禹重科技® ÜZONGLAB

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可视化的风味分析

--- GC-IMS技术在风味研究中的应用

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食品中风味物质浓度通常较低，大多数风味物质的含量在ppb级别，目前的检测技术如GC、GC-MS需要顶空固相微萃取的方式进行样品前处理，因萃取头材质的不同，仪器检测到的结果并非样品原有真实的气味；此外风味成分在食品加工、贮藏、提取分离过程中的变化也是目前检测技术亟待解决的问题；从食品风味的角度来看，并非所有仪器检测到的挥发性有机物都是同等重要的，有时我们只需找出有差异的成分进行分析即可。

FlavourSpec®风味分析仪结合了气相色谱的高分离度和离子迁移谱的高灵敏度，无需样品前处理，直接顶空进样分析固体或液体样品的顶空成分，检出限可达到ppbv级别。仪器可对单一化合物/标记物进行定性定量分析，亦可对样品的挥发性有机物进行快速与结果导向的分析，其原理如图1所示。

扫描二维码观看
GC-IMS技术原理介绍视频

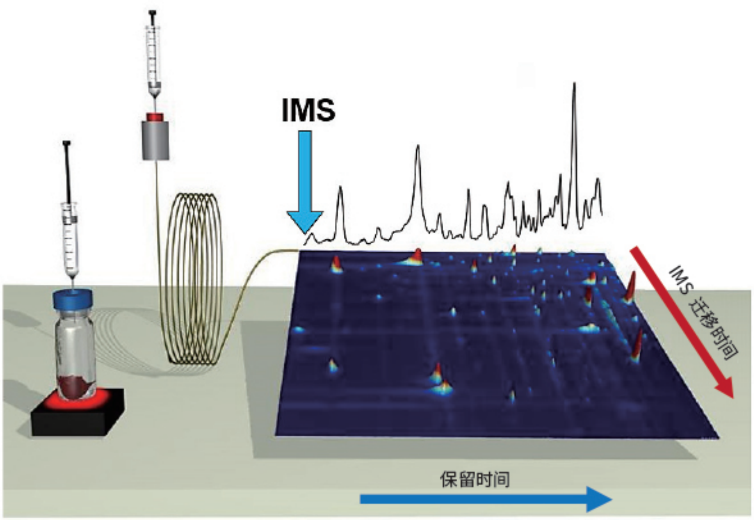


图1. 气相色谱-离子迁移谱原理示意图

GC-IMS技术在风味分析中的特点

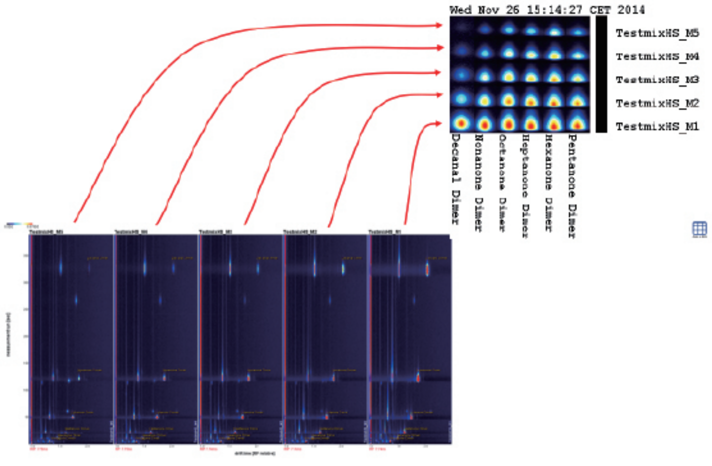
- 无需富集浓缩，超高灵敏度，痕量挥发性有机物“尽在掌握”；
- 无真空系统，开机稳定时间短，20min便可工作；
- 数据处理简单易操作；
- 可视化的软件，得到“看得见”的数据：

1）Reporter插件在得到数据后可快速查看样品间风味物质的差异；

2）GalleryPlot插件可得到产品/原材料的指纹图谱，数据直观可视化、风味差异“看得见”，适用于食品加工工艺的优化选择；

3）PCA插件，用于聚类分析，帮助产品品控或分级，建立分类模型后可用于产品原产地的保护。

示例：M1-M5为不同浓度混标溶液（5ppb、20ppb、50ppb、75ppb、100ppb），经过仪器快速分析后，软件直接给出样品之间对比的气相离子迁移谱图，如图2左下图所示；将样品中的挥发性有机物选取后，处理得到样品的挥发性有机物指纹图谱，如图2中右上图所示。



在指纹谱图中：

- 每一个点代表一种挥发性有机物，点的颜色深浅表示物质信号峰强度的高低；
- 每一行代表一个样品中选取的挥发性有机物信号峰；
- 每一列代表同一挥发性有机物在不同样品中的信号峰强度；

