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The profile of volatile compounds in the outer and inner parts of broiled pork neck is strongly influenced by the acetic-acid marination conditions



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ABSTRACT

Raw pork neck cutlets were marinated in an aqueous solution of acetic acid (pH 4, 24 h, 4 °C) without (M) or with 1% (w/w) of glucose. The control (K) was formed by non-treated raw pork neck. The cutlets were then broiled (185 °C, 30 min). In all K cutlets, significant higher amounts of volatile compounds (VCs) were developed after broiling than the other samples. Significant more aldehydes and alcohols were present in the inner parts than in the surface. The correlation between surface and internal layers was high only for aldehydes, Marinating decreased the differences among VCs and led to the standardization of the processed meat. The addition of glucose to the marinade led to more volatile aldehydes, carboxylic acids, esters, furan, pyran, pyrazine, pyrrol and pyridine derivatives than in M samples. Several (53) specific VCs explained the differences among the surface samples related to the marinating process. However, only 16 VCs explained the variance among the inner parts.

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1. Introduction

The heating of pork meat causes modifications of several food components, including the lipid fraction. Chain reactions, such as the nonenzymatic browning process (Maillard reaction, Amadori rearrangement and Strecker degradation) can lead to the formation of specific profiles of volatile compounds (Song et al., 2011; Yaylayan, 1999; Yu & Zhang, 2010). The extent of these reactions depends on the characteristics of the matrix — the raw meat tissue (Lu, Li, Yin, Zhang, & Wang, 2008; Meinert, Schäfer, Bjergegaard, Aaslyng, & Bredie, 2009; Meinert, Tikk, et al., 2009) and the methods of preheating and heating treatments

Corresponding author. E-mail address: e.boselli@univpm.it (E. Boselli). (Calkins & Hodgen, 2007; Rivas-Cañedo, Juez-Ojeda, Nuñez, & Fernández-García, 2011). The types and the concentration of the compounds formed ex novo affect both the acceptance of the products (Moon, Cliff, & Li-Chan, 2006; Tikk, Haugen, Andersen, & Aaslyng, 2008) and the food safety aspects (Jing & Kitts, 2004; Litchfield, Thorpe, & Baynes, 1999). During the heat treatment of raw meat by using frying, baking, grilling and/or the broiling processes, unpredictable profiles of volatile compounds can be formed because the thermal shock (Mottram, 1998) occurs much more intensively on the surface than in the internal layers with respect to boiling or stewing. Therefore, a complex distribution of ex novo formed compounds will be obtained in the different parts of meat. These compounds are the products of the amino acids reactions, fat and phospholipids degradation, as well as the Maillard reaction.

It is well documented that the pH significantly influences the Maillard reaction (Ames, 1998), so the simplest and low-cost method to stabilize the pH of the meat before the heat treatment could be marinating in a solution of an organic acid (Birk et al., 2010). Data are lacking in the literature regarding the effects of a meat marinade in an acetic acid solution on the profile of volatile compounds and non-enzymatic browning of the product. Similarly, there is a lack of investigations analyzing the differences in the formation of volatile compounds in the superficial and internal layers of the formulated meat products, although the differences seem to be obvious. The differences in the profile of volatile compounds between external and internal parts of the marinated and cured loin was investigated previously (Muriel, Antequera, Martín, & Ruiz, 2004), but the authors did not take in account the heated meat.

The advantages of marinating, such as the lowering of the pH of meat tissues, might slow the thermal reactions and thus the development of the flavour. In fact, the higher pH of the matrix, the higher amount of pyrazine and pyridine derivatives is formed in the products (Meynier & Mottram, 1995; Müller & Rappert, 2010). In addition, pyrazine and pyridine derivatives show a low sensory threshold (Meynier & Mottram, 1995; Shipar, 2006). Another important group of compounds of the meat flavour are the furanic compounds, whose formation is pH-dependent too. The addition of sugar to acetic acid in the marinade could improve the profile of the volatile compounds and extend the presence of sensorially desirable compounds. The influence of sugars on the formation of the flavour of meat marinated in acidic solution is not well known. The addition of sugar can cause osmotic dehydration as well as dewatering of the food matrix leading to a decrease of the net mass of the product (Aguilera & Stanley, 1999).

The objective of this study was to investigate the effects of two different marinating solutions (an acetic acid solution with or without addition of glucose) used before broiling on the volatile compounds of pork neck. Broiling was chosen because it does not need any addition of fat, that could influence the types of formed volatile compounds. The profile and concentration of the main groups of volatile compounds and individual components on both the surface and the internal layers were determined. The correlation between the amount of each group of volatile compounds formed on the surface and internal parts of meat was discussed.

