



# *Changes in aroma and sensory profile of food ingredients smoked in the presence of a zeolite filter*

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## Changes in Aroma and Sensory Profile of Food Ingredients Smoked in the Presence of a Zeolite Filter

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During smoking, formation of desirable smoky compounds and carcinogenic polycyclic aromatic hydrocarbons (PAH) are inextricably linked. We have previously developed a zeolite filter technology (PureSmoke Technology or PST) that reduces the PAH content of a smoke stream, particularly reducing the concentration of benzo[a]pyrene, a known carcinogen, by up to 93%. The aim of this work was to determine whether there were changes in the volatile and sensory profiles of ingredients smoked using PST compared to the traditional smoking process (Trad). Smoked tomato flakes (either PST or Trad) were added to either low-fat or full-fat cream cheese for sensory profiling and consumer preference tests, and volatile analysis was carried out using solid phase microextraction (SPME) followed by gas chromatography-mass spectrometry (GC-MS). The sensory analysis showed a significant decrease ( $p < 0.01$ ) in bitterness

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when the PST was employed and a significant decrease in overall smoky aroma and flavor ( $p < 0.001$ ), which resulted in an increase in the perception of cheesy aroma and flavor. This was consistent with a decrease in many of the smoky aroma compounds, particularly the guaiacols. However, consumer preference tests showed that there was no adverse effect on the flavor of the products, and there was even a tendency for the PST product to be preferred to the Trad product ( $p = 0.096$ ). The smoke compounds were quantitated and compared in smoked tomato paste. Odor activity values (OAVs) calculated from the literature thresholds suggested that guaiacol and 4-alk(en)yl-substituted guaiacols are likely to be among the most highly odor-active compounds in these smoked ingredients.

## Introduction

The use of smoke for preservation has become secondary to its use in creating unique smoky aromas and flavors in foods. Smoked ingredients are used widely by the food industry to impart a characteristic smoky flavor to rubs, dips, marinades, soups, and snacks. The volatile components of aqueous smokes have been studied extensively<sup>1</sup> as have the smoky aroma compounds in various fish<sup>2,3</sup> and cheeses.<sup>4-6</sup> The highly desirable smoky flavor is generated by the burning of wood chips, of varying origin, at high temperatures (400–1000 °C). Phenolic compounds such as syringol and guaiacol are essential for the sensory characteristics of smoke, and the gas chromatography–olfactometry (GC–O) of smoked fish has shown many more important compounds that contribute to the smoky aroma.<sup>7,8</sup>

However, the smoking process also results in the formation of polycyclic aromatic hydrocarbons (PAHs). These PAHs are a series of fused benzene ring structures, and many of these are classified as Class 2 carcinogens. One of these PAHs, benzo[a]pyrene, is a known carcinogen, and epidemiological evidence has implicated smoked foods in an increased risk of cancer in humans.<sup>9,10</sup> In 2015, Griffiths, Baines, and Parker-Gray<sup>11</sup> developed a filtration technology based on zeolites (PureSmoke Technology or PST) whereby up to 93% of the PAHs could be removed from a smoke stream.<sup>12</sup> Comparison of the headspace of oils smoked either traditionally (Trad) or through the filter (PST) showed that generally, the low molecular weight aroma compounds were not removed by the filter. However, many of the components of smoke most likely to contribute to the smoky flavor were partially removed by the filter, particularly the guaiacols and

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9 the eugenols. Preliminary sensory testing of smoked tomato ketchup suggested  
10 that the PST product had a sweeter aroma than the Trad product. Differences in  
11 the smoky, rubbery, and tar aroma and flavor were not observed, despite a  
12 decrease in smoky aroma compounds; these differences were possibly masked by  
13 the intensity of the neat ketchup. The changes in flavor warrant further  
14 investigation since it is important to establish that PST does not adversely affect  
15 the flavor of the product. In this study, we used tomato flakes that were Trad or  
16 PST smoked. The flakes were finely ground and added to cream cheese for  
17 sensory, consumer, and instrumental analysis.  
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## 19 20 **Materials and Methods**

### 21 22 **Materials**

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26 Tomato flakes were purchased from Camstar Ingredients (Eye, U.K.), and  
27 tomato paste was purchased from Silbury Marketing (Banbury, U.K.). They were  
28 smoked with oak chips obtained from Ashwood Smoking Chips (Kettering, U.K.)  
29 using either PST or Trad smoking. Portions of the smoked tomato flakes were  
30 ground with a pestle and mortar and sieved. The fraction collected from sieve size  
31 3 (355  $\mu\text{m}$ –1 mm) was added to either low-fat or full-fat cream cheese for volatile  
32 analysis, sensory profiling, and consumer testing. The tomato paste was used to  
33 quantitatively compare the aroma compounds derived from either PST or Trad  
34 smoking.

