









Volatile compounds in off-odor honey

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ABSTRACT: Climatic conditions in the mid-northern region of Mato Grosso State in Brazil are favorable for beekeeping. However, since 2011, the honey production chain has suffered losses because the production of off-odor honey has made it impossible to market the honey. Reports from beekeepers indicated a relationship between the off-odor in the honey and the nectar of *Borreria verticillata* (L.) G. Mey (Rubiaceae). In this study, the botanical origins and volatile profiles of ten off-odor honeys (H1–H10) and flowers of *B. verticillata* were evaluated. Palynological and sensorial analyses of the honeys were performed; a scale from 1 to 4 was applied for the sensorial analysis, in which 1 indicates no off-odor and 4 indicates extreme off-odor. Analysis of volatile was performed by using headspace solid-phase microextraction and gas chromatography–mass spectroscopy methods. The honeys investigated were classified with very high to intense off-odors, except H4 and H5, which did not differ from the control honey (no off-odor). Palynological analyses showed that honeys H1–H4, H7, and H9 were monofloral from *B. verticillata*, whereas in H5, H6, H8, and H10 this pollen were accessory. However, there was no quantitative correlation between the *B. verticillata* pollen content and the off-odor attributes of the honeys. Skatole was identified in all of the honeys except H4, H5, and the control honeys, suggesting that skatole contributed to the off-odor attributes of the products. However, further studies are required to investigate the origin of the skatole because it is not transferred directly from *B. verticillata* flowers to the honey.

Key words: *Apis mellifera*, melissopalynology, gas chromatography, headspace solid-phase microextraction.

Compostos voláteis em mel com odor indesejável

RESUMO: As condições climáticas da região Centro-Norte do Estado de Mato Grosso são favoráveis a apicultura, contudo ocorrem prejuízos nesta cadeia produtiva desde 2011 devido a produção de mel com odor indesejável, o que impossibilitou sua comercialização. Relatos dos apicultores apontaram relação da ocorrência do odor indesejável no mel com o néctar *Borreria verticillata* (L.) G. Mey (Rubiaceae). Neste estudo foi avaliado a origem botânica e o perfil de voláteis de méis (M1 até M10) com odor indesejável e das flores de *B. verticillata*. Foi realizada a análise polínica do mel e também sensorial, empregando-se uma escala de um a quatro pontos, em que um refere-se a nenhum odor desagradável e quatro, extremo odor desagradável. A análise de compostos voláteis no mel e nas flores de *B. verticillata* foi realizada utilizando microextração em fase sólida por headspace e cromatografia gasosa acoplada a detector por espectrometria de massas. Os méis investigados foram classificados desde muito a extremo odor desagradável, exceto os méis M4 e M5, que não diferiram do mel controle (sem odor indesejável). Os méis M1 até M4, M7 e M9 eram monoflorais de *B. verticillata*, enquanto M5, M6, M8 e M10 o pólen *B. verticillata* era acessório. Todavia, não foi observada correlação quantitativa entre o teor deste pólen e o atributo odor indesejável. O escatol foi identificado nos méis investigados, exceto em M4, M5 e mel controle. Estes resultados sugerem que o escatol contribuiu para o atributo odor desagradável do produto. Contudo, mais estudos devem ser conduzidos para investigar a origem do odor indesejável, porque o escatol não foi transferido diretamente das flores para o mel.

Palavras-chave: *Apis mellifera*, melissopalínologia, cromatografia gasosa, microextração de fase sólida por headspace.

INTRODUCTION

The mid-northern region of Mato Grosso State in Brazil, a transition region between the Amazon rainforest and the “cerrado” (savannah),

offers appropriate factors for successful apiculture, i.e., favorable climatic conditions, botanical variety, and the presence of Africanized bees (IMEA, 2017). However, since 2011, the honey production chain has suffered economic losses because some honey

produced has had an off-odor, which has hampered its marketing (IBGE, 2010, 2011, 2012).

Empirical observations of beekeepers indicated a relationship between the nectar of an herbaceous plant known as “vassourinha” (*Borreria verticillata* (L.) G. Mey (Rubiaceae) and the occurrence of undesirable odor in the honey, because the relocation of apiaries to regions without this plant was an efficient method to avoid or reduce the unwanted odor. However, the spread of this invasive species and the economic viability of relocating hives are problems still faced by this production sector.