图2. 混标样品的气相离子迁移谱图和指纹谱图

图2中左下图为混标样品挥发性有机物的气相离子迁移谱图；右上图为M1-M5混标中选取的戊酮、己酮、庚酮、辛酮、壬酮、癸醛的指纹谱图；

由指纹谱图可知：M5中6种醛酮含量最低，M1中醛酮含量最高，从M5至M1，醛酮含量逐渐升高，建立标准曲线后可对任一醛酮物质进行定量分析。

4) 软件内置NIST气相保留指数(RI)数据库和G.A.S.迁移时间(Dt)数据库，定性时采用二维定性，数据更精准，且两个数据库均可扩展，用户可建立行业专有数据库。

例如：鸡肉中定性出的部分物质如下表：

Count 编号	Compound 化合物	CAS号	Formula 分子式	MW 分子量	RI 保留指数	Rt [sec] 保留时间	Dt [RIPrel] 相对迁移时间
1	hexanal	C66251	C6H12O	100.2	796.0	200.11	1.2763
2	Heptanal	C111717	C7H14O	114.2	894.5	255.983	1.3444
3	n-Nonanal	C124196	C9H18O	142.2	1103.0	488.847	1.4932
4	Benzaldehyde	C100527	C7H6O	106.1	953.5	301.545	1.1439
5	methional	C3268493	C4H8OS	104.2	902.9	261.977	1.0835
6	1-Octen-3-ol	C3391864	C8H16O	128.2	978.8	323.506	1.1552
7	Ethyl methyl ketone	C78933	C4H8O	72.1	608.3	137.499	1.0589
8	Ethanol	C64175	C2H6O	46.1	455.2	103.337	1.043
9	Dimethyl ketone	C67641	C3H6O	58.1	494.1	111.101	1.1213
10	3-Methylbutanal	C590863	C5H10O	86.1	667.4	153.532	1.1825
11	1-Pentanol	C71410	C5H12O	88.1	769.2	188.801	1.2554
12	4-methyl-2-pentanone	C108101	C6H12O	100.2	735.6	176.061	1.1789
13	Acetoin	C513860	C4H8O2	88.1	713.5	168.144	1.0772
14	1-Hexanol	C111273	C6H14O	102.2	868.0	239.386	1.3274
15	Butyl methyl ketone	C591786	C6H12O	100.2	783.9	194.643	1.1916
16	acetophenone	C98862	C8H8O	120.2	1059.4	421.648	1.1873
17	octanal	C124130	C8H16O	128.2	1002.5	347.745	1.4239
18	Maltol	C118718	C6H6O3	126.1	1088.0	464.496	1.2261
19	(E)-2-octenal	C2548870	C8H14O	126.2	1054.0	414.02	1.3312
20	2-Ethylhexanol	C104767	C8H18O	130.2	1028.8	380.208	1.4177
21	methyl hexanoate	C106707	C7H14O2	130.2	920.8	275.385	1.2729
22	2-Heptanone	C110430	C7H14O	114.2	886.0	250.32	1.2614
23	Pentanal	C110623	C5H10O	86.1	695.7	162.027	1.1992
24	2-Pentanone	C107879	C5H10O	86.1	691.6	160.652	1.3624
25	(E)-2-hexen-1-ol	C928950	C6H12O	100.2	849.2	228.414	1.1794
26	2-pentyl furan	C3777693	C9H14O	138.2	987.7	331.679	1.25

GC-IMS技术在风味研究中的应用

- 地理标识性产品的保护
- 产品品种、品质的区分
- 产品真伪的鉴别
- 产品新鲜度和货架期的评估
- 产品加工适用性的选择
- 产品加工过程中风味的变化研究

FlavourSpec®风味分析仪

1.靶向区分广陈皮/陈皮，用于地理标识性产品的保护

“橘生淮南则为橘，生于淮北则为枳”，柑橘因种植的产地水土不同，得到的陈皮的挥发性有机物亦不同，直接称取0.2g陈皮粉置于顶空瓶中，经过仪器分析后，软件处理得到可视化的指纹图谱，如下图所示。

