
Le profil aromatique des produits de mangue de l'étude

Cette 1^{ère} partie est dédiée à la caractérisation du profil aromatique des produits de mangue frais et séchés de l'étude.

Dans un 1^{er} temps, une technique d'extraction des composés d'arôme de mangue a été mise en place pour étudier au mieux le potentiel aromatique des produits d'étude. Pour cela, la technique SAFE a été choisie et 2 protocoles ont été testés parmi ceux présentés dans la littérature. Seul le protocole le plus satisfaisant a été appliqué pour caractériser l'ensemble des produits de mangue utilisés pour la suite des travaux de thèse.

Dans un 2nd temps, l'étude des profils aromatiques des produits de mangue frais (purée, cubes) et séchés (poudre, cubes) par SAFE/GC-MS sur variété de mangue Kent a été réalisée. Les résultats obtenus ont été valorisés sous la forme d'un article accepté dans une revue à facteur d'impact.

4.1. Choix d'une technique d'extraction des composés d'arôme de mangue

4.1.1. Choix de la technique SAFE

L'extraction des COVs des produits de mangue par la technique SAFE a été choisie au détriment des autres méthodes d'extraction couramment menées sur ce fruit (SE, SDE, SPME, P&T, etc.). En effet, cette technique permet d'obtenir des extraits aromatiques représentatifs de l'arôme originel des matrices d'étude, sans génération d'artéfacts, à l'inverse des méthodes d'extraction classiques. La technique SAFE a notamment déjà été utilisée sur mangue par Munafo *et al.* (Munafo, Didzbalis, *et al.*, 2014; Munafo, Didzbalis, *et al.*, 2016) pour décrire le potentiel aromatique de plusieurs cultivars.

4.1.2. Choix et mise en place d'un protocole SAFE

Les travaux précédemment menés en SAFE mentionnent cependant deux techniques existantes : le SAFE en mode direct et le SAFE en mode indirect. Le SAFE direct consiste à réaliser l'extraction SAFE des COVs directement sur la matrice d'étude, tandis que, le SAFE

indirect demande dans un 1^{er} temps d'extraire les COVs à l'aide d'un solvant organique, puis l'extrait aromatique organique obtenu est traité en SAFE. Aucuns travaux ne reportent la méthode SAFE la plus adaptée à une application sur fruits, l'une et l'autre étant tout autant utilisées.

Deux protocoles SAFE direct et indirect, inspirés de la littérature, ont ainsi été testés sur de la purée de mangue fraîche élaborée à l'UMR Qualisud. Les composés d'arôme extraits ont ensuite été analysés en GC-MS. Les résultats SAFE/GC-MS obtenus en mode direct et indirect sont reportés dans le tableau ci-contre (Tableau 1).

D'un point de vue praticité du protocole, le SAFE en mode direct est plus simple à réaliser. Cette méthode demande moins de matériel (verrerie, appareils) et évite les étapes d'extraction liquide-solide au solvant en amont du SAFE souvent longues et fastidieuses à réaliser. Cependant, d'un point de vue temps d'extraction des composés d'arôme, le SAFE en mode direct est plus long à réaliser. En effet, l'extraction sous vide avec un solvant organique est plus rapide (1h15 à 1h30 pour le protocole SAFE indirect mis en place) qu'avec un solvant aqueux (2h à 2h30 pour le protocole SAFE direct mis en place) du fait que le point d'ébullition du solvant est plus faible que celui de l'eau.

D'un point du vue résultats GC-MS, les chromatogrammes obtenus par SAFE direct sont plus lisibles et facilement exploitables. En effet, des composés polluants peuvent être extraits en SAFE indirect, en plus des COVs, du fait des nombreuses étapes expérimentales intermédiaires du protocole.

En ce qui concerne la composition des extraits aromatiques obtenus, le SAFE direct et indirect permettent d'obtenir des résultats qualitatifs similaires. Des différences quantitatives significatives (ANOVA) sont cependant observées pour la moitié des composés extraits.

Tableau 10. Comparaison des techniques SAFE/GC-MS en mode direct et indirect sur purée de mangue fraîche.

Composés	IR DBWAX ^a	Quantité relative (µg/kg MF) ^b				ANOVA ^c
		SAFE direct		SAFE indirect		
		Moy	CV	Moy	CV	
<i>Monoterpènes</i>						
β-pinène	1088	9.3	8.1	27.1	37.1	n.s.
δ-3-carène	1133	7183.1	6.6	7665.0	6.7	n.s.
α-phellandène	1146	74.2	20.1	91.6	1.5	n.s.
β-myrcène	1151	251.6	8.6	278.6	3.0	n.s.
α-terpinène	1160	45.2	17.3	72.7	10.1	***
Limonène	1181	215.2	6.8	1024.0	6.3	**
β-phellandène	1189	83.2	10.2	93.0	8.1	n.s.
γ-terpinène	1228	8.6	12.0	11.7	5.0	n.s.
β-ocimène	1239	15.0	16.9	20.2	6.8	n.s.
p-cymène	1251	35.3	23.1	54.3	13.4	**
Monoterpène inconnu	1259	38.7	11.9	47.7	7.1	*
α-terpinolène	1264	386.0	12.9	438.3	4.0	n.s.
Total		8345.4	7.3	9824.2	6.5	*
<i>Sesquiterpènes</i>						
α-copaène	1467	4.9	5.1	9.0	18.1	n.s.
α-gurjunène	1502	12.5	16.5	15.3	15.8	n.s.
β-caryophyllène	1563	205.4	11.9	153.6	18.5	n.s.
α-caryophyllène	1633	114.9	10.7	66.9	18.9	*
Germacrène D	1669	8.2	5.5	9.2	8.5	n.s.
Total		345.9	11.4	254.0	18.0	n.s.
<i>Alcools</i>						
Pentan-3-ol	1102	8.6	16.1	18.0	7.7	**
Pentan-2-ol	1114	32.5	7.7	88.0	10.5	**
(E)-hex-1-ène-3-ol	1361	8.9	10.7	12.0	34.7	n.s.
(Z)-hex-1-ène-3-ol	1373	3.7	21.3	4.3	38.3	n.s.
Total		53.7	10.5	122.4	13.5	***
<i>Aldéhydes</i>						
Hexanal	1075	5.4	5.5	31.2	15.0	**
Heptanal	1176	t	t	18.6	30.4	*
Nonanal	1381	17.9	5.9	76.3	12.4	*
& hexénol (isomère)						
(E)-non-2-énal	1514	7.8	3.4	t	t	***
Total		31.1	5.2	126.1	15.7	**
<i>Furanes</i>						
2,5-diméthyl-4-méthoxy-3(2H)-furanone (Mésifurane, MDMF)	1572	8.3	13.9	6.8	18.6	n.s.
<i>Lactones</i>						
γ-butyrolactone	1586	21.8	10.0	34.0	2.9	**
γ-hexalactone	1657	8.2	10.9	8.7	9.8	n.s.
δ-hexalactone	1747	24.1	31.5	12.7	20.2	n.s.
δ-octalactone	1932	40.9	5.6	15.9	26.3	**
Total		95.0	13.7	71.3	12.0	*
Total des composés extraits		8879.5	7.6	10404.7	7.0	*

Légende :

^aIR, Indices de rétention expérimentaux des composés obtenus sur colonne DB-WAX.

^bQuantité relative des composés d'arôme en μg équivalent de nonan-4-ol par kg de matière fraîche de purée fraîche de mangue (semi-quantification, $\mu\text{g/kg MF}$) : (Moy) quantité relative moyenne sur les 3 répétitions (en $\mu\text{g/kg MF}$); (CV) coefficient de variation (en %).

^cANOVA, analyse de la variance avec Test de Tukey (HSD, honestly significant difference) avec un risque α de 5% : (n.s.) différence non significative ; (*) différence significative avec $\alpha < 5\%$; (**) différence significative avec $\alpha < 1\%$; (***) différence significative avec $\alpha < 0.1\%$.

Symboles : (\$) composés coélués sur colonne DBWAX, (n.f.) composés non détecté en GC-MS ; (t) composés à l'état de trace ($3 < \mu\text{g/kg MF}$)

Ainsi, des différences quantitatives significatives ont été observées en faveur du SAFE indirect pour des terpènes (α -terpinène, limonène, p-cymène et un monoterpène inconnu) ; des alcools (pentan-3-ol et pentan-2-ol) ; des aldéhydes (hexanal, heptanal, nonanal) et la γ -butyrolactone. L'utilisation d'un solvant organique peut permettre d'isoler plus de composés en amont de l'extraction SAFE. À l'inverse, l' α -caryophyllène, le (*E*)-non-2-ène et la δ -octalactone sont plus extraits avec le SAFE direct.

Sur les totaux par classes chimiques, les monoterpènes, les alcools et les aldéhydes sont davantage représentés en SAFE indirect, à l'inverse, les lactones sont davantage présentes en SAFE direct. Sur le total des composés extraits, le SAFE indirect permettrait d'extraire plus de composés (10.4 mg/kg MF) que le SAFE direct (8.9 mg/kg MF).

Enfin, les 2 protocoles ont une répétabilité similaire d'après les coefficients de variation. La difficulté de concentrer les extraits à volumes identiques et le souci de détection des composés en GC-MS peut expliquer les coefficients de variation un peu élevés pour certains COVs.

Les deux techniques SAFE permettent d'obtenir des extraits aromatiques satisfaisants (nature et quantité des COVs extraits) et avec une bonne répétabilité. Cependant, le SAFE direct présente des avantages pratiques d'un point de vue protocole (simple, rapide) et exploitation des résultats (chromatogramme propre, pic facilement intégrable). Ainsi, bien que le SAFE indirect puisse permettre d'extraire plus de COVs en quantité, c'est la technique du SAFE direct qui a été choisie et préférée pour le reste de l'étude.

4.2. Le profil aromatique des produits de mangue frais et séchés de l'étude

4.2.1. Application du protocole SAFE direct aux produits d'étude

Le protocole SAFE direct validé précédemment a été appliqué sur les différents produits de mangue. Pour caractériser le profil aromatique des produits de mangue frais (purée, cubes) et séché (poudre, cubes), les extractions SAFE direct ont été réalisées respectivement sur la purée de mangue fraîche et sur la poudre de mangue séchée.