35 Two types of cream cheese were used in order to provide four samples for  
36 sensory profiling, rather than just two. Tubs (180 g) of Philadelphia Original (21%  
37 fat) and Philadelphia Light (11% fat) (Mondelez, Uxbridge, UK) were purchased  
38 from one local supermarket, ensuring that the tubs for each product were from one  
39 batch. Ground smoked tomato flakes were added to the cheese (2.5% w/w), mixed  
40 thoroughly, and returned to the container to equilibrate overnight before tasting  
41 or analysis. Thus, four samples of cream cheese with smoked tomato flakes were  
42 prepared: low-fat Trad, low-fat PST, full-fat Trad, and full-fat PST. All sensory  
43 references were purchased from a local supermarket.

44 2-Octanol, 2,3-butanedione (diacetyl), 3-hydroxybutanone (acetoin), acetic  
45 acid, benzeneacetaldehyde, 6-methyl-5-hepten-2-one, 2-acetylpyrrole, phenol, 5-  
46 butyl-4-methyloxolan-2-one (whiskey lactone, mix of two isomers), 4-methyl-2-  
47 methoxyphenol (4-methylguaiacol), 4-ethyl-2-methoxyphenol (4-ethylguaiacol),  
48 2-methoxy-4-propylphenol (4-propylguaiacol), 4-ethenyl-2-methoxyphenol (4-  
49 vinylguaiacol), 2,6-dimethoxyphenol (syringol), 2-hydroxy-3-methyl-2-  
50 cyclopenten-1-one (cyclotene), and 2,6-dimethylphenol were purchased from  
51 Sigma-Aldrich (Poole, U.K.). 2-Furaldehyde (furfural), 1-(2-furyl)ethanone (2-  
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9 acetylfuran), 5-methyl-2-furaldehyde (5-methylfurfural), 4-methylphenol (*p*-  
10 cresol), 2-methylphenol (*o*-cresol), and 2-methoxyphenol (guaiacol) were  
11 obtained from Fisher Scientific (Loughborough, U.K.). 2-Methoxy-4-[(1*E*)-1-  
12 propen-1-yl]phenol ([*E*]-isoeugenol) containing 1% of the *Z*-isomer and 4-allyl-  
13 2-methoxyphenol (eugenol) were purchased from Givaudan (Milton Keynes,  
14 U.K.). 2-Isopropyl-5-methylphenol (thymol) was purchased from Mane (London,  
15 U.K.).  
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### 18 **Volatile Analysis of Cream Cheese with Added Smoked Tomato Flakes**

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20 Samples of cream cheese (20 g) were incubated in a Duran bottle at 40 °C for  
21 30 min. A triple-phase Stabilflex fiber (PDMS/Carboxen/DVB, 11 mm, from  
22 Supelco, Poole, U.K.) was exposed to the headspace for a further 30 min to extract  
23 the volatile compounds. Gas chromatography-mass spectrometry (GC-MS)  
24 analysis was conducted using a 5972 MS coupled to an Agilent Technologies  
25 5890 GC (Agilent, Santa Clara, CA, U.S.A.). Each extraction was injected in  
26 splitless mode onto a J&W DB-WAX column (30 m × 250 μm × 1 μm film  
27 thickness) (Agilent, Santa Clara, CA), and the following temperature program was  
28 employed: 2 min at 40 °C, then raised to 250 °C at a rate of 5 °C/min. The flow  
29 rate of the helium carrier gas was 0.9 mL/min. Mass spectra were measured in  
30 electron ionization mode at 70 eV. The scan range was from *m/z* 29–300. Samples  
31 (20 g) of unsmoked tomato flakes, unsmoked tomato paste, and unflavored full-  
32 fat and low-fat cheese were also analyzed for comparison purposes. Volatiles  
33 were identified by comparing each mass spectrum with the spectrum of the  
34 authentic compounds analyzed in our laboratory. To confirm the identification,  
35 the linear retention index (LRI) was calculated for each volatile compound using  
36 the retention times of a homologous series of C<sub>6</sub>–C<sub>25</sub> *n*-alkanes and by comparing  
37 the LRI with those of authentic compounds analyzed under similar conditions.  
38 Samples were also analyzed on a non-polar DB5 column (30 m × 250 μm × 1 μm  
39 film thickness) (Agilent, Santa Clara, CA) using the same temperature program  
40 to further confirm their identity. Rather than adding an internal standard into a  
41 semi-solid cheese, an external standard of 2-octanol was injected every six  
42 samples. The deviation in the peak areas was no greater than 10% and there was  
43 no observed trend.  
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### 45 **Sensory Profiling of Cream Cheese with Added Smoked Tomato Flakes**