2. Materials and methods

2.1. Pork neck samples

The pork neck was obtained from a meat processing plant the day after the slaughter. After trimming the visible external fat, the meat was chopped into cutlets of about 1.5 cm of thickness. The chops were divided into three different groups. One aliquot was subjected to heat treatment without additional preparation (control sample, K). The second group of the cutlets was marinated into a solution obtained from deionized water and edible acetic acid (initial concentration accounted 10% v/v) (M samples). The final pH of the marinade was 4. The third portion of cutlets was immersed in the same solution added with glucose (1% w/w of the total mass of marinade) (Mg samples). The cutlets were immersed in the solution in a glass container which was tightly closed. All the samples were kept for 24 h at 4 °C until broiling. About ten cutlets out of each group were prepared. The cutlets were broiled inside the chamber of a gastronomic roaster Philips HD4454/A. The roaster was preheated to 185 \pm 5 °C. At this temperature, the cutlets were broiled for 30 min (15 min for each side), to reach a final temperature of 70 °C in the internal parts. The temperature during the process was controlled by a thermocouple. No additional fat was used. The ready products were subjected to further analysis, after cooling to room temperature.

For each pork neck sample, two subsamples were obtained. An outside layer (s, surface sample), with a thickness of about 1–1.5 mm, was

shorn from the cutlets by using a scalpel. The remaining part of the cutlet was the internal part (i, internal part). Each sample was minced with a grinder prior to the analysis.

2.2. Analysis of volatile compounds

The samples of ground meat were prepared in triplicates and weighed (0.5 g) in vials. An internal standard (IS) (1,2-dichlorobenzene, 99%, Fluka) was added to each sample (1.306 µg) as a methanol solution. The meat samples were kept thermostatically for 20 min prior to the adsorption of volatile compounds. Next, the SPME (solid phase microextraction) fiber (75 µm CAR/PDMS carboxen/polydimethylsiloxane) was used for head space analysis with a manual holder. The adsorption lasted 30 min at a temperature of 60 °C. Afterwards, the fiber was injected into GC. A GC/MS chromatograph (mod. GCMS-QP 2010, Shimadzu) was equipped with a ZB-WAX Plus column $(30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \text{ }\mu\text{m})$. The injector temperature was 200 °C; the oven temperature increased from 40 to 220 °C at a rate of 4 °C/ min. The helium pressure was 35.2 kPa with a flow rate of 22.8 ml/ min. The temperature of the ion source of the MS was 170 °C. The identification of the compounds was performed by comparing each mass fragmentation spectrum with the NIST 147, PAL 600 and NIST 02 libraries. The linear retention indexes (LRI) were calculated on the basis of the analysis of standard hydrocarbons C₈-C₂₀ (Fluka) and confirmed with previously published database (El-Sayed, 2015; Mottram, 2015) and publications (Boulanger & Crouzet, 2001; Kocsis, Amtmann, Mednyánszky, & Korány, 2002). The amount of volatile compounds was calculated by dividing the total area of each peak by the area of the IS, to obtain the relative "index of amount" or "concentration". The final amount of the IS was assumed to be 1000.

2.3. Statistical analysis

The data were elaborated by using the Statistica software (ver. 10.0). The coefficient of determination (R²) was calculated and the corresponding ranges of relations were considered: 0 < R² < 0.1 slight, $0.1 \le R² < 0.3$ weak, $0.3 \le R² < 0.5$ mean, $0.5 \le R² < 0.7$ high, $0.7 \le R² < 0.9$ very high and $0.9 \le R² < 1$ complete correlation. The analysis of variance (ANOVA) was used to calculate statistical significance (95% confidence level) between samples. The Factor Analysis was used to calculate significant relations between variables and samples, and exclude not significant variables, and Principal Components Analysis (PCA) was used to project and classify significant variables and to point out groupings and differences among the samples.

3. Results

3.1. The pH of raw samples before broiling

The pH of both solutions used for the marinates reached 4. The pH of the samples decreased from 5.77 in control samples, to 5.52 and 5.20, in the M and Mg samples, respectively. The immersion of the meat in the solution of acetic acid with glucose led to the migration of water out of the cutlets ('dewatering' according to Aguilera & Stanley, 1999).

3.2. Profile of volatile compounds in the surface and internal layers of broiled pork neck

The groups of volatile compounds found in the heat-treated cutlet were aldehydes, ketones, alcohols, carboxylic acids, esters, furan and pyran derivatives (F&P), pyrazine, pyrrole and pyridine derivatives (P&P&P), benzene derivatives and others (Table 1).

In all cases, significantly more aldehydes and alcohols were found in the internal part than in surface layers (p < 0.05). Adversely, ketones, carboxylic acids, esters, F&P, P&P&P, benzene derivatives and other compounds were identified in more amount in the s samples than in i

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