4.2.2. Impact du séchage sur le profil aromatique de la mangue fraîche

Les composés d'arôme de mangue séchée et l'impact du séchage sur le profil aromatique de la mangue fraîche n'ayant jamais été rapporté dans la littérature, les résultats obtenus ont été valorisés sous la forme d'un article.

a) Contexte de l'étude

Pour étudier l'impact du séchage sur les composés d'arôme de mangue fraîche, de la purée fraîche de mangue et de la poudre séchée de mangue ont été confectionnées à partir d'un lot de mangues homogène de variété Kent (32 mangues, en provenance du Pérou, importation par bateau). La sélection des fruits s'est faite suivant l'aspect général des fruits et suivant leur indice de fermeté (Indice Durofel). Les propriétés physicochimique des produits ont été relevées ($^{\circ}\text{Bx}$, pH, AT, aw, MS). La technique du SAFE direct a été appliquée et les composés d'arôme ont été analysés en GC-MS. Les composés ont été quantifiés grâce à l'utilisation d'un étalon interne, le nonan-4-ol. Des tests statistiques (ANOVA avec test de Tukey et un risque α de 5 %) ont été opérés pour pouvoir comparer les profils aromatiques de la mangue fraîche et séchée pour chaque composé d'arôme, les différences significatives montrant l'impact du séchage. L'OAV des composés a été calculée en considérant la quantité des composés d'arôme et les seuils de détections odorants de ces composés dans l'eau mentionnés dans la littérature. À noter que les composés dont l'OAV est supérieure à 1 peuvent être considérés comme impactant pour l'arôme du produit.

b) Résultats obtenus sur l'impact du séchage sur l'arôme de mangue fraîche

Les résultats SAFE/GC-MS obtenus sur l'impact du séchage sur le profil aromatique de la mangue fraîche sont présentés dans l'article ci-contre "*Aroma compounds in fresh and dried mango fruit (Mangifera indica L. cv. Kent): impact of drying on volatile composition*". Cet article a été soumis et accepté dans l'International Journal of Food Science and Technology (2016).

Original article

Aroma compounds in fresh and dried mango fruit (*Mangifera indica* L. cv. Kent): impact of drying on volatile composition

Adeline Bonneau,^{1,2} Renaud Boulanger,² Marc Lebrun,² Isabelle Maraval² & Ziya Gunata^{1*}

¹ UMR Qualisud, University of Montpellier, 34095, Montpellier Cedex 5, France

² UMR Qualisud, CIRAD, 34398, Montpellier Cedex 5, France

(Received 31 August 2015; Accepted in revised form 25 November 2015)

Summary Volatile compounds from fresh and dried mango were extracted by the solvent-assisted flavour evaporation (SAFE) technique and analysed by GC-MS. Forty-one and fifty five volatile compounds were identified in fresh and dried mango, respectively. Monoterpenes, followed by sesquiterpenes, lactones and alcohols were the major compounds. Drying induced substantial losses of several compounds. The total amount of volatiles decreased by about 59%. These losses could be mainly attributed to the evaporation of the volatiles during drying, the extent of which seemed to increase with the hydrophobicity and Henry's law constant of the compounds. However, new compounds appeared and enrichment of some compounds was observed after drying. Limonene, β -myrcene, δ -3-carene, β -caryophyllene, γ -butyrolactone and 3-methylbutyl butanoate were found to be flavour contributors in both products on the basis of the odour activity values (OAVs). Mesifuran displayed high OAV only in fresh fruit while hexanal and heptanal only in dried mango.

Keywords Dried fruit, fresh fruit, mango, odour activity value, solvent-assisted flavour evaporation.

Introduction

Mango (*Mangifera indica* L.) is a climacteric drupe fruit with a fleshy and juicy mesocarp representing the edible part. Its annual production reaches 30 MT, being the most tropical fruit produced after banana. Its distinct and attractive flavour is highly appreciated by consumers. This fruit has nutritional benefits due to its high fibre, vitamin C and β -carotene content (Tharanathan *et al.*, 2006; Hassan *et al.*, 2013).

Mangoes are mainly consumed fresh. The fresh fruit has a short shelf-life, being quickly perishable, thus causing texture, flavour and taste deterioration. To extend the storage life and meet consumer demand, mangoes are processed into juice, puree, jam and dried fruit. Consumers' food preferences are closely linked with the sensorial characteristics of the products. Flavour is considered to be a key feature affecting consumers' preference. The volatile composition of fresh mango fruit from numerous cultivars has been widely investigated. Several hundred volatile compounds occurring in free form (Pino *et al.*, 2005; Pandit *et al.*, 2009a; Munafo *et al.*, 2014) and more than sixty compounds as glycosidically bound conjugates have been

detected (Lalel *et al.*, 2003c). Monoterpenes, sesquiterpenes, aldehydes, esters, ketones, lactones, alcohols, fatty acids, aliphatic hydrocarbons and shikimate derivatives were identified (Pino *et al.*, 2005; Pandit *et al.*, 2009a; Munafo *et al.*, 2014). Mono- and sesquiterpenes were found to constitute 70–90% of all volatile compounds (Pino *et al.*, 2005; Pandit *et al.*, 2009a). Odour-active compounds in fresh fruits from several mango cultivars have been identified by calculating the flavour dilution factor (FD) of the volatile compounds through gas chromatography-olfactometry (GC-O) analysis (Pino, 2012; Munafo *et al.*, 2014). Monoterpenes like limonene, δ -3-carene, β -myrcene, (*Z*) and (*E*)- β -ocimene; sesquiterpenes like β -caryophyllene; esters like ethyl butanoate, ethyl-2-methylbutanoate and ethyl-3-methylbutanoate; aliphatic aldehydes like (*E,Z*)-2,6-nonadienal, (*E*)-2-nonanal and (*E*)-3-hexenal; furane and furanone derivatives like 2,5-dimethyl-4-methoxy-3(2H)-furanone (mesifuran) and 4-hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF); lactones like γ -octalactone; and norisoprenoidic ketones like β -ionone and β -damascenone were detected with the highest FD factors. They were therefore considered as being amongst the most odour-active compounds. Volatile compounds exhibiting high FD factors from mango fruit were generally found to have high odour activity

*Correspondent: E-mail: zgunata@univ-montp2.fr

2 Impact of drying on volatile composition A. Bonneau et al.

values (OAVs) (Pino, 2012). OAV is the ratio of the volatile concentration in the matrix to its odour threshold determined in the matrix or in a medium close to the matrix. Hence, it gives some clues on the possible impact odorants in the matrix. Interestingly, volatile compounds with high OAVs were often found to be important in the flavour of the product (Grosch, 2001).

In this study, the effect of drying on the volatile composition of mango fruit (cv. Kent) was studied. The effect of mango drying on fruit physicochemical characteristics and texture has been studied (Caparino *et al.*, 2012). However, no reports were found to be available on changes regarding the volatile composition. Drying of nectarines (Sunthonvit *et al.*, 2007), lulo fruit (Forero *et al.*, 2015) and plums (Sabarez *et al.*, 2000) caused significant changes in the volatile composition compared to the fresh fruit. The Kent cultivar was chosen for this study as it is one of the main cultivars cropped worldwide. The fruit was dried in a pilot unit in conditions similar to those implemented in the industry. The solvent assisted flavour evaporation (SAFE) technique was chosen to extract volatile compounds from fresh and dried fruits. So far, only one published study has used this technique to characterise aroma-active compounds from several mango cultivars (Munafò *et al.*, 2014). Other extraction techniques, such as liquid-liquid extraction (LLE), solid-liquid extraction (SLE) (Pandit *et al.*, 2009a), simultaneous distillation-extraction (SDE) (Pino *et al.*, 2005) and solid-phase micro-extraction (SPME) (Lalel *et al.*, 2003a,b), have been used for the recovery of volatile compounds from mangoes. The SAFE technique is operated under high vacuum at a low extraction temperature, leading to the preparation of aroma extracts that represent the flavour of the original product (Munafò *et al.*, 2014). In the current study, volatile compounds isolated by SAFE from fresh and dried mango were analysed by GC-MS. Observed changes in volatile composition as well as the contribution of volatile compounds to the flavour of both products through their OAVs are discussed.

Materials and methods

Chemical reagents

Chemical solvents, pentane, dichloromethane and methanol were from Sigma-Aldrich (St. Louis, MO, USA). 4-nonanol (internal standard) was from Alfa Aesar (Karlsruhe, Germany). 2-methyl-1-propanol, 1-butanol, β -pinene, α -phellandrene, heptanal, β -phellandrene, γ -terpinene, α -terpinolene, α -caryophyllene, 3-hydroxy-2-butanone, 4-hydroxy-4-methyl-2-pentanone, nonanal, benzaldehyde and PEA were from Sigma-Aldrich (Steinheim, Germany); hexanal, limonene, 2-methyl-1-butanol, 5-methylfurfural, 1-octanol,

β -caryophyllene and β -ionone from Fluka Analytical (Steinheim, Germany); 2-pentanol, 3-methyl-1-butanol, butyl butyrate, isoamyl butyrate, (Z)-3-hexen-1-ol, furfural, γ -butyrolactone and γ -hexalactone from Interchim (Montluçon, France); δ -3-carene, β -myrcene and δ -octalactone from Fabster (Angerville, France); o-xylene from Alfa Aesar; (E)-3-hexen-1-ol from Sarsynthex (Merignac, France); and 1-hexanol from SBI Sanofi (Grasse, France). Series of n-alkanes, C₈ to C₂₀, were from Sigma-Aldrich (St. Louis, MO, USA).

Fresh and dried mango samples

Twenty-two mango fruits (cv. Kent, Peruvian origin) were purchased at a local market in Montpellier, France. Fruits were selected at mature and ripe stage on the basis of their similar aspects, that is size, shape, weight, skin (no dehydration, no damage or fungal infections), texture or firmness (determined by touch by an experienced CIRAD researcher). Fruits were placed in a climatic chamber (KBF 720 E2.1; Binder GmbH, Tuttlingen, Germany) at 25 °C and 90% relative humidity for 2 days to obtain fully ripe fruits. The degree of mango ripening was estimated on the basis of the firmness using a durometer (Durofel; Setop Giraud Technologie, Cavaillon, France) equipped with a 0.10 cm² probe. Two measurements were performed on each mango cheek.