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47 A panel of nine trained assessors (90% women aged 35–60), each with a  
48 minimum of six months' experience, was used to develop a quantitative sensory  
49 profile for describing the sensory characteristics of the four different samples of  
50 cream cheese. Following an initial collection of terms, reference materials were  
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provided to help assessors standardize the terms and reach a consensus vocabulary. The references included a range of smoked foods (smoked haddock, smoked mackerel, kippers, and smoked cheese) as well as smoky bacon snacks, burnt wood, burnt paper, and burnt matches. The final vocabulary consisted of 5 aroma terms, 18 taste/flavor terms, 2 mouthfeel terms, and 1 after-effect term. The quantitative sensory assessment took place in individual sensory booths (under red light) at  $22 \pm 0.5$  °C. Assessors were provided with a glass of warm water and unsalted crackers (Carr's of Carlisle, Carlisle, U.K.) for palate cleansing between samples. Samples (~2–3 g) were presented to the assessors on a plastic teaspoon in a balanced order and randomly allocated. The assessors were asked to smell, taste, and swallow the samples and score them on appearance, odor, taste, flavor, and mouthfeel attributes. After a 45-s pause, they scored the samples for after-effects. The intensity of each attribute was recorded on a 150 mm unstructured line scale (scaled 0–100) and all data were collected using Compusense @Hand (Compusense Inc., Guelph, Ontario, Canada). A duplicate assessment was carried out in a separate session.

### **Consumer Preference of Cream Cheese with Added Smoked Tomato Flakes**

Consumer testing was carried out as described by IFT-SED<sup>13</sup> in individual sensory booths. A total of 115 naïve consumers (70% women aged 19–63; mean age of 32) carried out a paired preference test. They were served two samples of full-fat cream cheese; approximately 2–3 g of cheese was placed on the tip of a plastic teaspoon. One cheese sample contained 2.5% Trad smoked tomato flakes and the other contained 2.5% PST smoked tomato flakes as described for sensory profiling purposes. The consumers tasted the two samples in a balanced and randomly allocated order and were asked to select their preferred sample.

### **Quantitation of Volatiles in Smoked Tomato Paste**

The aroma compounds generated during the smoking process were quantified in tomato paste using solid phase microextraction (SPME) followed by GC-MS analysis. External calibration curves were prepared with unsmoked tomato paste containing a cocktail of standards at appropriate concentrations. Single standards were prepared in methanol (or acetone for compounds in cocktail C) to form stock solutions (200 mg/L), from which four standard cocktails were prepared in high-performance liquid chromatography (HPLC) water containing analytes at 10 mg/L unless otherwise indicated. Cocktail A contained a mixture of guaiacol, 4-methylguaiacol, 4-ethylguaiacol, syringol (30 mg/L), and cyclotene (30 mg/L). Cocktail B contained a mixture of phenol, 2-methylphenol, 4-methylphenol, 2,6-dimethylphenol, eugenol, and (*E*)-isoeugenol. Cocktail C contained a mixture of



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furfural, 5-methylfurfural, and 2-acetylfuran. Cocktail D contained a mixture of 4-vinylguaiacol (5 mg/L) and whiskey lactone (5 mg/L). Five serial dilutions (1:1) of these cocktails were prepared with HPLC grade water. Samples for calibration were made up of  $1.00 \pm 0.01$  g of tomato paste, 1.0 mL of cocktail, and 1  $\mu$ L of thymol (internal standard of 5 g/L). All vials were mixed using a Velp F202A0175 Wizard Vortex Mixer at 3000 rpm for 30 s and analyzed in triplicate by SPME at each dilution. For the smoked tomato paste samples (Trad and PST),  $1.00 \pm 0.01$  g was diluted with 1 mL of water; 1  $\mu$ L of internal standard was added and the samples were analyzed in triplicate under the same conditions as the standards.

SPME GC-MS was carried out using a DVB/Carboxen/PDMS Stableflex fiber (11mm, SupelCo, Poole, U.K.) and GC-MS was performed on an Agilent 7890-5975C GC-MS equipped with a Zebron ZB-5MSi column (30 m  $\times$  0.25 mm i.d.  $\times$  1  $\mu$ m film thickness). Samples were equilibrated at 40  $^{\circ}$ C for 10 min with intermittent stirring prior to exposing the fiber for 10 min at 40  $^{\circ}$ C. The fiber was desorbed in the injection port for 20 min and the volatile compounds were analyzed. Helium was the carrier gas at 1.2 mL/min. After desorption, the oven was maintained at 40  $^{\circ}$ C for 5 min, then raised to 250  $^{\circ}$ C at 4  $^{\circ}$ C/ min. Mass spectra were recorded in electron ionization mode at 70 eV and at a source temperature of 230  $^{\circ}$ C. A scan range of  $m/z$  29–400 with a scan time of 0.69 s was used and the data were controlled and stored by the ChemStation system.

Good linearity was observed for all compounds except 4-vinylguaiacol and whisky lactone which were close to the limit of detection for the method, and their calibrations were based on fewer points ( $R^2$  for these were 0.61 and 0.84, respectively). Otherwise,  $R^2$  was always greater than 0.9 and generally greater than 0.95. For (*Z*)-isoeugenol, the calibration curve for (*E*)-isoeugenol was used. For the unresolved 3- and 4-methylphenols, the calibration curve for 4-methylphenol was used.

