [14].

Figure 3 shows the setup in the depot of the Centraal Museum Utrecht for capturing the VOCs emitted by the three garments. Part of the scarf from the Viktor & Rolf women's ensemble is placed under a bell jar together with the Magic Chemisorbers. To prevent the edge of the bell jar from coming into contact with the garment, the garment is covered with tissue paper. To sample the Pennel et Flipo rain jacket, the bell jar was placed directly over one of the sleeves. As there were only two bell jars available, the Barbara Farber children's raincoat was sampled by placing the Magic Chemisorbers in one of the pockets. The time to capture VOCs was set to two hours.

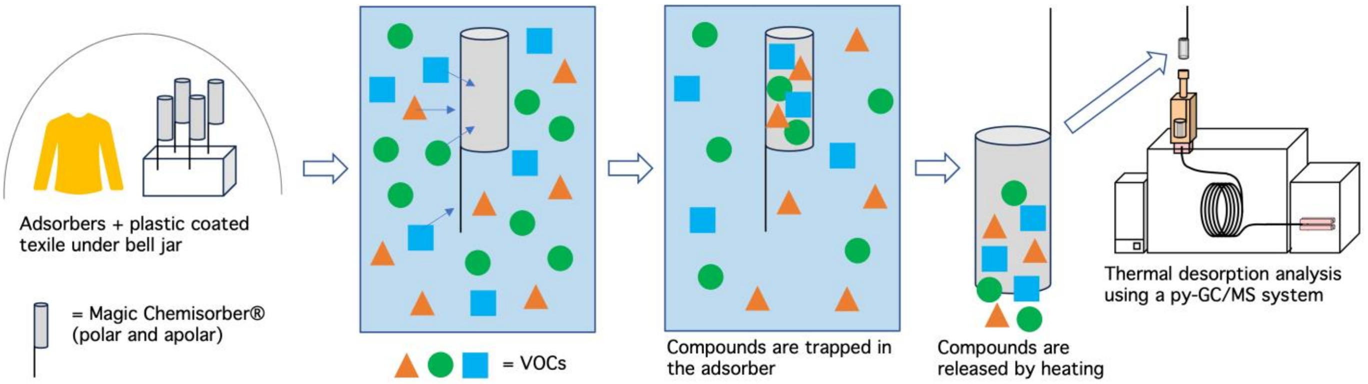

Figure 2. Schematic depiction of the method to capture and analyse VOCs near plastic coated garments.

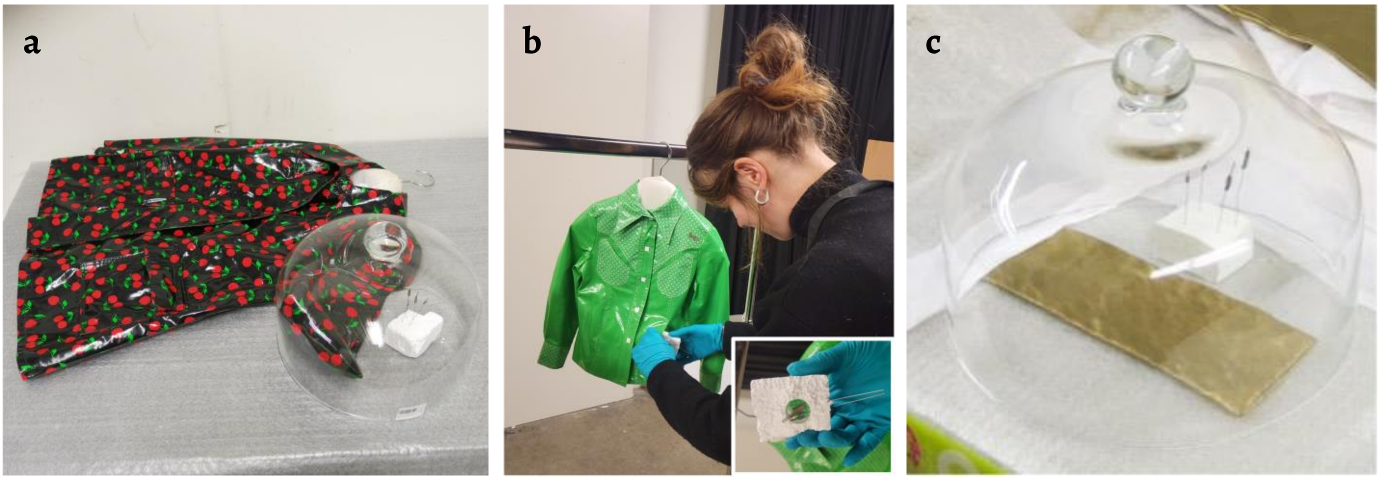

Figure 3. Sampling of the compounds in the air by the Magic Chemisorbers: *a)* Girl's raincoat (1972); *b)* Children's raincoat (1974); *c)* Woman's ensemble *L'apparence du Vide*.