Ripened fruits were manually washed with chlorine solution (NaClO, 100 ppm) at ambient temperature for 10 min and then rinsed abundantly with water. Materials (peeler, knife, mandolin, bowls, cutter table, jars) were washed with chlorine solution (NaClO, 100 ppm) followed by rinsing with water. Fruits were peeled, and cheeks were obtained by slicing the fruit lengthwise with a mandolin. Rectangular fruit slices from each mango were obtained (around 0.8 width $\times$ 0.8 height $\times$ 8 length in cm), gathered and divided into two equal batches. One batch was used to analyse the volatile profile of fresh mango fruit, while the other batch was subjected to a drying process prior to volatile analysis. Fresh fruit was processed into puree in a laboratory blender. The resulting puree was rapidly divided in fractions of around 80 g, immediately frozen under liquid nitrogen and kept at -80 °C until analysis. The other sliced mango batch was processed at the CIRAD experimental unit in Montpellier to obtain dried mango. Drying was performed with a pilot unit (UTA dryer, Villeneuve-sur-Lot, France) at 60 °C, 40% relative humidity and constant air flow. Drying was stopped after 7 h when the fresh fruit had lost around 80% of its initial weight, corresponding to less than 0.6 water activity (a_w), that is the industry standard safety value for dried fruit storage. Dried rectangular mango slices were cut into small sections (around 0.8 width $\times$ 0.8

height $\times$ 1 length in cm) and ground to a powder under liquid nitrogen with a ball crusher (Dangoumill 300; Prolabo, Fontenay-sous-Bois, France). The powder was rapidly separated in fractions of around 80 g and stored at -80°C until analysis.

Physico-chemical characterisation of mango samples

Fresh and dried mangos were characterised for their total soluble solids, pH, titratable acidity, water activity and dry matter.

Total soluble solids were determined with a hand-held refractometer (ATAGO® PAL-3; Atago Instruments, Tokyo, Japan). pH was measured with pH titrator (Titroline 96; Schott-Geräte GmbH, Mainz, Germany). Titratable acidity (TA) was evaluated using a pH titrator (Titroline 96; Schott-Geräte GmbH) and 0.1 M NaOH solution (Sigma-Aldrich, St. Louis, MO, USA). TA was expressed in % mEq citric acid, which is the major organic acid in mango fruit (Tharanathan *et al.*, 2006). Water activity (a_w) was measured with an a_w -meter (AquaLab 4TE; Decagon Devices, Pullman, WA, USA) at 25°C . Dry matter (DM) was determined by the AOAC procedure (method 934.06/37.1.10) (AOAC, 2000) with a vacuum oven operated at 70°C for 48 h (Heraeus RVT 360; Heraeus GmbH, Hanau, Germany). DM was expressed in %.

All measurements with puree and powdered dried mango were performed in three replicates. Prior to $^{\circ}\text{Bx}$, pH and TA analysis, distilled water was added to the dried mango powder.

Extraction of aroma compounds

Volatile compounds were extracted using the solvent-assisted flavour evaporation (SAFE) technique (Munafó *et al.*, 2014). Sixty grams of mango product (fresh puree or dried mango powder) were placed in a 500 mL flask containing 100 mL of ultrapure water and 1 mL of internal standard solution (4-nonanol at $30.0\ \mu\text{g mL}^{-1}$ in water:methanol 99:1, v/v). A magnetic stir bar and PTFE boiling stones were placed in the flask to regulate the agitation and distillation. The water bath temperature was held at 45°C . The volatile fraction was isolated by high vacuum (10^{-3} mbar) and collected in a flask cooled with liquid nitrogen. Distillation lasted for 90 min, when high vacuum had been reached. The distillate was recovered, thawed at room temperature and extracted three times with a pentane-dichloromethane mixture (2:1, v/v) at ambient temperature. The solvent volume was 100 mL for the first extraction and then 50 mL. Organic extracts were combined and dried over anhydrous sodium sulphate. They were first concentrated to approximately 400 μL at 45°C using a Kuderna Danish column, followed by concentration to around 200 μL under a gentle

nitrogen stream. Each mango product was extracted in triplicate.

Analysis of aroma compounds

An Agilent 6890 series GC (Agilent Technologies, Palo Alto, CA, USA) equipped with a DB-WAX polar column (30 m $\times$ 0.25 mm, 0.25 μm phase film thickness, Agilent J&W GC column) coupled to an Agilent 5973 mass spectrometer detector (Agilent Technologies) was used. Hydrogen was used as carrier gas at $1.5\ \text{mL min}^{-1}$ at constant flow. One microlitre of aroma extract was injected (on-column injection at 45°C). Five minutes after injection, the oven temperature increased by $2^{\circ}\text{C min}^{-1}$ to 115°C and then $10^{\circ}\text{C min}^{-1}$ to 250°C . The oven was held at the final temperature for 10 min. The mass spectrometer was operated in electron impact (EI) ionisation mode at 70 eV and at a scan range of 35 to 350 m/z.

Volatile compounds were identified through their mass spectra (NIST version 2.0; National Institute of Standards and Technology, Gaithersburg, MD, USA), their retention indices (RIs) and the injection of standards, when available. The RI of the compounds was determined by linear interpolation following the injection of n-alkanes (C_8 - C_{20}). Volatile compounds were semi-quantified as equivalent to 4-nonanol (internal standard).

Statistical analysis

ANOVA was performed with an honestly significant difference (HSD) in a Tukey test at a 95% significance level using XLSTAT software (version 2014.1.01; Addinsoft, Paris, France).

Results and discussion

Preparation and characterisation of fresh and dried mango samples

The flavour composition of climacteric fruits like mango is greatly influenced by the degree of ripeness (Pandit *et al.*, 2009b). Moreover, it is very usual to find mangoes of different degrees of ripeness in the same batch. To obtain homogenous batches of mangoes in terms of ripeness, mango fruit samples were matured in a climatic chamber at 25°C and 90% relative humidity for 2 days. The degree of mango ripening was estimated by a nondestructive method using a Durolfel penetrometer. The latter has already been used on several fruits, including mangos, to assess the degree of ripeness (Valente *et al.*, 2013).

Two cheeks from each mango were subjected to Durolfel analysis. More than 90% of the fruits presented Durolfel indices (DI) between 60 and 80 before

4 Impact of drying on volatile composition A. Bonneau et al.

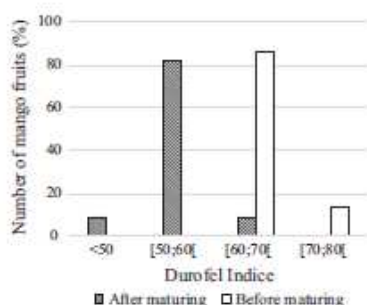


Figure 1 Distribution of mango fruits according to the Durofel indice before and after maturing in a climatic chamber.

maturing (Fig. 1). Note that DI is an arbitrary value ranging from 0 to 100. Maturing conditions were successful for decreasing the firmness as DI was between 50 and 60 for 80% of the fruits. Consequently, maturation in climatic chamber enabled us to obtain a quite homogenous fruit batch regarding ripeness. Fruits were divided into two batches, one for the fresh fruit samples and the other for dried fruit preparation.

The data on total soluble solids ($^{\circ}\text{Bx}$), pH, titratable acidity (TA), water activity (a_w) and dry matter (DM) of fresh and dried mango fruit were in agreement with those reported in earlier studies (Pott *et al.*, 2005; Tharanathan *et al.*, 2006) (Table 1). Dried mango fruit presented a water activity (0.5132) in agreement with the values (≤ 0.6) for microbiological safety of dried fruits (Pott *et al.*, 2005).

Changes in volatile constituents from fresh to dried mango

Fresh fruits were processed into puree to obtain homogenous samples prior to the recovery of volatile compounds by the SAFE technique. To better extract volatile compounds from dried mango, the samples were powdered prior to SAFE. Volatile levels were

expressed in dry matter (DM) to highlight changes following fruit drying.

Fresh fruit volatile composition of cv. Kent mangoes was dominated by monoterpene hydrocarbons (90.2%), and sesquiterpene hydrocarbons accounted only for about 3.0% (Fig. 2). This is in agreement with a previous study where cv. Kent was classified amongst monoterpene-dominant cultivar groups, in contrast to sesquiterpene-dominant groups (Pandit *et al.*, 2009a). A total of thirty nine volatile compounds were identified, including 27 that were positively identified (Table 2). These included eleven monoterpene hydrocarbons, four sesquiterpene hydrocarbons, nine alcohols, three ketones, two esters, one aldehyde, one furan, five lactones and three nonterpene hydrocarbons (Flavornet, 2004; Pino *et al.*, 2005; Pino & Mesa, 2006; Pandit *et al.*, 2009a; NIST, 2011; Pherobase, 2014; The LRI and Odour Database).

The total amount of volatiles was 27.4 mg kg^{-1} DM. δ -3-carene was the most abundant monoterpene (79.8%). This compound was also reported to be the major aroma compound in cv. Kent (Pino & Mesa, 2006), and in other cultivars like Haden, Tommy Atkins and Keitt (Andrade *et al.*, 2000; Pino & Mesa, 2006). β -caryophyllene was predominant sesquiterpene compound. Lactones, mainly γ -butyrolactone (2.6%), and alcohols, mainly 1-butanol (0.6%), were amongst other quantitatively important constituents. Most of the compounds detected have already been reported in fresh cv. Kent mangoes. Thirteen compounds were not previously detected in cv. Kent, but they have been found in other mango cultivars. They included β -phellandrene, m- and o-xylene, 2-methyl-1-propanol, 3-pentanol, 1-pentanol, (E)-3-hexen-1-ol, 3-hydroxy-2-butanone, mesifuran, 5-methylfurfural, α -methyl- γ -butyrolactone, γ -hexalactone, δ -hexalactone and δ -octalactone.

Three compounds, that is 2-pentanol, 2-decanol and 4-hydroxy-4-methyl-2-pentanone, were observed for the first time in the volatile composition of mango. These compounds have already been detected in other fruits. 2-pentanol and 2-decanol were found in Brazilian plum (De Sousa Galvão *et al.*, 2011), and

Table 1 Main analysis of fresh and dried mangoes

Mango products	$^{\circ}\text{Bx}^a$	pH ^b	TA ^c	a_w ^d	DM ^e
Fresh fruit	17.2	3.9	0.7	0.9764	18.2
Dried fruit	82.9	4.6	1.1	0.5132	87.5

^aTotal soluble solids ($^{\circ}\text{Bx}$) $\pm 0.1^{\circ}\text{Bx}$.