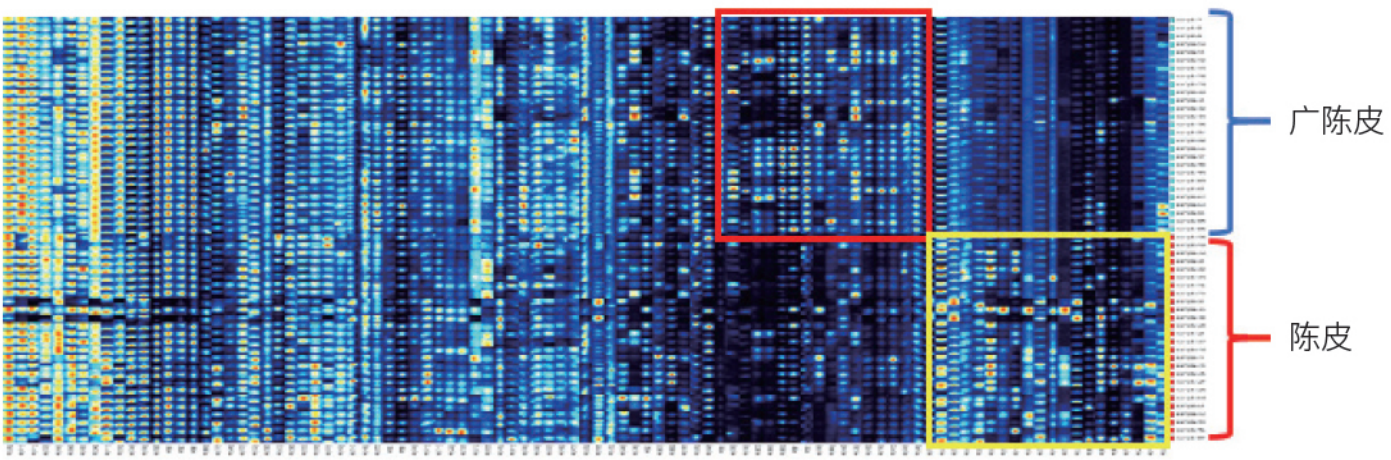


图3.广陈皮和陈皮中挥发性有机物指纹图谱

其中红框中的物质在广陈皮中含量很高，黄框中的物质在陈皮中含量很高。分别采用SE-54、1701和WAX三种不同极性的色谱柱进行分析，分别找到区别两种陈皮的12种、12种和14种特征挥发性有机物。

以SE-54色谱柱为例，12种特征标记物指纹图谱如图4，据此特征标记为做聚类分析结果如图5所示。

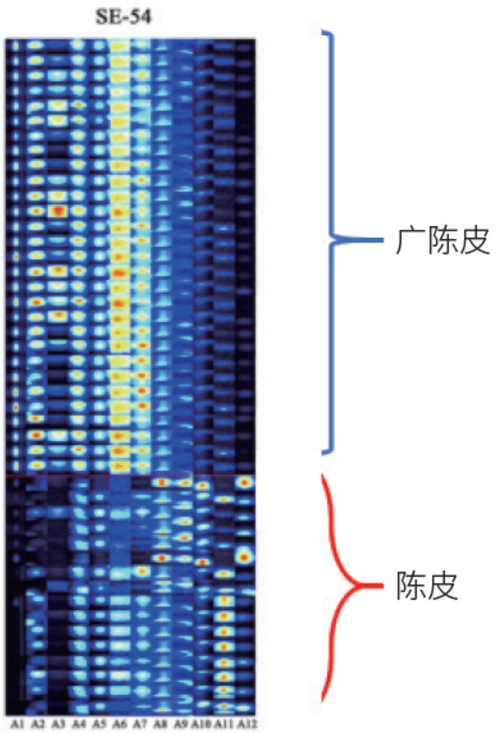


图4. SE-54色谱柱找到的12种特征标记物指纹图谱

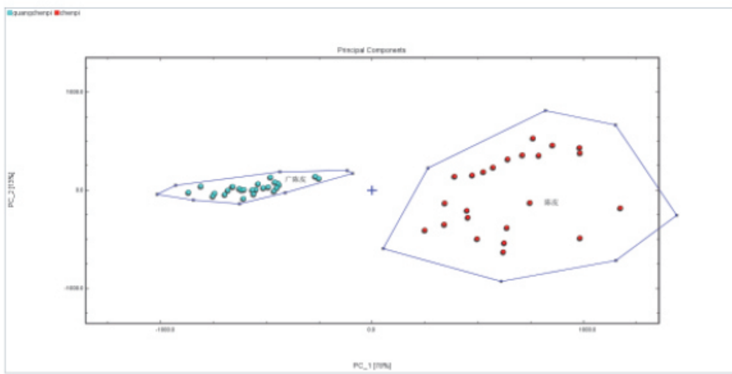


图5. 广陈皮与陈皮的PCA分析图

从图5可知，广陈皮样品聚集在PCA的左侧，其他产地的陈皮在图谱的右侧。根据挥发性有机物信息建立真实产地的陈皮分类模型后，可以用于广陈皮的道地区分。

Reference: Rapid discrimination of Citrus reticulata ‘Chachi’ by headspace-gas chromatography-ion mobility spectrometry fingerprints combined with principal component analysis. **Food Research International**. 131 (2020) 108985.

