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THE COMPOSITION OF VOLATILE COMPOUNDS, PARAMETERS OF COLOUR AND SENSORY QUALITY ATTRIBUTES OF ROASTED PORK NECK SHOULDER DERIVED FROM POLISH MEAT

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Abstract: The aim of the experiment was to investigate the composition of volatile compounds being formed while roasting pork shoulder derived from Polish meat, to measure the colour of the meat surface corresponding to the a specified profile of volatile compounds and to evaluate the sensory quality attributes of the meat. The major composition of volatile compounds determined by GC/MS method was hexanal and nonanal. The colour of the meat was determined in L*a*b* system, and the following average results were obtained: L* 53.56, a* 9.03, b* 18.98. These parameters corresponded with the colour of the surface which was determined by a sensory analysis as 4.17 in 10-degree scale. The specific surface colour of the meat was used for determining the profile of volatile compounds. The considerable content of hexanal and nonanal affected the sensory evaluation of the meat – taste and smell determined as fatty were evaluated to be rich in their intensiveness.

Key words: pork, meat, volatile compounds, colour measurement, roasting, GC/MS

Introduction

Pork is the most popular meat consumed in Poland by choice thanks to its attractive sensory quality attributes. Breaded and fried pork loin was the culinary piece of meat consumed in Poland most frequently until recently. There has been a tendency to prepare pork shoulder by means of equipment such as grill or griddle for years.

The attractiveness of meat as the foodstuff is related to the content of the compounds which are formed as a result of heating [1, 2]. The substrates which are necessary for them to be formed are water-soluble compounds: free sugars, sugar phosphates, nucleotide-bound sugars, free amino acids, peptides, nucleotides and other nitrogen compounds and lipids compounds – especially unsaturated fatty acids and phospholipids [3].

The main directions of changes which lead to the formation of meat flavour compounds is the thermal degradation of lipids and Maillard's reactions between reducing sugars and amino acids. The degradation of lipids leads to the formation of aliphatic hydrocarbons, aldehydes, ketones, alcohols, carboxylic acids and esters, as well as oxygenated heterocyclic compounds – lactones and alkylfurans [3].

Many various compounds (volatile and non-volatile) are formed as a result of Maillard's reactions, which impart specific attributes to the heated food. The mechanism of Maillard's reactions and directions of changes are thoroughly discussed in the literature [4–8]. Furfural and furanone derivatives, hydroxyketones and dicarbonyl compounds are formed at the early stage of the reactions. Furans, pyrazines, pyrroles, oxazoles, thiophenes, thiazoles and other heterocyclic compounds are formed at the following stage of the reactions due to many transformations [3].

The presence of heterocyclic compounds (pyrazines, pyrroles and oxazoles) makes meat products display the specific flavour defined as „roasted flavour". Sulphuric compounds have lower odour thresholds than compounds containing nitrogen. At the same time the presence of sulphuric compounds in meat is generally smaller than nitrogen derivatives, and they are usually formed in the cooking process. That is why the content structure of nitrogen containing compounds in relation to sulphuric compounds can indicate the conditions which were applied while heated.

The derivatives of phospholipids decomposition are responsible for the flavour defined as meaty.

The type of compounds, which are formed when meat is heated, is related to the raw material chemical composition [2]. The content of volatile compound precursors is connected with such factors as the way in which the animal is fed [9–11], post-mortem treatment, the animal's age and genetic features as well as its sex [12]. The same has been ascertained by Melton [1].

The following essential factor which decides on the type of compounds being formed is the method used for heating, mainly the duration and temperature of the process. The first effect to indicate the changes is the brown colour on the meat surface and the results of sensory evaluation – if the surface becomes too brown and as far as sensory quality attributes are concerned, the flavour of burning can be sensed – you can expect that potentially harmful compounds are present there [13–15]. They can be formed when too drastic conditions of the treatment were used. The composition and structure of the compounds being formed under different conditions are not completely known. So studies should be conducted to find out the interdependences between the content of volatile and non-volatile compounds, which are formed in the meat heating process, and the colour of its surface and sensory quality attributes of the product under study. Recognizing such interdependences would make it possible to standardize process conditions and to develop beneficial chemical composition of the product.