### Statistical Analysis

The data for the volatile analyses were analyzed with XLStat (AddinSoft, Paris, France, 2015.6.01) using one-way analysis of variance (ANOVA), and post-hoc multiple pairwise comparisons were carried out using the Fisher's least significant difference (LSD) test with the significance level set at  $p = 0.05$ . Two-way ANOVA was used to determine the significance of fat type and smoke technology. For the sensory data, SENPAQ version 3.2 (Qi Statistics, Reading, U.K.) was used to carry out the two-way ANOVA where main effects were tested against the sample by using assessor interaction. Multiple pairwise comparisons were done using the Fisher's LSD at  $p = 0.05$ . Principal component analysis (PCA) was carried out on the sensory data in XLStat with the volatile data used

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9 as supplementary data. Results from the paired preference test were evaluated  
10 using the binomial model in V-Power (Jesionka; macro for Microsoft Excel).  
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## 12 13 **Results**

### 14 15 16 17 **Flavor Changes in Smoked Tomato Flakes Added to Cream Cheese**

#### 18 19 20 21 *Volatile Analysis*

22  
23 Twenty compounds were selected for comparison between samples based on  
24 published GC–O data,<sup>8</sup> their abundance, and their relevance to cheese and smoke  
25 flavor (Table 1). Comparing just the peak areas of the Trad and the PST in full-  
26 fat cheese, all the smoke-derived compounds had smaller peak areas in PST  
27 compared to those in the Trad, 11 of these being significant at  $p < 0.05$  and the  
28 remainder at  $p < 0.1$ . These were all compounds that were observed in smoked  
29 oil<sup>12</sup> including furfurals (2), guaiacols (5), eugenols (3), 4-methylphenol, 2-  
30 acetylpyrrole, syringol, and cyclotene. In the low-fat cheese, the same trends were  
31 observed, except for 2-acetylpyrrole and (*Z*)-isoeugenol where the differences  
32 were either not significant or not consistent between the two cheeses. This  
33 decrease in smoke-derived compounds is similar to the decrease observed by  
34 Parker et al.<sup>12</sup> where sunflower oil was either Trad smoked or filtered through  
35 zeolite. However, in the smoked oils, furfural and 5-methylfurfural did not  
36 decrease when the filter was employed.  
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**Table 1. Relative Peak Areas of Selected Volatile Compounds Detected in the Headspace of Low-Fat or Full-Fat Cream Cheese Containing 2.5% w/w Trad or PST Ground Tomato Flakes.**

Compound	LRI DB5	LRI Wax	Full-Fat Cheese		Low-Fat Cheese		S <sup>a</sup>	S <sup>b</sup>
			Trad	PST	Trad	PST		
2,3-Butanedione	586	961	26 <sup>c</sup>	29	29	33	*	*
2-Heptanone	892	1167	3.3a	3.4a	2.5b	3.0a	ns	**
3-Hydroxybutanone	708	1283	75b	75b	80b	112a	*	**
6-Methyl-5-hepten-2-one	986	1330	9.6b	12b	12b	22a	***	***
Acetic acid	602	1458	25c	26c	35b	46a	**	***
2-Furfural	833	1462	5.0b	2.9c	6.2a	3.5c	***	*
5-Methylfurfural	965	1571	2.3a	0.9c	2.5a	1.3b	***	*
Benzenacetaldehyde	1048	1640	2.1a	2.5b	3.4a	3.9a	ns	***
Cyclotene	1030	1830	2.2a	0.3c	1.7b	0.3c	***	***
Guaiacol	1094	1859	18a	4.7c	19a	7.1b	***	*
4-Methylguaiacol	1198	1956	14a	3.2b	16a	6.1b	***	*
2-Acetylpyrrole	1061	1970	4.4a	3.0b	3.9a	4.3a	*	ns
4-Ethylguaiacol	1286	2029	9.4a	1.9b	11a	3.9b	***	*
4-Methylphenol	1072	2090	2.2a	0.6c	2.3a	0.9b	***	*
4-Propylguaiacol	1375	2108	1.9a	0.3b	1.9a	0.7b	***	ns
Eugenol	1365	2166	0.9	0.3	0.7	0.5	*	ns
4-Vinylguaiacol	1322	2195	0.3b	0.1b	1.5a	0.3b	**	**
(Z)-Isoeugenol	1417	2254	0.5	0.1	0.3	0.3	*	ns
Syringol	1357	2263	4.6a	1.1b	4.7a	2.3b	***	ns
(E)-Isoeugenol	1460	2348	1.2a	0.2b	1.3a	0.6b	***	ns

<sup>a</sup>Significance, obtained from ANOVA, that there is a difference between the Trad and PST samples where ns = no significant difference ( $p > 0.05$ ); \*sig is  $0.01 < p \leq 0.05$ ; \*\*sig is  $0.001 < p \leq 0.01$ ; and \*\*\*sig is  $p \leq 0.001$ .