Honey aroma compounds are derived from the transfer of volatile substances from plants, metabolic transformations of plant constituents by bees, the metabolism of bees, changes that occur during honey processing, and the action of microorganisms (NAEF et al, 2004; BARONI et al., 2006; RIBEIRO et al., 2008; DE-MELO et al., 2018).

Volatile-profile studies have identified chemical markers in monofloral honeys (BARONI et al., 2006; SEISONEN et al., 2015). Changes in volatiles have been reported in honeys subjected to heating, with a consequent alteration of flavor, intensifying the bitter and roasted tastes, and development of the appearance and taste of medicine (CASTRO-VÁZQUEZ et al., 2012; RIBEIRO et al., 2012). KRUŽÍ et al. (2017) reported a methodology for investigating volatile compounds in honeys to evaluate the quality and the occurrence of adulterations.

However, there are no scientific reports of undesirable volatiles in fresh honeys. This study evaluated the botanical origin and the volatile profiles of off-odor honeys produced in the mid-northern region of Mato Grosso State, Brazil.

MATERIALS AND METHODS

Ten samples (H1–H10) reported by beekeepers as off-odor honeys were collected, in three replicates of each, from apiaries in different cities of the mid-northern region of Mato Grosso State: Nova Ubiratã, Sinop, Tabaporã, Santa Carmen, and Ipiranga do Norte. Honeys H1 and H2 were from the 2015 harvest, H3 from 2016, H4 and H5 from 2017, and H6–H10 from 2018 (Table 1). Honeys H1–H3 were collected in honeycombs and transported in thermal boxes to the Food Technology Laboratory of the Federal University of Mato Grosso (UFMT), Campus of Sinop, where the honeys were extracted from the combs. Samples H4–H10 were centrifuged and delivered to this laboratory by beekeepers. The honeys were stored in a refrigerator until the

palynological, sensorial, and volatile-compound analyses were performed.

In June 2015, samples of the plant known as “vassourinha” (*Borreria verticillata* (L.) G. Mey) were collected in three replicates and sent to the Biological Collection of the Southern Amazon (ABAM), UFMT, in Sinop, for botanical identification. A voucher specimen was deposited at ABAM under the registration number CNMT-5423. In addition, flowers of *B. verticillata* with receptacles and stems were collected in three replicates and transported in thermal boxes to the Food Technology Laboratory, UFMT. Flowers were removed from the receptacle with tweezers and kept in a freezer until analysis of the volatiles by gas chromatography coupled with mass spectrometry (GC/MS).

Palynological analysis followed the methodology described by BARTH (1989), with polliniferous species (e.g., wind-pollinated like *Cecropia* sp.) counted separately (LOUVEAUX et al. 1978). Residues were used to assemble permanent slides that were cataloged and kept in the Pollen Reference Collection of the Paleontology Laboratory of UFMT, Cuiabá Campus.

Ten volunteer evaluators, older than 18 years old, were selected for sensory analysis based on the discriminative power and reproducibility of each individual for the identification of undesirable honey odors (PIANA et al.; 2004; KRUŽÍK et al., 2017).

The evaluators received three honeys with random codes from crop years 2015–2018, one of which was a control sample (a honey without an undesirable odor) and two of which had off-odors, and the intensity of the odor attributes was evaluated by using a numerical scale from very intense off-odor (4 on the scale) to no odor (1 on the scale). The evaluators also described the odor (PIANA et al., 2004; INSTITUTO ADOLFO LUTZ, 2008).

Headspace solid-phase microextraction (HS-SPME) was performed with a commercial polydimethylsiloxane/divinylbenzene fiber (65 µm, Supelco, Bellefonte, PA, USA). The fiber was exposed to the sample headspace in 20 mL vials for SPME with 1.0 g of honey sample in a saline solution (KCl). The extraction was carried out at 40 °C and 500 rpm for 30 min with the fiber exposed in the headspace. Thermal desorption of the compounds was performed directly on the injector for 10 min.

