Actual salt content in salted minced pork and beef as determined by AAS and NIR methods

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Abstract

The aim of this article was to evaluate the amount of salt dosed in various proportions into minced pork and beef. Determination of salt was conducted by the atomic absorption spectrometry (AAS) method and, for comparison, by the near infrared spectroscopy (NIR) method. The meat (beef neck and pork topside) was ground through a plate with a hole size of 8 mm. Salt doses were from 0 to 2.0%, at intervals of 0.2%. Each sort of meat was divided into two parts of 350 g: 1) with initial grinding (coarse grinding), and 2) with subsequent grinding through a plate with a hole size of 3 mm (fine grinding). The salt content in fresh pork meat (0% salt) ranged between 0.13 and 0.16%, whereas the content in beef ranged from 0.14 to 0.19% (as analysed by the AAS method). The proportion of added salt had a statistically significant effect on the salt content in meat. No significant differences were found between coarse and fine grinding and between pork and beef meat. The predicted natural salt content in meat was 0.15 and 0.19% for pork, and 0.20 and 0.21% for beef. The proportion of analysed salt was then determined as 0.99–1.06 times the dosed salt. The authors do not recommend the use of the NIR method, which provides reliable results for the main chemical components of meat, i.e. protein and fat, for the determination of the proportions of salt in meat.

Beef, pork, sodium determination, AAS, NIR

According to the definition given in Regulation (EC) 853/2004 (Annex I; Article 1.10), 'fresh meat' is meat, including meat packed in a vacuum or in a protective atmosphere, for the preservation of which no treatment other than chilling, freezing or quick-freezing has been used (Regulation 2004). Once salt is added to it, it can no longer be marketed as fresh meat. Minced meat (Regulation 853/2004, Annex I, Article 1.13) may contain less than 1% salt (Berger et al. 2023). Meat preparations (Article 1.15 of the above regulation) are defined as fresh meat, including meat that has been reduced to fragments, which has had foodstuffs, seasonings or additives added to it or which has undergone processes insufficient to modify the internal muscle fibre structure of the meat and thus to eliminate the characteristics of fresh meat (Regulation 2004). Salt is considered a food, for which reason it can be contained in meat preparations and its proportion is not limited in any way by this regulation (unlike minced meat).

In the case of meat preparations, however, Regulation (EU) 1169/2011 imposes the obligation of the provision of nutritional information, which also includes the amount of salt. According to this regulation, salt is understood as the salt equivalent content calculated according to the formula: salt = sodium × 2.5 (Regulation 2011). This is because sodium makes up approximately 40% of the NaCl molecule (39.3% to be precise, chlorides account for 60.7%; Feiner 2006). Sodium is a chemical element that has a number of functions in the human and animal bodies. In beef and pork, Wood (2017) reports its

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content as 63 mg/100 g of raw lean meat. Simple conversion results in slightly less than 160 mg of salt/100 g of meat, i.e. 0.16%. This must be taken into account in the recipes for processed meat, so that the dosed amounts of salt correspond to the percentages that appear in the final products and which the manufacturer must state in the nutritional information on the packaging.

Volhard titration methods based on chloride determination can no longer be used to determine the proportion of salt in meat products in the EU (FSIS 2009). The atomic absorption spectrometry (AAS) method or the inductively coupled plasma mass spectrometry (ICP-MS) method are used most often (Perez-Palacios et al. 2022). NIR hyperspectral imaging methods are often used for rapid determination of essential nutrients (proteins, fat, water) in meat (Jo et al. 2024). Devices based on the principle of these techniques are used at manufacturing companies. In addition to basic nutrients, quality managers also evaluate the results for the salt content determined by NIR analysers when checking the quality of meat.

The aim of this article was to evaluate the amount of salt dosed in various proportions into minced pork and beef. Salt determination was conducted by the AAS method and, for the purposes of comparison, by the NIR method.

