

Identification of Predominant Odorants in Thai Desserts Flavored by Smoking with “Tian Op”, a Traditional Thai Scented Candle

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“Tian Op”, a traditional Thai scented candle, is used for the smoking and flavoring of sweets, cakes, and other desserts for the purpose of adding a unique aroma to the final product. Gas chromatography–olfactometry, aroma extract dilution analysis, and GC-MS were applied to identify the potent odorants in two types of traditional Thai desserts (“num dok mai” and “gleep lum duan”) prepared using a Tian Op smoking process. On the basis of the results of AEDA and calculated odor-activity values, the predominant odorants in the Tian Op flavored desserts were vinyl ketones (C₅–C₉), *n*-aldehydes (C₅–C₁₁), (*E*)-2-unsaturated aldehydes (C₈–C₁₁), and ω -1-unsaturated aldehydes (C₈ and C₉). Sensory studies of model mixtures confirmed the importance of *n*-aldehydes, ω -1-unsaturated aldehydes, and guaiacol as predominant odorants; however, the results showed that vinyl ketones and (*E*)-2-unsaturated aldehydes, despite having high odor-activity values, may be of only minor importance in the typical aroma profiles of traditional Tian Op smoked desserts.

KEYWORDS: Tian Op; beeswax; candle; smoke; flavor; aroma; odorant; gas chromatography–olfactometry; aroma extract dilution analysis

INTRODUCTION

Jasmine (*Jasminum adenophyllum*), pandan leaves (*Pandanus amaryllifolius* Roxb.), and “Tian Op” have been used for centuries to flavor various kinds of Thai desserts. Tian Op is a traditional Thai scented candle that is used for smoking of sweets, cakes, and other desserts for the purpose of adding a unique aroma or scent to the final product. The main ingredients of Tian Op are beeswax (sometimes beeswax is the only component), benzoin, dried kefir lime peel (*Citrus hystrix* DC), sandalwood, and borneo camphor (*Pogostemon calstin* Benth). Each ingredient is believed to add to the aroma complexity of the smoke emitted from the Tian Op candle when it is burned. Depending upon the type and quantity to be processed, the dessert is “smoked” in a closed container for 30–60 min and from three to five times until the desired flavor is achieved. “Num dok mai” (low fat) and “gleep lum duan” (high fat) are two types of traditional desserts that are flavored using the Tian Op smoking process. In general, higher fat containing desserts require less smoking than low-fat products, because fat more effectively absorbs the mainly hydrophobic volatile compounds. Some potential drawbacks to the traditional Tian Op smoking

process are that flavor quality and intensity may not be the same for every piece of dessert within the smoking chamber and, furthermore, a black carbonaceous residue is deposited on the surface of the dessert.

Limited studies have been conducted on the volatile components of the ingredients of Tian Op. The main components of beeswax are nonvolatile high molecular weight waxes, including 14% hydrocarbons, 35% monoesters, 14% diesters, 3% triesters, 4% hydroxy monoesters, 8% hydroxyl polyesters, 1% acid esters, 2% acid polyesters, 12% free acids, 1% free alcohols, and 6% unidentified compounds (1). Ferber and Nursten (2) reported that *p*-cymene, limonene, *cis*- and *trans*-linalool oxide, α -terpineol, guaiacol, phenol, cresol, octanal, nonanal, decanal, and benzaldehyde were the major volatile components of molten (65 °C) beeswax. Benzoin is the resin of the styrax tree (*Styrax benzoin* Dryand) and contains coniferyl cinnamate, sumaresiloic acid, benzoic acid, cinnamic acid, styrene, vanillin, and benzaldehyde as its main chemical components (3). (*Z*)- α -Santalol and (*Z*)- β -santalol are the main volatile components of sandalwood essential oil (4). Kefir lime peel oil is composed of 2.5% α -pinene, 0.25% camphene, 30.6% β -pinene, 22.6% sabinene, 1.4% myrcene, 29.2% limonene, 1.3% cineole, 0.1% γ -terpinene, 0.1% *p*-cymene, 0.1% terpinolene, 0.6% (*E*)-sabinene hydrate, 4.2% citronellal, 0.6% copaene, 0.5% linalool, 0.5% β -cubebene, 4.2% terpinen-4-ol, 0.3% caryophyllene, 0.2% citronellyl acetate, 0.2% α -terpineol,

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0.4% citronellol, 0.3% δ -cadinene, 0.1% geraneol, 0.1% nerolidol, and 0.3% elemol (5).

To improve the Tian Op smoking process, or possibly for the development of an alternative Tian Op flavoring, a better understanding of the flavor chemistry of Tian Op smoke and Tian Op smoked desserts is needed. The objective of the present study was to apply gas chromatography–olfactometry, aroma extract dilution analysis, and sensory evaluation techniques to identify the potent odorants in two types of traditional Thai desserts (num dok mai and gleep lum duan) prepared using a Tian Op smoking process.

MATERIALS AND METHODS

Chemicals. Unless otherwise stated, all chemicals and reagents were obtained from Sigma-Aldrich Co. (St. Louis, MO). 1-Nonen-3-ol was purchased from Lancaster (Windham, NH), and 1-decen-3-ol, 7-octen-1-ol, 8-nonen-1-ol, and 9-decen-1-ol were obtained from TCI America (Portland, OR). 2-Acetyl-1-pyrroline was synthesized using the procedure described by Fuganti et al. (6). 1-Hepten-3-one, 1-nonen-3-one, 1-decen-3-one, 7-octenal, 8-nonenal, 9-decenal, and (*E*)-2-undecenal were synthesized from their corresponding alcohols by oxidation with pyridinium chlorochromate using a published procedure (7). (*Z*)-2-Nonenal was synthesized from (*Z*)-2-nonen-1-ol (Bedoukian Research Inc., Danbury, CT) by oxidation with Dess–Martin periodinane (0.3 M in dichloromethane; Aldrich Chemical Co.) following the procedure described by Meyer and Schreiber (8). Sandalwood essential oil (East Indian) was obtained from New Directions (Brampton, ON, Canada).

Beeswax was obtained from Sayun Bee Farm (Chang Mai, Thailand). Composition and properties reported by supplier were as follows: yellow color, saponification value of 87–110 mg of KOH/g, acid value of 17–24 mg of KOH/g, ester value of 70–86 mg of KOH/kg, melting point of 62–65 °C, and impurities of 0.1%. Kefir lime fruit was purchased from a local market (Pathumtanee, Thailand) and the peel immediately removed and dried for 36 h at 35 °C in a tray dryer to a moisture content of 12%. The dried peel was milled, sieved through a 0.25 mesh sieve, and stored in a desiccator until needed. Benzoin was purchased from a local market in Bangkok, Thailand (and originated from Chiang Kwang, Laos). Sandalwood, originating from the Chumphorn Province in Thailand, was obtained from the Thai Public Health Ministry (Bangkok, Thailand), then milled, sieved through a 0.25 mesh sieve, and stored in a desiccator until needed.

Wheat flour, rice flour, sugar, and palm oil were obtained from a local market (Bangkok, Thailand). Odorless deionized–distilled water was prepared by boiling glass-distilled water in an open flask until its volume was reduced by one-third of the original volume.