As the project progressed it became clear that more explicit results were achieved if the object was sampled under the bell jar for 72 hours, instead of two hours as used in the first series. This is because the longer sampling time allows more accumulation of evaporating volatiles in the bell jar headspace. It was therefore decided to reanalyse the smelliest garment, *L'apparence du vide* by Viktor & Rolf, with a sampling time of 72 hours.

Results

The obtained results from the analyses of the material samples and captured volatiles are described below. For a clearer representation and interpretation of the volatile compounds emitted by the objects, the results of the polar and apolar chemisorber are presented combined. The analysis of the blank measurements showed that the polystyrene mount did not emit or adsorb VOCs.

Raincoats by Pennel et Flipo, and Barbara Farber

The coats date from the 70s and they both appear in good condition: there are no signs of degradation on the surface and no blooming or weeping is visible. The coats have a slight sweet smell, vaguely reminiscent of cheap inflatables, like swimming rings and beach balls.

FTIR analysis of the material of both raincoats, the girl's raincoat from Pennel et Flipo and the children's raincoat from Barbara Farber, show that the raincoats are very similar in terms of material composition. The presence of bands at 1430 cm^{-1} (CH_2-Cl angular deformation), 1260 cm^{-1} (CH-Cl out of plane angular deformation), 960 cm^{-1} (C-H out of plane trans deformation) and 875 cm^{-1}, 610 cm^{-1} (both C-Cl stretching) allows identification of Polyvinyl Chloride. The addition of a large band at 1724 cm^{-1} (C=O stretching), is due to the presence of plasticiser [15]. Therefore both coatings consist of plasticised Polyvinyl Chloride (PVC-P). In addition, the backing shows a broad band around 3330 cm^{-1} (O-H stretching), 2895 cm^{-1} (C-H stretching), 1029 cm^{-1} (C-O stretching and O-H deformation), and 895 cm^{-1} (linkage between monosaccharides) indicating cellulose (cotton), as stated on each jackets' label [16].

The py-GC/MS analysis of the material of the raincoats shows that the main plasticiser is di(2-ethylhexyl)phthalate (DEHP). This is a widely used plasticiser for PVC-P [17]. In both raincoats plasticisers diisobutyl phthalate (DIBP) and dibutyl phthalate (DBP) are present in trace amounts.

The material thermal desorption analysis (TD-GC/MS) of samples taken from the raincoats, show DEHP plasticiser as main off-gassing compound. Traces of di-isobutyl phthalate (DIBP) and dibutyl phthalate (DBP) were also detected.

The combined results of the polar and apolar Magic Chemisorbers shows the plasticisers DEHP, DIBP and DBP.

***L'apparence du vide* by Viktor & Rolf**

The gold-coloured coating has a very rancid smell and excretes a liquid drenching the tissue paper it is wrapped in. First issues were detected in autumn 2018 after which the collection manager decided to replace the tissue paper. However, during the survey in January 2019, which was only a few months later, the tissue paper had absorbed even more liquid.

The coating (outside) on the Viktor & Rolf garment is characterised with FTIR as a polyurethane ester (PUR ester) due to the presence of the bands at 3294 cm^{-1} (N-H stretching), 1728 cm^{-1} (C=O stretching), 1534 cm^{-1} (N-H deformation), 1231 cm^{-1} and 1173 cm^{-1} (C-N stretching) and 1064 cm^{-1} (C-O-C stretching) [18].The substrate (inside) is characterised as nylon due to the specific amide bands. At 3295 cm^{-1} (N-H stretching), 1635 cm^{-1} (amide I), 1538 cm^{-1} (amide II), and 1261 cm^{-1} (amide III) [19].

Using py-GC/MS, the polyurethane coating is identified as an aromatic PUR ester, consisting of toluene diisocyanate (TDI) and methylene diphenyl diisocyanate (MDI) as the isocyanate building blocks, and polyethylene glycol and polypropylene glycol compounds as the polyol building blocks. Toluenediamine (TDA) and methylene diphenyl diamine (MDA), decomposition products of TDI and MDI, are also detected.