^bpH ± 0.1 .

^cTitratable acidity (TA) was expressed in % mEq citric acid.

^dWater activity (a_w) was expressed unitless ± 0.0001 at 25 $^{\circ}\text{C}$.

^eDry matter (DM) was expressed in %.

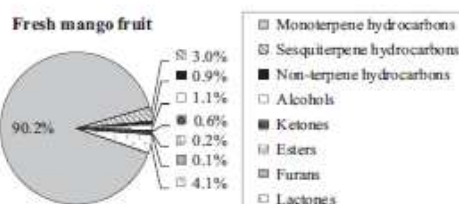


Figure 2 Distribution (%) of chemical class of volatile compounds in fresh mango.

Table 2 Aroma compounds in fresh and dried mango fruit

Compound	CAS no.	LogP ^a	HLC ^b	R ² DBWAX		ID ^d	Relative quantities (µg kg ⁻¹ DM) ^e		Significance ^f
				Lit	Exp		Fresh fruit	Dried fruit	
Monoterpene hydrocarbons									
β-pinene*	127-91-3	4.350	6.37E-02	1113	1105	(A)	20.3	16.8	n.s.
δ-3-carene*	13466-78-9	4.610	8.18E-02	1148	1143	(A)	2182.4	7511.0	**
α-phellandrene*	99-83-2	4.620	1.20E-01	1166	1155	(A)	223.3	43.6	***
β-myrcene*	123-35-3	4.880	6.22E-02	1150	1159	(A)	430.5	234.2	**
α-terpinene*	99-86-5	4.750	5.03E-02	1178	1169	(B)	113.9	17.6	**
Limonene*	138-86-3	4.830	5.67E-02	1188	1189	(A)	580.2	174.6	**
β-phellandrene ^o	555-10-2	4.700	1.43E-01	1209	1197	(A)	229.4	61.4	**
γ-terpinene*	99-85-4	4.750	5.70E-02	1238	1235	(A)	23.9	8.8	***
(E)-β-ocimene*	3779-61-1	4.800	2.39E-01	1242	1246	(B)	23.9	17.6	n.s.
Unknown monoterpene ^g	-	-	-	-	1265	(C)	132.0	19.6	**
α-terpinolene*	586-62-9	4.880	4.67E-02	1275	1270	(A)	1067.0	201.4	**
Total							2468.9	8306.8	
Sesquiterpene hydrocarbons									
α-copaene*	3856-25-5	5.360	4.05E-02	1488	1468	(B)	35.1	15.9	*
α-gurjunene*	489-40-7	6.180	8.60E-02	1529	1504	(B)	62.6	15.7	***
β-caryophyllene*	87-44-5	6.300	1.67E-01	1570	1565	(A)	525.1	210.9	**
α-caryophyllene*	6753-98-6	6.950	2.95E-01	1640	1634	(A)	199.2	85.3	**
Total							822.0	327.8	
Norisoprenoid									
β-ionone*	14901-07-6	4.420	4.06E-04	1912	1906	(A)	n.d.	t	n.s.
Nonterpene hydrocarbons									
Toluene*	108-88-3	2.540	5.01E-03	1042	1034	(A)	165.5	73.8	*
m-xylene ^o	108-38-3	3.090	4.46E-3	1138	1131	(B)	46.2	9.1	**
p-xylene ^o	95-47-6	3.090	3.33E-03	1182	1174	(A)	19.6	20.6	n.s.
Total							231.3	103.5	
Alcohols									
2-methyl-1-propanol ^o	78-83-1	0.770	1.35E-05	1077	1082	(A)	n.d.	112.5	***
3-pentanol ^o	584-02-1	1.260	2.64E-05	1110	1112	(B)	11.5	9.0	*
2-pentanol	6032-29-7	1.260	2.15E-05	1117	1124	(A)	43.0	30.6	*
1-butanol*	71-36-3	0.840	9.89E-06	1138	1146	(A)	175.6	48.9	**
3-methyl-1-butanol ⁴⁵	123-51-3	1.260	1.07E-05	1206	1213	(A)	18.2 ⁵	710.8 ⁵	**
8-2-methyl-1-butanol ⁴⁵	137-32-6	1.260	1.64E-05	1206	1213	(A)			
1-pentanol ^o	71-41-0	1.330	1.47E-05	1244	1253	(B)	n.d.	19.1	***
2-decanol	1120-06-5	3.710	2.72E-05	n.f.	1285	(C)	46.3	t	**
(E)-2-penten-1-ol	1576-96-1	1.120	6.52E-06	1316	1313	(B)	n.d.	5.6	***
(Z)-2-penten-1-ol*	1576-96-0	1.120	6.52E-06	1326	1321	(B)	n.d.	52.8	***
1-hexanol*	111-27-3	1.820	1.72E-05	1354	1365	(A)	15.07	31.6	**
(E)-3-hexen-1-ol ^o	928-97-2	1.610	7.72E-06	1356	1365	(A)	t	13.0	*
(Z)-3-hexen-1-ol*	928-96-1	1.610	7.72E-06	1378	1376	(A)	t	7.2	**
1-octanol ^o	111-87-5	2.810	2.08E-05	1557	1560	(A)	n.d.	6.4	***
Total							309.6	1047.0	
Aromatic alcohol									
2-phenylethanol*	60-12-8	1.570	1.78E-07	1893	1888	(A)	n.d.	16.9	***
Aldehydes									
Hexanal*	66-25-1	1.800	3.58E-04	1075	1078	(A)	t	26.8	***
Heptanal*	111-71-7	2.290	4.53E-04	1183	1180	(A)	n.d.	55.7	*
Nonanal ⁴⁵	124-19-6	3.270	8.02E-04	1392	1382	(A)	n.d. ⁵	26.6 ⁵	**
8-hexenal ⁵ (isomer)	-	-	-	-	1382	(C)			
(E,Z)-2,4-heptadienal	4313-02-4	1.860	2.19E-05	1455	1449	(B)	n.d.	12.8	**
(E,E)-2,4-heptadienal	4313-03-5	1.860	2.19E-05	1483	1473	(B)	n.d.	14.4	***
Total							t	136.3	
Aromatic Aldehyde									
Benzaldehyde*	100-52-7	1.710	2.31E-05	1496	1495	(A)	n.d.	6.6	***

6 Impact of drying on volatile composition A. Bonneau et al.

Table 2 (Continued)

Compound	CAS no.	LogP ^a	HLC ^b	RI ^c DBWAX		ID ^d	Relative quantities ($\mu\text{g kg}^{-1}$ DM) ^e		Significance ^f
				Lit	Exp		Fresh fruit	Dried fruit	
Ketones									
2-heptanone*	110-43-0	1.730	3.44E-04	1160	1116	(B)	30.4	t	***
3-hydroxy-2-butanone ^g	513-86-0	-0.360	2.79E-07	1272	1271	(A)	113.5	204.3	*
4-hydroxy-4-methyl-2-pentanone	123-42-2	-0.340	1.15E-07	1385	1347	(A)	10.5	18.2	**
Total							154.3	222.5	
Esters									
Butyl butanoate*	109-21-7	2.830	1.08E-03	1228	1214	(A)	30.2	11.6	**
3-methylbutyl butanoate*	106-27-4	3.250	1.79E-03	1267	1261	(A)	13.1	30.3	*
Total							43.3	41.9	
Furans									
Mesifuran (MDMF) ^g	4077-47-8	0.620	1.66E-06	1584	1577	(B)	16.9	t	***
2-pentylfuran*	3777-69-3	3.870	5.22E-03	1231	1227	(B)	n.d.	5.2	***
2-furfural*	98-01-1	0.830	5.48E-06	1456	1447	(A)	n.d.	62.8	***
5-methylfurfural ^g	620-02-0	1.380	3.41E-06	1560	1551	(A)	n.d.	4.0	*
Total							16.9	72.0	
Lactones									
α -methyl- γ -butyrolactone ^g	1679-47-6	0.110	7.70E-07	1576	1557	(B)	15.4	n.d.	***
γ -butyrolactone*	96-48-0	-0.310	7.47E-08	1619	1593	(A)	712.8	971.4	*
γ -hexalactone ^g	695-06-7	0.600	7.70E-07	1694	1664	(A)	140.8	9.8	***
δ -hexalactone ^g	823-22-3	0.600	8.68E-07	1770	1747	(B)	155.9	8.4	***
δ -octalactone ^g	698-76-0	1.590	1.41E-06	1964	1927	(A)	87.3	3.2	**
Total							1112.1	992.8	
Total volatiles ($\mu\text{g kg}^{-1}$ DM)							27358.5	11274.0	

^aLogP(o/w) octanol-water partition coefficient (at 25 °C) (EPI SUITE software version 4.11).

^bHLC Henry's law constant ($\text{atm.m}^3 \text{mol}^{-1}$ at 25 °C) (EPI SUITE software version 4.11). HLC is the product of the coefficient activity of the targeted compound in water and its saturated vapour pressure.

^cRI retention index of aroma compounds from databases (Flavornet; Pherobase; The LRI and Odour Database).

^dID identification (A) mass spectra database from NIST (NIST, 2011), RI and injection of standard (positive identification), (B) mass spectrum, RI (tentative identification), (C) only mass spectrum.

^eSemi-quantification of aroma compounds vs. an internal standard (4-nonanol) and expressed in ng per g of dry matter (ng g^{-1} DM) in fresh and dried fruit.

^fSignificance, analysis of variance (ANOVA) with the honestly significant difference (HSD) in a Tukey test at different significance levels (XLSTAT software): (n.s.) no significant difference between dried and fresh fruit ($\alpha > 5\%$); (*) significant difference with $\alpha < 5\%$; (**) significant difference with $\alpha < 1\%$; (***) significant difference with $\alpha < 0.1\%$.

Symbols: (\$) coelution of aroma compound; (n.d.) aroma compound was not detected; (t) aroma compound was found in trace quantity

(t < $3.0 \mu\text{g kg}^{-1}$ DM); (-) data are missing; (n.f.) data are not found in the literature; volatile compounds previously found in literature in mango cv. Kent (*) or in mango cultivars others than cv. Kent (g) (Pino *et al.*, 2005; Pino & Mesa, 2006; Pandit *et al.*, 2009a).