Preparation of Tian Op Candle. Tian Op candle was made from beeswax (24.25 g), dried kefir lime peel (0.25 g), sandalwood (0.25 g), and benzoin (0.25 g). Beeswax was heated in a stainless steel pot until it was soft and pliable. The other ingredients were thoroughly blended with the softened beeswax, and then the mixture was rolled into a flat sheet (0.3 cm thick \times 15 cm width). A cotton wick (Songkhla, Thailand) was then placed on the sheet and then the candle was formed by rolling the sheet into a cylindrical shape. At least 1.5 cm of the wick was left exposed from each end of the candle. The Tian Op candle was then bent into a half-circle shape.

Num Dok Mai (NDM) Preparation. Water (30% of total weight) and sugar (10% of total weight) were mixed and then heated until the sugar was completely dissolved. After cooling to room temperature, rice flour (60% of total weight) was added and the mixture stirred until evenly distributed. The dough was transferred to a glass bowl (20 cm diameter \times 4.5 cm height) and steamed for 10 min. Three separate batches of NDM were prepared.

Gleep Lum Duan (GLD) Preparation. Wheat flour (60% of total weight) and sugar (10% of total weight) were mixed, and then palm oil (30% of total weight) was slowly added while mixing. After thorough mixing, the dough was molded into \sim 2 cm³ cubes and baked on a flat sheet in an oven at 120 °C for 15 min. Three separate batches of GLD were prepared.

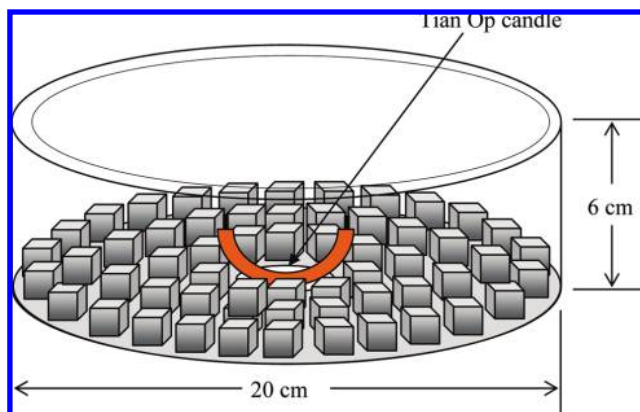


Figure 1. Apparatus used for Tian Op smoking of “num dok mai” and “gleep lum duan”.

Smoking Process. NDM and GLD from above were cut into small cubes (1 cm³). For smoking, 500 g of cubes, in a single layer, was placed into a 1-L cylindrical glass vessel along with a Tian Op candle (Figure 1). The candle was lit, then the vessel was sealed with a glass cover, and the smoking process was allowed to continue for 30 min.

Isolation of Volatile Compounds. Prior to extraction, desserts were homogenized at high speed for 1 min using a blender (702R, Hamilton Beach Inc., Washington, NC). One hundred grams of either the ground untreated dessert (NDM or GLD) or the ground smoked dessert (S-NDM or S-GLD) plus 50 μ L of an internal standard solution (500 μ g/mL of 2-methyl-3-heptanone in methanol) and 1 mL of a butylated hydroxytoluene solution (100 μ g/mL of BHT in ether) was soaked for 1 h in diethyl ether (50 mL) in a 500-mL glass bottle sealed with a PTFE-lined cap. The mixture was shaken on an orbital shaker (DS-500, VWR International Inc., West Chester, PA) at high speed (200 rpm) for 10 min, and then the solvent layer was removed. The remaining mixture was extracted as above two more times with 50 mL of ether. The pooled solvent extract was evaporated to 50 mL using a Vigreux column in a 45 °C water bath and then subjected to a high-vacuum distillation (5×10^{-5} Torr operating vacuum) cleanup step (9) for 3 h to remove the nonvolatile residue, with the sample kept at room temperature for the first 1.5 h and then warmed to 50 °C using a water bath. The resulting aroma extract was concentrated under a gentle stream of nitrogen gas to 10 mL, dried over 2 g of anhydrous sodium sulfate, and then further concentrated to 200 μ L under a nitrogen gas stream. Samples were prepared in triplicate (one extraction per each separate sample of control or smoked dessert) and kept at -70 °C until analysis.

Aroma Extract Dilution Analysis (AEDA). AEDA was conducted to determine the relative potency of individual odorants according to the method previously described (10, 11). Stepwise dilutions (1:3; 1 part aroma extract to 2 parts solvent) were prepared with diethyl ether. Each dilution was kept in a 2-mL amber vial equipped with PTFE-lined screw cap at -70 °C until analysis. The gas chromatography–olfactometry (GCO) system consisted of a 6890 GC (Agilent Technologies Inc., Palo Alto, CA) equipped with a FID, an on-column injector, and an olfactory detection port (DATU Technology Transfer, Geneva, NY). Each aroma extract was injected by cool on-column mode ($+3$ °C temperature tracking mode) into a polar capillary column (Stabilwax, 15 m \times 0.32 mm i.d.; 0.5 μ m film; Restek, Bellefonte, PA) or a nonpolar column (RTX-5SLIMS, 15 m \times 0.32 mm i.d.; 0.5 μ m film; Restek). Column effluent was split 1:5 between the FID and olfactory detection port using deactivated fused silica tubing, with both detector temperatures held at 250 °C. The GC oven temperature was programmed from 35 to 225 °C at a rate of 10 °C/min with initial and final hold times of 5 and 15 min, respectively. Helium was used as a carrier gas at a constant flow rate of 2.2 mL/min. Other conditions of GCO and AEDA have been previously described (11).

Gas Chromatography–Mass Spectrometry (GC-MS). Each aroma extract (1 μ L) was injected by cool on-column method ($+3$ °C temperature tracking mode) into a 6890 GC/5973N MSD (Agilent Technologies Inc.). Separations were performed using either a polar capillary column (Stabilwax-DA, 30 m \times 0.25 mm i.d.; 0.5 μ m film;

Table 1. GC-MS Response Factors and Recovery Factors for Selected Volatile Compounds

no. ^a	compound	mass ion ^b	response factor ^c	recovery factor ^d	
				NDM	GLD
3	1-penten-3-one	55	0.206	0.661	0.860
6	1-hexen-3-one	55	0.185	0.856	0.889
11	1-hepten-3-one	70	0.543	0.664	0.705
15	1-octen-3-one	97	1.43	0.682	0.814
21	1-nonen-3-one	70	0.269	0.629	0.480
2	pentanal	58	1.15	0.641	0.685
5	hexanal	56	0.568	0.847	0.907
9	heptanal	70	0.674	0.860	0.853
14	octanal	84	1.18	0.824	0.680
20	nonanal	98	1.97	0.700	0.478
25	decanal	112	2.36	0.606	0.291
29	undecanal	82	1.64	0.604	0.148
32	dodecanal	82	1.05	0.396	0.0254
22	(<i>E</i>)-2-octenal	70	0.778	0.726	0.582
27	(<i>E</i>)-2-nonenal	70	0.867	0.685	0.372
30	(<i>E</i>)-2-decenal	70	0.755	0.916	0.298
34	(<i>E</i>)-2-undecenal	70	1.42	0.894	0.102
8	2-methyl-(<i>E</i>)-2-pentenal	98	0.792	0.477	0.489
19	7-octenal	93	2.96	0.756	0.694
23	8-nonenal	93	2.88	0.762	0.539
35	(<i>E,E</i>)-2,4-decadienal	81	0.210	0.490	0.254
31	phenylacetaldehyde	120	0.930	0.242	0.326
36	2-methoxyphenol (guaiacol)	124	0.267	0.850	0.525
40	vanillin	152	0.304	0.703	0.145

^a Numbers correspond to those in **Tables 3–5**. ^b Mass ion chosen for quantitative analysis. ^c Response factor is relative to 2-methyl-3-heptanone (mass ion = 128). ^d Recovery factor determined using either unsmoked NDM or unsmoked GLD as sample matrix.

