



Characterization by gas chromatography-olfactometry of the most odour-active compounds in Italian balsamic vinegars with geographical indication



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ABSTRACT

Odour-active compounds in three traditional balsamic vinegars from Modena (TB) and seven balsamic vinegars from Modena (PGI) were determined by gas chromatography-olfactometry (GC-O) using frequency of detection methodology (modified frequency, MF, %). The main odour compounds (mean MF > 60%) were 2,3-butanedione (75%), acetic acid (70%), furan-2-carbaldehyde (62%), 1-(furan-2-yl)ethanone (62%), 2-methylpropanoic acid (66%), butanoic acid (78%), 3-methylbutanoic acid (83%), 2-phenylethyl acetate (65%), 2-hydroxy-3-methylcyclopent-2-en-1-one (61%), 2-phenylethan-1-ol (84%), 3-hydroxy-2-methylpyran-4-one (60%), (5-formylfuran-2-yl)methyl acetate (68%), 2-phenylacetic acid (69%) and 4-hydroxy-3-methoxybenzaldehyde (86%). All odour impact compounds were grouped into 7 categories according to their aromatic character: cheesy-butter-lactic, sweet, flower, empyreumatic, fruity, chemical and miscellaneous. Balsamic vinegars from Modena showed lower values for the sweet category whereas for the miscellaneous and chemical categories they exhibited higher values than those found in traditional balsamic vinegars from Modena.

A principal component analysis showed that both types of vinegars from Modena could be clearly differentiated based on olfactometric data.

1. Introduction

In the European Union (EU) the term “geographical indication” is commonly employed to indicate a regulation that intends to standardizes the production, marketing and certification of specific food products whose quality derive from their production area and for which, specific elaboration processes are clearly set up. There are three different formats for geographical indications and traditional specialities in the EU (protected designation of origin, PDO; protected geographical indication, PGI; and traditional speciality guaranteed, TSG). Their main objective is to promote and to protect the denomination of high quality agricultural products and foodstuffs (European Commission, 2017).

The vinegars identified as traditional balsamic vinegar from Modena (TB) and Reggio-Emilia (TBVRE) from two protected designations of origin (PDO) are produced in Italy (European Commission, 2000). They are exclusively produced at those geographical areas (Modena and Reggio-Emilia) and specific grape varieties are grown for the purpose.

Both of them are obtained from cooked and concentrated must that experiences a dynamic aging process (in barrels made of different types of woods, such as oak, chestnut, mulberry, or juniper, and decreasing size) where alcoholic and acetic fermentation take place simultaneously. The aging process can last at least for 12 years (*affinato* type) or even 25 years (*extravecchio* type). This kind of vinegar is usually produced by families that support a tradition that comes from several generations and the final product is usually quite valuable due to this long time production process. Balsamic Vinegar from Modena (PGI) is another Italian vinegar, but in this case with protected geographical indication (PGI) (European Commission, 2009). It is obtained from the Modena geographical area but through a different production process. For the production of Modena balsamic vinegar, a certain portion of cooked must is added to wine vinegar and then it is aged for a period that ranges from 2 months to 3 years in single wood barrels. This is generally an industrial product and it often combines cooked wine must vinegar with caramel and other thickeners to obtain a similar density

Abbreviations: EU, European Union; PDO, protected designations of origin; PGI, protected geographical indication; TSG, traditional speciality guaranteed; TB, traditional balsamic vinegar of Modena; TBVRE, traditional balsamic vinegar of Reggio-Emilia; GC-O, gas chromatography-olfactometry; MF, modified frequency; MS, mass spectrometry; SPE, solid phase extraction; ANOVA, analysis of variance; PCA, principal component analysis; Che, cheesy-butter-lactic; Swe, sweet; Flo, flower; Emp, empyreumatic; Fru, Fruity; Chem, Chemical; Mis, Miscellaneous

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Table 1
Volatile compounds detected. LRI values, odour descriptors and identification type.