2.快速捕捉挥发性有机物差异，用于橄榄油等级的区分

根据GB 23347-2009橄榄油、油橄榄果渣油国家标准，橄榄油主要分为初榨橄榄油、精炼橄榄油、果渣油三大类，橄榄油的等级不同，其价格不同。

根据挥发性有机物的信息，可以对橄榄油进行等级区分，图谱如下图所示。

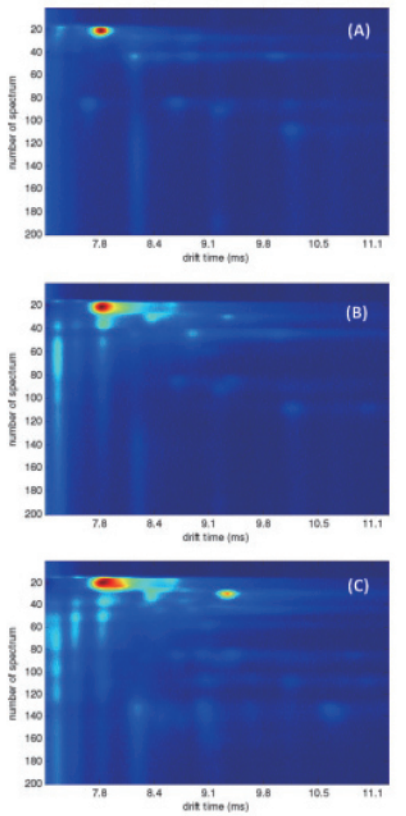


Fig. 5. Topographic plot of GC–IMS spectra. (A) Pomace olive oil, (B) olive oil and (C) extra virgin olive oil.

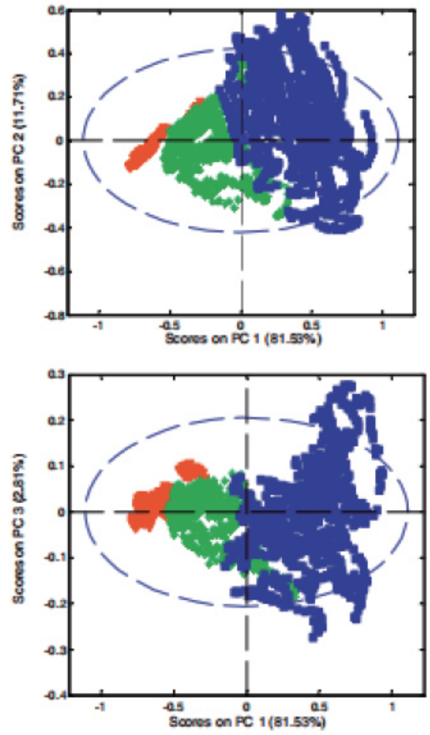


Fig. 3. PCA results for mean-centered data for the three types of oil as obtained with UV–IMS. Samples: Pomace olive oil (red triangles); olive oil (green asterisks); extra virgin olive oil (blue squares). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

图6.不同等级橄榄油的气相离子迁移谱图和PCA图

注：样品信息：A.果渣橄榄油、B.橄榄油、C.特级初榨橄榄油。

从上图知：特级初榨橄榄油（EVOO)产品中风味物质明显多于橄榄油（OO）和果渣橄榄油（POO）；本文中采用两种技术（GC-IMS和UV-IMS）对EVOO、OO、和POO进行分类，橄榄油等级区分的识别率高达95%。

Reference：Direct classifiication of olive oils by using two types of ion mobility spectrometers. Anal. Chim.**Acta （2011）.DOI: 10.1016/j.aca.2011.03.007.**

