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ORIGINAL PAPER

CHANGES IN MACRO- AND MICROELEMENTS IN FRESHWATER FISH DURING FOOD PROCESSING

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Abstract

Fish meat is a valuable dietary component owing its exceptionally high nutritional value, as it contains high quality protein, fat (including n-3 acids) and a variety of vitamins and minerals. The aim of this study was to determine the content of ash and minerals (macro- and microelements) in three freshwater fish species, and quantitative changes in smoked and marinated products. The research material originated from three species, i.e. common carp (*Cyprinus carpio*), rainbow trout (*Oncorhynchus mykkis*) and northern pike (*Esox lucius*). The following parameters were determined in samples of raw as well as processed fish meat: ash (using the gravimetric method, according to PN-ISO 936:2000) and the minerals Na, K, Ca, Mg, P, Fe, Zn, Mn, Cu (using inductively coupled plasma optical emission spectrometry ICP-OES). The statistical analysis of the results did not show significant differences in the ash content in meat of the three fish species. The ash content in processed fish meat differed significantly from that in raw fish, thus indicating changes in concentrations of the minerals. The differences depended on the applied processing technology. As for the mineral content, the examined fish species achieved high nutritional quality, with the highest content of magnesium, iron and zinc in common carp, potassium and phosphorus in rainbow trout and calcium in pike. The levels of the minerals in processed fish meat differed significantly from those in the raw material, especially in carp products (higher amounts of almost all the determined elements). Smoking as well as marinating resulted in a considerable increase in the sodium contet, and the highest values were obtained for marinated rainbow trout. Monitoring the content of macro- and microelements in fish meat is important for the consumer.

Keywords: freshwater fish, smoking, marinating, macroelements, microelements.

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INTRODUCTION

Fish meat is a valuable dietary component because of its exceptionally high nutritional value and desirable sensory attributes. It provides high quality protein and fat as well as a wide variety of vitamins. It is also a rich source of minerals (Khora 2013).

All minerals found in the human body are divided into two main groups: macroelements and microelements (Grzebisz 2011). Five macroelents, such as sodium (Na), potassium (K), magnesium (Mg), calcium (Ca), phosphorus (P), and four microelements, such as iron (Fe), manganese (Mn), copper (Cu), zinc (Zn) are crucial for the regulation of health functions in the human organism (Hosseini et al. 2014). Ca, P and Mg are major minerals involved in bone formation and health. Fe is the most abundant trace element in the human body and its insufficient daily intake results in anaemia. Mg, Mn and Zn are responsible for the regulation of the activity of several enzymes (Śmigielska et al. 2005, Taskaya et al. 2009). For the consumer, it is therefore important to know the content of macro- and microelements in fish meat.

Several studies have shown that the chemical composition of fish tissue can be affected by many factors, including genetic make-up, place of origin, availability and quality of food and the season. Moreover, variation in the chemical composition of fish is closely related to its migrations and changes associated with the spawning season (Sant'Ana et al. 2010, Usydus et al. 2011).

There is evidence that the content of macro- and microelements in fish depends on its species and feeding type (Łuczyńska, Brucka-Jastrzębska 2006). Differences in the chemical composition of fish muscle tissue, especially in terms of ash and minerals, can affect the processing and composition of fish products (Sant'Ana et al. 2010).

On the other hand, the nutritive value of fish can be affected by processing or cooking methods (gokoglu et al. 2004). These effects depend on the extent and duration of processing. Main factors resulting in a decrease of the food mineral content are separation processes, such as concentration or leaching, spill as well as a change of acidity. The impact of industrial and home-made processing on the mineral content of food is associated with mechanical treatment and leaching of water-soluble compounds as well as changes in the bioavailability of minerals. This bioavailability is usually significantly increased during hydrothermal processes, although the advantages obtained in this way can be offset by the quantitative losses of these compounds (Gawęcki 2011).

The aim of this study was to determine the content of ash and minerals (macro- and microelements) in freshwater fish flesh, as well as the quantitative changes during the production of smoked and marinated products.

