



# Ageing and retail display time in raw beef odour according to the degree of lipid oxidation



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## ABSTRACT

This study aims to assess the changes in beef aroma over time when steaks from pre-aged knuckles are stored in retail display under high oxygen conditions for 15 or 22 days in vacuum conditions. Odorous volatile compounds were analysed by solid-phase microextraction/gas chromatography-mass spectrometry. Results were grouped as *low*, *medium* and *high* oxidative groups according to thiobarbituric acid reactive substances values after 9 days of display. The intensity of off-odours in the raw meat increased with ageing and display time and oxidative groups. Based on correlations between the variables and regressions of the compounds through display, eight compounds were proposed as odour shelf-life markers. Among them, five were most sensitive and precise in all oxidative groups: 1-hexanol in meat aged for 15 days, ethyl- octanoate and 2-pentylfuran in meat aged for 22 days, and pentanoic and hexanoic acids in the two studied ageing times.

## 1. Introduction

Beef is usually aged for 7–21 days, in order to improve tenderness and homogeneity, the optimum period being dependent on several factors, including the muscle type and the breed under consideration (Monsón, Sañudo, & Sierra, 2005; Thompson, 2002). In addition, ageing could affect other quality parameters, such as odour, taste and flavour in the cooked meat, due to changes in the concentrations of precursors (Koutsidis et al., 2008; Meinert et al., 2009), and also the development of odorous compounds in the raw product coming from chemical and enzymatic reactions and bacterial action (Casaburi, Piombino, Nychas, Villani, & Ercolini, 2015; Estévez, Morcuende, Ventanas, & Cava, 2003; Insausti, Beriain, Gorraiz, & Purroy, 2002). The mentioned processes occur even when ageing is conducted under refrigeration, vacuum and dark conditions. Nevertheless, after cooking the meat could reach high flavour acceptability scores (Colle et al., 2015; Pérez-Juan et al., 2014; Smith et al., 2008). In fact, ageing up to 21 days improves the flavour in some beef breeds (Monsón et al., 2005).

Commonly, after ageing the meat is packed and preserved in retail display for some days until purchase. It has been shown that steaks displayed without oxygen improve their eating quality (Aaslyng, Tørrngren, & Madsen, 2010), but at commercial level steaks are still

mostly sold in oxygen-permeable overwrap or in packs with high oxygen levels due to colour issues. Previous studies found unacceptable rancid flavour developed in beef *longissimus dorsi* displayed for around nine days in high oxygen packs (Campo et al., 2006; Resconi et al., 2012), but shorter or extended periods of odour shelf-life could occur, depending on numerous factors, such as the antioxidant status of the meat. In a study with lambs, for example, dietary supplementation of vitamin E led to lower levels of lipid oxidation derived compounds, such as 2-heptanone and 1-penten-3-ol in the cooked meat (Rivas-Cañedo et al., 2013), which may affect the odour perception of the meat.

The effect of time on the beef aroma development, when ageing the meat or when stored in retail display under high oxygen conditions, has been previously studied using cooked samples (Campo et al., 2006; Gorraiz, Beriain, Chasco, & Insausti, 2002; Ma, Hamid, Bekhit, Robertson, & Law, 2012; O'Quinn et al., 2016; Resconi et al., 2012; Stetzer, Cadwallader, Singh, McKeith, & Brewer, 2008; Watanabe et al., 2015), but less has been published regarding raw meat (Insausti et al., 2002; La Stora et al., 2012; Saraiva et al., 2015). Most studies have used cooked meat matching the usual way of consumption, but also because a richer variety and intensity of aromas is developed (Resconi, Escudero, & Campo, 2013). The odour of the raw meat in the store while buying is becoming less important, since the

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**Table 1**

F-value and significance of ageing (15 or 22 days), display time (0, 5, 7 or 9 d) and oxidative group<sup>1</sup> (low, medium or high) effects and their interactions in volatile compounds analysed by SPME-GCMS, odour intensity and TBARS value from raw knuckle steaks.

