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Is authentication of the geographic origin of poultry meat and dried beef improved by combining multiple trace element and oxygen isotope analysis?

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ABSTRACT

Data available on contents of up to 72 different trace elements and the oxygen isotope ratio of 78 poultry breast and 74 dried beef samples were analysed to determine whether the accuracy of the prediction of the geographic origin is improved by combining promising methods. Validation was performed by determining the origin of a smaller sub-group using a statistical model established from the data of the second, larger, sub-group. As expected, the combined data proved useful for the determination of the geographic origin of meat samples. However, combining data did not clearly reduce the percentage of incorrectly classified individual samples compared to the two approaches applied separately. In poultry, cross-validation and validation resulted in 83% and 50% correct classifications, respectively. The corresponding values in dried beef were 73% and 43%. In conclusion, compared to element signature data alone, combining both methods did not improve predictions of origin.

1. Introduction

Legislation in the EU and in Switzerland demands a better traceability of food. Therefore, meat and meat products have to be labeled with their country of origin (2000/13/EG; Eidgenössisches Departement des Inneren, 2005). The development of appropriate tools to authenticate these origins independent from paper traceability increases consumers' safety and contributes to the protection of producers against potential frauds and misrepresentation.

In recent years several studies were conducted to determine the geographic origin of meat (Boner & Förstel, 2004; Hegerding, Seidler, Danneel, Gessler, & Nowak, 2002; Piasentier, Valusso, Camin, & Versini, 2003; Renou et al., 2004; Shintu, Caldarelli, & Franke, 2007). In a large research programme, various analytical tools considered to be of high potential for this purpose (Franke, Gremaud, Hadorn, & Kreuzer, 2005) were tested. Determination of various minerals and trace elements (Franke et al., 2007a, 2007b) and the oxygen isotope ratio (δ^{18}O) (Franke et al., 2008) have been shown to be useful for geographic authentication of poultry meat and dried beef. Still classifications to countries of origin of the raw meat or country of processing (dried beef) is not possible with certainty, especially in case of neighbouring origins and when meat is processed at the same plant. Characteristic profiles of elements may be related to the environment of the animals, e.g. through specific geologic profiles or through (anthropogenic) pollution (Franke et al., 2007a) while distance from the ocean and other external factors are responsible for variations in δ^{18}O (Boner & Förstel, 2004; Franke et al., 2008). The applicability of both methods is limited, either by uncontrollable feed supplementation with, sometimes essential, elements or when processing such as drying modifies the isotope ratio. Initial differences in oxygen isotope ratio between European and South American meat (Boner & Förstel, 2001; Boner & Förstel, 2004; Förstel & Lickfett, 2002) may be masked (Franke et al., 2008).

The power to discriminate between samples of different geographic origin might be increased by combining techniques based on different principles and the limitations outlined above could be overcome. In the present study, therefore, available data of elements (Franke et al., 2007a, 2007b) and δ^{18}O (Franke et al., 2008) were combined for overall statistical analyses to compare the accuracy of geographic authentication with that found when applying the methods independently.

2. Materials and methods

2.1. Samples

A total of 78 frozen poultry breasts were obtained from Brazil (n = 14), France (n = 13), Germany (n = 15), Hungary (n = 16), and Switzerland (n = 20) between February 2004 and December 2005. This basically included two sample sets, the first comprising 22

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samples from these countries (Phase I), the second 56 samples (Phase II). The authenticity of all samples had been certified with valid custom documents, specifying place and date of slaughter. The samples were vacuum-sealed in vapour-impermeable plastic bags and kept frozen at −25 °C until analysed.

Concerning the dried beef, 74 samples (21 being collected in Phase I), having been prepared either from *Musculus biceps femoris* or *Musculus semitendinosus*, were either directly collected from the production site (samples processed in Switzerland) or purchased from producers in Australia ($n = 8$), Austria ($n = 6$), Canada ($n = 8$), and USA ($n = 5$) between May 2004 and February 2006. The Austrian samples had been produced from Brazilian meat, whereas all other non-Swiss samples had meat from the country of processing as their origin. Part of the Swiss samples had been produced in the Swiss canton of Valais using Swiss meat ($n = 15$) and the rest in the canton of Grisons using Swiss ($n = 16$) and Brazilian meat ($n = 16$). All dried beef samples were produced by curing and various sequences of air drying and pressing. Previous studies (Franke et al., 2007) showed they had final dry matter contents of 46 ± 5%, equivalent to about 40% water loss. Slight variations in recipes (e.g. amount of salt, kind of herbs) and technology (salt application, curing, drying, pressing, use of moulds, storing, packaging, etc.) may have occurred within the same type of product, depending on the producer. The samples were vacuum-sealed in vapour-impermeable plastic bags and kept at −5 °C until analysed.

The individual poultry and dried beef samples were independent of each other as they were either obtained from different producers or obtained from different production batches when originating from the same producer. No detailed information on conditions of animal fattening, meat handling and processing were available, as would be also the case in future routine control of declared geographic origins.