4-hydroxy-4-methyl-2-pentanone in Malaysian soursop (Cheong *et al.*, 2011).

In contrast to previous findings regarding volatile compounds from cv. Kent, some potent odorants were not detected, such as β -ionone, β -damascenone and (E,Z)-2,6-nonadienal. Various factors can impact the aromatic profile of mango fruits, that is environmental conditions (Kulkarni *et al.*, 2012), fruit ripening conditions (Lalel *et al.*, 2003a; Pandit *et al.*, 2009b), storage conditions (Beaulieu & Lea, 2003) or intrafruit aroma heterogeneity (Lalel *et al.*, 2003b). Besides, extraction techniques to isolate volatile compounds can, through chemical and enzymatic reactions, lead to the generation of volatile compounds that were not present in

the initial product. β -ionone, a potent flavour compound, was detected in fresh mango fruits from several cultivars, including cv. Kent (Pino & Mesa, 2006; Pino, 2012), whereas it was absent in the extracts assessed in the present study. In the aforementioned studies, this compound could have been generated at the elevated temperature used to extract volatiles by the SDE technique through oxidative cleavage of β -carotene (Kanasawud & Crouzet, 1990). Fruits from some mango cultivars were reported to contain β -ionone on the basis of the analysis of aroma extracts obtained by the SAFE technique (Munafò *et al.*, 2014). As this technique involves quite gentle extraction conditions, β -ionone may not be considered as a

compound generated during extraction process. The occurrence of carotene oxygenase, leading to β -ionone via the cleavage of β -carotene, was reported in several fruits, such as quince, nectarine and star fruit (Baldermann *et al.*, 2005). To our best knowledge, no data have been reported yet regarding the presence of carotene oxygenase in mango cultivars. Some aldehydes, such as heptanal, nonanal and heptadienal isomers, were not detected in fresh fruit in the present study. They could also be generated through chemical and enzymatic reactions during flavour extract preparation from fatty acids under oxygen and heat (Belitz *et al.*, 2009b).

Noteworthy changes occurred in the volatile profile of mango fruit following drying (Table 2). A total of fifty four volatile compounds were identified, including 36 that were positively identified. These included eleven monoterpene hydrocarbons, four sesquiterpene hydrocarbons, one norisoprenoid, fifteen alcohols, one aromatic alcohol, three ketones, two esters, five aldehydes, one aromatic aldehyde, four furans, four lactones and three nonterpene hydrocarbons. The levels of fifty compounds were significantly different ($P \leq 0.05$) between fresh and dried mango. New compounds were generated and substantial losses occurred.

The total amount of volatiles decreased by about 58.8%. It was $27.4 \text{ mg kg}^{-1} \text{ DM}$ in fresh fruit compared to $11.3 \text{ mg kg}^{-1} \text{ DM}$ in dried fruit. Monoterpene hydrocarbons (73.7%) were again the major volatile compounds (Fig. 3), with δ -3-carene being the most abundant (66.6%). Alcohols (9.3%), mainly 3-methyl-1-butanol and 2-methyl-1-butanol (6.3%), and lactones (8.8%) were amongst the predominant compounds.

Under drying, considerable losses occurred with regard to almost all terpenoids, except for β -pinene and (*E*)- β -ocimene. The total monoterpene concentration was 24.8 mg kg^{-1} in fresh mango but 8.3 mg kg^{-1} in dried mango and that of sesquiterpenes was 0.8 mg kg^{-1} in fresh mango and 0.3 mg kg^{-1} in dried mango. The losses could be explained by the physico-chemical properties of terpenoids detected in

mango. They are all hydrocarbons and readily prone to losses by evaporation because of their high Log *P* values and high Henry's law constant (Table 2). Clear declines in terpenoids were also observed during dried nectarine preparation in drying conditions similar to those used in the present work (Sunthonvit *et al.*, 2007). Note that the aforementioned paper reported only the total terpenoid amount without consideration of the behaviour of individual compounds.

Regarding aliphatic alcohols, the 3-pentanol, 2-pentanol, 1-butanol and 2-decanol concentration significantly decreased while that of 3-methyl-1-butanol, 2-methyl-1-butanol, 1-hexanol and (*Z*)-3-hexen-1-ol increased following drying. Some compounds appeared, that is 2-methyl-1-propanol, 1-pentanol, (*E*) and (*Z*)-2-pentene-1-ol and 1-octanol. In dried grapes, an increase in alcohols such as 2-methyl-1-propanol has also been observed (Franco *et al.*, 2004). Alcohol losses may be explained by their evaporation during drying. The increase or generation of alcohols could be due to acidic hydrolysis of the corresponding glycosides favoured by the temperature applied during drying (60 °C). Importantly, aliphatic alcohols such as 1-hexanol and (*Z*)-3-hexen-1-ol have been identified in the glycosidic fraction of mango fruits (Lalel *et al.*, 2003c). Acceleration of acido-catalysed hydrolysis of glycosides under elevated temperatures is well documented in the literature (Winterhalter & Skouroumounis, 1997). The Henry's law constant was quite similar for the different aliphatic alcohols detected in the current study. Consequently, it can be assumed that the extent of their losses by evaporation during drying was similar. Losses of these compounds during drying may have been largely counterbalanced through the release of alcohols from glycosides as their total amount was 1.6 mg kg^{-1} in dried fruits but 0.3 mg kg^{-1} in fresh fruits.

2-phenylethanol was not detected in fresh fruit but appeared following drying. Hydrolysis of relevant glycosidic conjugates may explain the formation of this compound. This alcohol has been detected in the glycosidically bound fraction of mango fruits (Adedeji *et al.*, 1992; Lalel *et al.*, 2003c). Similarly in dried grapes, generation of 2-phenylethanol was also observed under drying (Franco *et al.*, 2004).

In contrast to fresh fruit, benzaldehyde was found in dried mango. It may be formed by benzyl alcohol oxidation. This alcohol was not detected in fresh fruit. However, it could be generated by hydrolytic release of its glycoconjugate during drying. Glycosidically bound benzyl alcohol was already reported in mango fruits (Adedeji *et al.*, 1992; Lalel *et al.*, 2003c). Benzaldehyde was also observed in dried nectarines (Sunthonvit *et al.*, 2007) and plums (Sabarez *et al.*, 2000). Its formation in dried nectarines was explained by the hydrolysis of amygdalin, its glucosidic precursor,

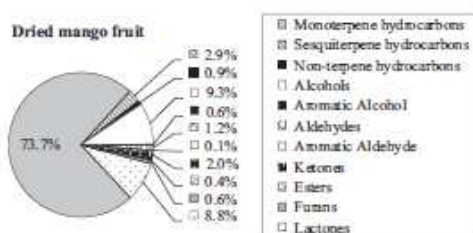


Figure 3 Distribution (%) of chemical class of aroma compounds in dried mango.

during drying (Sabarez *et al.*, 2000). No data were available regarding the occurrence of amygdalin in mango. This glucoside is often reported in stone fruits (Aubert & Milhet, 2007). Hence, its occurrence in mango may not be excluded.

Amongst norisoprenoid compounds, β -ionone was only observed in dried mango, but at trace levels (less than $3 \mu\text{g kg}^{-1}$). The detection of β -ionone in dried mango may be linked to the oxidative cleavage of β -carotene accelerated by the temperature applied during drying. In model studies, it was demonstrated that β -carotene degradation under heat treatment generates β -ionone (Kanasawud & Crouzet, 1990). Heating of mango pulp also generated this compound (Sakho *et al.*, 1985). Drying of nectarines also led to its formation (Sunthonvit *et al.*, 2007).

Several aliphatic aldehydes, such as heptanal, nonanal and 2,4-heptadienal isomers, appeared under drying. Drying induced an increase in the hexanal level. Drying nectarines and peppers has also led to the formation of aliphatic aldehydes (Sabarez *et al.*, 2000; Sunthonvit *et al.*, 2007). Aldehyde generation was probably due to the degradation of unsaturated fatty acids during drying. It is well documented that oleic acid degradation leads to the formation of nonanal, that of linolenic acid to hexanal and heptanal, and that of linolenic acid to 2,4-heptadienal isomers (Belitz *et al.*, 2009b). δ -hexalactone, γ -hexalactone, δ -octalactone and α -methyl- γ -butyrolactone levels dropped dramatically following drying, while that of γ -butyrolactone increased. The latter was the major lactone in fresh and dried mango. Similarly, in nectarines (Sunthonvit *et al.*, 2007) and grapes (Franco *et al.*, 2004) some lactones were reported to decrease while others increased following drying. Lactone losses may be due to thermal degradation rather than their evaporation. Indeed, Henry's law constant values of lactones were amongst the lowest of all volatile compounds from mango. The increase in the γ -butyrolactone level could be initiated by the hydrolytic release of 4-hydroxybutanoic acid from relevant glycoconjugated precursor during drying. Further interesterification of this hydroxy fatty acid yields γ -butyrolactone. Two lactones were detected in the glycosidically bound fraction of fresh mango from cv. Kensington Pride (Lalé *et al.*, 2003c). Glycoconjugated hydroxy fatty acid precursors of lactones have also been reported in quince fruit (Lutz & Winterhalter, 1992).

Heat treatment markers, that is 5-methylfurfural, furfural and 2-pentylfuran, were found in dried mango. Generation of 5-methylfurfural and furfural in dried grapes (Franco *et al.*, 2004), that of furfural in dried plums (Sabarez *et al.*, 2000) and that of 2-pentylfuran in dried nectarines (Sunthonvit *et al.*, 2007) has also been observed. These compounds are well-known products of Maillard or caramelisation reactions. Their

formation in the present study may have involved Maillard reactions rather than caramelisation reactions as the temperature applied was at a moderate level. Furthermore, 2-pentylfuran could have been generated from linoleic acid under drying (Mandin *et al.*, 1999). Mesifuran was the only furan observed in fresh mango but was significantly degraded during drying.

Amongst the two esters detected, the 3-methylbutyl butanoate level increased from fresh fruit to dried fruit, while the level of butyl butanoate decreased. Nonterpene hydrocarbon levels dropped following drying, probably due to their evaporation. Indeed, they had a quite high Henry's law constant. On the other hand, 3-hydroxy-2-butanone and 4-hydroxy-4-methyl-2-pentanone levels increased following drying. Hydrolysis of their possible glycosidic precursors may explain this enrichment.