MATERIALS AND METHODS

Research material

The research material consisted of 3 species of fish, farmed in the Carp Valley (Zator, Malopolska, Poland), common carp (*Cyprinus carpio*) called the Carp of Zator, rainbow trout (*Oncorhynchus mykkis*), and pike (*Esox lucius*). The fish farming was based on the semi-extensive system and nutrition. At least 70% of grain (wheat, barley, triticale, maize) used for feeding was obtained from the region.

A total of 6 fish specimens from each species were collected during the early spring season of 2012. The age of common carp was 2 years, and its weight was 1.5-2.0 kg; the age of rainbow trout was 0.5 years and its weight was 0.5-1.0 kg; the pike was 3 years old and weighed 2-3 kg. The fish were killed, washed, gutted, cleaned and divided into parts. The samples were then stored at - 80°C for no more than a month.

The samples were used to prepare smoked (common carp, rainbow trout, northern pike) and marinated (common carp, rainbow trout) products. They were produced according to traditional recipes.

Fish smoking

Pieces of fish meat were arranged in layers in a large bowl and cured in spices (salt, pepper, granulated onion and garlic, thyme and marjoram). After cold storage (4 - 8°C) for 12 h, the fish meat pieces were washed, dried and finally subjected to warm smoking for 4-6 h depending on the size (smoke was produced by burning wood from fruit trees, adding alder wood in the last stage).

Preparation of marinated products

Fish pieces were spiced with salt, pepper and seasoning for fish, coated in flour and bread crumbs, and then deep-fried in rapeseed oil until golden brown. Next, the fried pieces were loosely placed in jars and covered with hot vinegar marinade (vinegar and water in the ratio 1:3, sugar, mustard seeds, bay leaf, allspice and slices of carrots and onions). The filled jars were boiled for approximately 10 minutes.

Chemical analyses

A total of 18 samples of the raw fish meat (3 species) and 15 samples of fish products (smoked or marinated) underwent determinations. The ash content was tested using the gravimetric method, according to PN-ISO 936:2000 standard. Prior to mineral analysis, a 0.5 g of a homogenized sample was added to 5 ml of nitric acid (65%, Suprapur, Merck, Germany) and 1 ml hydrochloric (30% Suprapur, Merck, Germany). Wet mineralization was performed in a microwave system Multiwave 3000 (Anton Paar, Graz, Austria). The operational conditions were: power 800 W, a reaching time of 10 min, a hold time of 20 min, ventilation time of 15 min. Finally the macro- (Ca, Mg, K, Na, P) and the microelement (Fe, Zn, Mn, Cu) levels were measured using a spectrometer ICP-OES 7300 DualView (Perkin Elmer, USA), at the following wavelenghts: Ca 317.933 nm, Mg 285.213 nm, K 766.490 nm, Na 589.592 nm, P 213.617 nm, Fe 238.204 nm, Zn 206.200 nm, Mn 257.610 nm, Cu 327.393 nm. The detection limits were: Ca – 0.01 mg L^{-1}, Mg – 0.0016 mg L^{-1}, K – 0.02 mg L^{-1}, Na – 0.069 mg L^{-1}, P – 0.076 mg L^{-1}, Fe – 0.0046 mg L^{-1}, Zn – 0.0059 mg L^{-1}, Mn – 0.0014 mg L^{-1}, Cu – 0.0097 mg L^{-1}.

The quality of all the analyses was verified by making parallel analyses of Certified Reference Material IAEA-407 (Fish Homogenate). The recovery for the applied method was: Ca – 104.90% , Mg – 106.80%, K – 98.55%, Na – 98.32%, Fe – 105.48%, Zn – 102.36%, Mn – 95.74%, Cu – 103.05%.

Statistical analysis of the results

All the results were subjected to statistical analysis, using Statistica ver. 12.0 computer program. Normality of the results and homogeneity of variances were calculated using the Saphiro-Wilk test. Significance of differences was established using the Tukey's *post hoc* test. Significance of difference was established for $\alpha = 0.05$.