The aim of the investigations was to investigate the composition of volatile compounds being formed while roasting pork shoulder derived from Polish meat, to measure the colour of the surface corresponding to the specific profile of volatile compounds and to evaluate sensory quality attributes of the meat.

Materials and Methods:

Materials for investigations

Pork shoulder purchased in a Warsaw markets was used as a material for the experiment. The pork shoulder was sliced crosswise into chops 1.5 cm thick. The chops were cooked on a griddle (Spomasz PG-20). Electric heaters were located directly under the bottom of the griddle. The griddle was////////*– oiled with a rapeseed oil layer and then heated up to 200°C. The meat was placed onto the griddle and roasted for 20 minutes; it was turned over from time to time. The meat was evaluated after heating in terms of sensory quality attributes, colour and the content of volatile compounds were analysed. Samples were prepared and investigated three times.

Investigation of volatile compounds composition

About 1 g of the investigated meat sample was placed into a 4 ml vial closed with a septa nut and heated to 40°C for 30 minutes. The SPME fiber (CAR/PDMS type) was exposed to meat headspace. After 30 min, the SPME fiber was withdrawn from the vial and promptly introduced into the GC injector under the conditions described below.

GC/MS analyses were performed using Agilent 6890 GC coupled to an Agilent 5975 quadrupole mass detector. The SPME fiber was desorbed in GC injector at 230°C for 5 minutes in splitless mode and chromatographic separation was carried out on a 3 m x 0.25 mm x 0.25 μm film thickness HP-5MS (5% Phenyl Methyl Siloxane) capillary column. The GC oven temperature changes was programmed to stabilize from 40°C (held for 5 min.) up to 250°C at a rate of 5°C/min. Helium was used as a carrier gas at a constant flow of 0.9 ml/min. Mass spectra were recorded in EI mode at 70 eV, scanning the 20-500 m/z range. The identification of the isolated volatile compounds was achieved by comparing obtained mass spectra of unknown peaks with those stored in the NIST.02 (US National Institute of Standards and Technology) and Wiley.7n. mass spectral libraries.

Instrumental colour measurement

A chromameter Minolta CR-310 was used for instrumental colour measurement. The samples were measured against white reference standard utilising D65 light in L*a*b* measuring system. The meat was examined after heating at several spots on both sides of the sample and the results were averaged. The top layer was investigated, then it was cut off with a scalpel and the subsurface layer was measured.

Sensory analysis

Sensory analysis was carried out in accordance with the Polish standard PN-ISO 6564:1999 Sensory analysis – Methodology – Flavour profile methods. The research was done by the sensory panel of a trained ten-person team. The following sensory attributes were evaluated: burning flavour (scale span: little intensive – 0, very intensive – 10), fatty flavour (scale span: little intensive – 0, very intensive – 10), meaty taste, fatty taste, burnt taste (all had limiting designations little intensive – 0, very intensive – 10); general quality („I do not like it at all" – 0, „I like it very much" – 10), colour (pale – 0, intense brown – 10).

Statistical analysis

Statistical analysis was calculated using a Statistica 10.0. An analysis of variance was applied to determined differences between samples, on confidential level p=0.05.

Results and discussion:

Composition of volatile compounds

The total number of 80 volatile compounds were obtained as a result of SPME-GC-MS analysis. 39 compounds out of this number were identified with the probability of at least 80 %.

The types of individual compounds and corresponding areas below the peak [%] are shown in Table 1.

Hydrocarbons

Most of all volatile hydrocarbons in roasted pork were toluene and heptane. The content of toluene, also observed

Table 1: Composition of volatile compounds identified by means of GS/MS technique in roasted pork shoulder (as area %).