Potential flavour contributors in fresh and dried mango

Significant changes in the composition of volatiles from fresh to dried mango could induce differences in flavour perception. OAVs were determined to gain insight into the contribution of the volatile compounds to the flavour of fresh and dried mango (Table 3). The amount of volatiles in fresh and dried mango, measured as fresh weight equivalents, was considered. The odour threshold of the compounds in water was derived from the literature. Eight compounds were found to be potential contributors in both products, as their OAVs were over 1. Amongst them, five and four terpenoids are observed in fresh and dried mango, respectively. Four of them were common in both products. α -phellandrene presented an OAV of slightly higher than 1 only in fresh mango. Limonene, which develops citrus, green and ethereal notes, had the highest OAV amongst terpenoids, that is 10.6 and 16.2 in fresh and dried mango, respectively. It was followed in decreasing order of OAVs by β -myrcene, δ -3-carene, β -caryophyllene and α -phellandrene in both products. These compounds confer citrus, green, terpenic, ethereal, woody and resinous notes to mango. The data presented here were in agreement with previous studies. δ -3-carene, α -phellandrene, β -myrcene, limonene and β -caryophyllene were reported with OAV > 1 in fresh fruit from cv. Kent (Pino & Mesa, 2006). In other mango cultivars, they were suggested to be flavour contributors, thanks to their high flavour dilution (FD) factor or to the high OAVs of aroma extracts issued from SAFE (Munafó *et al.*, 2014) or SDE techniques (Pino, 2012). β -pinene, α -terpinene, β -phellandrene, γ -terpinene, (*E*)- β -ocimene and α -terpinolene did not seem to contribute to the flavour of cv. Kent as their OAVs were less than 1. By contrast, in some other mango cultivars, (*E*)- β -ocimene and α -terpinolene were considered to be potent odorants, with citrus

Table 3 Aroma compounds in fresh and dried fruit and their odour potential

Compounds	Odour threshold ^d	Odour description ^e	OAV Exp ^a		OAV Lit ^{bc}	FD Lit ^e
			Fresh fruit	Dried fruit		
Monoterpene hydrocarbons						
β -pinene	140 ^{A,c}	Woody, pine, resin, turpentine, green	<0.1	0.1		
δ -3-carene	770 ^A	Citrus fruit, citrus peel, lemon, orange	5.2	8.5	5.01 ^A ; 80 ^B	128 ^B
α -phellandrene	40 ^B	Citrus, herbaceous, juniper, turpentine, spice	1.0	1.0	39.81 ^A	
β -myrcene	15 ^{A,c}	Woody, resinous, musty, balsamic, ethereal	5.2	13.7	25.12 ^A ; 28 ^B	64 ^B ; 4-128 ^d
α -terpinene	85 ^C	Lemon, ethereal, chemical	0.2	0.2		
Limonene	10 ^{A,c}	Citrus, lemon, orange, green, ethereal	10.6	15.3	1.26 ^A ; 47 ^B	128 ^B
β -phellandrene	500 ^B	Herbaceous, turpentine, terpenic, minty	<0.1	0.1		
γ -terpinene	260 ^C	Citrus, herbaceous, turpentine, terpenic	<0.1	<0.1		
(E)- β -ocimene	34 ^d	Citrus, herbaceous, sweet	0.1	0.5		4-128 ^d
α -terpinolene	200 ^{A,b}	Woody, anisic, sweet	1.0	0.9	1 ^A ; 230 ^B	512 ^B
Sesquiterpene hydrocarbons						
α -copaene	n.f.	Woody, earthy, spicy	–	–		
α -gurjunene	n.f.	Woody, earthy, balsamic	–	–		
β -caryophyllene	64 ^{A,b}	Woody, green, spicy, terpenic	1.5	2.9	2 ^A ; 72 ^B	64 ^B
α -caryophyllene	160 ^A	Woody, oily	0.2	0.5		
Norisoprenoids						
β -ionone	0.007 ^A	Violet, raspberry, floral, seaweed, artificial	n.d.	t	2511.89 ^A ; 450 ^B	256 ^B ; 32-512 ^d
Nonterpene hydrocarbons						
Toluene	n.f.	Chemical, synthetic, solvent	–	–		
m-xylene	n.f.	Plastic, green, pungent	–	–		
o-xylene	n.f.	Fatty, geranium	–	–		
Alcohols						
2-methyl-1-propanol	40 000 ^A	Wine, licorice, alcoholic, chemical	n.d.	<0.1		
3-pentanol	n.f.	Fruit, green	–	–		
2-pentanol	n.f.	Green, fruity, chemical, sweet	–	–		
1-butanol	500 ^{A,f}	Fruit, floral, medicinal, sweet	<0.1	<0.1		
3-methyl-1-butanol ^g	300 ^{A,c}	Balsamic, whisky, malt, pungent, onion, cheese	^s	^s		
β -2-methyl-1-butanol ^g	300 ^A	Fatty, wine, malty, onion				
1-pentanol	4000 ^B	Balsamic, green, sweet, pungent	n.d.	<0.1		
2-decanol	n.f.	Aniseed, coconut	–	t		
(E)-2-pentanol-1-ol	n.f.	Mushroom	n.d.	–		
(Z)-2-pentanol-1-ol	720 ^C	Green, plastic, rubber	n.d.	<0.1		
1-hexanol	500 ^A	Fatty, green, grass, resin, flower, sweet	<0.1	<0.1		
(E)-3-hexen-1-ol	110 ^C	Green, moss, fresh	t	0.1		
(Z)-3-hexen-1-ol	70 ^A	Green, grass, fruity	t	<0.1		
1-octanol	190 ^B	Fatty, green, toasted, burnt, chemical	n.d.	<0.1		
Aromatic Alcohol						
2-phenylethanol	1100 ^A	Floral (rose, lilac), honey, spicy, sweet	n.d.	<0.1		4-128 ^d
Aldehydes						
Hexanal	4.5 ^{A,h,i}	Fatty, grass, green, tallowy	t	5.2	15.85 ^A ; 2 ^B	32 ^B ; 8 ^d
Heptanal	3 ^A	Fatty, rancid, citrus, green, dry fish, heavy	n.d.	16.2	6.31 ^A	
Nonanal ^g	1 ^{A,b}	Fatty, wax, citrus, green, floral, sweet, soapy	n.d.	^s	19.95 ^A ; 10 ^B	32 ^B
(E,Z)-2,4-heptadienal	n.f.	Fatty, fried, tallowy	n.d.	–		
(E,E)-2,4-heptadienal	n.f.	Fatty, nutty, hay	n.d.	–		
Aromatic Aldehyde						
Benzaldehyde	350 ^{A,f}	Bitter almond, burnt sugar	n.d.	<0.1		
Ketones						
2-heptanone	140 ^{A,c}	Fatty, toasted, nutty, gravy, blue cheese	<0.1			
3-hydroxy-2-butanone	8000 ^A	Fatty, butter, cream, rancid	<0.1	<0.1		
4-hydroxy-4-methyl-2-pentanone	n.f.	n.f.	–	–		

Table 3 (Continued)

Compounds	Odour threshold ^f	Odour description ^g	OAV Exp ^a		OAV Lit ^h	FD Lit ⁱ
			Fresh fruit	Dried fruit		
Esters						
Butyl butanoate	110 ^{c,e}	Fruity, sweet, fresh	<0.1	<0.1		
3-methylbutyl butanoate	0.13 ^a	Fruity, banana, apricot, sweet	18.4	204.0		
Furans						
Mesifuran (MDMF)	0.03 ^a	Caramel, sweet	102.8	t	3 ^b	32 ^b ; 4–128 ^d
2-pentylfuran	6 ^{a,g}	Fatty, butter, warm, sweet	n.d.	0.8		
2-furfural	3000 ^{a,f}	Almond, caramel, honey, roasted, fatty	n.d.	<0.1		
5-methylfurfural	1110 ^f	Almond, caramel, coffee, roasted, warm	n.d.	<0.1		
Lactones						
α -methyl- γ -butyrolactone	n.f.	Woody	–	n.d.		
γ -butyrolactone	35 ^f	Caramel, sweet	3.7	24.3		
γ -hexalactone	260 ^h	Coumarin, sweet	0.1	<0.1		
δ -hexalactone	n.f.	n.f.	–	–		
δ -octalactone	400 ^{f,h,g}	Sweet	<0.1	<0.1		32–512 ^d

^fOT odour threshold in water (ppb) found in the literature with references: (a) (Pino & Mesa, 2006), (b) (Pino, 2012), (c) (Boonbumrung *et al.*, 2001), (e) (Ferreira *et al.*, 2000), (f) (Belitz *et al.*, 2009a), (g) databases (Leffingwell & Associates, 2008; The LRI and Odour Database), (h) (Du *et al.*, 2010), (i) (Castro-Vazquez *et al.*, 2006), (j) (Tamura *et al.*, 2001).

^gOdour description found in the literature with databases (Flavornet; Pherobase; The LRI and Odour Database).

^aOAV experimental odour activity value obtained by dividing the concentration of each aroma compound by its OT in water.

^hOAV odour activity value (unitless) in the literature with references: (a) (Pino & Mesa, 2006), (b) (Pino, 2012).

ⁱFD flavour dilution factor (unitless) in the literature with references: (b) (Pino, 2012), (d) (Munafó *et al.*, 2014).

Symbols: (\$) coelution of aroma compound, (n.f.) data not found in the literature, (n.d.) aroma compound was not detected in mango product, (t) aroma compound was found in trace quantity (OAV could not be estimated), (–) odour threshold not found in the literature (OAV could not be estimated), (–\$) coelution of aroma compound (OAV could not be estimated).

and green notes due to the high FD factors in aroma extracts determined by SAFE (Munafó *et al.*, 2014) or SDE techniques (Pino, 2012) (Table 3). Odour thresholds of α -copaene and α -gurjunene had not been previously reported, which hindered determination of their OAVs.