Table 2. Attributes, Definitions, and References for the Sensory Descriptive Analysis of Unsmoked and Smoked Num Dok Mai and Gleep Lum Duan

attribute	definition	reference
starchy	aroma associated with starchy grains	raw rice or wheat grains
cooked rice	aroma of cooked rice	cooked rice
baked wheat	aroma of baked wheat	baked wheat
sweet aromatic	aroma associated with the aroma of cane sugar	sugar
smoky	aroma associated with wood smoke	burnt cotton
floral	aroma associated with flowers	yland-yland flower (<i>Cananga odorata</i>)
citrusy	aroma associated with lime peel	fresh lime peel
waxy	aroma associated with beeswax	beeswax

Restek) or a nonpolar column (RTX-5MS, 30 m × 0.25 mm i.d.; 0.5 μm film; Restek). The oven temperature was programmed from 35 to 225 °C at a rate of 6 °C/min with initial and final hold times of 5 and 10 min, respectively. Helium was used as carrier gas at a constant rate of 1.0 mL/min. The MSD conditions were as follows: capillary direct interface temperature, 280 °C; ionization energy, 70 eV; mass range, 35–300 amu; electron multiplier voltage (Autotune + 200 V); scan rate, 5.27 scans/s.

Compound Identification. Compound identification was based on matching retention indices (on two different GC column phases) and mass spectra of unknowns with those of authentic standards. A homologous series of *n*-alkanes was used for the determination of retention indices according to the method of van den Dool and Kratz (12).

Quantitative Analysis. The concentrations of a selected positively identified odorant were based on its area response ratio [extracted ion chromatogram area of compound (see **Table 1**)/extracted ion (*m/z* 128) chromatogram area of the internal standard (2-methyl-3-heptanone)]. Concentrations were corrected by use of MS response and recovery factors (**Table 1**) determined by spiking/recovery experiments using unsmoked NDM and GLD as sample matrices and by applying the same extraction and analysis methods described earlier. Determinations

Table 3. Formulations of Stock Solutions Used To Prepare Aroma Models

no. ^a	compound	% purity ^b	amount of compound (mg) per 10 mL of stock solution	
			S-NDM model	S-GLD model
Group 1, Vinyl Ketones ^c				
3	1-penten-3-one	99.0	3.80	31.1
6	1-hexen-3-one	95.0	3.64	33.1
11	1-hepten-3-one	95.7	5.50	69.5
15	1-octen-3-one	96.1	4.04	32.9
21	1-nonen-3-one	98.7	5.53	36.9
Group 2, <i>n</i> -Aldehydes ^c				
2	pentanal	99.1	21.0	61.1
5	hexanal	99.1	10.7	33.5
9	heptanal	97.4	10.8	25.9
14	octanal	99.0	15.8	55.9
20	nonanal	98.4	27.5	98.3
25	decanal	95.9	22.6	71.5
29	undecanal	96.9	43.3	99.3
32	dodecanal	95.8	24.7	201
Group 3, 2-Alkenals ^c				
22	(<i>E</i>)-2-octenal	97.2	9.10	46.9
27	(<i>E</i>)-2-nonenal	96.9	7.61	60.1
30	(<i>E</i>)-2-decenal	97.7	26.4	133
34	(<i>E</i>)-2-undecenal	99.5	62.0	443
Group 4, ω -Alkenals ^c				
19	7-octenal	98.4	17.9	35.8
23	8-nonenal	98.1	10.4	19.0
Phenylacetaldehyde ^c				
31	phenylacetaldehyde	95.4	34.4	78.6
2-Methoxyphenol ^{d,e}				
36	2-methoxyphenol (guaiacol)	99.5	15.5	22.5
Vanillin ^d				
40	vanillin	99.9	21.5	155

^a Numbers correspond to those in **Tables 1, 4**, and **5**. ^b Purity determined by GC analysis. ^c Stock solution was prepared in *n*-heptane. ^d Stock solution was prepared in methanol. ^e Levels adjusted on the basis of preliminary sensory evaluation.

were performed in triplicate on three separate batches of smoked desserts. Concentrations are expressed in micrograms per kilogram (ppb) on a wet sample basis.

Determination of Odor Detection Thresholds. Prior to threshold determinations compounds were purified by flash chromatography on silica gel 60 using a 95:5 pentane/ether mobile phase to a GC purity of >98%. ASTM procedure E679-91 (13) was used to determine orthonasal odor detection thresholds in odor-free water for 2-methyl-(*E*)-2-pentenal (**8**; 98.2% purity by GC), 7-octenal (**19**; 98.4% purity), 8-nonenal (**23**; 98.1% purity), and (*E*)-2-undecenal (**34**; 99.5% purity) and in fresh canola oil for 2-methyl-(*E*)-2-pentenal, 1-nonen-3-one (**21**; 98.7% purity) and vanillin (**40**; 99.9% purity). Stock solutions were prepared in methanol. Aliquots of the stock solutions were dissolved in the matrix and presented to panelists in 125-mL Teflon squeeze bottles as previously described (14). Panelists (11) were given each concentration (1:3 dilution series) along with two matrix blanks containing the same volume of methanol used in preparing the sample solutions. A group of seven series was tested in ascending order. The individual best estimate threshold was calculated as the geometric mean of the last concentration with an incorrect response and the first concentration with a correct response using the criteria previously described (13). The group best estimate threshold (BET) was calculated as the geometric mean of the individual BETs.

Sensory Descriptive Analysis. Panelists were selected on the basis of their ability to discriminate odor and flavor differences and to communicate their perceptions by use of duo-tri and ranking/rating tests (15). The 12-member panel (2 males, 10 females, 25–33 years old) received approximately 50 h of training during which they selected terms and references to describe the aroma attributes of the unsmoked and smoked desserts (**Table 2**) and learned to use an aroma intensity scale anchored by 0 = “none” on the left and 3 = “very” on the right