No.	Compound	Peak area [%]	No.	Compound	Peak area [%]
Hydrocarbons			**Ketones**		
1.	heptane	3.13	**22.**	2-butanone-3-hydroxy	1.75
2.	eicosane	0.03	**23.**	2-heptanone	0.33
3.	tridecane	0.06	**24.**	2,3-octanedione	3.34
Branched hydrocarbons			**Alcohols**		
4.	heptane-3-methylene	0.13	**25.**	1-butanol	0.11
5.	1-hexene-4-methyl	0.03	**26.**	1-hexanol	1.01
6.	toluene	5.63	**27.**	1-heptanol	0.59
7.	benzene-1,2,3,4-tetramethyl	0.01	**28.**	1-octanol	1.32
8.	3,4-diethylphenol	0.02	**29.**	1-nonanol	0.10
Carboxylic acids			**30.**	1-penten-3-ol	0.27
9.	hexadecanoic acid	0.08	**31.**	1-octen-3-ol	4.76
Aldehydes			**32.**	2-octen-1-ol	0.37
10.	hexanal	26.92	**33.**	1-butanol-3-methyl	0.22
11.	heptanal	3.04	**34.**	3,5-octadien-2-ol	0.07
12.	2-heptenal (E)	0.57	**Polihydrooxide alcohols**		
13.	octanal	4.97	**35.**	2,3-butanediol	0.51
14.	2,4-heptadienal (E,E)	0.29	**Cyclic compounds**		
15.	nonanal	10.85	**36.**	cyclohexene-4-ethenyl-1,4-dimethyl	0.07
16.	2-octenal (E)	0.46	**37.**	3-penten-2-on-4-(2,6,6-trimethyl-2-cyklohexen-1-yl)	0.06
17.	2,4-decadienal (E,E)	0.18	**38.**	cyclopentaneacetic acid-3-oxo-2-pentyl-methyl ester	0.31
18.	2,4-decadienal (E,Z)	0.4	**Furans**		
19.	2-dodecenal	0.33	**39.**	furan-2-pentyl	0.72
20.	benzaldehyde	0.56			
21.	benzaldehyde-3-ethyl	0.32			

and confirmed by other authors, can be related to the thermal degradation of phenylalanine [16]. The other hydrocarbons were present in trace quantities.

There are many factors which are critical so that certain types of volatile compounds are formed as a result of meat heating. Mottram [3] says that exposing meat to a high temperature, first of all, results in an oxidation process of fat contained in the meat and added thereto, which among others results in formation of hydrocarbons. Meinert et al. [2] specified pentane and octane in the volatile hydrocarbons determined in fried pork. Ahn and Olson [17] found the presence of toluene, 3-ethyl-4-methyl hexane, 2,2,8 – trimethyl decane, 2,2,4,6,6-pentamethyl heptane, 3,5-dimethyl octane, 2,5-dimethyl undecane and 2,8-dimethyl undecane in radiation-preserved pork. Wettasinghe et al. [18] identified 20 hydrocarbons in beef, pork and poultry as well as in other various compositions, including saturated, unsaturated and branched hydrocarbons. Poligne et al. [16] identified the presence of 10 aliphatic and 5 aromatic hydrocarbons while examining a product made of pork and processed through salting, drying and being smoked – mainly methyl derivatives of benzene. Fleming-Jones and Smith [19] examined volatile compounds content in various groups of market products purchased in shops in the USA. They identified the presence of benzene which is harmful to people's health and other harmful volatile compounds after heating of the purchased samples (including meat as a raw material). The authors explained that their presence was due to the natural environmental pollution resulting from combustion processes in the first place.