3.直观看出蜂蜜风味的差异，用于蜜源的识别及掺伪区分

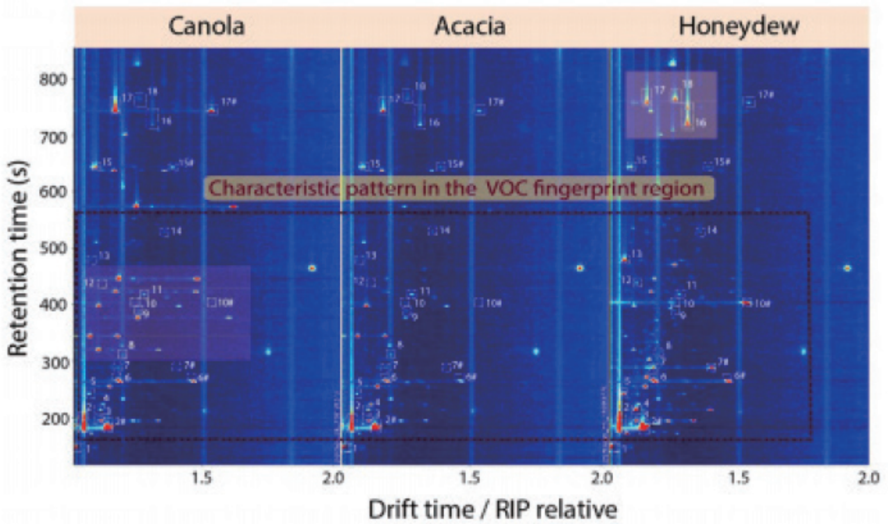


图7. 不同蜜源蜂蜜挥发性有机物的气相离子迁移谱图

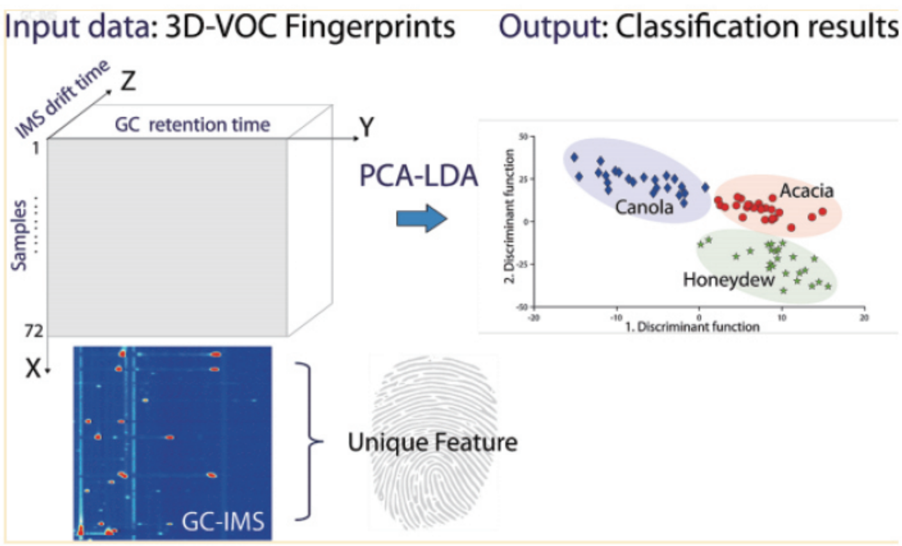


图8. 不同蜜源蜂蜜的PCA分析图

由上两图知：油菜花、金合欢花与蜜露蜂蜜挥发性有机物图谱差异明显；HS-GC-IMS技术可以作为^{1}H NMR蜂蜜样品溯源的可靠辅助工具。

Referenc: Volatile-Compound Fingerprinting by Headspace-Gas-Chromatography Ion-Mobility Spectrometry (HS-GC-IMS) as aBenchtop Alternative to 1H NMR Profiling for Assessment of the **Authenticity of honey. Analytical Chemistry DOI: 10.1021/acs.analchem.7b03748（2017）**