The material thermal desorption analysis (TD-GC/MS) of a sample of the Viktor & Rolf garment shows diethylene glycol, and a compound related to the polyol part of the polyurethane ester, consisting of dimers and trimers of adipic acid and glycol fragments (poly-adipic-glycol compound), as main off-gassing compounds. It also shows the presence of diisopropylnaphthalene-isomers.

The results of the compounds captured by polar and apolar Magic Chemisorbers show diethylene glycol and the poly-adipic-glycol compound as main compounds. Furthermore, butanoic acid and diisopropylnaphthalene-isomers have been captured.

Figure 4 depicts the chromatograms of the material thermal desorption analysis (Figure 4a) and compounds captured by the polar Magic Chemisorber (Figure 4b), the latter is chosen because all main emitted compounds are captured by this type of sorbent. Table 1 presents the results of the main compounds emitted by *L'apparence du vide* using material thermal desorption, and captured with both polar and apolar Magic Chemisorbers.

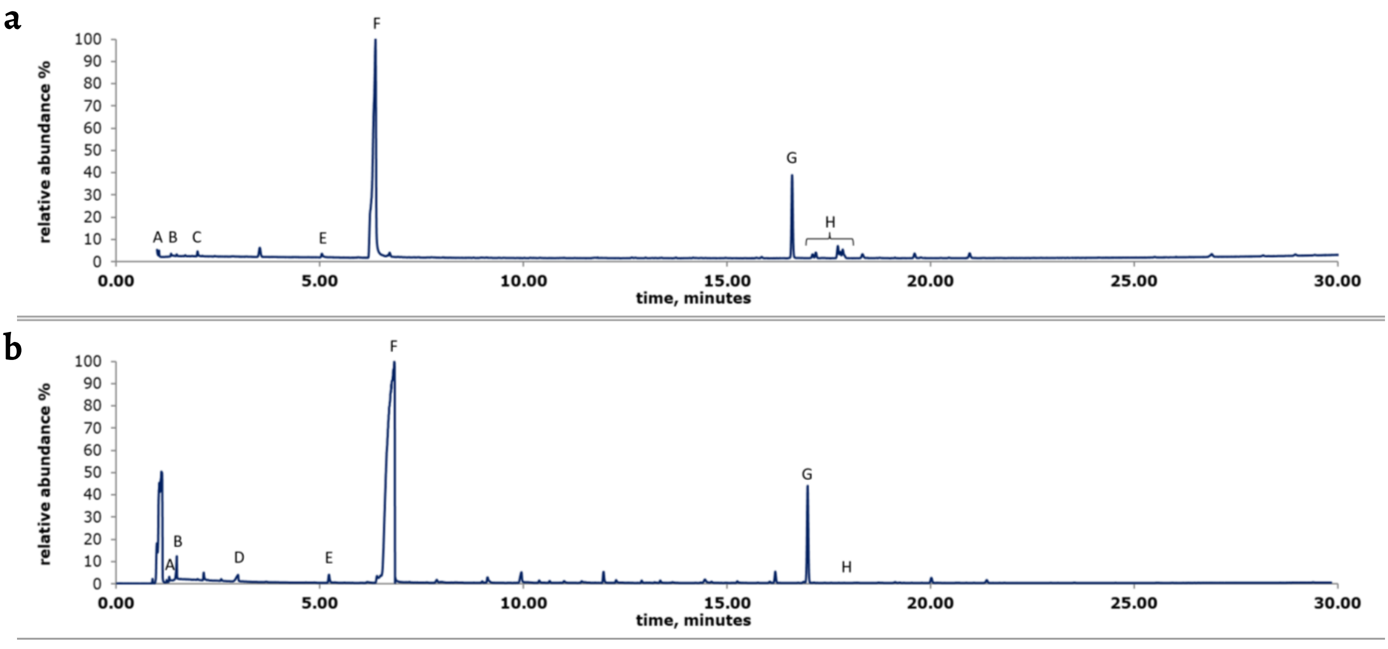

Figure 4. The result of the thermal desorption analysis of: *a)* a small sample of *L'apparence du vide* and *b)* the compounds captured by the polar Magic Chemisorbers after 72h. The main compounds are listed in Table 1.

Table 1. Off-gassed compounds from *L'apparence du vide* identified with thermal desorption and captured with both polar and apolar Magic Chemisorbers.