Aldehydes

The presence of 12 aldehydes were identified in the meat under investigation, including hexanal which was the most abundant aldehyde. Hexanal makes the products have quality attributes defined as „grass-like" ones. Poligne et al. [16]

say that hexanal and nonanal impart an unpleasant rancid pungent odour. A considerable content of nonanal, octanol and heptanal was identified in the meat under investigation apart from hexanal. The other aldehydes were present in smaller quantities.

Fat exposed to high temperature undergoes dehydration, decarboxylation, hydrolysis, oxidation and bond cleavage between carbon atoms [20]. Hydrocarbons, β-keto acids, methylketones, lactones and esters are specified as main products of fat changes.

However, they are just aldehydes which are the most important compounds being formed during fat decomposition and answerable for meat's aromatic quality attributes. Mottram [3] says that aldehydes comprising 6-10 carbon atoms are the main compounds initiating meat aromatic flavour treated under various conditions. They impart the flavour determined as „green", „tallowy" and „fatty".

2,4-decadienal is an important aldehyde in particular, which brings about the fried food aroma determined as „fat-fried food". The content and type of aldehydes present in the treated meat are dependent on the type of fatty acids present in the raw material, quantity and „stability" of peroxides being formed which lead to formation of aldehydes. The factors connected with the type of heat treatment – the temperature and duration of the process and the oxidation degree of fatty acids under given conditions are equally important [20]. Mainly octanal, nonanal and 2-undecenal are formed during oxidation of oleic acid; however, hexanal, 2-nonenal and 2,4-decadienal are formed during oxidation of linoleic acid. The two fatty acids out of unsaturated fatty acids are specified as the most typical ones in the case of pork.

A similar composition of aldehydes characterising cooked pork was presented in Shahidi [20] and discussed by Meinert et al. [2]. Hexanal was the dominant aldehyde in both cases. The differences between the types of the formed aldehydes specified by various authors are to be explained by various initial chemical composition of raw materials and various types of heat treatment (cooking, frying, roasting, drying, smoking) with the consumption of various doses of heat. The composition of aldehydes in pork and mixed products manufactured with the addition of pork which are treated under various conditions was presented among others by Wettasinghe et al. [18], Poligne et al. [16], Elmore et al. [21].

Ketones

It was identified that 2,3-octanedione was the most abundant ketone. The like details were shown in Shahidi [20]. There was also a considerable amount of 2-butanone-3-hydroxy when compared with the other ketones. The presence of 2,3-octanedione was also identified by Meinert et al. [2], Elmore et al. [21], who examined pork which was subjected to autoclaving, and by Wettasinghe et al. [18] in mixed meat comprising pork, beef and poultry; however, no such presence was identified in pork which underwent salting, drying and smoking [16]. These authors identified the presence of 2-heptanone in the product which they examined; it was also present in sterilised pork [21]. Wettasinghe et al. [18] found that the content of 2-butanone-3-hydroxy was different depending on the type of meat. This compound was also identified in autoclaved pork [21]. 2-butanone-3-hydroxy are characterised by a fragrance note determined as „buttery".

In general it is considered that ketones give meat a buttery aroma to cooked meats [18].

Alcohols

11 compounds were identified in volatile alcohols, including 5 saturated alcohols, 4 unsaturated alcohols, 1 branched alcohol (methyl derivative of butyl alcohol) and 1 dihydroxide alcohol (2,3-butanediol). 1-octen-3-ol, 1-octanol, 1-hexanol were identified to be the most abundant compounds in alcohols in the roasted pork shoulder. 1-octen-3-ol is a characteristic compound of cooked pork. It is also characteristic of cooked mushrooms. Peroxide of arachidonic acid is its precursor in pork [20].