4.直观看出不同储存时间样品风味的变化，用于评估鸡蛋的新鲜度和货架期

不同存储时间鸡蛋的风味变化如下图所示：

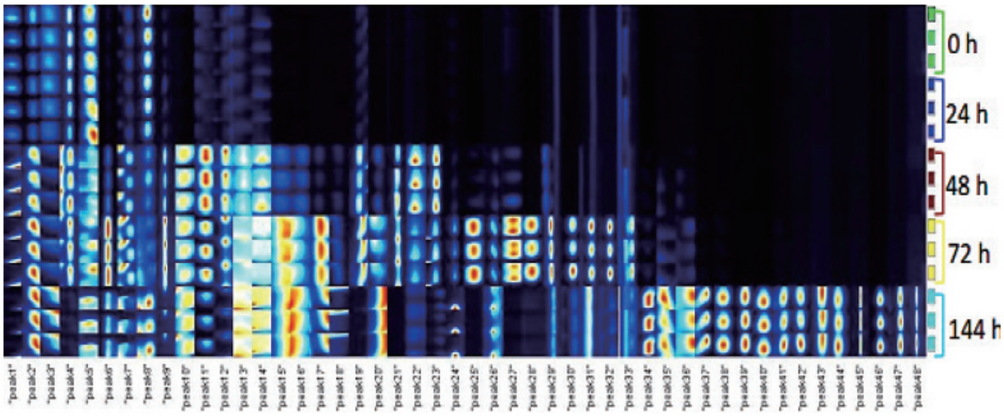


图9.不同存储时间的鸡蛋挥发性有机物指纹图谱

鸡蛋在室温下放置0-144h过程中气味逐渐发生变化；0-24h过程中，鸡蛋风味几乎不变，说明此阶段鸡蛋品质没有明显变化。

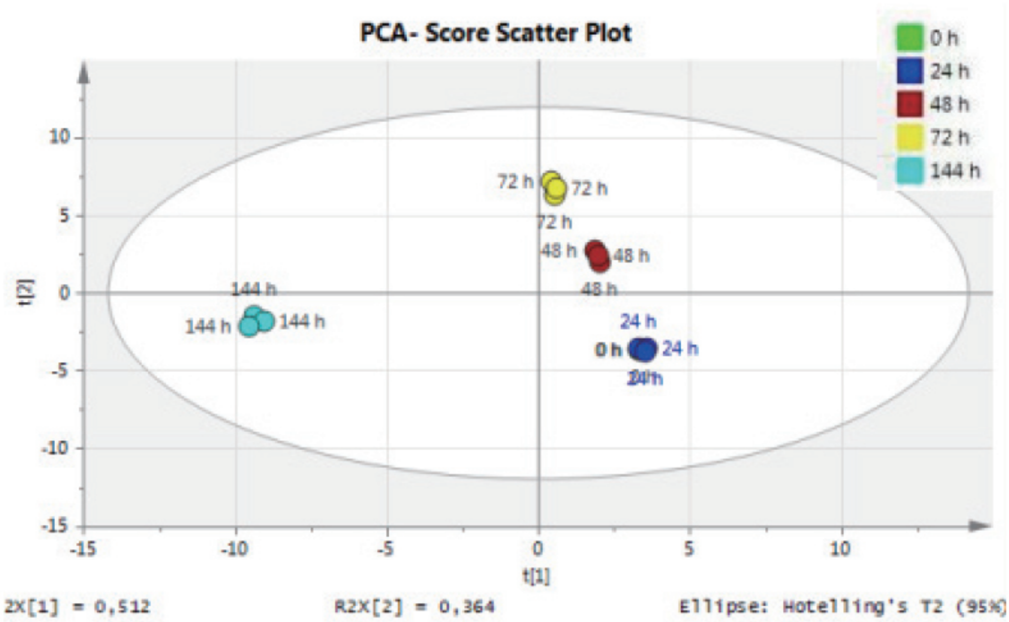


图10.不同存储时间的鸡蛋的PCA分析

建立不同储存时间鸡蛋的分类模型后，可用于快速判断鸡蛋的新鲜度。

Reference: Ion mobility spectrometry coupled to gas chromatography: A rapid tool toassess eggs freshness. **Food Chemistry, 271 (2019) 691-696.**