Label	Compound name	Thermal desorption	Polar & apolar chemisorber
A	Acetone	×	×
B	Acetic acid	×	×
C	Triethyl amine	×	
D	Butanoic acid		×
E	1,4-Dioxan-2-ol	×	×
F	Diethylene glycol	×	×
G	Poly-adipic-glycol component	×	×
H	Diisopropylnaphthalene-isomers	×	×

Discussion and conclusion

The odour of a plastic is always the result of evaporating organic substances, either from additives such as plasticisers and processing aids, or due to the degradation of plastics, where the large polymer molecules break down into smaller volatile compounds. The odour perceived by the human nose is not only dependent on the concentration of different smelly compounds but also on the sensitivity of the human nose. It is possible that a substance, present in very low concentrations, can be easily detected by the human nose, but remains below the detection limit of the method.

Raincoats by Pennel et Flipo, and Barbara Farber

The plastic coating of the raincoats is characterised as PVC-P. Plasticised PVC often expels an odour that can be described as cheap inflatables, like swimming rings and beach balls [1-3]. This smell is noticeable with both raincoats, though, the odour is not that strong. Di(2-ethylhexyl)phthalate (DEHP) is the main plasticiser of the PVC-P coating of the raincoats, together with small amounts of diisobutyl phthalate (DIBP) and dibutyl phthalate (DBP). DEHP has almost no odour, DBP has a slight aromatic odour, and DIBP has a mild odour. This is consistent with the observation that the odour of both raincoats was only slightly perceptible.

The analysis of the captured compounds by the Magic Chemisorbers, with sampling time of two hours, confirms that both raincoats emit the plasticisers DEHP, DIBP and DBP. Both coats appear in good condition: the coating is still flexible. Nevertheless, the plasticisers are evaporating, which indicates that the material will eventually become less flexible and the coating may shrink due to the loss of plasticiser [20]. These are both well-known degradation phenomena for PVC-P [17].

For future research it is interesting to explore the possibility of monitoring the off-gassing of plasticisers, which could help to decide on the most suitable environmental conditions to store these types of garments, thus avoiding the loss of plasticiser.

L'apparence du vide by Viktor & Rolf

The Viktor & Rolf ensemble is coated with an aromatic PUR-ester. The smell of *L'apparence du vide* is rancid, like stale butter. The strong rancid smell of the suit is most likely the result of the off-gassing of butanoic acid and naphthalene derivatives. The origin of these compounds is not clear. According to literature naphthalene derivatives can be used to make monomers of high-performance polyester fibres, moulded plastics and other advanced polymer materials and can exhibit characteristic smells [21-24]. Butanoic acid is known to give off a rancid odour even in low concentrations. It would be interesting to further investigate whether aromatic PUR ester coatings emit these compounds more often.

The main off-gassing compounds, captured by the Magic Chemisorbers, are diethylene glycol and a poly-adipic-glycol component. Both are formed due to degradation by hydrolysis of the soft segment (polyol part) of the PUR ester [25-26].

Magic Chemisorbers

This pilot study shows that polar (PEG) and apolar (PDMS) Magic Chemisorbers are useful sorbents to trap VOCs off-gassing from plastic coatings. Practice has shown that the chemisorbers are robust and can be used several times due to the process of capture and desorption and storage under an atmosphere. An added value for the use of Magic Chemisorbers is that one pyrolysis-GC/MS instrument and set-up is used for all applications (pyrolysis, desorption, and passive sampling), allowing the results to be compared easily and reliably. Ongoing research at the Cultural Heritage Agency of the Netherlands will investigate the long-term efficiency and reusability of the Magic Chemisorbers.

Although a sampling time of two hours is sufficient for the Magic Chemisorbers to capture the volatiles emitted by the sampled object in the bell headspace, this time is too short for emissive volatiles from the objects to equilibrate with the space in the bell jar. It was therefore decided to sample for 72 hours, during which time the object was placed under the bell jar together with the Magic Chemisorbers.

The analytical procedure described can be used to gain insight into which compounds the material off-gasses through non-destructive sampling. This can provide information about the odours that are released, but also about degradation compounds. It is a promising method that should be further investigated and hopefully can be used as a non-invasive technique in the near future.

The safety concerns raised when opening a packaging of a smelly plastic object are justifiable, but also a complex topic and complicated to test. Further research is necessary to be able to provide answers to these questions.

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