Table 4. Potent Odorants in Unsmoked and Smoked Num Dok Mai and Gleep Lum Duan

no. ^a	compound	odor ^b	RI ^c			FD factor ^d		
			WAX	RTX5	NDM	GLD	S-NDM	S-GLD
1	1-buten-3-one	plastic, painty	961	— ^e	nd ^f	nd	3	9
2	pentanal	painty	980	701	nd	nd	<3	9
3	1-penten-3-one	plastic, pungent	1021	680	nd	nd	3	81
4	unknown	potato	1042	—	nd	nd	3	27
5	hexanal	green, cut grass	1081	799	3	3	27	81
6	1-hexen-3-one	plastic, pungent	1096	777	nd	nd	6561	2187
7	unknown	potato	1158	—	nd	nd	27	27
8	2-methyl-(E)-2-pentenal	dark chocolate	1165	831	nd	nd	81	243
9	heptanal	melon, citrus, orange	1181	902	nd	nd	9	27
10	unknown	dark chocolate	1186	—	nd	nd	9	27
11	1-hepten-3-one	plastic, pungent	1196	876	nd	nd	2187	2187
12	(Z)-4-heptenal	stale, crabby	1240	901	nd	nd	nd	27
13	styrene	plastic	1241	895	nd	nd	27	81
14	octanal	orange oil	1282	1002	<3	nd	81	729
15	1-octen-3-one	mushroom	1296	976	nd	9	6561	729
16	unknown	potato	1303	—	nd	nd	<3	9
17	(E)-2-heptenal	pungent, fruity	1333	956	nd	nd	3	27
18	2-acetyl-1-pyrroline ^g	popcorn	1340	921	3	27	<3	9
19	7-octenal	fresh, melon	1354	996	nd	nd	243	729
20	nonanal	pungent, green	1388	1104	nd	nd	27	27
21	1-nonen-3-one	mushroom, pungent	1398	1078	nd	nd	243	729
22	(E)-2-octenal	fresh, melon, green	1436	1058	nd	nd	243	2187
23	8-nonenal	stale, fatty, plastic	1445	1095	nd	nd	6561	2187
24	3-(methylthio)propanal (methional)	potato	1462	906	3	3	nd	nd
25	decanal	pungent, green, soapy	1495	1208	nd	nd	9	9
26	(Z)-2-nonenal	hay, stale	1507	1151	nd	<3	243	729
27	(E)-2-nonenal	hay, stale, fatty	1535	1162	<3	3	243	729
28	(E,Z)-2,6-nonadienal	cucumber	1587	1155	3	3	nd	27
29	undecanal	green, pungent	1600	1303	nd	nd	27	27
30	(E)-2-decenal	pungent, soapy	1641	1264	nd	nd	27	27
31	phenylacetaldehyde	rosy, styrene	1653	1049	nd	9	nd	3
32	dodecanal	green, pungent, soapy	1701	1404	nd	nd	3	9
33	unknown	hay, fatty	1735	—	9	3	81	27
34	(E)-2-undecenal	cilantro, green, soapy	1754	1366	nd	nd	729	81
35	(E,E)-2,4-decadienal	fatty, fried	1817	1317	nd	9	27	81
36	2-methoxyphenol (guaiacol)	smoky	1862	1090	<3	<3	729	729
37	4-hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF) ^g	burnt sugar, caramel	2034	1071	3	9	243	81
38	(Z)- α -santalol ^h	incense, woody, cedar	2350	1674	nd	nd	3	nd
39	(Z)- β -santalol ^h	incense, woody, fragrant	2434	1716	nd	nd	9	3
40	vanillin	vanilla	2576	1404	3	nd	729	81

^a Numbers correspond to those in Tables 1, 3, and 5. ^b Odor quality as perceived during GCO. ^c Retention indices were calculated from GCO data; WAX, Stabilwax column. ^d Flavor dilution (FD) factor determined on Stabilwax column; NDM, unsmoked num dok mai; GLD, unsmoked gleep lum duan; S-NDM, smoked NDM; S-GLD, smoked GLD. ^e —, not available. ^f nd, not detected. ^g Compound was tentatively identified on the basis of comparison of its odor property and retention indices with reference compound. ^h Compound was identified on the basis of mass spectra and retention indices determined for (Z)- α -santalol or (Z)- β -santalol in authentic sandalwood oil and by comparison to literature (4).

with 0.5 unit increments as previously described (16). For evaluation, samples were presented in PTFE bottles coded with three-digit random numbers as previously described (11). Samples were evaluated in duplicate. Differences among samples were evaluated by analysis of variance with means separation using SPSS version 11 software (SPSS Inc., Chicago, IL).

Preparation of Aroma Models. Prior to use, all odorants used in model studies, except 36 and 40, were distilled under vacuum ($\sim 5 \times 10^{-3}$ Torr). Compound purities are provided in Table 3. Aroma models were prepared by spiking an unsmoked NDM or GLD matrix with various odorant combinations. This was accomplished by first shredding 1 kg of the unsmoked dessert matrix in a food processor. A 100-g portion of the shredded material was then spiked with 10 μ L of each of the stock solutions (indicated in Table 3) to give the complete model. Omission models were prepared in the same manner, except that each time a different odorant or group of odorants was omitted. Prior to conducting any sensory analyses, the aroma model mixtures were allowed to equilibrate for >24 h at room temperature in tightly capped glass jars equipped with PTFE-lined caps.

Sensory Comparison of Aroma Models. Triangle difference tests (15, 17) were employed to determine if panelists could detect differences between each of the complete aroma models and model mixtures in which single odorants or groups of odorants had been

omitted. Forty untrained panelists participated in the sensory evaluation testing. Aroma model mixtures were served in three-digit coded PTFE bottles (as described above) in random order with all six possible combinations being presented to the panel (15). Panelists were told that two samples were identical and one was different. They were then instructed to evaluate the odor of each sample from left to right, with the option of going back to repeat the evaluation if needed, and to select the sample that was odd or different among the three. Results were analyzed according to the method of Roessler et al. (17).

RESULTS AND DISCUSSION

Sensory Attributes of Tain Op Smoked Desserts. The two types of unsmoked matrices (NDM and GLD) had markedly different sensory aroma profiles (Figure 2). Although waxy and floral notes were detected at moderate intensities in both matrices, the low-fat matrix (NDM) contained pronounced starchy, cooked rice and sweet aromatic notes which were either found at low intensity or not detected (i.e., cooked rice) in the high-fat matrix (GLD). Meanwhile, panelists detected a pronounced baked wheat note in GLD, but not in NDM. It is

Table 5. Concentrations and Odor-Activity Values (OAV) of Selected Volatile Components of Unsmoked and Smoked Num Dok Mai and Gleep Lum Duan