Materials and Methods

Preparation of meat samples

Beef (boneless neck) and pork (topside muscles) meat was dispatched directly from the producer (Steinhauser s.r.o., Tišnov, Czech Republic) in a refrigerated state 2–3 days after slaughter during the period June–July 2023. The meat was ground in a Kenwood PRO 2000 Excel electric food mincer (Kenwood, London, United Kingdom) through a plate with 8-mm holes. The ground meat was divided into portions weighing 700 g (Radwag PS 3000 scale; Radwag váhy s.r.o., Šumperk, Czech Republic). Doses of salt from 0 to 2.0%, at intervals of 0.2% (Boeco BBI-41 scales; Boeco, Hamburg, Germany), were added to each portion. The salt was mixed into the ground meat with a metal whisk (MPRO NZ 34 cm whisk; Makro Cash & Carry CR s.r.o., Prague, Czech Republic). A total of 11 portions of 700 g each were prepared. The individual portions were placed in polypropylene bowls (227 × 178 × 50 mm) and wrapped without atmosphere modification in a T-190 sealing machine (MetalPack, Maso-profit s.r.o., Brno, Czech Republic) with a top PET/PP 185-mm foil (thickness 52 μm; Maso-profit s.r.o., Brno, Czech Republic) and a sealing temperature of 180 °C. The wrapped batches were stored at 1 °C for 24 h. The following day, each batch was divided into two parts of 350 g each: 1) with initial grinding (coarse grinding), and 2) with subsequent grinding in a Kenwood PRO 2000 Excel device with a plate with 3-mm holes (fine grinding). The samples prepared in this way were sent to laboratories for determination of the salt content. The experiment was performed in duplicate, i.e. two series of pork and two series of beef were prepared at an interval of one month.

The AAS method of measuring the sodium concentration

Homogenised samples were taken for the determination of sodium, after which mineralisation by wet digestion was performed with a mixture of 6 ml nitric acid (67% v/v) and 1 ml hydrogen peroxide (30% v/v). Pressure digestion was performed in two stages with a maximum temperature of 200 °C and microwave power of up to 1000 W (Ethos SEL, Milestone, Italy). The sodium content was determined by the FAAS flame atomisation method on a high-resolution continuum source atomic absorption spectrophotometer (ContrAA 700, Analytik Jena AG, Jena, Germany). The oxidising flame was acetylene-air (Air Products spol. s.r.o., Děčín, Czech Republic). For calibration, a series of 5 solutions was prepared from a standard sodium solution of a concentration of 1 g/l. All determinations were performed three times, with the average of the three measurements used as the resulting value. The repeatability of the AAS method for two different salt concentrations is as follows: $0.37 \pm 0.01\%$, RSD 2.99%; $1.87 \pm 0.08\%$, RSD 4.29%. The values obtained were processed by Aspect CS software, version 2.2.2.0 (Analytik Jena AG, Jena, Germany). The correctness of the method was validated using standard reference material 1566 b (Oyster Tissue, National Institute of Standards and Technology, USA). The reference material was digested and measured using the same method as used for the samples. The declared sodium content of the reference material was $0.3297 \pm 0.0053\%$. The sodium content was converted into the salt content according to Regulation (EU) No. 1169/2011 of the European Parliament and of the Council on the provision of food information to consumers, according to the formula: salt = sodium × 2.5.

Determination of the proportions of salt using NIR spectroscopy

The proportion of salt was determined by the NIR spectroscopy method using a FoodScan™ 2 Meat Analyser (Foss, Hilleroed, Denmark). Samples of minced meat were loaded into an analysis dish with a diameter

of ca 15 cm and a height of ca 1 cm, which amounts to approximately 150 g of sample. The duration of the analysis was around 2 min following the insertion of the dish into the NIR analyser. The determined salt content was then read from the device's display, which was evaluated as an average value from 18 individual measurements taken at different places of the meat in the analysis dish. The device's evaluation software included calibrations for determining various parameters, including the proportion of salt, created on the basis of analyses of several thousand samples of pork and beef meat and various types of meat products. The repeatability of the NIR method for two different salt concentrations is as follows: $0.576 \pm 0.012\%$, RSD 2.08%; $1.398 \pm 0.015\%$, RSD 1.07%.

Statistical evaluation of the results

General linear model – Analysis of Covariance – was used to assess the effect of meat, grinding as well as their interaction and salt concentration on the proportion of salt analysed by AAS method. Type of meat and type of grinding were included as categorical factors, added salt as a continuous predictor. Linear regression model was used to determine the relationship between the added salt and the proportion of salt analysed by AAS method. In order to assess the differences between the AAS and NIR methods, salt proportions were aggregated by averaging the values for the given salt concentrations and paired t-test was applied. $P < 0.05$ was considered significant in all tests. Statistical analyses were performed using Statistica, version 14 (TIBCO Software Inc., Santa Clara, USA). The principle of statistical processing of the results is based on the authors' article Biffin et al. (2020).