All the above-mentioned alcohols were also present in volatile alcohols characteristic of the pork presented in Shahidi [20], whereas Meinert et al. [2] identified the presence of as few as 3 alcohols in fried pork. They were methylalcohol, ethanol and 2-methyl-3-buten-2-ol, which were identified neither in roasted pork nor in volatile acids presented in Shahidi [20]. Only the presence of one alcohol – 1-butanol-3-methyl was reported in the case of salted, dried and smoked meat [16]. Moreover, Wettasinghe et al. [18] identified as many as 31 alcohols in mixed meat. They included 1-hexanol, 1-octanol, 1-octen-3-ol and 2,2-butanediol. Among others the content of 1-heptanol, 1-octen-3-ol and 1-ethanol was identified in sterilised meat [21].

Straight-chain alcohols are responsible for attributes defined as greenish, woody and fatty-floral quality attributes [18].

Cyclic compounds

The presence of 2 cyclic unsaturated compounds (branched derivatives of cyclohexane) and of one methyl ester – cyclopentane derivative was identified in the meat under experiment. Cyclic compounds can mainly be formed as a result of either hydrogenation of aromatic compounds due to cyclization of chain compounds or as a result of cycloaddition of unsaturated compounds. Elmore et al. [21] identified the presence of only one cyclic compound, which

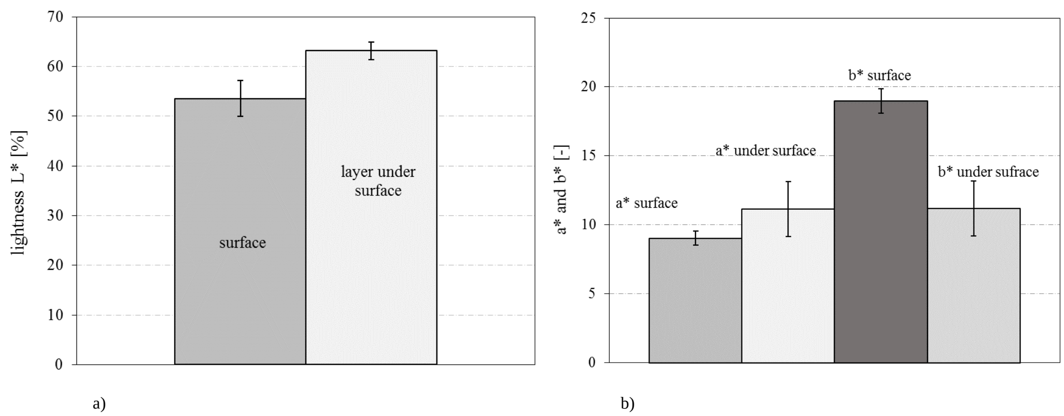

Fig. 1: The colour of the surface and layer under the surface of pork shoulder after roasting; a). lightness L* [%], b). a* and b* [-].

is 5-ethyl-1-formylcyclopentene in pork subjected to sterilisation. Wettasinghe et al. [18] identified 1 cyclic ketone (pentane derivative), 1 cyclic alcohol, 1 cyclic hydrocarbon and 2 cyclic compounds comprising nitrogen in mixed meat.

Furans

The presence of one furan compound was reported in the pork shoulder under investigation. It was furan-2-pentyl which is a characteristic compound of pork. Its presence in cooked meat and cured meat products has been reported by many authors [16,18,20,21]. Furans impart sensory quality attributes to products determined as meaty [3].

Results of instrumental measurement of roasted pork neck shoulder colour

The colour of the pork's surface was determined by means of instrumental measurement. In order to examine the influence of heat treatment on the colour of meat surface, the surface colour of the sample under investigation and the layer colour after having removed the top layer were analysed. Such a determination permitted to decide which colour factor is the most essential indicator of change due to the heating process (Fig. 1 a) and b)).

Lightness L* of the surface of the meat (of the pork shoulder) subjected to heat averaged to 53.56%. This value corresponded with the colour sensory evaluation of 4.17 (shown at Fig. 2), whereas the value considered to be very dark equals 10. Such a value of the lightness affirms that the product is neither too light nor too dark.

After having removed the outer layer, the lightness was 63.16%. Analysis of variance stated that the layer under the surface was significant lighter than the surface (sensory evaluation of the layer under the surface was not investigated).