no. ^a	compound	concn ^b (μg/kg)				odor threshold ^c (μg/kg)	OAV ^d			
		NDM	GLD	S-NDM	S-GLD		NDM	GLD	S-NDM	S-GLD
3	1-penten-3-one	nd ^e	nd	38.0 (±38%)	311 (±37%)	1.3 [27] 5.5 [27]	— ^f	—	29.2	239
6	1-hexen-3-one	nd	nd	36.4 (±50%)	331 (±39%)	0.024 [28] 5 [29]	—	—	1520	13800
11	1-hepten-3-one	nd	nd	55.0 (±47%)	695 (±45%)	0.04 [30] 7 [29]	—	—	1380	17400
15	1-octen-3-one	nd	nd	40.4 (±57%)	329 (±52%)	0.005 [31] 10 [32]	—	—	5050	41130
21	1-nonen-3-one	nd	nd	55.3 (±24%)	369 (±76%)	0.008 [33] 6 ^h	—	—	6910	46100
2	pentanal	2.43 (±15%)	12.8 (±76%)	2100 (±40%)	6110 (±38%)	12 [34] 240 [35]	0.2	1.1	175	509
5	hexanal	69.0 (±12%)	94.2 (±73%)	1070 (±38%)	3350 (±51%)	4.5 [27] 120 [27]	15.3	20.9	238	744
9	heptanal	nd	nd	1080 (±38%)	2590 (±25%)	3 [27] 250 [27]	—	—	360	863
14	octanal	21.3 (±31%)	25.3 (±63%)	1580 (±31%)	5590 (±63%)	0.7 [34] 320 [35]	30.4 <1	36.1	2260	7990
20	nonanal	69.5 (±32%)	220 (±28%)	2750 (±31%)	9830 (±64%)	1 [27] 1000 [27]	69.5	220	2750	9830
25	decanal	15.2 (±18%)	76.1 (±36%)	2260 (±59%)	7150 (±68%)	2 [34] 6700 [35]	7.6	38.1	1130	3580
29	undecanal	nd	nd	4330 (±85%)	9930 (±70%)	5 [36] 6800 [35]	<0.1	<0.1	0.3	1.1
32	dodecanal	87.4 (±43%)	170 (±67%)	2470 (±75%)	20100 (±75%)	2 [37] 3000 [35]	43.7	85.0	1240	10050
22	(E)-2-octenal	nd	nd	91.3 (±49%)	469 (±56%)	3 [27] 500 [27]	—	—	30.4	156
27	(E)-2-nonenal	3.90 (±32%)	29.9 (380%)	76.1 (±74%)	601 (±59%)	0.08 [27] 150 [27]	48.8	374	951	7510
30	(E)-2-decenal	0.93 (±25%)	14.8 (±45%)	264 (±44%)	1330 (±65%)	0.3 [27] 2100 [27]	3.1	49.3	880	4430
34	(E)-2-undecenal	nd	nd	616 (±71%)	4430 (±77%)	3.4 ^g 150000 [35]	—	—	180	1290
8	2-methyl-(E)-2-pentenal	15.1 (±25%)	31.8 (±24%)	35.5 (±2%)	91.6 (±59%)	580 ^g 3500 ^h	<0.1	<0.1	<0.1	0.2
19	7-octenal	nd	nd	179 (±31%)	358 (±51%)	21.5 ^g 510 [38]	—	—	8.3	16.7
23	8-nonenal	nd	nd	104 (±31%)	190 (±62%)	0.03 ^g 1600 [38]	—	—	3470	6330
35	(E,E)-2,4-decadienal	2.16 (±14%)	31.9 (±48%)	14.3 (±54%)	162 (±74%)	0.07 [27] 135 [27]	30.9	456	204	2314
31	phenylacetaldehyde	2.87 (±10%)	57.7 (±72%)	34.4 (±26%)	78.6 (±19%)	4 [27] 22 [27]	0.7	14.4	8.6	19.7
36	2-methoxyphenol(guaiacol)	0.48 (±18%)	0.75 (±59%)	105 (±55%)	175 (±36%)	3 [34] 16 [39]	0.2	0.3	35.0	58.3
40	vanillin	212 (±92%)	35.7 (±88%)	2150 (±90%)	1550 (±95%)	25 [40] 95 ^h	8.5	1.4	86.0	62.0

^a Numbers correspond to those in **Tables 1, 3, and 4**. ^b Average concentration (± percent relative standard deviation) of data from triplicate samples; NDM, unsmoked num dok mai; GLD, unsmoked gleep lum duan; S-NDM, smoked NDM; S-GLD, smoked GLD. ^c Orthonasal odor threshold in water or in oil (italics). Reference is provided in brackets. ^d Odor-activity value = concentration divided by odor detection threshold. ^e nd, not detected. ^f Not available. ^g Orthonasal odor detection threshold in water determined in present study (see Materials and Methods). ^h Orthonasal odor detection threshold in canola oil determined in present study (see Materials and Methods).

noteworthy that both matrices received only low intensity ratings for the citrusy attribute, and neither had a perceivable smoky note.

As a result of the Tian Op smoking process, the intensities of the citrusy, floral, and smoky notes increased in both S-NDM and S-GLD. In addition, the smoking process caused an increase in the sweet aromatic attribute in S-GLD, but this note remained at about the same high intensity in S-NDM. Intensities of the remaining sensory attributes (i.e., starchy, waxy cooked rice, and baked wheat) were not affected by the smoking process. The end result was that S-NDM and S-GLD were nearly the same with respect to intensities of the citrusy, smoky, floral, and sweet aromatic notes imparted by the Tian Op smoking process.

Major Volatile Components. Treatment of NDM and GLD with Tian Op smoke resulted in large increases in hydrocarbons

and oxygenated compounds in the smoked desserts (S-NDM and S-GLD, respectively) (data not shown). For both S-NDM and S-GLD, the major volatile compounds were *n*-alkanes (C₇–C₁₄), α-1-alkenes (C₇–C₁₄), and *n*-aldehydes (C₅–C₁₂). Branched alkanes, α-1,ω-1-alkadienes, unsaturated aldehydes, methyl ketones, and vinyl ketones were some of the minor volatile constituents identified. The occurrence of the homologous series of *n*-alkanes, α-1-monoalkenes, and α-1,ω-1-dialkenes is in agreement with volatile patterns reported for the pyrolysis GC-MS analysis of beeswax (18). It was previously demonstrated that the formation of α-1-monoalkenes in beeswax during pyrolysis is the result of either radical alkyl chain degradation or ester scission of the ester-bound fatty acids (19). Meanwhile, methyl ketones and *n*-aldehydes (homologous series from C₄ to C₁₇) were reported as major degradation products of the thermal degradation of tristearin in air (20). These

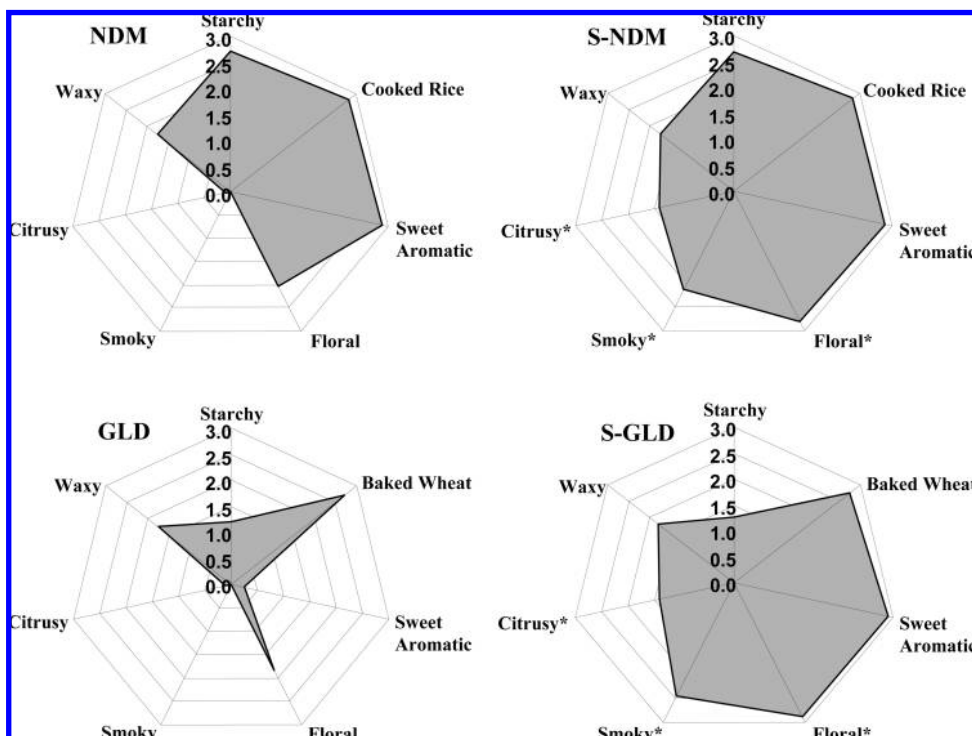


Figure 2. Sensory descriptive aroma profile comparison of unsmoked and smoked (S-) num dok mai (NDM) and gleep lum duan (GLD). Attributes marked with an asterisk are significantly different between unsmoked and smoked desserts ($p \leq 0.05$).