5.精准快速捕捉松茸加工前后风味的变化，用于加工适用性的优化选择

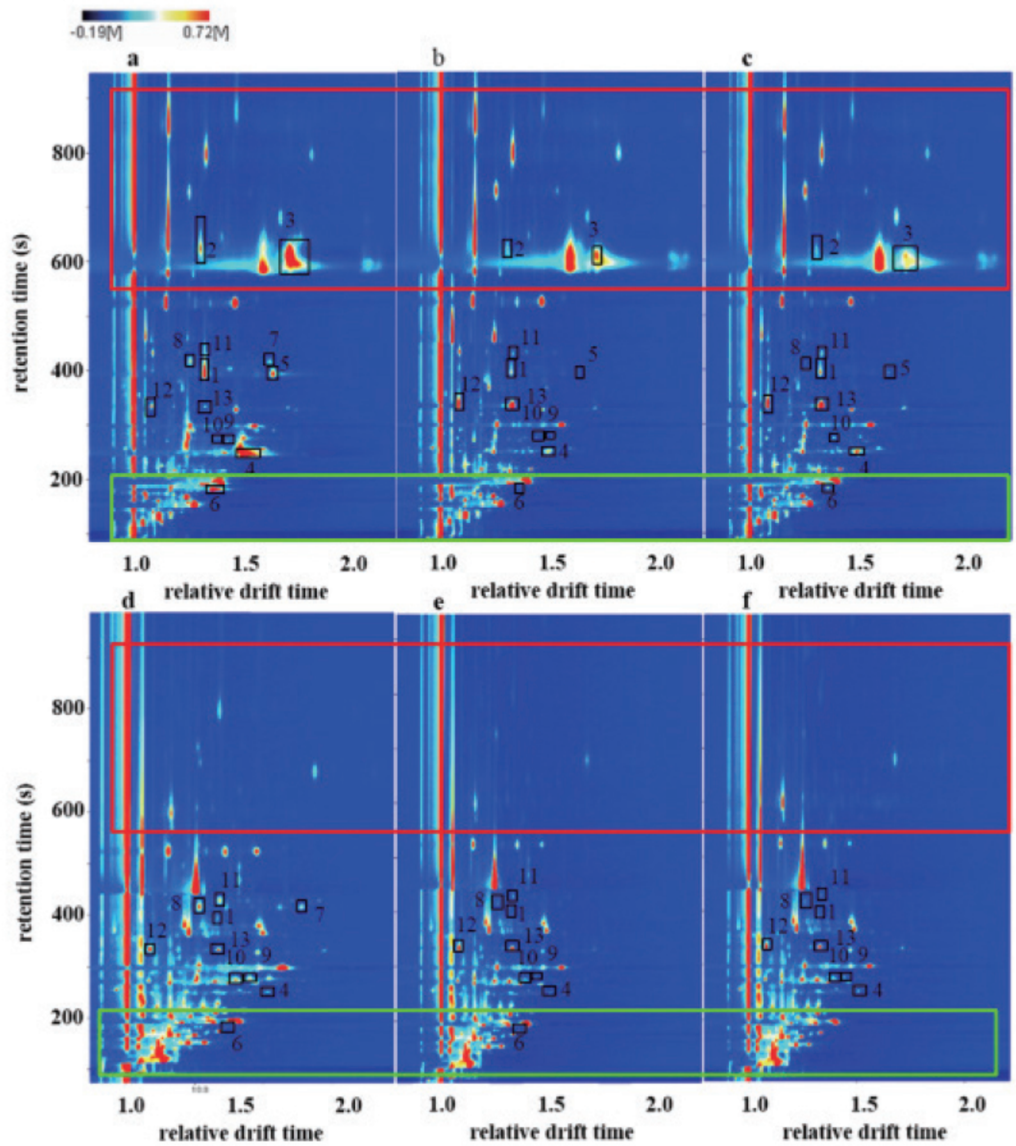


图11. 不同部位松茸烤干前后气相离子迁移谱图

注：a. 菌伞、b.菌柄、c.柄底；d.烤干菌伞、e.烤干菌柄、f.烤干柄底；

经谱图对比可知，松茸烤干后，不同部位的松茸样品红框中的标出的物质如1-Octene-3-ol（蘑菇醇）等消失。

将样品中挥发性有机物选取出来形成指纹图谱，如下图所示：

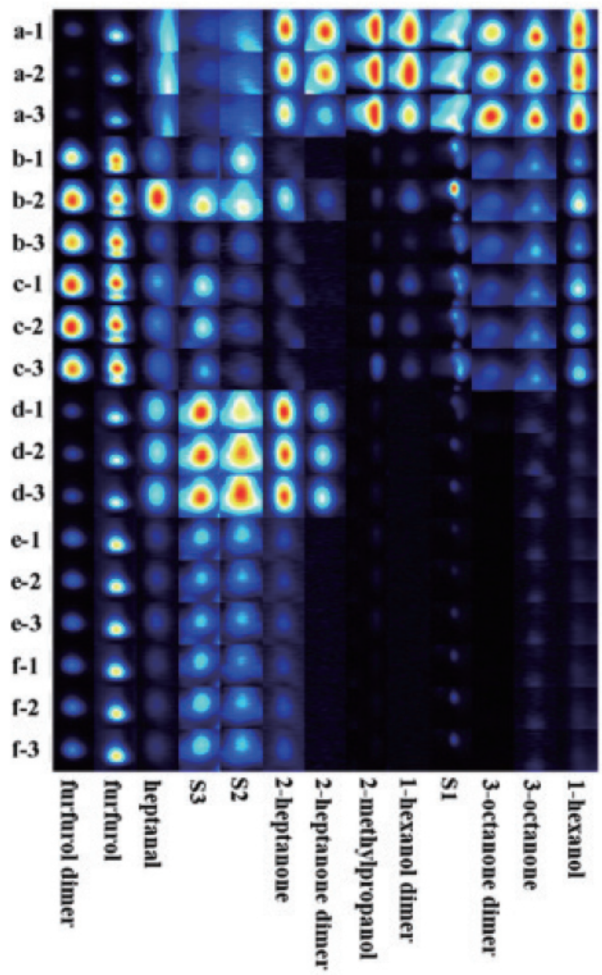


图12. 不同部位松茸烘干前后挥发性有机物指纹图谱

由左图知：

松茸部位不同，风味成分各不相同；菌伞中含量最高的成分在烤干后均消失，这是导致烤干前后松茸风味不同的主要原因。

根据加工前后不同部位松茸风味变化对比，结合感官评价及理化指标的数据，可用于选择最好的食材，亦可用于加工工艺的优化。

Reference: Characteristic volatiles fingerprints and changes of volatile compounds in fresh and dried Tricholoma matsutake Singer by HS-GC-IMS and HS-SPME-GC–MS. **Journal of Chromatography B, 1099. (2018) 46-55.**