Results and Discussion

Salt contents in meat as determined by the AAS method

The salt contents in fresh pork (0% added salt) ranged between 0.13 and 0.16% (Table 1). Macharáčková et al. (2021) measured around 0.12% salt in samples of fresh pork loin, while the authors Perez-Palacios et al. (2022) measured $0.25 \pm 0.05\%$. In both cases the samples were analysed by the AAS method and the salt contents were calculated from the sodium concentration.

The values in beef also fell within a range that was in agreement with the results for the salt contents in pork meat (Table 1). The salt contents in fresh beef (0% NaCl) ranged from 0.14 to 0.19% and corresponded to the average value of 0.16% reported by Wood (2017).

After adding the maximum proportion of salt (2%), the salt values in the meat analysed by the AAS method ranged from 1.68 to 2.42%. The proportion of added salt showed a significant effect on the salt content in meat (ANCOVA: effect of added salt: $F(1,233) = 7661.693$, $P < 0.001$). The type of meat (pork and beef), the method of grinding (coarse and fine) and even the interaction of these two factors had no significant effect on the salt content in the meat (ANCOVA: effect of type of meat: $F(1,233) = 0.001$, $P = 0.977$, effect of type of grinding: $F(1,233) = 0.268$, $P = 0.605$, effect of meat/grinding interaction: $F(1,233) = 0.374$, $P = 0.541$).

The predicted salt content without added salt was 0.19% for coarse grinding and 0.15% for fine grinding meat for pork, and 0.21 and 0.20% for beef, as evident from the results of regression models. The increase in the analysed salt content in the meat then corresponded to the proportion of added salt, with the coefficients of 1.00 and 1.06 for pork and 0.99 and 1.00 for beef. (Table 2)

Salt contents in meat as determined by NIR spectroscopy

Determination using the NIR spectroscopy method is extremely fast (around 2 min), which provides a considerable time advantage during analysis. Table 3 presents the mean values for salt contents determined using the NIR method in pork and beef meat, and a statistical comparison with the results of the AAS method. The salt content determined by the NIR method for fine ground pork ($P = 0.015$) and coarse ground beef ($P = 0.0003$) differed significantly from the results produced by the AAS method, the differences for coarse ground pork and fine ground beef were non-significant (Table 3).

Requirement 4.2.1.3 of the standard IFS Food version 8 states, among other things, that specifications relating to manufactured products must be up-to-date, unambiguous, and in accordance with legal requirements and, if defined, customer requirements (IFS 2023).

Table 1. Results for the salt percentage (mean ± standard error) in samples of minced pork and beef analysed by the AAS method (N = 4–6).

Salt added (%)	Salt in pork (topside)		Salt in beef (boneless neck)	
	Coarse grinding (8 mm)	Fine grinding (3 mm)	Coarse grinding (8 mm)	Fine grinding (3 mm)
0.00	0.14 ± 0.01	0.14 ± 0.01	0.16 ± 0.01	0.15 ± 0.01
0.20	0.38 ± 0.02	0.37 ± 0.01	0.41 ± 0.02	0.39 ± 0.01
0.40	0.53 ± 0.01	0.54 ± 0.01	0.57 ± 0.03	0.48 ± 0.01
0.60	0.74 ± 0.02	0.74 ± 0.01	0.79 ± 0.01	0.79 ± 0.01
0.80	0.95 ± 0.05	0.97 ± 0.02	1.00 ± 0.03	1.11 ± 0.02
1.00	1.28 ± 0.05	1.27 ± 0.03	1.28 ± 0.02	1.27 ± 0.01
1.20	1.52 ± 0.05	1.49 ± 0.03	1.51 ± 0.01	1.50 ± 0.01
1.40	1.64 ± 0.03	1.60 ± 0.04	1.67 ± 0.05	1.66 ± 0.04
1.60	1.97 ± 0.09	1.87 ± 0.03	1.83 ± 0.01	1.80 ± 0.02
1.80	2.04 ± 0.07	2.06 ± 0.05	1.92 ± 0.02	1.94 ± 0.03
2.00	1.94 ± 0.10 [A]	2.18 ± 0.06 [B]	2.04 ± 0.13	2.08 ± 0.03

[A,B] Salt contents in meat with different types of grinding marked with a different superscript capital letter differ significantly (Tukey *post hoc* test).