2.2. Chemical analysis

Samples were analysed for $\delta^{18}O$ as described in detail by Franke et al. (2008) and for element content as outlined in Franke et al. (2007a, 2007b). Briefly, water for oxygen isotope analysis, extracted from approx. 10 g of meat, had been obtained, using still frozen samples to avoid drip loss and thus possible shifts in $\delta^{18}O$, by azeotropic distillation with toluene (130 °C, 18 h) using the Bidwell-Sterling apparatus as outlined by Schäfer (1967). In these water samples, the oxygen isotope ratio was determined with the help of an IRMS (Delta-Plus XL, Finnigan, Bremen, Germany). For element analysis, samples of dried beef (0.4–0.5 g) and poultry (0.8–1 g) were prepared by micro-wave assisted digestion with nitric acid under pressure. The digested material was analysed using a sector field ICP-MS (Element 2, Finnigan MAT, Bremen, Germany) using a CertiPUR® Rhodium ICP Standard (Merck, Darmstadt, Germany).

2.3. Statistical analysis

Poultry samples were grouped according to their geographic origin, while dried beef samples were grouped according to both raw meat origin and place of processing. The results for the reference material, analysed for elements in both sampling phases, were compared using the Wilcoxon–Mann–Whitney test (probability >0.01) to exclude all those elements from statistical analysis where concentrations varied with time (cf. Franke et al., 2007a). Also elements below the detection limit in at least one of the two sampling phases were excluded from further statistical analysis.

Analysis of variance (ANOVA) was performed with each single variable and, in case of significance ($p < 0.05$), Bonferroni-adjusted pairwise comparison was performed in order to identify the origins being separated from the others by individual variables. Different to Franke et al. (2007a), this was done on the combined dataset of both sampling phases. For $\delta^{18}O$, results were taken from Franke et al. (2008). Additionally, $\delta^{18}O$ and element analyses were combined and linear discriminant analysis (LDA) in stepwise backward elimination (probability f to enter/to remove = 0.15) was performed. By this way, (i) cross-validation, jackknifed-type, classification matrices were built using all samples (cross-validation) and (ii) the origin of the samples from Phase 1 was predicted using the data of Phase 2 samples (validation). All statistical analyses were performed by Systat (version 11, Systat Software Inc., Richmond, California, USA).

3. Results and discussion

3.1. General

According to the results of the comparison based on the Wilcoxon–Mann–Whitney test, the elements ^{45}Sc, ^{53}Cr, ^{67}Zn, ^{68}Zn, ^{104}Pd, ^{128}Te, ^{141}Pr, ^{151}Eu, ^{153}Eu, ^{161}Dy, ^{163}Dy, ^{169}Tm, ^{171}Yb, ^{172}Yb, ^{203}Tl, ^{209}Bi, ^{238}U were excluded, for the reasons described in Franke et al. (2007a). These elements are mostly rare in nature and of low concentration in meat.

3.2. Poultry meat

In the univariate mode of analysis, poultry meat groups from any country of origin could be distinguished from that of other countries by using at least one element (Table 1). Differentiation between Swiss and Hungarian poultry samples was almost impossible as just ^{75}As was useful. Possible reasons for the discriminating power of the elements As, Rb, Sr and Tl found to be significantly different among countries have been discussed earlier (Franke et al., 2007b).

The cross-validation classification matrix gave a mean classification rate of 83%, based on the elements ^{82}Se, ^{85}Rb, ^{88}Sr, ^{155}Gd, ^{205}Tl, ^{23}Na, ^{51}V, and on $\delta^{18}O$ (Table 2). Only samples from Brazil could be classified completely correctly, but one French sample was misclassified as being Brazilian. German samples were classified correctly with the exception of one sample which was classified as being French. Between Hungarian and Swiss samples

Table 1
Univariate differentiation of poultry breast meat of various origins[a]

Country	Brazil	France	Germany	Hungary	Switzerland	Variable
n	14	13	15	16	20	
Brazil		X	X	X	X	^{85}Rb
	–	X	0	X	X	$^{86,88}Sr$
		X	X	0	0	^{205}Tl
		0	X	X	X	$\delta^{18}O$
France	X		X	0	0	$^{86,88}Sr$
	X	–	0	X	X	^{205}Tl
	0		X	X	X	$\delta^{18}O$
Germany	0	X		0	0	$^{86,88}Sr$
	X	0	–	X	X	^{205}Tl
	X	X		X	X	$\delta^{18}O$
Hungary	X	X	X		X	^{75}As
	0	X	X	–	0	^{205}Tl
	X	0	0		0	$^{86,88}Sr$
	X	X	X		0	$\delta^{18}O$
Switzerland	0	X	X	0		^{205}Tl
	X	0	0	0	–	$^{86,88}Sr$
	X	X	X	0		$\delta^{18}O$

[a] X = differentiation possible; 0 = no differentiation possible. Results for $\delta^{18}O$ taken from Franke et al. (2008).