An Agilent gas chromatograph (GC-MS 7890A/5975C) equipped with a VF-Wax MS capillary column (30 m × 0.25 mm internal diameter × 0.25 µm, Agilent J&W, Santa Clara, CA, USA) was used for analysis of volatile compounds in the honeys

Table 1 - Skatole contents and sensory panel scores for the off-odor attribute in *Apis mellifera* honey from some cities in the mid-north region of Mato Grosso State, Brazil.

Honey	Apiary locality	Geographical coordinates	Haverst	Skatole (mg.kg ⁻¹)	Score ¹
Control ²	Santa Carmen	11°54'37.6"S 55°17'05.6"W	2016	ND ³	1.3 a ⁴
H1	Nova Ubiratã	13° 36' 53" S 54° 49' 14" W	2015	NA ⁵	3.4 c
H2	Sinop	11°52'10.0"S 55°37'08.7"W	2015	NA	3.6 c
H3	Sinop	11°52'10.0"S 55°37'08.7"W	2016	NA	3.7 c
H4	Tabaporã	11°19'8" S 56°14'56" W	2017	ND	1.6 a
H5	Santa Carmen	11°54'37.6"S 55°17'05.6"W	2017	ND	1.4 a
H6	Sinop	11°53'51"S 55°27'36"W	2017	0.484 d ²	3.6 c
H7	Sinop	11°53'51"S 55°27'36"W	2018	0.425 d	3.0 b
H8	Sinop	11°51.683' S 55° 34.406' S	2018	0.328 c	3.7 c
H9	Sinop	12°08.874' S 55°30.406' W	2018	0.155 b	2.8 b
H10	Ipiranga do Norte	12°25'13"S 56°3'41" W	2018	0.079 a	2.9 b

¹Scores for the off-odor attribute from 1 to 4, i.e. from no off-odor to very intense.

²Control honey free from off-odor.

³ND = Not detected.

⁴Averages followed by the same letter in the columns do not differ significantly from each other (Scott-Knott test $p > 0.05$).

⁵NA = Not analyzed.

and the flowers of *B. verticillata*. Ultrapure helium at 0.8 mL min⁻¹ was used as the carrier gas. The injector temperature was set at 250 °C, and the temperature of the ion source was 230 °C with an electron energy of 70 eV. The oven temperature program was: 45 °C (held for 2 min), 40 °C min⁻¹ to 300 °C (held for 3 min). Identifications of compounds were performed by comparing the mass spectra obtained with the NIST Mass Spectral Search program, version 2.0, 2011. The skatole measurement was performed for samples H4–H10 with a 98% purity standard (Sigma–Aldrich, St. Louis), by using a calibration curve of 0.01 to 0.6 mg kg⁻¹ (BIANCHIN et al., 2014).

The experimental design was completely randomized and the averages of the scores were compared with the Scott–Knott test ($p > 0.05$).

RESULTS AND DISCUSSION

We reported pollen from *Borreria verticillata* (L.) G. Mey (*Rubiaceae* – *Rubioideae* – *Spermacoceae*) in all ten of the honey samples that were analyzed. In samples H1–H4, H7, and H9, this pollen type was predominant, and the honeys were classified as monofloral; in samples H5, H6, H8, and H10, *B. verticillata* pollen was accessory, with a percentage in the range of 16–45% (BARTH, 1989; RAMÍREZ-ARRIAGA, et al., 2011).

B. verticillata is a nectariferous plant (BARTH, 2004; MAIA-SILVA et al., 2012), and

its monofloral honey has been classified as being of low quality because of its sensorial attributes (BARTH, 1998). ALMEIDA-MURADIAN et al. (2014) described the odor of a bifloral honey of *B. verticillata* and *Hyptis* (*Lamiaceae*) in the following terms: “animal, resin, wax, fruit, malt, coffee, caramel, wood, and smoke smells”, i.e., a mixture of pleasant and unpleasant odors.

The honeys investigated herein contained skatole, except samples H4 and H5 (Table 1). The prior volatilization of the skatole of samples H4 and H5 is a possibility, because the beekeepers only delivered the honey from the harvest of 2017 in February of 2018 and they confirmed that the products had been stored in plastic buckets at room temperature (BURDOCK, 2010).