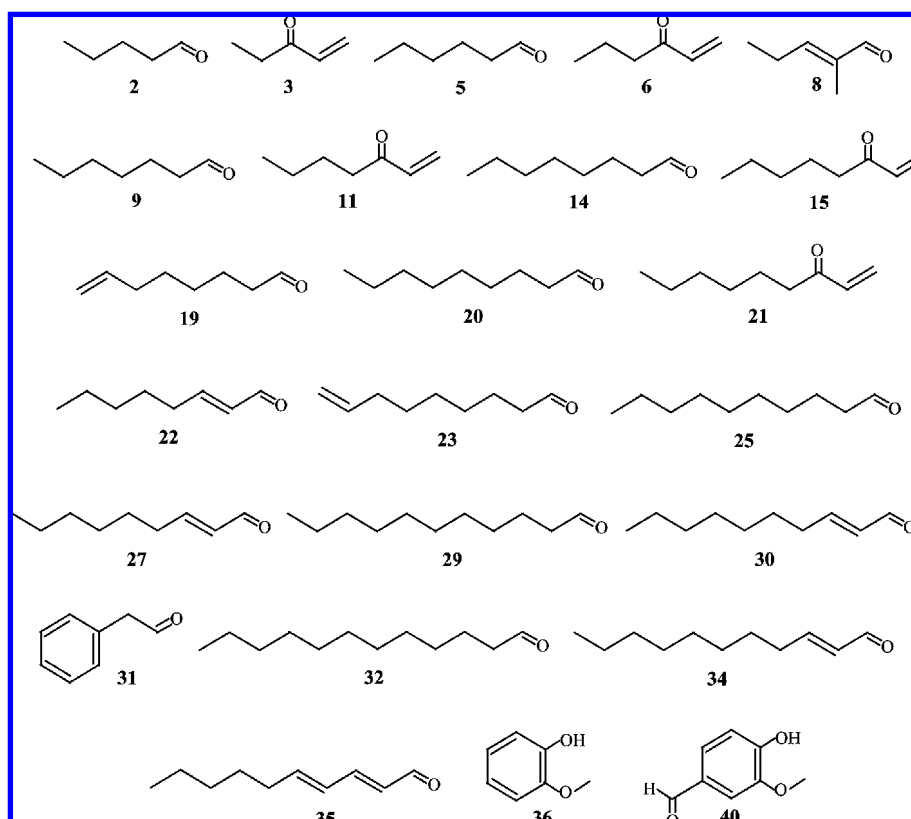


Figure 3. Structures for odorants listed in Table 5.

researchers concluded that there was no specific oxidation pattern and that all of the fatty acid methylene carbon atoms were susceptible to oxidation at high temperatures with preferential attack occurring near the center of the molecule. Similar results were observed for the thermal/oxidative degradation of methyl palmitate (21).

AEDA. The predominant odorants of the unsmoked and smoked desserts were identified by means of GCO and AEDA (Table 4). A combined total of 14 odorants were found at low flavor dilution (FD) factors (≤ 27) in the two unsmoked desserts matrices. Eight common odorants were detected in both matrices, with more odorants being detected in GLD (12) than

Table 6. Triangle Difference Test Comparison of Complete Aroma Models against Smoked Desserts and Omission Model Mixtures^a

comparison of complete models against	correct responses (significance) ^b	
	S-NDM	S-GLD
smoked desserts	11 (NS)	8 (NS)
model with vinyl ketones omitted	10 (NS)	12 (NS)
model with <i>n</i> -aldehydes omitted	30 (*)	32 (*)
model with (<i>E</i>)-2-unsaturated aldehydes omitted	12 (NS)	10 (NS)
model with ω -1-aldehydes omitted	28 (*)	25 (*)
model with phenylacetaldehyde omitted	8 (NS)	10 (NS)
model with 2-methoxyphenol (guaiacol) omitted	29 (*)	26 (*)
model with vanillin omitted	9 (NS)	10 (NS)

^a Model compositions are described under Materials and Methods and in **Table 3**. ^b Number of correct responses ($n = 40$). Level of significance in parentheses: NS, not significantly different ($p > 0.05$); *, significantly different ($p \leq 0.05$).

in NDM (10). The majority of the odorants in NDM and GLD were either lipid-derived (e.g., **5**, **14**, **15**, **26–28**, and **35**) or thermally generated compounds (e.g., **18**, **24**, **36**, **37**, and **40**) originating from the ingredients or produced during steaming/baking.

The treatment of the NDM and GLD with Tian Op smoke caused a large increase in both the number and potencies of the odorants found in the smoked desserts. A combined total of 39 odorants were detected by AEDA in the smoked desserts, with 36 found in S-NDM and 38 in S-GLD. With the exception of 2-acetyl-1-pyrroline (**18**), methional (**24**), and phenylacetaldehyde (**31**), all of the odorants detected in the unsmoked dessert matrices increased in potency as a result of the smoking process. S-NDM and S-GLD were similar with respect to the potent odorants identified (35 common odorants detected). On the basis of their high overall FD factors (≥ 243), the predominant odorants of both S-NDM and S-GLD were vinyl ketones (C_5 – C_9), *n*-aldehydes (C_5 – C_{11}), (*E*)-2-unsaturated aldehydes (C_8 – C_{11}), and ω -1-unsaturated aldehydes (C_8 and C_9). In particular, 1-hexen-3-one (**6**), 1-hepten-3-one (**11**), 1-octen-3-one (**15**), 8-nonenal (**23**), and guaiacol (**36**) had the highest FD

factors (≥ 729) in both S-NDM and S-GLD. Odorants with FD factors ≥ 729 in at least one of the two smoked desserts included octanal (**14**), 7-octenal (**19**), 1-nonen-3-one (**21**), (*E*)-2-octenal (**22**), (*Z*)-nonenal (**26**), (*E*)-2-nonenal (**27**), (*E*)-2-undecenal (**34**), and vanillin (**40**).

The majority of the above-mentioned potent odorants were most likely produced as a result of the thermal oxidation of the beeswax components as discussed earlier. Saturated and (*E*)-2-unsaturated aldehydes were reported as the most abundant odor-active compounds formed during heating of triolein and trilinolein (**22**). Vinyl ketones (1-hepten-3-one, 1-octen-3-one, and 1-nonen-3-one), along with aldehydes [hexanal, octanal, nonanal, and (*E*)-2-nonenal] were reported as being primarily responsible for the off-odor associated with thermally oxidized polyethylene (**23**). 7-Octenal was previously reported as a volatile decomposition product of thermally oxidized triolein (**24**). Sanders et al. (**25**) reported that 8-nonenal contributed a “plastic” off-odor to high-density polyethylene packaging. In addition to the above, some odorants may have originated from other ingredients of the Tian Op candle, such as vanillin from benzoin (**3**) and (*Z*)- α -santalol and (*Z*)- β -santalol from sandalwood (**4**). Likewise, guaiacol may have been derived from the pyrolysis of any of the lignin-containing ingredients (e.g., cotton wick or sandalwood) (**26**).