The values a* and b* were the following parameters of colour, which were analysed. These values give evidence

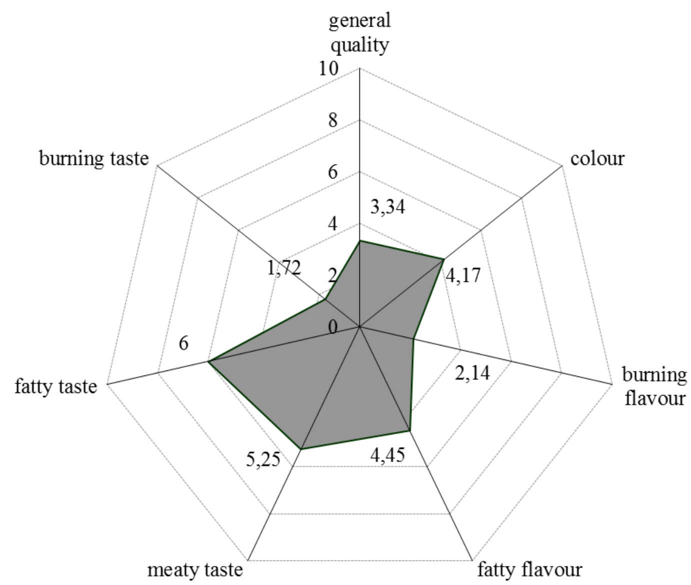

Fig. 2: Sensory profile of roasted pork shoulder.

of red (a*) and yellow (b*) colour in the final image of a given product colour. In the case of the pork shoulder under investigation, the outer layer of the product after roasting process was characterized by a* parameter value at a level of 9.03 and b* value equal to 18.98. The values of both parameters for layer under surface were: a* 11.14 and b* 11.17, respectively. Upon the basis of the results it can be ascertained that the distinguishing factor which imparted the characteristic colour to the outer layer of the roasted product was b* parameter, whose value was significantly different from b* value for subsurface layer (the a* value changed insignificantly).

Sensory profile of roasted pork shoulder

Fig. 2 presents intensity values of the attributes evaluated for the meat under investigation after heating. The highest value was reported in the case of fatty taste (6.0).

This determination was connected with the taster sensing an intensiveness in the heated fat taste while consuming the meat, and it should be interpreted as negative. This distinguishing factor dominated the general profile of the meat under examination. The other factor in terms of its high intensiveness was the taste determined as meaty. The average value of the evaluation was 5.25. Unfortunately, the fatty taste was more intensive than the meaty one. The fatty smell (its numerical value was 4.45) was also essential for the general evaluation of the meat. Its value was most probably related to the high fatty taste intensiveness. The smell determined as burned (2.14) and the burning taste (1.72) were of less significance in the general profile of the meat after heating . The smell determined as burnt (2.14) and the burnt taste (1.72) were of less significance in the general profile of the meat after heat treatment process.

The following essential factor, which was examined, was colour; in the case of colour the limiting designations ranged between pale (0), and intensely brown (10). The value of the colour was evaluated as 4.17.

Conclusions

The greatest contribution to volatile compounds identified in the roasted pork shoulder was made by aldehydes (Fig. 3), including hexanal and nonanal, which speaks to the fact that the taste determined as fatty was the quality attribute evaluated most intensively, and the fatty smell was also sensed intensively.