The monofloral honeys H1, H2, and H3 were classified as having intense to very intense off-odors (Table 1); although the scores for H6 and H8 were similar despite the fact that *B. verticillata* pollen was accessory. Meanwhile, for H7, H9, and H10, average scores of 2.9 were given, with no significant difference between them, even though *B. verticillata* pollen was dominant in H7 and H9 but was accessory pollen in H10. Therefore, the off-odor honey contained *B. verticillata* pollen, but there is no quantitative correlation between the frequency of this pollen and the odor attributes.

Samples H6 and H7 presented the highest contents of skatole, followed by H8, and these honeys

were classified as having intense to very intense off-odors (Table 1). Samples H1–H3 showed similar odor classification and, as shown in table 2, exhibited the largest average percentages for the relative area of skatole (10.2% ± 2.3%) in comparison with those of the other honeys investigated (3.5% ± 2.8%). Skatole was not identified in the volatile compound profiles of the control honey (without an unpleasant odor); skatole was also not detected in the H4 and H5 honeys, and their scores for unpleasant odor attributes did not differ from that of the control honey (Table 1, Figure 1). These results suggested that skatole contributed to the off-odor characteristics of the product.

In the off-odor honey descriptions, evaluators reported an “animal feces smell”, “pigsty odor”, and “smell of swine feces”; these odor descriptions can be explained by the presence of

skatole in the volatile profile, because this compound presents a fecal odor (SIEFARTH & BUETTNER, 2014; MAHMOUD & BUETTNER, 2016).

The major volatile compounds in *B. verticillata* flowers were toluene, linalool, and benzaldehyde with relative abundances of 17.5%, 15.1%, and 7.2%, respectively. Styrene was transferred from the flowers of *B. verticillata* to the honeys, with the exception of H9 (Tables 2 and 3). Benzaldehyde was observed in the flowers and in samples H1–H4, H6, and H10, whereas toluene was only transferred from the flowers of *B. verticillata* to samples H1–H3 (Tables 2 and 3). However, there was no transfer of skatole, possibly because this substance originated from the metabolic processes of bees or from microorganisms associated with bees or with *B. verticillata* (BARONI et al., 2006; NAEF et al., 2004; RIBEIRO et al., 2008).

Table 2 - Relative abundance in percentages of major volatile compounds identified in *Borreria verticillata* flowers and in off-odor honey from 2015 to 2017 harvests in mid-northern region of Mato Grosso state, Brazil.

Compound	Flower ¹	H1 ²	H2	H3	H4	H5
Toluene	17.5±3.5 ³	16.0±4.3	17.5±3.5	18.3±5.1	-	-
Ethylbenzene	-	3.2±0.7	4.9±1.1	2.6±0.8	-	-
5-methyl-hexanone	-	-	2.3±0.3	-	-	-
Styrene	3.6±1.0	6.9±1.9	7.8±1.8	8.9±2.4	1.4±0.1	1.9±0.4
Rose oxide (cis)	6.1 ±2.1	-	-	-	-	-
1-Octen-3-ol	5.2±1,3	-	-	-	-	-
Tetramethylbenzene	-	1.0±0.5	-	-	-	1.3±0.2
Benzaldehyde	7.2±0.9	4.1±1.3	5.1±1.2	2.7±0.3	1.3±0.2	-
Salicylic acid	6.0±2.1	-	-	-	-	-
D-Limonene	-	2.0±0.7	-	-	-	-
Cyclotetrasiloxane	-	1.4±0.2	-	-	-	0.3±0.0
Gamma-Terpinene	-	-	-	1.3±0.2	-	-
Tetrasiloxane	-	-	-	-	-	-
Linalool	15.1±3.9	-	-	-	-	-
Hotrienol	-	-	2.3±0.4	2.9±0.5	-	-
Linalool oxide	-	-	-	1.0±0.1	-	5.4±1.2
Δ ³ -Carene	-	3.0±0.4	-	-	-	3.0±0.8
Benzoic acid	-	3.4±1.2	-	-	-	4.7±1.3
Benzeneacetaldehyde	-	-	4.3±1.2	3.6±0.1	3.2±0.2	-
Benzeneacetic acid	-	3.3±0.6	-	-	-	32.7±5.3
Pentanoic acid	-	4.3±1.5	5.5±1.9	7.0±2.1	-	-
Cyclohexasiloxane	-	-	-	-	-	1.4±0.4
Neroloxide	-	-	-	2.0±0.7	5.8±1.2	-
Skatole	-	11.3±4.2	10.0±3.2	9.8±3.1	-	-
Benzeneacetic acid	-	-	6.3±1.8	3.0±1.1	2.4±0.4	-
Cyclohexasiloxane	-	1.03±0.4	-	-	1.6±0.1	-
Benzenepropanoic acid	-	-	-	-	1.5±0.2	11.5±4.3

¹Flower of *B. verticillata*.