<sup>b</sup>Significance, obtained from ANOVA, that there is a difference between the full-fat and the low-fat cheese samples.

<sup>c</sup>Mean peak areas  $\times 10^4$  AU ( $n=3$ ); means in the same row that are not labelled with the same letters are significantly different ( $p = 0.05$ ).

In the full-fat cheese, the smoking technology had no impact on the compounds that were already present in the unflavored cream cheese (2,3-butanedione, 2-heptanone, 3-hydroxybutanone, and acetic acid); however, in the low-fat cheese, there was a tendency for these compounds to be higher in the PST sample than the Trad sample. This was also the case for 6-methyl-5-heptene-2-one which is a carotenoid-derived compound found in the unsmoked tomato flakes.

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Although the primary aim of the experiment was not to compare flavor release in full-fat and low-fat cream cheese, the data for the PST cheese showed a significant increase in flavor release for nine compounds in the low-fat cheese compared to the full-fat cheese, with a similar trend for another nine. This is consistent with current understanding of the role of fat content with regard to flavor release. This has mainly been demonstrated in dairy yogurts<sup>14</sup> or ice cream,<sup>15</sup> but for hydrophobic aroma compounds with relatively high  $\log p$  values, it is well established that a decrease in fat content will promote partitioning into the headspace and increase flavor release. However, with the Trad cheese, this effect was greatly diminished, and only three compounds (acetic acid, furfural, and 4-vinylguaiacol) showed a significant increase in the low-fat cheese.

### Sensory Analysis

The sensory data showed that there were significant differences between the cheese samples for 12 of the 25 attributes (Table 2). One key difference was the significant reduction in the bitter taste when the PST was applied, which is perhaps a result of the filter holding back non-volatile bitter compounds in the tar fraction that does not pass through the filter. The compounds associated with smoke were consistently higher in Trad compared to PST, particularly the overall smoky aroma, flavor and aftertaste, the bonfire aroma and flavor, and the diesel flavor. This is consistent with the instrumental volatile data that showed a higher concentration of typical smoky aroma compounds in the Trad samples. On the other hand, the cheesy aroma was significantly higher in the PST samples. In full-fat cheese, the change in smoke technology did not significantly alter the concentration of the compounds associated with the cheese suggesting that these compounds were masked by the high levels of smoke compounds in the full-fat Trad product, which makes them more prominent in the full-fat PST sample. In low-fat cheese, the compounds associated with cheese did increase in the PST product, which is consistent with an increase in aroma in this product. Either or both of these mechanisms could explain the increase in cheesy aroma in the PST products.

When these data are viewed on a PC plot, the correlations are clear (Figure 1). All the smoky aroma compounds are positioned to the far right of PC1: an area associated with the low-fat Trad product which a) contains more smoky compounds than PST, and b) is based on the low-fat cheese which promotes the release of more smoky compounds. PC1 separates the Trad from the PST (with more cheesy and dairy notes associated with PST), which is consistent with less masking from the smoke volatiles. Interestingly, 2,3-butanedione and 3-hydroxybutanone which have buttery creamy and dairy notes were highly correlated with the sour yogurt note as was acetic acid, which contributes the

“sour” to this attribute. The sun-dried tomato flavor attribute is associated with 6-methyl-5-hepten-2-one, a carotenoid-derived compound found in the tomato flake.

**Table 2. Mean Panel Scores for Sensory Attributes Found in Low-Fat or Full-Fat Cream Cheese Containing 2.5% w/w Trad or PST Ground Tomato Flakes.**

<i>Sensory Attribute</i>	<i>Full-Fat Cheese</i>		<i>Low-Fat Cheese</i>		<i>Sig<sup>a</sup></i>
	<i>Trad</i>	<i>PST</i>	<i>Trad</i>	<i>PST</i>	
<i>Aroma</i>					
Overall smokiness	33a <sup>b</sup>	16b	41a	23b	***
Bonfire	25b	12c	33a	15c	***
Ash	4.4ab	1.3b	6.9a	2.0b	*
Cheesy	19bc	33a	17c	26ab	**
<i>Taste</i>					
Sweet	18	19	16	18	ns
Salty	19	18	21	20	ns
Umami	30	27	30	32	ns
Bitter	7.6ab	5.3b	9.7a	7.7b	*
Sour	13	15	13	15	ns
<i>Flavor</i>					
Overall smokiness	40a	20c	46a	30b	***
Bonfire	28a	13b	36a	20b	***
Paprika	14	12.8	15	12.3	ns
Ash	3.7b	1.7b	8.1a	2.2b	**
Smoked fish	19a	8.5b	20a	15a	**
Diesel	4.2a	0.6b	4.1a	0.8b	*
Sundried tomato	16	17	15	20	ns
Dairy	24	29	23	26	ns
Sour yogurt	13	13	13	16	ns
Spicy	13	10	16	13	ns
Smoked bacon	17	11	20	15	ns
Cheesy	27ab	32a	20b	28ab	*
Balanced	44	46	44	46	ns
<i>After-effects</i>					
Mouthcoating	34	30	31	31	ns
Warming	9.4	6.6	10	7.8	ns
Smoked food	26a	14b	30a	15b	***