Mesifuran developing a caramel and sweet odour gave the highest OAV (102.8) in fresh mango. It has been reported amongst flavour contributors in fresh mango when considering the FD factor or OAV (Pino, 2012; Munafó *et al.*, 2014). Its participation in the flavour of dried mango can be excluded because of its drastic loss during drying. 4-Hydroxy-2,5-dimethyl-3 (2H)-furanone (HDMF), which was reported to be amongst most potent odorants in some mango cultivars thanks to the high FD factor, was not detected in the current study. Furan compounds, 5-methylfurfural, furfural and 2-pentylfuran generated in dried mango were not detected at levels exceeding their odour threshold. However, their potential participation in the roasted and caramel odour of this product should not be overlooked as the synergetic effect of odorants at subthreshold concentrations in a mixture has been reported (Laska & Hudson, 1991). γ -butyrolactone and 3-methylbutyl butanoate presenting high OAVs

and developing caramel, sweet and fruity notes may intervene in the flavour of fresh and dried mango. 3-methylbutyl butanoate displaying fruity and sweet attributes was the volatile compound with the highest OAV (204.0) in dried mango.

Amongst aldehydes, that is heptanal and hexanal, which appeared following drying and presenting fatty, grass and green notes, are possible odour-active compounds in dried mango as they yielded high OAVs.

OAVs of some compounds like β -ionone, 2-decanol and 2-heptanone could not be determined as they were detected at trace levels. Amongst them, β -ionone is probably the best candidate for influencing the flavour of dried mango due to its very low odour threshold ($0.2 \mu\text{g kg}^{-1}$) (Pino, 2012). Furthermore, some compounds, such as 3-methyl-1-butanol with 2-methyl-1-butanol, were coeluted, thus hindering the determination of their concentration.

2-Phenylethanol and δ -octalactone reported as active-aroma compounds in some mango cultivars (Munafó *et al.*, 2014) and yielded low OAVs in cv. Kent in the current study (Table 3).

Despite losses of volatile compounds during drying, OAVs of several compounds were found to be higher in dried mango than in fresh mango. This may be

attributable to the increased dry matter following the drying process.

Conclusion

Significant losses of volatile constituents of fresh mango occurred following the drying process in the conditions similar to those used in the industry. Drying reduced the amount of many of volatile compounds, including monoterpenes, sesquiterpenes, aliphatic alcohols and lactones, which are considered as impact odorants. Their decline may be explained by their evaporation and degradation during the drying process. Meanwhile, some aliphatic alcohols and an aromatic alcohol, that is 2-phenylethanol, appeared and the concentration of some other aliphatic alcohols also increased. The hydrolysis of relevant glycosides under high temperature during drying was suggested to induce such flavour compound enrichment. A follow-up of glycosidically bound volatiles in mango under drying conditions could provide evidence in support of this assumption. As expected, dried mangoes contained thermally generated compounds, that is 2-furfural, 5-methylfurfural and 2-pentylfuran. When OAVs of the volatile compounds were examined, eight compounds could be considered as potent odorants in each mango product. Limonene, β -myrcene, δ -3-carene, β -caryophyllene, γ -butyrolactone and 3-methylbutyl butanoate were common potent odorants in fresh and dried fruit. In contrast to fresh mango, aliphatic aldehydes, hexanal and heptanal gave high OAVs in dried mango. On the other hand, amongst furan derivatives, only mesifuran exhibited a high OAV and only in fresh mango.

Sensory studies based on aroma reconstitution and omission tests using volatile compounds with an OAV of over 1 but also including some others presenting OAVs of less than 1 would help gain further insight into the flavour contributors. Indeed, an additive effect has been found for mixtures containing volatile compounds at concentrations below their odour threshold (Escudero *et al.*, 2004).

Acknowledgments

Staff of the CIRAD pilot unit are gratefully thanked for mango processing, Marc Valente and Jacques Joas from UMR Qualisud for their helpful suggestions for mango fruit physiology and Karine Gourrat from INRA Dijon for her advice regarding the SAFE technique.

References

Adedeji, J., Hartman, T.G., Lech, J. & Ho, C.T. (1992). Characterization of glycosidically bound aroma compounds in the African

- mango (*Mangifera indica* L.). *Journal of Agricultural and Food Chemistry*, **40**, 659–661.
- Andrade, E.H.A., Maia, J.G.S. & Zoghbi, M.D.G.B. (2000). Aroma volatile constituents of Brazilian varieties of mango fruit. *Journal of Food Composition and Analysis*, **13**, 27–33.
- AOAC (2000). *Official Methods of Analysis of AOAC International*. 2200 pages. Pp. 912–914. Gaithersburg, MD: Association of Official Analytical Chemists, Inc.
- Aubert, C. & Milhet, C. (2007). Distribution of the volatile compounds in the different parts of a white-fleshed peach (*Prunus persica* L. Batsch). *Food Chemistry*, **102**, 375–384.
- Baldermann, S., Naim, M. & Fleischmann, P. (2005). Enzymatic carotenoid degradation and aroma formation in nectarines (*Prunus persica*). *Food Research International*, **38**, 833–836.
- Beaulieu, J.C. & Lea, J.M. (2003). Volatile and quality changes in fresh-cut mangos prepared from firm-ripe and soft-ripe fruit, stored in clamshell containers and passive MAP. *Postharvest Biology and Technology*, **30**, 15–28.
- Belitz, H.-D., Grosch, W. & Schieberle, P. (2009a). Aroma compounds. In: *Food Chemistry* (edited by Springer). Pp. 340–402. Berlin: Springer-Verlag Berlin Heidelberg.
- Belitz, H.-D., Grosch, W. & Schieberle, P. (2009b). *Food Chemistry*. 1070 pages. Pp. 158–247. Berlin: Springer-Verlag Berlin Heidelberg.
- Boonbumrung, S., Tamura, H., Mookdasanit, J. *et al.* (2001). Characteristic aroma components of the volatile oil of yellow keaw mango fruits determined by limited odor unit method. *Food Science and Technology Research*, **7**, 200–206.
- Caparino, O.A., Tang, J., Nindo, C.I., Sablani, S.S., Powers, J.R. & Fellman, J.K. (2012). Effect of drying methods on the physical properties and microstructures of mango (Philippine 'Carabao' var.) powder. *Journal of Food Engineering*, **111**, 135–148.
- Castro-Vazquez, L., Diaz-Maroto, M.C. & Perez-Coello, M.S. (2006). Volatile composition and contribution to the aroma of spanish honeydew honeys. Identification of a new chemical marker. *Journal of Agriculture and Food Chemistry*, **54**, 4809–4813.
- Cheong, K.W., Tan, C.P., Mirhosseini, H. *et al.* (2011). Optimization of equilibrium headspace analysis of volatile flavor compounds of malaysian soursop (*Annona muricata*): comprehensive two-dimensional gas chromatography time-of-flight mass spectrometry (GC $\times$ GC-TOFMS). *Food Chemistry*, **125**, 1481–1489.
- De Souza Galv  o, M., Narain, N., do Socorro Porto dos Santos, M. & Nunes, M.L. (2011). Volatile compounds and descriptive odor attributes in umbu (*Spondias tuberosa*) fruits during maturation. *Food Research International*, **44**, 1919–1926.
- Du, X., Finn, C.E. & Qian, M.C. (2010). Volatile composition and odour-activity value of thornless 'Black Diamond' and 'Marion' blackberries. *Food Chemistry*, **119**, 1127–1134.
- Escudero, A., Gogorza, B., Melus, M., Ortin, N., C  cho, J. & Ferreira, V. (2004). Characterization of the aroma of a wine from Maccabeo. Key role played by compounds with low odor activity values. *Journal of Agriculture and Food Chemistry*, **52**, 3516–3524.
- Ferreira, V., L  pez, R. & C  cho, J.F. (2000). Quantitative determination of the odorants of young red wines from different grape varieties. *Journal of the Science of Food and Agriculture*, **80**, 1659–1667.
- Flavornet (2004). <http://www.flavornet.org/flavornet.html>. Accessed 10 August 2015.
- Forero, D.P., Orrego, C.E., Peterson, D.G. & Osorio, C. (2015). Chemical and sensory comparison of fresh and dried lulo (*Solanum quitoense* Lam.) fruit aroma. *Food Chemistry*, **169**, 85–91.
- Franco, M., Peinado, R.A., Medina, M. & Moreno, J. (2004). Off-vine grape drying effect on volatile compounds and aromatic series in must from Pedro Ximenez grape variety. *Journal of Agricultural and Food Chemistry*, **52**, 3905–3910.
- Grosch, W. (2001). Evaluation of the key odorants of foods by dilution experiments, aroma models and omission. *Chemical Senses*, **26**, 533–545.