Compound	LRI	Odour descriptor from literature	Odor descriptor from sniffers	Identification	m/z^a	Previously identified in vinegar
2,3-Butanedione	970	Butter	Butter-like odor	ST, MS, OD	43, 86	6
2-Methylpropyl acetate	1004	Fruity, floral		ST, MS	43, 56, 73	1, 3, 4, 5, 6
2-Butanol	1014	Pleasant, fruity		ST, MS	45, 41, 59	
2-Methyl-1-propanol	1083	Alcohol, wine	Sweet, musty, winey	ST, MS, OD	43, 41, 74	1, 5, 6
3-Methylbutyl acetate	1120	Banana, fruity	Banana, fruity	ST, MS, OD	43, 70, 55	1, 3, 4, 5, 6, 7
3-Methyl-1-butanol	1229	Alcohol	Choking alcohol	ST, MS, OD	55, 43, 70	1, 2, 3, 4, 5, 6, 7
2-Methyloxolan-3-one*	1024	Sweet, toasted	Sweet, solvent, brown, nutty	MS, OD	43, 72, 100	
3-Hydroxy-2-butanone	1283	Buttery	Milk product	ST, MS, OD	45, 43, 88	1, 2, 3, 4, 5, 6, 7
2-Hexanol	1308	Chemical, winey, cauliflower	Alcohol	ST, MS, OD	45, 69	6
Ethyl 2-hydroxypropanoate	1335	Sweet, fruity, acidic	Food, sweet, acid	ST, MS, OD	45, 75	1, 2, 4, 5, 6, 7
Acetic acid	1407	Pungent, vinegar	Acid, vinegar	ST, MS, OD	43, 45, 60	3, 4, 5, 6, 7
Furan-2-carbaldehyde	1423	Almond	Almond, food	ST, MS, OD	96, 95, 39	1, 2, 3, 4, 5, 6
3-(Acetyloxy)butan-2-yl acetate*	1452	Toasted maize, fried chicken, burned	Food	MS, OD	43, 87	2, 3
1-(Furan-2-yl)ethanone*	1468	Sweet, balsamic, almond, cocoa, caramel	Fruity, sweet	MS, OD	95, 110	2, 3
Propanoic acid	1524	Pungent, rancid	Cheese	ST, MS, OD	74, 73, 45	2, 5, 6
Butane-2,3-diol	1528	Fruity, creamy, buttery	Fruity	ST, MS, OD	45, 43, 57	5, 7
2-Methylpropanoic acid	1582	Rancid butter, cheese	Cheese	ST, MS, OD	43, 41, 73	2, 4, 5, 6
5-Methylfuran-2-carbaldehyde	1567	Spicy-sweet, warm, caramel		ST, MS	110, 109, 53	1, 2, 3, 6
1-Hydroxypropan-2-yl acetate*	1599			MS	43, 87, 75	2
Butanoic acid	1627	Rancid butter	Cheese	ST, MS, OD	60, 73	2, 3, 5, 6, 7
Dihydrofuran-2(3H)-one	1628			ST, MS	42, 86, 56	1, 2, 5
3-Acetyloxypropyl acetate*	1660			MS	43, 61	5
3-Methylbutanoic acid	1690	Rancid, cheese	Cheese	ST, MS, OD	60, 87, 43	1, 2, 3, 4, 5, 6, 7
Diethyl butanedioate	1711	Faint, pleasant		ST, MS	101, 129	1, 2, 3, 4, 5, 6, 7
5-Methyl-(3H)-furan-2-one*	1720	Sweet, nutty, tobacco		MS	98, 55, 43	6
Benzyl acetate	1755	Sweet, fruity, floral	Floral	ST, MS, OD	108, 91, 150	1, 2, 3, 4
Ethyl phenylacetate	1810	Floral, honey, sweet	Floral	ST, MS, OD	91, 164	1, 2, 3, 4
(E)-But-2-enoic acid	1820	Milk product, roasted, burnt	Milk product	ST, MS, OD	86, 69, 41	6
2-Phenylethyl acetate	1849	Sweet, rosy-fruity, honey-like	Rose	ST, MS, OD	104, 43, 91	1, 2, 3, 4, 5, 7
2-Hydroxy-3-methylcyclopent-2-en-1-one*	1854	Sweet, caramel, maple sugar, coffee	Toasted	MS, OD	112, 69, 55	2, 3, 6
Hexanoic acid	1865	Cheesy, goaty, sweat	Disagreeable, acid	ST, MS, OD	60, 73	1, 2, 3, 4, 5, 6, 7
Phenylmethanol	1912	Slightly pungent, faint aromatic, fruity	Sweet	ST, MS, OD	79, 108, 107	1, 2, 3, 4, 5, 6
2-Phenylethan-1-ol	1944	Mild, warm, rose, honey-like	Rose	ST, MS, OD	91, 92, 122	1, 2, 3, 4, 5, 6, 7
3-Hydroxy-2-methylpyran-4-one*	1990	Caramel-butterscotch, fruity	Sweet	MS, OD	126, 71, 43	2, 3, 6
Methyl furan-2-carboxylate*	2033			MS	95, 126	6
5,6-Dihydro-4-methyl-(2H)-pyran-2-one*	2089	Sweet	Sweet	MS, OD	82, 54, 112	2, 6
Octanoic acid	2101	Fatty, rancid, vegetable	Unpleasant, acid, rancid	ST, MS, OD	60, 73, 101	1, 3, 4, 5, 6, 7
(5-Formylfuran-2-yl)methyl acetate	2222	Sweet	Sweet, toasted	ST, MS, OD	126, 79, 109	2, 3, 6
3,5-Dihydroxy-2-methyl-(4H)-pyran-4-one*	2280	Toasted, caramel	Toasted, aromatic plant	MS, OD	142, 68, 43	2, 6
4-Oxopentanoic acid	2301	Caramel	Caramel, sweet	ST, MS, OD	43, 56	7
Diphenylmethane	2350			ST, MS	105, 77, 182	3
Furan-2-carboxylic acid	2380	Fatty, musty, waxy	Acid, milk product	ST, MS, OD	112, 95	2
5-(hydroxymethyl)furan-2-carbaldehyde	2509	Fatty, bitter, musty		ST, MS	97, 126	2, 3, 4, 6, 7
2-Phenylacetic acid	2555	Sweet, animal, honey-like	Sweet	ST, MS, OD	91, 136	2, 5, 7
4-Hydroxy-3-methoxybenzaldehyde	2564	Sweet, creamy, vanilla	Chocolate	ST, MS, OD	151, 152, 81	1