Table 2. Regression equations of the salt content in minced pork and beef as analysed by the AAS method depending on the amount of added salt.

Meat	Grinding	Intercept estimate ± SE	P	Slope estimate ± SE	P	R²
Pork	Coarse	0.187 ± 0.040	< 0.001	1.002 ± 0.034	< 0.001	0.941
Pork	Fine	0.148 ± 0.019	< 0.001	1.057 ± 0.016	< 0.001	0.986
Beef	Coarse	0.212 ± 0.024	< 0.001	0.985 ± 0.021	< 0.001	0.975
Beef	Fine	0.201 ± 0.020	< 0.001	0.996 ± 0.017	< 0.001	0.982

P – *P*-value; SE – standard error, R^2 – coefficient of determination

Table 3. Results for the salt percentage (mean ± standard error) in samples of minced pork and beef analysed by the NIR method (N = 2).

Salt added (%)	Salt in pork (topside)		Salt in beef (boneless neck)	
	Coarse grinding (8 mm)	Fine grinding (3 mm)	Coarse grinding (8 mm)	Fine grinding (3 mm)
0.00	0.28 ± 0.04	0.25 ± 0.00	1.41 ± 0.04	0.99 ± 0.07
0.20	0.40 ± 0.03	0.42 ± 0.01	1.42 ± 0.05	1.17 ± 0.14
0.40	0.60 ± 0.04	0.56 ± 0.04	1.44 ± 0.10	1.29 ± 0.03
0.60	0.72 ± 0.05	0.74 ± 0.02	1.47 ± 0.07	1.37 ± 0.02
0.80	0.94 ± 0.06	0.85 ± 0.00	1.59 ± 0.12	1.49 ± 0.04
1.00	1.06 ± 0.03	1.02 ± 0.11	1.72 ± 0.14	1.48 ± 0.00
1.20	1.32 ± 0.11	1.26 ± 0.07	1.78 ± 0.12	1.59 ± 0.05
1.40	1.53 ± 0.05	1.29 ± 0.18	1.92 ± 0.11	1.65 ± 0.01
1.60	1.52 ± 0.13	1.60 ± 0.13	2.12 ± 0.07	1.61 ± 0.03
1.80	1.86 ± 0.10	1.63 ± 0.13	2.23 ± 0.13	1.76 ± 0.04
2.00	1.97 ± 0.11	1.91 ± 0.09	2.33 ± 0.16	1.79 ± 0.05
Statistical comparison with the results of the AAS method	P = 0.116	P = 0.015	P = 0.0003	P = 0.060

P– *P*-value

This also applies to the salt content in ground meat, meat preparations, and meat products. A reliable method of evaluating the salt content is to determine the sodium concentration using AAS with subsequent conversion to the salt content.

The use of the NIR method, which provides reliable results for the principal chemical components of meat, i.e. proteins and fat (Kartakoullis et al. 2019; Maduro Diaz et al. 2021), is a great help in terms of significant time savings during analyses, but it is not so reliable in terms of accuracy of the results. Deviations from the actual salt content in food ingredients or products can be significant. Results more accurate than those provided by the AAS method cannot be expected without thorough calibration of the NIR analysers for the tested product. Such calibration is, however, demanding for the preparation of a large number of samples and is difficult to perform under the conditions in force at production plants. For this reason, NIR method is not recommended by the authors for determination of salt contents.

In the case of the device used in this study, the incorporated calibrations were created by the supplier of the device on the basis of several thousand results from analyses of pork and beef meat and various types of meat products. The instrument supplier offers the option of adjusting the installed calibrations based on the specific values of stipulated parameters for the given samples analysed in the given operation, which is crucial to the accuracy of the results determined by the NIR analyser.

In conclusion, it is evident from the results presented that with careful preparation, batches of minced meat or meat preparations made from ground meat can be prepared with a relatively precise salt content. When dosing salt, manufacturers must take into account the natural sodium concentration in fresh meat, which corresponds to approximately 0.15% salt.

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