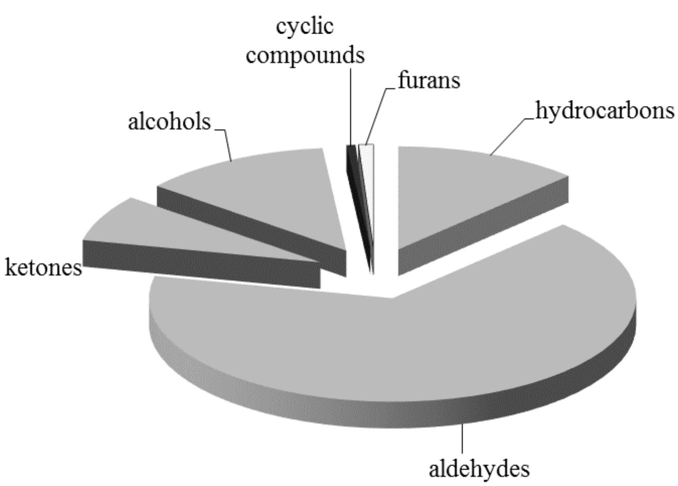

Fig. 3: Structure of individual groups of volatile compounds in roasted pork shoulder.

A large share of alcohols in the compounds also contributed remarkably to the sensing of the fatty taste. The meaty taste intensiveness was evaluated to be not much lower due to the content of 2,4-decadienal. The attributes determined as meaty resulted also from the presence of ketones and furan-2-pentyl. The rapeseed oil could partly have influenced such sensory quality attributes, which was used to smear the griddle before the treatment. However, the oil layer was very thin.

The content of nitrogen containing compounds was the most essential group of compounds responsible for the characteristic flavour of the roasted meat. No presence of typical nitrogen containing compounds such as pyrazines was affirmed in the pork shoulder under investigation. The presence of a few nitrogen containing compounds was identified; however, because of their trace content, they were identified with too little probability and that is why they were not listed in Table 1. There were no sulphuric compounds in the meat under investigation either. Such a composition of volatile compounds were most probably caused by the conditions of the process – the griddle temperature 200°C, and the short duration of the process (20 minutes). Sulphuric compounds are mainly formed in the meat subjected to cooking [3]. Their presence was not affirmed under the conditions which the experiment was conducted.

The least intensive attribute in the sensory evaluation was the aroma and taste determined as burnt. No presence of volatile compounds was identified either, which would be responsible for formation of just such a sensory note.

The intensiveness of the fatty taste and fatty smell had an impact on the general meat evaluation which was 3.34.

The colour of the meat surface after heating is an essential discriminant suggesting the intensity of the changes which were brought about. The colour of the surface depends on Maillard's reaction of non-volatile compounds which is a subject of interest to many researchers. However, it is complicated to identify them correctly in a ready-made product. That is why the measurement of the colour can be a criterion of the changes which were brought about in the product under the specific conditions of the heating process. The distinguishing factor which significantly changed due to the conducted process was lightness L* and parameter b*. Parameter a* was of smaller significance, which was affirmed by the comparison of its values for the outer layer and the subsurface layer. At the same time it can be assumed that the parameters L* approximating to 53% and b* within the limits of 19[-] impart such a colour of the surface which is determined as 4.17 in 10-degree scale with the limiting designations ranging between pale (0), and intensely brown (10) by means of sensory evaluation. That also explains the smallest sensory evaluation value, which was found for the burning taste and burnt smell (the colour of the surface was not considerable). Olsson et al. [14] found various values of L*a*b* upon the meat surface depending on the temperature of the treatment process while examining the colour of fried pork loin. For a temperature of 200°C they were as follows: L* – 56, 57.5 [%], a* – 10, 10.1 [-] and b* – 22.4, 23 [-] (for the meat with genotype

RN- and RN, respectively). They determined the results of L*a*b* values as „surface colour".

Summarized – the pork neck shoulder cooked on a griddle at a temperature of 200°C was mainly characterised by the taste and smell determined as fatty. It was connected with the presence of aldehydes – hexanal and nonanal, whose content was absolutely predominant in other volatile compounds.

The presence of nitrogen containing compounds was not identified unequivocally in volatile compounds, nor were sulphuric derivatives. The meat with the above composition of volatile compounds was characterized by the colour parameters equal to: L* – 53.56, a* – 9.03, b* – 18.98. It was a specific surface colour.

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