<sup>a</sup>Significance, obtained from ANOVA, that there is a difference between the mean scores where ns = no significant difference ( $p > 0.05$ ); \*sig is  $0.01 < p \leq 0.05$ ; \*\*sig is  $0.001 < p \leq 0.01$ ; and \*\*\*sig is  $p \leq 0.001$ .

<sup>b</sup> Mean panel scores (n = 9 in duplicate); means in the same row not labelled with the same letters are significantly different ( $p = 0.05$ ).

Overall the sensory profiling showed a decrease in smoky notes and a decrease in bitterness when the PST was applied. As a result, more notes from the cheese and the tomato flakes were perceived by the panelists.

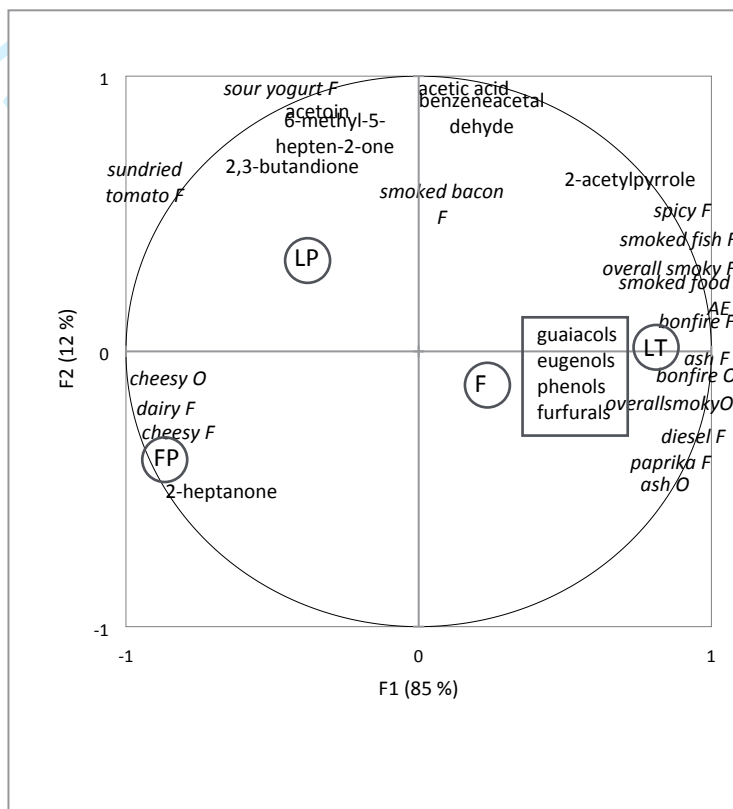


Figure 1. PC1 vs. PC2 of sensory attributes (*italics*) for cream cheese with added smoked tomato flakes (F=full-fat, L=low-fat, T=traditional process, P=PST) with volatile data overlaid.

### Consumer Analysis

Sensory profiling, however, is not hedonic, so we recruited a panel of 115 naïve volunteers to indicate which product they preferred when given a choice of full-fat Trad or full-fat PST. Of these volunteers, 65 preferred the PST product

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9 and 50 preferred the Trad (ratio of 57:43). This indicates that there is no adverse  
10 effect on flavor when the PST is applied and that there is tendency for the PST to  
11 be preferred to Trad at  $p = 0.096$ . Thus, the removal of PAHs from smoke using  
12 PST does not adversely affect the flavor and may also improve it. Other food  
13 ingredients are under test.  
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### 16 **Quantitative Comparison in Tomato Paste of Aroma Compounds** 17 **Generated during either Trad or PST** 18