	LRI	Quan ion	Ageing	Display	Oxidative Group	A × D	A × OG	D × OG	A × D × OG
<b>Acids</b>									
<i>Butanoic acid</i>	1632	60	ns	5.84 <sup>***</sup>	ns	5.29 <sup>***</sup>	3.47 <sup>*</sup>	3.75 <sup>***</sup>	ns
<i>Pentanoic acid</i>	1744	60	34.3 <sup>***</sup>	124 <sup>***</sup>	45.2 <sup>***</sup>	5.27 <sup>***</sup>	ns	4.15 <sup>***</sup>	2.93 <sup>**</sup>
<i>Hexanoic acid</i>	1851	60	4.34 <sup>*</sup>	155 <sup>***</sup>	35.0 <sup>***</sup>	ns	4.39 <sup>*</sup>	7.02 <sup>***</sup>	3.42 <sup>**</sup>
<i>Heptanoic acid</i>	1975	60	6.46 <sup>*</sup>	85.7 <sup>***</sup>	7.76 <sup>***</sup>	5.84 <sup>***</sup>	3.69 <sup>*</sup>	2.17 <sup>*</sup>	3.16 <sup>**</sup>
<i>Octanoic acid</i>	2051	60	ns	11.2 <sup>***</sup>	ns	11.4 <sup>***</sup>	ns	ns	2.51 <sup>*</sup>
<i>Nonanoic acid</i>	2116	60	17.0 <sup>***</sup>	4.87 <sup>**</sup>	ns	4.62 <sup>**</sup>	ns	ns	3.16 <sup>**</sup>
<i>Decanoic acid</i>	2200	60	28.0 <sup>***</sup>	ns	ns	ns	3.88 <sup>*</sup>	ns	ns
<b>Alcohols</b>									
<i>1-Hexanol</i>	1359	69	10.9 <sup>***</sup>	84.1 <sup>***</sup>	81.8 <sup>***</sup>	ns	3.45 <sup>*</sup>	8.35 <sup>***</sup>	3.08 <sup>**</sup>
<i>1-Octen-3-ol</i>	1449	57	ns	15.1 <sup>***</sup>	5.13 <sup>**</sup>	15.0 <sup>***</sup>	ns	ns	3.72 <sup>***</sup>
<b>Aldehydes</b>									
<i>Hexanal</i>	1052	57	ns	ns	ns	4.74 <sup>**</sup>	ns	ns	ns
<i>Heptanal</i>	1188	71	ns	6.85 <sup>***</sup>	6.31 <sup>**</sup>	ns	9.26 <sup>***</sup>	3.03 <sup>**</sup>	ns
<i>Nonanal</i>	1402	98 + 120	ns	2.98 <sup>*</sup>	ns	ns	ns	ns	ns
<i>(E)-2-Heptenal</i>	1344	97	9.98 <sup>**</sup>	10.3 <sup>***</sup>	7.71 <sup>***</sup>	ns	ns	ns	2.44 <sup>*</sup>
<i>(Z)-2-Octenal</i>	1420	83	4.37 <sup>*</sup>	21.8 <sup>***</sup>	13.9 <sup>***</sup>	ns	ns	ns	2.14 <sup>*</sup>
<i>(E)-2-Nonenal</i>	1562	93	9.64 <sup>**</sup>	10.6 <sup>***</sup>	7.06 <sup>***</sup>	2.80 <sup>*</sup>	ns	ns	2.94 <sup>**</sup>
<i>(E,E)-2,4-Nonadienal</i>	1741	81	7.11 <sup>**</sup>	3.50 <sup>*</sup>	4.02 <sup>*</sup>	ns	ns	ns	ns
<i>(E)-2-Undecenal</i>	1781	83	ns	49.5 <sup>***</sup>	45.8 <sup>***</sup>	ns	4.10 <sup>*</sup>	3.91 <sup>***</sup>	ns
<i>(E,Z)-2,4-Decadienal</i>	1799	81	ns	15.8 <sup>***</sup>	ns	ns	ns	ns	2.58 <sup>*</sup>
<i>(E,E)-2,4-Decadienal</i>	1854	81	5.37 <sup>*</sup>	21.1 <sup>***</sup>	10.4 <sup>***</sup>	ns	ns	ns	2.61 <sup>*</sup>
<i>Phenylacetaldehyde</i>	1681	91 + 120	3.88 <sup>*</sup>	3.36 <sup>*</sup>	ns	ns	ns	ns	ns
<b>Esters</b>									
<i>Ethyl hexanoate</i>	1236	88	ns	44.5 <sup>***</sup>	15.0 <sup>***</sup>	3.44 <sup>*</sup>	9.68 <sup>***</sup>	2.72 <sup>*</sup>	6.37 <sup>***</sup>
<i>Ethyl octanoate</i>	1438	88	ns	115 <sup>***</sup>	36.5 <sup>***</sup>	ns	23.6 <sup>***</sup>	2.17 <sup>*</sup>	2.62 <sup>*</sup>
<b>Ketones</b>									
<i>2-Butanone</i>	668	72	ns	3.51 <sup>*</sup>	ns	ns	ns	ns	ns
<i>2-Heptanone</i>	1192	99	3.93 <sup>*</sup>	58.4 <sup>***</sup>	10.4 <sup>***</sup>	ns	ns	ns	ns
<i>3-Octanone</i>	1265	99	ns	20.3 <sup>***</sup>	3.30 <sup>*</sup>	ns	6.76 <sup>***</sup>	2.96 <sup>**</sup>	ns
<i>2-Octanone</i>	1293	58	ns	35.8 <sup>***</sup>	5.55 <sup>**</sup>	ns	7.63 <sup>***</sup>	ns	ns
<i>2-Nonanone</i>	1400	142	ns	16.3 <sup>***</sup>	ns	3.49 <sup>*</sup>	3.28 <sup>*</sup>	ns	2.88 <sup>**</sup>
<i>1-Octen-3-one</i>	1311	70	ns	20.7 <sup>***</sup>	7.85 <sup>***</sup>	4.59 <sup>**</sup>	ns	ns	2.52 <sup>*</sup>
<i>3-Octen-2-one</i>	1427	111	ns	8.09 <sup>***</sup>	3.06 <sup>*</sup>	2.96 <sup>*</sup>	ns	ns	ns
<i>3-Nonen-2-one</i>	1535	125	ns	4.77 <sup>**</sup>	ns	ns	ns	ns	ns
<i>2,3-Octanedione</i>	1332	99	ns	5.33 <sup>***</sup>	ns	5.62 <sup>***</sup>	ns	ns	ns
<b>Others</b>									
<i>2-Pentylfuran</i>	1235	138	11.0 <sup>**</sup>	38.7 <sup>***</sup>	33.5 <sup>***</sup>	ns	ns	4.32 <sup>***</sup>	ns
<b>Odour intensity</b>									
<i>Raw meat odour</i>			84.7 <sup>***</sup>	57.9 <sup>***</sup>	22.9 <sup>***</sup>	3.24 <sup>*</sup>	5.02 <sup>**</sup>	ns	3.19 <sup>**</sup>
<i>Rancid odour</i>			54.4 <sup>***</sup>	184 <sup>***</sup>	70.4 <sup>***</sup>	ns	ns	5.29 <sup>***</sup>	ns
<i>Metallic odour</i>			ns	17.8 <sup>***</sup>	4.60 <sup>*</sup>	3.18 <sup>*</sup>	ns	ns	2.96 <sup>*</sup>
<i>Sweet odour</i>			23.1 <sup>***</sup>	14.4 <sup>***</sup>	ns	2.87 <sup>*</sup>	ns	ns	2.54 <sup>*</sup>
<i>Musty odour</i>			ns	39.2 <sup>***</sup>	14.0 <sup>***</sup>	12.6 <sup>***</sup>	13.4 <sup>***</sup>	ns	3.54 <sup>**</sup>
<i>Cooked meat odour</i>			4.56 <sup>*</sup>	51.1 <sup>***</sup>	ns	6.53 <sup>***</sup>	3.77 <sup>*</sup>	3.31 <sup>**</sup>	2.13 <sup>*</sup>
<i>Sulfurous odour</i>			11.6 <sup>***</sup>	18.8 <sup>***</sup>	7.37 <sup>***</sup>	ns	3.66 <sup>*</sup>	ns	2.62 <sup>*</sup>
<b>TBARS value</b>			20.5 <sup>***</sup>	210 <sup>***</sup>	146 <sup>***</sup>	4.46 <sup>**</sup>	ns	20.5 <sup>***</sup>	2.23 <sup>*</sup>