RESULTS AND DISCUSSION

The content of ash and minerals in raw fish

The samples for analysis of raw fish were made from homogenized fish flesh and the content of ash and minerals were determined directly in flesh of the different fish species. The ash content in the investigated raw fish ranged from 1.220% to 1.260%. The statistical analysis showed no significant differences between the fish species (Table 1). Compared to our results, a slightly lower ash content in the flesh of carp (0.74-1.17 g 100 g^{-1}, depending on the location of a fish farm) was reported by Tkaczewska and Migdał (2012), and significantly lower values (0.6 g 100 g^{-1} in carp and 0.8 g 100 g^{-1} in trout) were found by Usydus et al. (2011). Much higher amounts of ash (1.8-2.2 g 100 g^{-1}) were presented by Liu et al. (2004). Significant differentiation in the ash level in fish muscles depending on the site and conditions of farming as well as the chemical composition of fish feeds was found.

The content of all the examined minerals (except Ca and Mn) in raw fish was significantly different depending on the fish species (Table 2). Common carp and rainbow trout were abundant in Mg (301.6 mg kg^{-1}, 284.4 mg kg^{-1}, respectively) and K (3402.4 mg kg^{-1}, 3750.4 mg kg^{-1}, respectively). However, the levels of Na (655.6 mg kg^{-1}), Fe (6.490 mg kg^{-1}) and Zn (8.320 mg kg^{-1}) in

Table 1

The ash content (mean ± SD) in fish and fish products according to a species and technological process (%)

Sample	Fish species		
	common carp	rainbow trout	northern pike
Raw*	1.260 ± 0.040^{Aa}	1.230 ± 0.080^{Aa}	1.220 ± 0.080^{Aa}
Smoked*	1.650 ± 0.120^{b}	1.700 ± 0.150^{b}	1.340 ± 0.150^{a}
Marinated**	2.000 ± 0.050^{c}	1.440 ± 0.170^{a}	na

* n = 6 for each fish species, ** n = 3 for the process, ^{A} – mean values with the same letter superscript are not significantly different (α = 0.05) – differentiating factor: fish species, $^{a, b, c}$ – mean values with the same letter superscript are not significantly different (α = 0.05) – differentiating factor: technological process, na – not assayed

the flesh of common carp were approximately twice as high as in the flesh of rainbow trout (Table 2). Grela et al. (2010) found distinctly lower amounts of Fe (3.68-3.84 mg kg^{-1}) and a comparable content of Zn (7.38-8.35 mg kg^{-1}) in the muscle tissue of common carp. In a study conducted in Japan, lower levels of Zn and Fe were detected in samples of wild and farmed carp (Alam et al. 2002). In turn, a higher content of Fe in the muscle of carp from Lake Tokat (Turkey) was reported by Mendil and Uluozlu (2007). Common carp (from farm ponds supplied with water from the Rudawa River) studied by Łuszczek-Trojnar et al. (2011) had the Zn content from 10-17 mg kg^{-1}.

The results showed that the content of Mg in common carp muscles was more than twice as high as the values presented in the Tables of Composition and Nutritional Value of Food (Kunachowicz et al. 2005). The K and P levels in the muscles of rainbow trout were considerably lower than the values published by Kunachowicz et al. (2005). The pike muscles were characterized by a high content of Ca, with its amount twice as high as the value published in the Tables of Composition and Nutritional Value of Food (Kunachowicz et al. 2005).

The bioaccumulation of the microelements (Zn, Cu, Fe, Mn) depends on several factors, such as the age of fish, its weight and body length (Farkas et al. 2003, Łuczyńska, Brucka-Jastrzębska 2006, Łuczyńska et al. 2009). The Fe level in the muscles of pike (3.490 mg kg^{-1}) was significantly lower than obtained by Grela et al. (2010), who reported it to be in the range of 7.16-7.48 mg kg^{-1}. Simultaneously, a lower average Zn content (4.430 mg kg^{-1}) was found in the muscles of this fish species than the data presented by Grela et al. (2010) and Amundsen et al. (1997). It was also shown that the Cu content was below the limit of quantification (Table 2). However, some amounts of this microelement were reported in the studies of other authors (Alam et al. 2002, Djedjibegovic et al. 2012, Subotic et al. 2013). The muscle tissue of farmed carp studied by Alam et al. (2002) had the content of Cu at the level 0.33 mg kg^{-1}. Moreover, Mendil and Uluozlu (2007) and Subotic et al. (2013) detected a relatively high Cu content in the muscles of carp