Quantitative Analysis. Selected positively identified odorants from **Table 4** were quantified in the unsmoked and smoked desserts (**Figure 3**; **Table 5**). The relatively high standard deviations are reflective of the fact that the average concentrations are based on the analysis of three totally independent samples for each type of dessert. NDM and GLD contained a low abundance of odorants **2**, **5**, **8**, **14**, **20**, **25**, **27**, **30–32**, **35**, **36**, and **40**. In both S-NDM and S-GLD, the *n*-aldehydes (**2**, **5**, **9**, **14**, **20**, **25**, **29**, and **32**) were in highest abundance, followed by vanillin (**40**), (*E*)-2-unsaturated aldehydes (**22**, **27**, **30**, and **34**), ω -1-unsaturated aldehydes, vinyl ketones (**3**, **6**, **11**, **15**, and **21**), guaiacol (**36**), (*E,E*)-2,4-decadienal (**35**), phenylacetaldehyde (**31**), and 2-methyl-(*E*)-2-pentenal (**8**). All odorants were found at higher abundance in S-GLD than in S-NDM. The GLD matrix

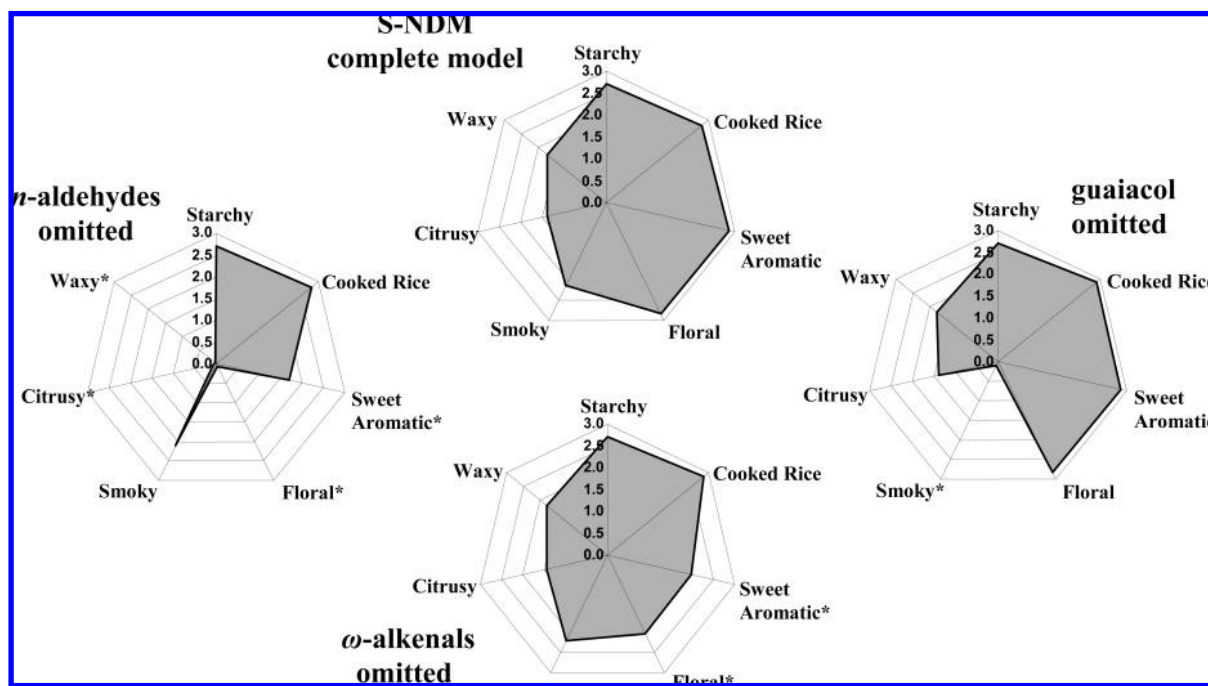


Figure 4. Sensory descriptive aroma profile comparison of smoked num dok mai (S-NDM), the complete S-NDM model, and selected NDM omission models. Attributes marked with an asterisk are significantly different between the omission model and the complete model ($p \leq 0.05$).

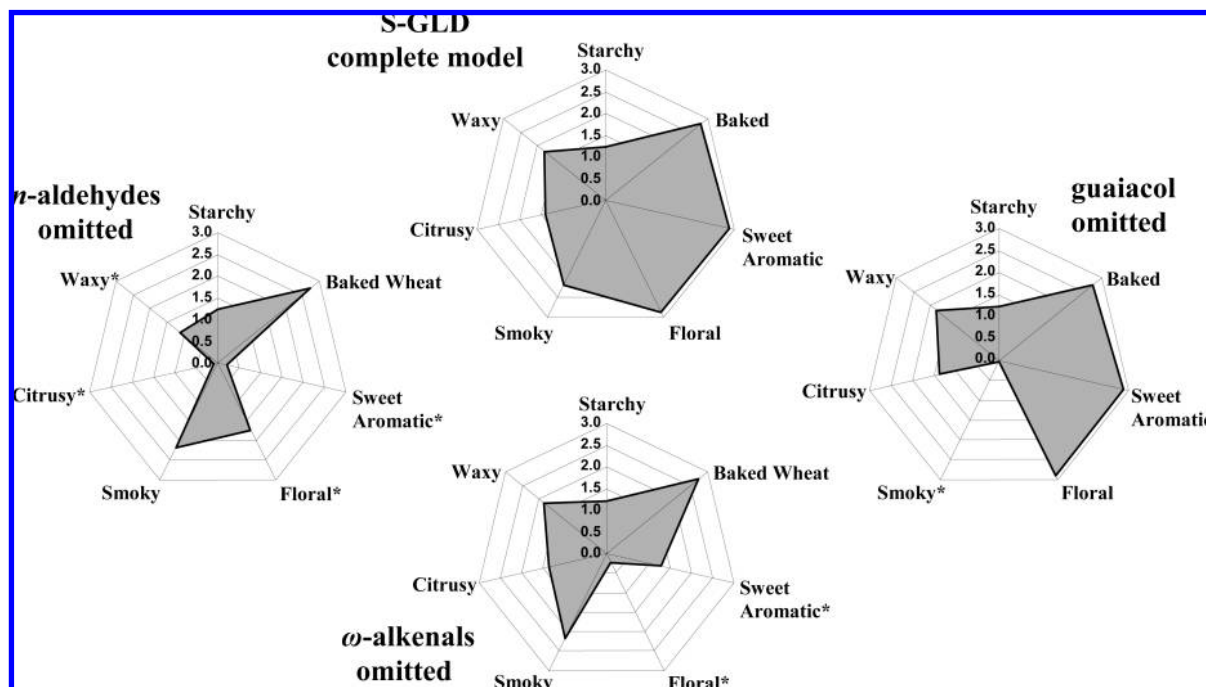


Figure 5. Sensory descriptive aroma profile comparison of smoked gleep lum duan (S-GLD), the complete S-GLD model, and selected S-GLD omission models. Attributes marked with an asterisk are significantly different between the omission model and the complete model ($p \leq 0.05$).

had a higher fat content than NDM, and because of its more lipophilic nature, the GLD matrix would be expected to more readily absorb the nonpolar odorants from the Tian Op smoke.