²Off-odor honey samples (H1 to H5). H1 and H2; H3; H4 and H5, respectively from 2015; 2016 and 2017 harvests.

³Average relative abundance ± standard deviation.

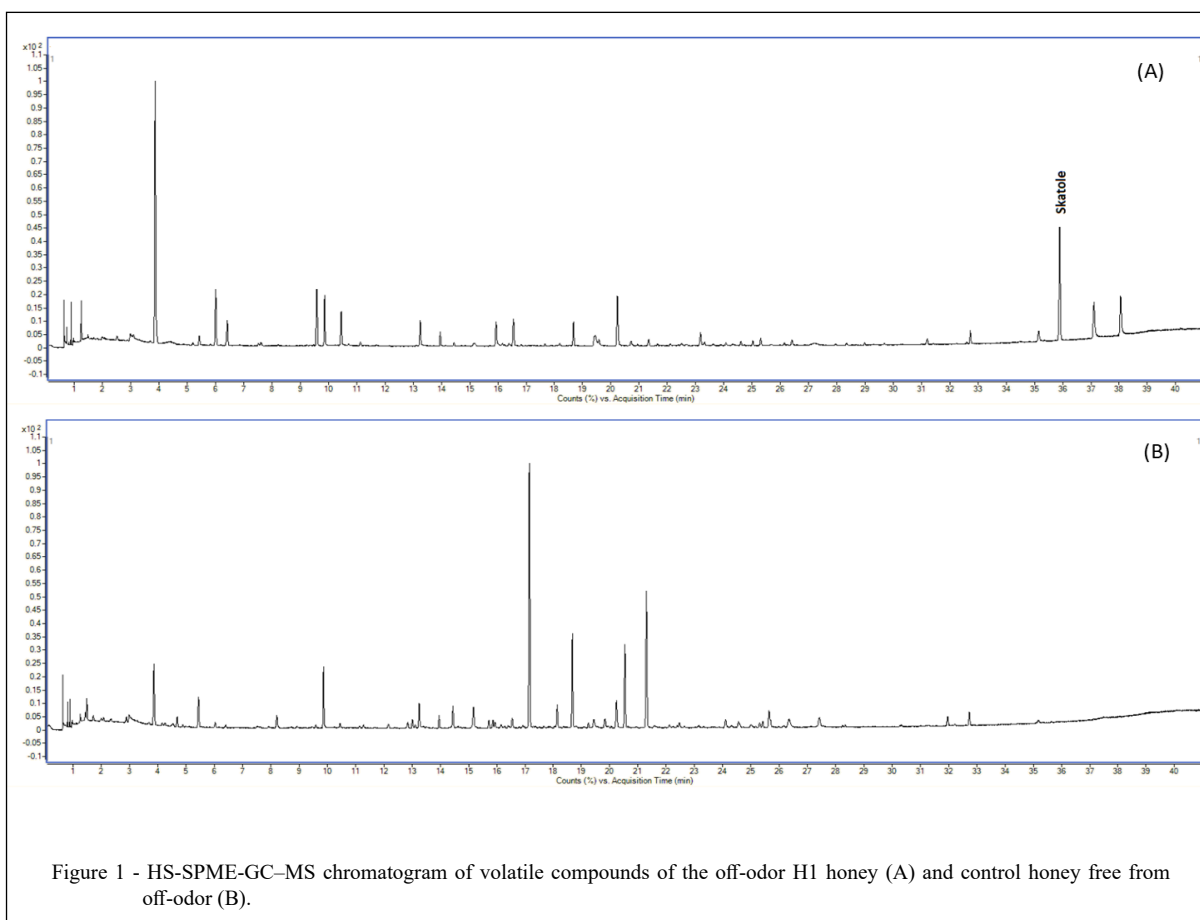


Table 3 - Relative abundance percent of major volatile compounds identified in off-odor honey (H6 to H10) from 2018 harvest in mid-northern region of Mato Grosso State, Brazil.