19 However, questions remain as to why the reduction in some of the aroma  
20 compounds may improve the flavor and which of these compounds are the key  
21 contributors to the less desirable aroma attributes such as ash and diesel. With this  
22 in mind, we quantitated the aroma compounds in tomato paste that had been  
23 smoked using either Trad or PST. Seventeen compounds were selected based on  
24 the literature where GC–O was used to determine odor-active compounds in  
25 smoked foods.<sup>8</sup> Table 3 shows the concentration of each compound found in the  
26 smoked tomato paste. Furfural and 5-methylfurfural were by far the most  
27 abundant compounds in the smoked tomato paste, but not necessarily the most  
28 odor-active. In order to estimate the odor activity of these compounds, literature  
29 thresholds in water were employed, where possible, to calculate odor activity  
30 values (OAV, the concentrations in ug/kg divided by the odor thresholds in  
31 ug/kg). Of those compounds where thresholds were available, guaiacol had the  
32 highest odor activity, which is consistent with data taken from Varlet et al.<sup>8</sup>  
33 showing that guaiacol had the highest average intensity by GC–O in smoked  
34 salmon, despite very different matrices. According to Table 3, the next most odor-  
35 active compounds are likely to be eugenol and 4-methyl-, 4-ethyl-, and 4-  
36 vinylguaiacols; in the smoked salmon,<sup>8</sup> 4-methylphenol, 2-acetylfuran, and (*E*-  
37 isoeugenol were the next most intense. No odor thresholds in water were available  
38 for (*E*-isoeugenol, but 4-methylphenol and 2-acetylfuran were much less odor-  
39 active if water thresholds are used. The odor threshold for whiskey lactone was  
40 determined in a water-ethanol mix and is therefore likely to be overestimated  
41 since the ethanol is likely to reduce the partitioning into the headspace. This would  
42 result in an underestimation of the OAV. Schranz et al.<sup>16</sup> have recently reported  
43 thresholds in air for many of these compounds. Applying these thresholds to  
44 compare OAVs (with arbitrary units) for selected compounds shows that guaiacol  
45 is again the most odor-active followed by 4-ethylguaiacol, 4-methylguaiacol, and  
46 (*Z*-isoeugenol. There are clearly major limitations associated with using literature  
47 thresholds from different matrices for calculating OAVs. What is clear though is  
48 the fact that the guaiacols, and perhaps also the eugenols, are important  
49 contributors to the aroma of the smoked tomato pastes. It is less likely that the  
50 furans, the phenols, and the whiskey lactone contribute, but this requires a full  
51 sensomics analysis to confirm the role of these compounds in smoky flavor and  
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identify the key differences that make Trad and PST smoke smell slightly different.

**Table 3. Concentration (ug/kg) and Approximated OAV of Aroma Compounds in Trad or PST Tomato Paste**

Compound	LRI DB5	Threshold <sup>a</sup> ug/kg	Concentration <sup>b</sup> ug/kg		OAV		S <sup>c</sup>
			Trad	PST	Trad	PST	
Guaiacol	1095	3	750	400	250	133	***
Eugenol	1366	6	140	70	23	12	***
4-Methylguaiacol	1200	90	670	360	7.4	4.0	***
4-Ethylguaiacol	1286	50	230	110	4.6	2.2	***
4-Vinylguaiacol	1323	18	3.3	1.7	3.3	1.7	ns
2-Furfural	836	3000	3070	2460	1.0	0.8	***
Cyclotene	1028	300	220	110	0.7	0.4	ns
3/4-Methylphenol	1073	55	20	10	0.4	0.2	***
Syringol	1358	1850	380	240	0.2	0.1	ns
Phenol	978	5900	650	400	0.1	0.1	***
Whiskey lactone <sup>d</sup>	1298	790	40	20	0.05	0.03	***
2-Methylphenol	1053	650	30	10	0.05	0.02	***
2-Acetylfuran	913	10000	110	70	0.01	0.01	***
5-Methylfurfural	967	na	3720	2890			***
2,6-Dimethylphenol	1025	na	50	20			***
( <i>E</i> )-isoeugenol	1461	na	770	360			***
( <i>Z</i> )-isoeugenol	1418	na	380	170			***

<sup>a</sup>Threshold in water (ug/kg)<sup>17</sup>; na = not available.

<sup>b</sup>Mean concentration ug/kg (n=3).

<sup>c</sup>Probability, obtained from ANOVA, that there is a difference between the Trad and PST samples where ns = no significant difference ( $p > 0.05$ ); and \*\*\*significant at  $p \leq 0.001$ .

<sup>d</sup>Odor threshold in water-ethanol mixture (6:4 by vol).<sup>18</sup>

### Overall Conclusion

A new technology (PST) has been developed to remove carcinogenic PAHs from smoke streams used for smoking food ingredients. The aim of this chapter was to determine whether the PST had a detrimental effect on flavor. Tomato flakes were smoked using either Trad or PST, and the smoked tomato flakes were presented in either full-fat or low-fat cream cheese. Using sensory profiling, we demonstrated that in both cases there was a small but significant reduction in



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bitterness when PST was used to filter the smoke prior to smoking the tomato flakes. There was also a significant reduction in the smoky aroma and flavor attributes, and this was confirmed using instrumental analysis. However, when the two full-fat products were compared in a consumer preference test (n = 115), there was no clear preference for either product. Thus, we conclude that when PST is employed to reduce the concentration of carcinogenic PAHs in products such as smoked tomato flakes (or other spices), it also can reduce some of the bitterness associated with the smoking process. There is a minor impact on the aroma profile, but this did not have an impact on consumer preference.

