Investigations into Off-flavours in PET bottled Mineral Water due to Sunlight Exposure

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Introduction

A specific mineral water off-odour, the so-called "sunlight" flavour produced after UV light exposure, is generally described as plastic-like, deep-fried and fatty, and is most pronounced in water packaged in plastic materials, e.g. polyethylene terephthalate (PET). However, it has also been reported to arise in glass containers, especially in bottles with polyolefin bottle caps [1]. In the literature, the primary reason for this off-flavour has been attributed to the oxidation of ingredients of the packaging materials, especially the lubricant agents [1,2]. The most extensively used lubricant is erucamide (ESA) because of its low cost and techno-functional properties. ESA is a C-22 fatty acid with a double bond at the C-13 position.

The aim of the present study was the characterization of the "sunlight" off-odour in mineral water, with identification of the major aroma components. Original commercial mineral water samples with the characteristic sunlight flavour were investigated. In addition, off-odour free water samples from glass bottles were filled into PET bottles and irradiated in two different ways: either in a specific UV SunTester set-up (replicating natural sunlight conditions), or by natural sunlight.

Samples were evaluated and described by sensory analyses. Chemical analyses were also performed, whereby odour concentrates of the samples were obtained by solvent extraction using dichloromethane and subsequent mild extract concentration, followed by analyses using high resolution (HR) gas chromatography (GC) with flame ionization detection (FID) in combination with GC-Olfactometry/mass spectrometry (GC-O/MS).

Materials and Methods

Description of Samples: The following samples were analyzed:

OS: Original sample in a PET bottle purchased from a local store; sample exhibited plastic-like off-flavour.

IS: Irradiated sample in a PET bottle, irradiated with artificial UV light.

sunS: Sunlight-exposed sample in a PET bottle.

Packaging Material: The bottles were closed by two part polypropylene (PP) screw caps with an ESA amount of 6420 ppm in the cap and 550 ppm in the inlay.

UV Irradiation of IS and sunS: For IS samples, UV irradiation was performed in an ATLAS SUNTEST XLS+ (ATLAS, Wiesbaden, Germany). The irradiation conditions were 250 W m^{-2} (λ=290 nm) for 24 hours at 35 °C. This irradiation time equates to 96 h exposure to natural sunlight (July midday sun in Germany). The mineral water was irradiated in 1 L PET bottles with a PP cap. For sunS samples the irradiation by natural sunlight was similarly carried out on 1 L PET bottles with PP caps. The irradiation time was 96 h (daily exposure of 7 hours).

Sensory Analysis: The panellists were asked to describe the samples (collection of sensory attributes) and to score the intensities on a scale from 0 (no perception) to 3 (strong perception). The samples were presented to the sensory panel in covered glass vessels (capacity 45 mL, i.d. 40 mm) for comparative retronasal evaluation. The results obtained in three different sessions were averaged and plotted in spider-web diagrams. The values obtained in different sessions and for the different assessors differed by no more than 10 %.

Solvent extraction of mineral water: 200 mL of mineral water was extracted three times, each with 80 mL of dichloromethane. After 30 min of extraction at room temperature the dichloromethane phases in each case were separated. Then, the combined dichloromethane phases (240 mL) were dried over anhydrous sodium sulphate and finally concentrated to a total volume of 100 µL at 50° C by means of Vigreux distillation and distillation [3].

Results

Retronasal Sensory Analysis: The descriptive analyses resulted in sensory detection of the characteristic plastic-like, fatty and deep-fried odour notes in all three samples, with a broad overlap in the respective sensory profiles (Figure 1). General sensory compliance between samples was rated as very high with a mean score of 2.5. However, the cardboard-like note only emerged in the irradiated samples which were generally more odour-intense than OS. The overall odour intensities of the samples were rated as high, with mean values of 2.5, 2.8 and 3.0 for OS, IS, and sunS samples, respectively.

GC-O and AEDA: Characterization of odour-active substances by means of HRGC-O with solvent extraction of the analytes from the liquid phase led to consistent olfactometric detection of 14 odour active substances in all samples (OS, IS and sunS), among them the predominantly fatty and plastic-like or metallic smelling compounds (Z)- and (E)-non-2-enal, (Z)- and (E)-dec-2-enal, and (E,Z)-nona-2,4-dienal. Green, citrusy and fatty odour notes originated from octanal and nonanal, while metallic and mushroom-like notes were due to *trans*-4,5-epoxy-(E)-2-decenal and oct-1-en-3-one.