<sup>1</sup> Low, medium and high oxidative groups reached < 1, 1–2 or > 2 mg MDA/kg muscle (TBARS values), respectively, on Day 9 of display in samples aged 15 days. LRI: linear retention index calculated for DB-WAXETR capillary column. A: ageing. D: display. OG: oxidative group. Quan ion: *m/z* used for quantification. ns:  $p > 0.05$ , \*:  $p \leq 0.05$ , \*\*:  $p \leq 0.01$ , \*\*\*  $p \leq 0.001$ .

meat is usually sold packaged. However, when opening the package at home, the odour should be agreeable for the consumer (Schindler, Krings, Berger, & Orlin, 2010). Furthermore, some compounds already present or developed in the raw meat, remain after cooking and could affect the flavour perception (Insausti et al., 2002; Rota & Schieberle, 2005; Schindler et al., 2010).

The aim of this study was to evaluate the effect of display time on the odour characteristics and the volatile compounds involved in raw knuckle beef steaks pre-aged under vacuum conditions for 15 or 22 days, according to the extent of lipid oxidation. Furthermore, it also aimed to identify shelf-life markers from the volatile compounds analysed. It is already known that lipid oxidation affects odour development; therefore samples were grouped by their oxidative potential, since different compounds may affect aroma properties throughout display.

## 2. Materials and methods

### 2.1. Samples, ageing and display conditions

This study used 48 crossbred entire young bulls 12.8–13.9 months with a cold carcass weight of 231–340 kg and an intramuscular fat content in *longissimus thoracis* muscle of 1.5–2.5%. The animals were raised on the same farm and fed concentrates (based on maize, barley and soya) and cereal straw *ad libitum*. After slaughtering in a commercial abattoir following standard procedures meeting welfare regulations, both knuckles from each animal were obtained, vacuum packaged and aged for either 15 days (left side) or 22 days (right side), in the dark at  $3 \pm 1$  °C. Then, 0.6-cm thick steaks were obtained and randomly allocated for each display time. Samples from each animal

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