ST: coincidence of Linear Retention Index with standard.

MS: matching mass spectra with those from Wiley 7 N Edition Library.

OD: coincidence of odour listed in literature.

* Tentatively identified.

^a Mass fragments employed for identification purposes. The first one used for quantification purposes. ¹Callejón, et al. (2008). ²Chinnici, et al. (2009). ³Marruffo-Curtido et al. (2012). ⁴Cejudo-Bastante et al. (2013). ⁵Charles, et al. (2000). ⁶Zeppa, et al. (2002). ⁷Pinu et al. (2016).

and flavour to those of the traditional product, but at a lower price.

All of these vinegars are unique oenological products based on their high quality and specific production processes. Therefore, the differences either in raw materials or specific production and aging processes determine the final product's aromatic profile that allows us to differentiate one vinegar from the other.

Aroma is a very important food feature to support its quality and to contribute to its acceptance. Aroma in oenological products is

determined by several hundreds of different volatile compounds that belong to different chemical families. Alcohols, aldehydes, esters, acids, monoterpenes, and other minor compounds usually comprise the aromatic profile of these products (Schreier, 1979). But only those which are odour-active compounds actually play an important role and characterize their aroma profile.

In order to check and characterize those volatile compounds which actually determine the aroma of a substance, gas chromatography

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