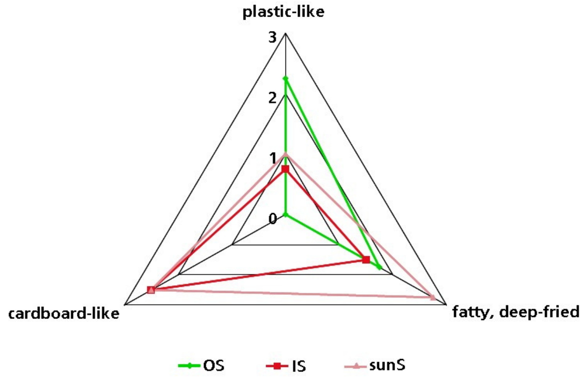

Figure 1. Comparative flavour profile analyses from retronasal sensory evaluation of the three samples. The data are displayed as mean numerical values of the sensory evaluation [4].

Table 1. Most odour-active volatiles (Flavour Dilution; FD ≥ 1) in solvent extracts from mineral water with the "sunlight" off-odour [4].

No.	Odorant [a)]	Odour Quality [b)]	RI value on	
			DB 5	FFAP
1	Octanal	citrusy, fatty	1002	1279
2	Oct-1-en-3-one	mushroom-like	980	1294
3	(*Z*)-Non-2-enal[d]	fatty	1145	1495
4	(*E*)-Non-2-enal	fatty	1160	1524
5	(*Z*)-Dec-2-enal	fatty, metallic	1249	1600
6	(*E*)-Dec-2-enal	fatty, plastic-like	1265	1631
7	(*E,Z*)-Nona-2,4-dienal[d]	fatty, plastic-like	1190	1642
8	(*E,E*)-Deca-2,4-dienal[d]	plastic-like, metallic, deep-fried	1320	1804
9	γ-Octalactone	coconut-like	1256	1911
10	*trans*-4,5-Epoxy-(*E*)-dec-2-enal	metallic-like	1390	1990
11	γ-Nonalactone	coconut-like	1364	2023
12	Unknown	plastic-like, fatty	nd	2195
13	Unknown	cardboard-like	nd	2516
14	Vanillin	vanilla-like	1396	2571

a) The compounds were identified by comparing it with the reference odorant based on the following criteria: retention indices on the capillaries named in the table, mass spectra obtained by MS/EI and MS/CI, odour quality and intensity perceived at the sniffing port.
b) Odour quality as perceived at the sniffing port.
c) nd = not determined.
d) The MS signal was at or below the detection limit of the instrument for this compound. In this case, the compound was identified by comparing it with the reference odorant based on the remaining criteria: retention indices on the capillaries named in the table, odour quality and intensity perceived at the sniffing port.

Conclusions

The substances hexanal, octanal, nonanal, decanal, (Z)-non-2-enal, (E)-non-2-enal, (Z)–dec-2-enal, (E)-dec-2-enal, (E,Z)-nona-2,4-dienal, (E,E)-deca-2,4-dienal, (E)-4,5-epoxy-(E)-dec-2-enal, non-1-en-3-one, oct-1-en-3-one, γ-octalactone and vanillin were identified in mineral water with "plastic" off-odour. A series of new compounds were detected and identified that have not been previously reported as plastic-like off-odour constituents in mineral water. Among these were, for example, (Z)-non-2-enal and (Z)-dec-2-enal, (E,Z)-nona-2,4-dienal, (E,E)-deca-2,4-dienal, and *trans*-4,5-epoxy-(E)-2-decenal. These compounds were present in all three sample types.

The present study shows that a series of compounds with high odour potencies that require precursor substances containing at least a di-unsaturated system are additionally formed. It is proposed here that these precursors are di- or tri-unsaturated fatty acid compounds such as linoleic and linolenic acid. Future studies will target the formation kinetics from the respective precursor systems, as well as the stability and further degradation of intermediate odour substances formed during these processes. The study addresses the question of whether the generated compounds are due to lubricant impurities and whether purification of the lubricant (e.g. ESA) might reduce the problem of "sunlight" off-odour in stored mineral water.

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References

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