6.发酵过程中风味的差异变化，用于优化发酵工艺及把控发酵时间

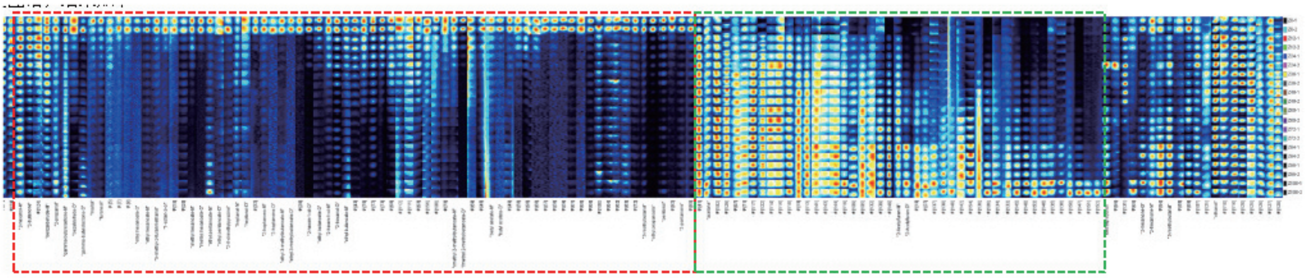


图13.不同发酵时间（每隔12h取样）红枣挥发性有机物的指纹图谱

红框中标出的物质随着发酵时间的延长，风味物质的种类和含量大幅减少；绿框中标出的物质随着发酵时间的延长，风味物质的种类和含量逐渐增多；结合感官评价和其他理化指标的检测，可用于发酵时间的优化和过程把控。

Reference：Content variations in compositions and volatile component in jujube fruits during the blacking process. **Food Sci Nutr. 2019;1-9.**

气相离子迁移谱（GC-IMS）技术在风味检测时，无需固相微萃取，快速捕捉样品的真实风味，检出限可达ppbv级别，软件给出直观可视的挥发性有机物指纹图谱，风味差异“看得见”，结合感官评价及其他理化指标的分析，可为地理标识性产品的保护、产品品种/品质的区分、产品新鲜度及货架期的评估、加工工艺的优化等提供科学的数据指导。

气相-离子迁移谱型号简介

FlavourSpec®风味分析仪

进样方式:顶空进样

应用领域:食品中风味物质分析
液体样品中挥发性有机物分析

BreathSpec®呼气分析仪

进样方式:气泵进样、定量环定量

应用领域:人体呼出气分析
细菌性感冒/病毒性感冒的快速区分
癌症(如肺癌、胃癌等癌症)的早期筛查
肺部真菌感染类型的快速区分
口腔加工学研究

GC-IMS环境气体分析仪

进样方式:气泵进样、定量环定量

应用领域:非破坏性食品风味分析
在线VOCs连续监测
公共安全在线监测
厂界异味溯源

附录

可视化的风味分析应用列表
---气相离子迁移谱(GC-IMS)在风味分析中的应用

一. 产地区分文章(节选)

1. 我国东北稻米溯源技术的研究. **农业部环境保护科研监测所研究生院**. 2018.

2. 一种基于香气物质指纹图谱的GC-IMS梨果产地区分方法. **专利公布号:CN 111398470 A. 山东省农业科学院农业质量标准与检测技术研究所**. 2020.

3. 新疆不同产区羊肉特征风味离子迁移色谱指纹谱的构建. **食品科学**. 2019.

4. 一种快速鉴定盐渍海参品质及产地的方法. **授权公告号: CN 108387648 B. 山东省海洋资源与环境研究院**. 2018.

5. 基于矿质元素与挥发性物质的“大红袍”花椒产地判别研究. **山西师范大学**. 2019.

6. 基于顶空气相色谱-离子迁移谱的不同产地咖啡挥发性有机物指纹图谱分析. **食品科学**. 2019.

7. 一种基于GC-IMS对不同产地烟草香气的评价方法. **专利公布号:CN 110907556 A 湖北中烟工业有限责任公司**. 2020.

8. Discrimination of Chinese Yellow Wine from Different Origins Based on Flavor Fingerprint. **Acta Chromatographica**. DOI: 10.1556/1326.2019.00613.

9. Rapid discrimination of Citrus reticulata ‘Chachi’ by headspace-gas chromatography-ion mobility spectrometry fingerprints combined with principal component analysis. **Food Research International**. 131 (2020) 108985. https://doi.org/10.1016/j.foodres.2020.108985.

10. Stable isotope signatures versus gas chromatography ion mobility spectrometry to determine the geographical origin of Fujian Oolong tea (Camellia sinensis) samples. **European Food Research and Technology** (2020) 246:955–964. https://doi.org/10.1007/s00217-020-03469-0

二. 品种区分文章(节选)

1. 一种不同企业生产的黄酒的气相离子迁移谱鉴别方法. **授权公告号:CN 109781918 B.浙江大学**. 2020.

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