Odor-Activity Values (OAVs). OAVs were calculated to estimate the potential sensory impact of each odorant based on the quantitative data (Table 5). Due to the complexities of the two dessert matrices, OAVs were calculated from both water- and oil-based odor detection thresholds. Except for some odorants with low extraction recovery factors (e.g., 25, 29, 30, 32, and 34; see Table 1), the calculated OAVs (Table 5) are in good agreement with FD factors determined by AEDA. The above-mentioned discrepancies can be explained by the fact that recovery factors were not taken into consideration in AEDA, whereas they were used in the quantification method. Therefore, for poorly recovered odorants, the OAVs, which are based on accurate quantitative data, should be more indicative of an odorant's actual potency as long as the odor detection thresholds are reasonably accurate.

The vinyl ketones (in particular 6, 11, 15, and 21), despite being present in relatively low abundance, had the highest OAVs in both S-NDM and S-GLD. This is due to the overall low odor detection thresholds (in both water and oil) of these odorants (especially 15 and 21). Some other odorants in low abundance and having low odor detection thresholds (in water), for example, 8-nonenal (23) and (*E,E*)-2,4-decadienal (35), had relatively high OAVs. Likewise, eight compounds found in high abundance, that is, *n*-aldehydes 14, 20, 25, 29, and 32 and (*E*)-2-unsaturated aldehydes 27, 30, and 34, also had particularly high OAVs on the basis of their odor detection thresholds in water. However, when their odor detection thresholds in oil were considered, the OAVs for the above-mentioned aldehydes were markedly lower. On the basis of the OAV concept, the above-mentioned odorants should make the greatest impact on the overall aroma of the smoked desserts. However, these data are only suggestive, and sensory studies of model mixtures are necessary to fully assess the actual contribution of these odorants to the overall aroma of S-NDM and S-GLD.

Omission Studies. Twenty-two odorants were selected for additional sensory studies on the basis of the results of GCO and AEDA and the calculated OAVs. Omission studies were performed by comparing the complete models (mixture of all 22 compounds in Table 3) against actual smoked desserts (S-NDM and S-GLD) and omission models in which one or several odorants were omitted. This strategy, where more than one odorant is omitted at a time, has been previously used when a large number of compounds were to be evaluated (41, 42). In the present study, omission experiments were conducted by omitting groups of odorants having similar odor properties or based on grouping a homologous series of compounds (e.g., *n*-aldehydes). To account for odorant–matrix interactions, models were prepared in either the unsmoked NDM or GLD matrix prior to analysis.

The composition of the complete model was based on the quantitative data in Table 5. Compounds 8 and 35 were excluded from the model because they were found at similar levels in both the unsmoked and smoked desserts. Furthermore, initial sensory evaluation of complete model mixtures for both S-NDM and S-GLD indicated that the smoky note in the complete models was lower than for the smoked desserts (data not shown). Therefore, it was necessary to adjust (increase) the level of guaiacol (36) in both complete models until the panel consistently scored the smoky note at the same intensities as in the smoked desserts. The final levels of guaiacol used in the sensory studies are indicated in Table 3.

Triangle difference testing was used to compare each of the complete models (S-NDM or S-GLD) against the actual smoked desserts and against each of their respective omission models (Table 6). Results showed that the actual smoked desserts could not be distinguished from their corresponding complete models. This was further supported by the fact that the aroma profiles determined by sensory descriptive analysis did not significantly differ between the smoked desserts and the complete models (see Figures 2 and 4 for a comparison between S-NDM and the S-NDM complete model and Figures 2 and 5 for a comparison between S-GLD and the S-GLD complete model).

Both types of model systems were similar in that only the *n*-aldehyde (group 2), the ω -1-unsaturated aldehyde (group 3), and guaiacol omission models could be distinguished from the complete models. The remaining omission models were not significantly different from the complete models. These data suggest that *n*-aldehydes, ω -1-unsaturated aldehydes, and guaiacol profoundly influence the overall aroma of the complete models, whereas the vinyl ketones, (*E*)-2-unsaturated aldehydes, and phenylacetaldehyde may make only a minor contribution. It was expected that omission of phenylacetaldehyde would have no appreciable impact on the aroma of the complete models, because the OAVs for this odorant were relatively low. On the other hand, the OAVs for both the vinyl ketones and (*E*)-2-unsaturated aldehydes indicated that they were potent odorants; thus, it was surprising that those omission models were not discriminated from the complete models.

Sensory descriptive aroma profiles for the S-NDM and S-GLD complete models and for selected omission models (those shown to be different from the complete models) are shown in **Figures 4** and **5**. Omission of *n*-aldehydes had a dramatic effect on the aroma profile, causing a large decrease in the intensity of the citrusy note and moderate decreases in waxy and floral notes. In addition, moderate (S-NDM) to large (S-GLD) decreases in the sweet aromatic note were observed. Declines in the intensities of the sweet aromatic and floral attributes were observed when the ω -1-unsaturated aldehydes were omitted. This effect was more pronounced for the S-GLD omission models. Omission of guaiacol caused nearly a complete loss of the smoky note in both models. As expected, there was no influence of any of the omissions on the intensities of the starchy, cooked rice or baked wheat attributes. The above findings confirm the importance of the *n*-aldehydes, the ω -1-unsaturated aldehydes, and guaiacol to the overall aroma of the Tian Op smoked desserts.

Human panelists have a limited capacity to identify components of complex odor mixtures. This is thought to be largely due to inhibition of olfactory receptor cells by odorants via competitive mechanisms (43). Mixture suppression or antagonism, in particular, by the highly abundant *n*-aldehydes, might explain the inability of the panel to detect the presence or absence of vinyl ketones. On the other hand, it is possible that odorants with quite dissimilar odor properties may be more easily detected in a mixture, which could explain why the presence or absence of the smoky note of guaiacol was readily detected in the models. Cross-adaptation between the various aldehydes, for example, *n*-aldehydes versus (*E*)-2-unsaturated aldehydes, might explain why (*E*)-2-unsaturated aldehyde omission models were not distinguishable from the complete models. This does not appear to be true for all of the aldehydes, because models in which the ω -1-unsaturated aldehydes were omitted were discriminated from the complete models. It is possible that the ω -1-unsaturated aldehydes do not cross-adapt with the other aldehydes in the models. Additional studies would be necessary to fully understand this relationship.

In summary, the results of GCO and AEDA indicated *n*-aldehydes, (*E*)-2-unsaturated aldehydes, ω -1-unsaturated aldehydes, and vinyl ketones as predominant odorants in two types of traditional Thai desserts prepared using a Tian Op candle smoking process. These findings were further supported by the quantitative data and calculated OAVs for selected odorants. Sensory studies of model mixtures confirmed the importance of *n*-aldehydes, ω -1-unsaturated aldehydes, and guaiacol as predominant odorants contributed by the smoking process; however, the results showed that vinyl ketones and (*E*)-2-

unsaturated aldehydes, despite their high odor potencies, may be of only minor importance in the typical aroma profiles of traditional Tian Op smoked desserts.

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Received for review August 29, 2008. Revised manuscript received December 10, 2008. Accepted December 13, 2008.

JF802674C