## References

1. Guillen, M. D.; Manzanos, M. J. Characteristics of Smoke Flavorings Obtained from Mixtures of Oak *Quercus sp.* Wood and Aromatic Plants *Thymus vulgaris* L. and *Salvia lavandulifolia* Vahl. *Flavour Fragrance J.* **2005**, *20*, 676–685.
2. Guillen, M. D.; Errecalde, M. C. Volatile Components of Raw and Smoked Black Bream *Brama raii* and Rainbow Trout *Oncorhynchus mykiss* Studied by Means of Solid Phase Microextraction and Gas Chromatography/Mass Spectrometry. *J. Sci. Food Agric.* **2002**, *82*, 945–952.
3. Guillen, M. D.; Errecalde, M. C.; Salmeron, J.; Casas, C. Headspace Volatile Components of Smoked Swordfish *Xiphias gladius* and Cod *Gadus morhua* Detected by Means of Solid Phase Microextraction and Gas Chromatography-Mass Spectrometry. *Food Chem.* **2005**, *94*, 151–156.
4. Guillen, M. D.; Ibargoitia, M. L.; Sopelana, P.; Palencia, G.; Fresno, M. Components Detected by Means of Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in the Headspace of Artisan Fresh Goat Cheese Smoked by Traditional Methods. *J. Dairy Sci.* **2004**, *87*, 284–299.
5. Guillen, M. D.; Palencia, G.; Ibargoitia, M. L.; Fresno, M.; Sopelana, P. Contamination of Cheese by Polycyclic Aromatic Hydrocarbons in Traditional Smoking. Influence of the Position in the Smokehouse on the Contamination Level of Smoked Cheese. *J. Dairy Sci.* **2011**, *94*, 1679–1690.
6. Majcher, M. A.; Jelen, H. H. Key Odorants of Oscypek, a Traditional Polish Ewe's Milk Cheese. *J. Agric. Food Chem.* **2011**, *59*, 4932–4937.
7. Varlet, V.; Knockaert, C.; Prost, C.; Serot, T. Comparison of Odor-Active Volatile Compounds of Fresh and Smoked Salmon. *J. Agric. Food Chem.* **2006**, *54*, 3391–3401.
8. Varlet, V.; Serot, T.; Cardinal, M.; Knockaert, C.; Prost, C. Olfactometric Determination of the Most Potent Odor-Active Compounds in Salmon Muscle *Salmo salar* Smoked by Using Four Smoke Generation Techniques. *J. Agric. Food Chem.* **2007**, *55*, 4518–4525.

9. Alonge, D. O. Carcinogenic Polycyclic Aromatic Hydrocarbons (PAH) Determined in Nigerian Kundi (Smoke-Dried Meat). *J. Sci. Food Agric.* **1988**, *43*, 167–72.
10. Committee on Diet, Nutrition and Cancer, Assembly of Life Sciences, National Research Council. *Diet, Nutrition and Cancer*; National Academy Press: Washington, DC, 1982.
11. Griffiths, H. D.; Baines, D. A.; Parker-Gray, J. K. Smoked Food, Method for Smoking Food and Apparatus Therefor. WO 2015007742 A1, Jan 22, 2015.
12. Parker, J. K.; Lignou, S.; Shankland, K.; Kurwie, P.; Griffiths, H. D.; Baines, D. A. Development of a Zeolite Filter for Removing Polycyclic Aromatic Hydrocarbons (PAHs) from Smoke and Smoked Ingredients While Retaining the Smoky Flavor. *J. Agric. Food Chem.* **2018**, *66*, 2449–2458.
13. Institute of Food Technologists. Sensory Evaluation Division Sensory Evaluation Guide for Testing Food and Beverage Products. *Food Technol.* **1981**, 50–59.
14. Brauss, M. S.; Linforth, R. S. T.; Cayeux, I.; Harvey, B.; Taylor, A. J. Altering the Fat Content Affects Flavor Release in a Model Yogurt System. *J. Agric. Food Chem.* **1999**, *47*, 2055–2059.
15. Ayed, C.; Martins, S. I. F. S.; Williamson, A.-M.; Guichard, E. Understanding Fat, Proteins and Saliva Impact on Aroma Release from Flavoured Ice Creams. *Food Chem.* **2018**, *267*, 132–139.
16. Schranz, M.; Lorber, K.; Klos, K.; Kerschbaumer, J.; Buettner, A. Influence of the Chemical Structure on the Odor Qualities and Odor Thresholds of Guaiacol-Derived Odorants, Part 1: Alkylated, Alkenylated and Methoxylated Derivatives. *Food Chem.* **2017**, *232*, 808–819.
17. Leffingwell, J. C.; Leffingwell, D. GRAS flavor Chemicals—Detection Thresholds. *Perfum. & Flavor.* **1991** *16*, 1–19.
18. Poisson, L.; Schieberle, P. Characterization of the Key Aroma Compounds in an American Bourbon Whisky by Quantitative Measurements, Aroma Recombination, and Omission Studies. *J. Agric. Food Chem.* **2008**, *56*, 5820–5826.