Compound	H6	H7	H8	H9	H10
Ethylbenzene	6.3±1.4 ¹	4.1±1.7	10.67±2.6	-	2.8±0.2
5-methyl-hexanone	-	-	-	-	-
Styrene	21.9±4.6	15.6±4.0	13.01±1.5	-	5.3±1.4
Tetramethylbenzene	-	1.4±0.2	-	-	1.9±0.1
Benzaldehyde	-	-	2.25±0.5	-	-
D-Limonene	-	2.6±0.6	-	20.2±4.3	49.0±3.5
Cyclotetrasiloxane	-	-	2.64±1.1	1.1±0.1	-
Gamma-Terpinene	1.2±0.2	1.3±0.3	-	1.6±0.3	-
Tetrasiloxane	-	-	1.39±0.4	-	-
Hotrienol	4.4±1.3	12.9±3.1	-	-	-
Benzoic acid	1.5±0.1	1.7±0.2	-	2.5±0.3	-
Benzeneacetaldehyde	-	-	-	-	-
Benzeneacetic acid	14.4±3.9	18.4±4.3	6.3±0.7	17.1±3.7	2.7±0.4
Cyclohexasiloxane	1.4±0.3	2.8±0.3	-	2.1±0.6	2.4±0.5
Neroloxide	-	-	-	-	-
Skatole	5.2±0.8	3.9±0.2	6.0±2.1	0.2±0.0	2.6±0.8
Benzenepropanoic acid	9.5±1.9	6.8±1.5	3.4±0.4	5.1±1.7	-

¹Average relative abundance percent ± standard deviation.

There are no reports of the presence of skatole in honey from Brazil or other countries (BARONI et al., 2006; DE MARIA & MOREIRA, 2003; KRUŽÍK et al., 2017; PATRIGNANI et al., 2018), but it has been described in off-flavor meat products.

In swine, skatole is one of the compounds associated with male odor, because it is a product of the degradation of tryptophan by lactic bacteria of the intestinal colon (CLAUS et al., 1994). There are no scientific reports of the amino acid profile of *B. verticillata* honey, but the high protein level (2,236 $\mu\text{g g}^{-1}$) of this honey stands out relative to those Sapindaceae, eucalyptus, and citrus honeys, which have protein contents of 1,203, 734, and 628 $\mu\text{g g}^{-1}$, respectively (AZEREDO et al., 2003).

In addition, skatole has been identified among off-flavor compounds in trout (*Oncorhynchus mykiss*) and Gibel carp (MAHMOUD & BUETTNER, 2016). However, there is no consensus for the origin of skatole in fish, and it was hypothesized that the river had been contaminated with swine feces. In this study, the hypothesis of bees collecting in a pigsty is unlikely, because there were no rural constructions within a radius of 3 km of the apiary investigated (BRASIL, 1985).

CONCLUSION

The off-odor honeys investigated contained *Borreria verticillata* (L.) G. Mey pollen. Six samples (H1–H4, H7, and H9) were monofloral honeys, whereas *B. verticillata* pollen was only one of the components in four others (H5, H6, H8, and H10). However, we did not observe a relationship between the *B. verticillata* pollen abundance and the odor characteristics of the honeys investigated.

Skatole was quantified and/or identified in honeys categorized as having an intense off-odor. However, further studies are required to investigate the origin of the skatole because it was not transferred directly from *B. verticillata* flowers to the honey.

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BIOETHICS AND BIOSECURITY COMMITTEE APPROVAL

The sensory analysis of this study was approved by the Júlio Müller Hospital Ethics Committee, Federal University of Mato Grosso (CAAE: 44328315.2.0000.5541, 06/26/2015), according to Resolution 466/2012 of the Ministry of Health that establishes the guidelines and rules regulating research involving human beings.

DECLARATION OF CONFLICT OF INTEREST

The authors declare no conflict of interest. The founding sponsors had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

AUTHOR'S CONTRIBUTIONS

BRF and SCCB performed chromatography analyses; CD performed pollen analyses; LCS performed botanical identifications; EAA performed statistical analyses; CW designed the research, coordinated the project, performed sensory analyses and wrote the manuscript. All authors were involved in writing and revision of the final draft.

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