12 Impact of drying on volatile composition A. Bonnesu et al.

- Hasan, F.A., Al-Sheraji, S.H. & Ismail, A. (2013). Dried mangoes: phytochemicals, antioxidant properties, and health benefits. In: *Dried Fruits: Phytochemicals and Health Effects*, (edited by C. Alasalvar & F. Shahidi) Pp. 457–470. Chichester: John Wiley & Sons Inc.
- Kanasawud, P. & Crouzet, J.C. (1990). Mechanism of formation of volatile compounds by thermal degradation of carotenoids in aqueous medium. 1. β -Carotene degradation. *Journal of Agricultural and Food Chemistry*, **38**, 237–243.
- Kulkarni, R.S., Chidley, H.G., Pujari, K.H., Giri, A.P. & Gupta, V.S. (2012). Geographic variation in the flavour volatiles of Alphonso mango. *Food Chemistry*, **130**, 58–66.
- Lalel, H.J.D., Singh, Z. & Tan, S.C. (2003a). Aroma volatiles production during fruit ripening of 'Kensington Pride' mango. *Postharvest Biology and Technology*, **27**, 323–336.
- Lalel, H.J.D., Singh, Z. & Tan, S.C. (2003b). Distribution of aroma volatile compounds in different parts of mango fruit. *Journal of Horticultural Science & Biotechnology*, **78**, 131–138.
- Lalel, H.J.D., Singh, Z. & Tan, S.C. (2003c). Glycosidically-bound aroma volatile compounds in the skin and pulp of 'Kensington Pride' mango fruit at different stages of maturity. *Postharvest Biology and Technology*, **29**, 205–218.
- Laska, M. & Hudson, R. (1991). A comparison of the detection thresholds of odour mixtures and their components. *Chemical Senses*, **16**, 651–662.
- Leffingwell & Associates. (2008). <http://www.leffingwell.com/odor-thre.htm>. Accessed 10 August 2015.
- Lutz, A. & Winterhalter, P. (1992). Isolation of additional carotenoid metabolites from quince fruit (*Cydonia oblonga* Mill.). *Journal of Agricultural and Food Chemistry*, **40**, 1116–1120.
- Mandini, O., Duckham, S.C. & Ames, J.M. (1999). Volatile compounds from potato-like model systems. *Journal of Agricultural and Food Chemistry*, **47**, 2355–2359.
- Munaf, J.P. Jr, Didzhalis, J., Schnell, R.J., Schieberle, P. & Steinhaus, M. (2014). Characterization of the major aroma-active compounds in mango (*Mangifera indica* L.) cultivars Haden, White Alphonso, Praya Sowoy, Royal Special, and Malindi by application of a comparative aroma extract dilution analysis. *Journal of Agriculture and Food Chemistry*, **62**, 4544–4551.
- NIST, National Institute of Standards and Technology (2011). <http://webbook.nist.gov/>. Accessed 10 August 2015.
- Pandit, S.S., Chidley, H.G., Kulkarni, R.S., Pujari, K.H., Giri, A.P. & Gupta, V.S. (2009a). Cultivar relationships in mango based on fruit volatile profiles. *Food Chemistry*, **114**, 363–372.
- Pandit, S.S., Kulkarni, R.S., Chidley, H.G. et al. (2009b). Changes in volatile composition during fruit development and ripening of 'Alphonso' mango. *Journal of the Science of Food and Agriculture*, **89**, 2071–2081.
- Pherobase. (2014). <http://www.pherobase.com/database/compound/compounds-index.php>. Accessed 10 August 2015.
- Pino, J.A. (2012). Odour-active compounds in mango (*Mangifera indica* L. cv. Corazón). *International Journal of Food Science and Technology*, **47**, 1944–1950.
- Pino, J.A. & Mesa, J. (2006). Contribution of volatile compounds to mango (*Mangifera indica* L.) aroma. *Flavour and Fragrance Journal*, **21**, 207–213.
- Pino, J.A., Mesa, J., Muñoz, Y., Martí, M.P. & Marbot, R. (2005). Volatile components from mango (*Mangifera indica* L.) cultivars. *Journal of Agriculture and Food Chemistry*, **53**, 2213–2223.
- Pott, I., Neidhart, S., Mühlbauer, W. & Carle, R. (2005). Quality improvement of non-sulphited mango slices by drying at high temperatures. *Innovative Food Science & Emerging Technologies*, **6**, 412–419.
- Saharez, H.T., Price, W.E. & Korth, J. (2000). Volatile changes during dehydration of d'Agen prunes. *Journal of Agricultural and Food Chemistry*, **48**, 1838–1842.
- Sakho, M., Crouzet, J. & Seck, S. (1985). Evolution des composés volatils de la mangue au cours du chauffage. *Lebensmittel-Wissenschaft und -Technologie*, **18**, 89–93.
- Sunthonvit, N., Szrednicki, G. & Craske, J. (2007). Effects of drying treatments on the composition of volatile compounds in dried nectarines. *Drying Technology*, **25**, 877–881.
- Tamura, H., Boonbumrung, S., Yoshizawa, T. & Varanyanond, W. (2001). The volatile constituents in the peel and pulp of a green Thai mango, Khiao Sawoei Cultivar (*Mangifera indica* L.). *Food Science and Technology Research*, **7**, 72–77.
- Tharanathan, R.N., Yashoda, H.M. & Prabha, T.N. (2006). Mango (*Mangifera indica* L.). "The King of Fruits" – an overview. *Food Reviews International*, **22**, 95–123.
- The LRI and Odour Database <http://www.odour.org.uk/index.html>. Accessed 10 August 2015.
- Valente, M., Prades, A. & Laux, D. (2013). Potential use of physical measurements including ultrasound for a better mango fruit quality characterization. *Journal of Food Engineering*, **116**, 57–64.
- Winterhalter, P. & Skouroumounis, G.K. (1997). Glycoconjugated aroma compounds: occurrence, role and biotechnological transformation. In: *Biotechnology of Aroma Compounds* (edited by T. Scheper). Pp. 73–105. Erlangen: Springer-Verlag Berlin Heidelberg.

c) Résumé et résultats principaux obtenus

La sélection des mangues, pour confectionner les produits d'étude, s'est opérée suivant l'aspect (calibre, remplissage du fruit, etc.) et l'indice de fermeté (indice durofel) des fruits. Ces précautions ont permis de constituer un lot de mangues homogène en termes de maturité et de maturation.

L'étude des COVs de la mangue fraîche (purée) et séchée (poudre) en SAFE/GC-MS a permis de mettre en évidence l'impact du séchage sur le profil aromatique du fruit frais. Les résultats obtenus démontrent que le séchage induit une perte importante des COVs originels du fruit frais, et parallèlement génère de nouveaux COVs.

Les profils aromatiques des produits de mangue frais et séchés, confectionnés à partir du cultivar Kent, comptent majoritairement des composés terpéniques. Le δ -3-carène est, de loin, le composé et le monoterpène le plus abondant des produits de mangue suivi de l' α -terpinolène, du limonène, du β -myrcène, de l' α -phellandrène et du β -phellandrène. Le β -caryophyllène et l' α -caryophyllène sont, quant à eux, les sesquiterpènes les plus abondants. La classe chimique des terpènes est significativement impactée par le séchage avec une perte importante de ces COVs.

Les lactones et les alcools sont les classes chimiques les plus présentes après les composés terpéniques. La γ -butyrolactone est la lactone la plus abondante dans la mangue Kent fraîche et séchée. Le butan-1-ol est davantage marqué dans la mangue fraîche tandis que le 2-méthylpropan-1-ol, le 3-méthyl et 2-méthylbutan-1-ol, et le (*z*)-pent-2-èn-1-ol sont davantage présents dans la mangue séchée. D'autres classes chimiques communes aux 2 produits comme les hydrocarbures non-terpéniques, les cétones et les esters définissent également le profil aromatique des produits de mangue. Tout comme les terpènes, une perte significative de lactones et d'alcools spécifiques ainsi que d'hydrocarbures non-terpéniques est observée. A l'inverse, le séchage génère l'apparition de nouveaux composés tels que les aldéhydes, les furanes, les alcools et aldéhydes aromatiques.

Les pertes en composés au cours du séchage sont avant tout expliquées par le potentiel volatil et hydrophobe des molécules (Log P, HLC). La génération de nouveaux composés est due à des réactions de dégradation de précurseurs d'arôme. Ainsi, la dégradation d'acides gras insaturés peut générer des aldéhydes ; l'hydrolyse acide de glycoconjugués peut donner des alcools, des acides et des acides-alcools précurseurs des lactones, les réactions de Maillard ou de caramélisation donnent des furanes.

Les résultats SAFE/GC-MS obtenus sur purée de mangue fraîche Kent sont globalement similaires aux données de la littérature pour ce cultivar (Pino & Mesa, 2006; Pino, Mesa, *et al.*, 2005). Les différences qualitatives et quantitatives observées entre les données expérimentales SAFE/GC-MS et les précédents travaux de la littérature sur Kent, peuvent venir soit de la méthode d'extraction utilisée (SE, SDE, SPME, P&T, etc.) ou bien de la matière première utilisée (origine géographique, degré de maturité/maturation, etc.)

Les composés aromatiques clés de la mangue sont décrits dans de nombreux travaux menés en GC-O (FC, FD/AEDA, notes olfactives, etc.) et mentionnés dans la littérature (Munafo, Didzbalis, *et al.*, 2014; Munafo, Didzbalis, *et al.*, 2016; Pino, 2012; Pino & Mesa, 2006). Ainsi, les OAVs des composés extraits ont été calculées et comparées aux données GC-O de la littérature sur mangue afin de mettre en évidence les composés aromatiques qui pourraient être clés dans l'arôme des produits de mangue frais et séchés. Ainsi, d'après le calcul des OAVs, le β -myrcène, le δ -3-carène, le β -caryophyllène, la γ -butyrolactone, et le 3-méthylbutyle butanoate peuvent contribuer à l'arôme de la mangue fraîche et séchée. Le mésifurane participerait, quant à lui, spécifiquement à l'arôme de la mangue fraîche, tandis que, l'hexanal et l'heptanal seraient spécifiques à l'arôme de la mangue séchée. Au regard des données de la littérature (Munafo, Didzbalis, *et al.*, 2014; Munafo, Didzbalis, *et al.*, 2016; Pino, 2012; Pino & Mesa, 2006), l' α -phellandrène, le β -ocimène, l' α -terpinolène, la β -ionone, le 2-phenyléthanol, le nonanal et la δ -octalactone peuvent aussi être des contributeurs de l'arôme de mangue en raison de leur valeurs OAV et FD importantes citées dans ces travaux. Les composés terpéniques (β -myrcène, δ -3-carène, β -caryophyllène, α -phellandrène, β -ocimène, α -terpinolène) peuvent contribuer à apporter des notes aromatiques citrus, vertes, terpéniques, boisées et résineuses au profil aromatique des produits de mangue. Le mesifurane, la γ -butyrolactone, δ -octalactone développent des notes sucrées et de caramel. Le 3-méthylbutyle butanoate contribue à la présence de notes fruitées ; les aldéhydes (hexanal et heptanal nonanal) à des notes grasses, vertes, et rances ; la β -ionone et le 2-phenyléthanol à des notes florales.

Ainsi, en affectant la quantité et la nature des COVs présents dans le profil aromatique des produits de mangue, le séchage tend aussi à impacter grandement la perception aromatique des produits.

4.3. Conclusion

La méthode de sélection des mangues mise en place pour obtenir des lots de fruits homogène a été appliquée à l'ensemble des lots de fruits de l'étude. Ces précautions sont notamment importantes pour obtenir d'une part, des produits frais de mangue (purée/cubes), et d'autre part, des produits séchés de mangue (poudre/cubes) qui puissent être comparables d'un point de vue aromatique mais différents d'un point de vue matriciel pour la suite des travaux à mener en *in vivo* sur la libération et sur la perception des COVs.

De même, la méthode SAFE/GC-MS mise en place a permis d'obtenir des résultats satisfaisants quant à la description du potentiel aromatique des produits de mangue. Cette méthode a été appliquée aux autres produits de mangue de l'étude confectionnés pour les travaux *in vivo* et *in vitro* à mener.