Table 2

The content of minerals (mean ± SD) in fish and fish products dependening on a fish species and technological process (mg kg^{-1})

Mineral	Common carp			Rainbow trout			Northern pike	
	raw*	smoked**	marinated**	raw*	smoked**	marinated**	raw*	smoked**
Macroelements								
Ca	382.7 ± 90.24^{Aa}	550.0 ± 18.49^{b}	450.2 ± 35.51^{a}	454.9 ± 92.07^{Ab}	193.8 ± 33.46^{a}	490.3 ± 11.30^{c}	556.2 ± 389.5^{Aa}	229.3 ± 46.24^{a}
Mg	301.6 ± 2.190^{Cb}	367.4 ± 4.030^{c}	121.5 ± 5.530^{a}	284.4 ± 14.59^{BCc}	224.3 ± 0.300^{b}	176.9 ± 2.440^{a}	209.3 ± 4.480^{Aa}	205.7 ± 9.630^{a}
K	3402.4 ± 63.93^{Bb}	4300.8 ± 21.09^{c}	968.9 ± 22.07^{a}	3750.4 ± 26.69^{Bc}	3242.8 ± 4.840^{b}	1183.0 ± 16.05^{a}	2352.1 ± 66.74^{Aa}	3209.6 ± 177.6^{b}
Na	655.6 ± 11.79^{Ba}	4055.2 ± 38.29^{c}	3914.7 ± 27.17^{b}	329.5 ± 5.770^{Aa}	1566.4 ± 34.29^{b}	4441.5 ± 24.39^{c}	381.3 ± 2.370^{Aa}	1600.8 ± 66.50^{b}
P	2248.6 ± 54.67^{ABb}	3327.6 ± 47.77^{c}	1025.9 ± 2.730^{a}	2595.2 ± 86.47^{Bc}	2149.6 ± 27.77^{b}	1788.2 ± 26.71^{a}	1884.7 ± 208.5^{ABa}	2037.3 ± 53.52^{a}
Microelements								
Fe	6.490 ± 0.720^{Ba}	10.78 ± 0.670^{b}	9.630 ± 0.130^{b}	3.790 ± 0.020^{Aa}	4.010 ± 0.660^{a}	13.44 ± 0.020^{b}	3.490 ± 0.010^{Aa}	4.150 ± 0.110^{b}
Zn	8.320 ± 0.310^{Bb}	11.49 ± 1.040^{c}	5.500 ± 0.020^{a}	4.470 ± 0.040^{Aa}	7.860 ± 0.660^{b}	6.200 ± 0.010^{c}	4.430 ± 0.020^{Aa}	12.59 ± 0.120^{b}
Mn	0.130 ± 0.050^{Aa}	0.390 ± 0.010^{b}	0.460 ± 0.000^{b}	0.160 ± 0.020^{Aa}	0.010 ± 0.010^{a}	1.680 ± 0.080^{b}	0.170 ± 0.070^{Aa}	0.040 ± 0.070^{a}
Cu*	<LOD	0.480 ± 0.130^{b}	0.050 ± 0.000^{a}	<LOD	0.050 ± 0.020^{a}	0.490 ± 0.010^{b}	<LOD	0.060 ± 0.010

* n = 6 for each fish species, ** n = 3 for each process, <LOD – result below LOD (LOD_{Cu} = 0.0097 mg^{-1} L), $^{A, B, C}$ – mean values with the same letter superscript not significantly different (a = 0.05) – differentiating factor: fish species, $^{a, b, c}$ – mean values with the same superscript are not significantly different (a = 0.05) – differentiating factor: technological process

(1.02-1.08 mg kg^{-1} and pike (1.15-1.17 mg kg^{-1}). Łuszczek-Trojnar et al. (2011) and Subotic et al. (2013) also found a high content of Mn in raw carp. In rainbow trout, 0.54 mg Cu kg^{-1} and 5.94 mg Zn kg^{-1} were detected by Drąg-Kozak et al. (2011). The concentrations of macroelements in the fish we investigated were in the following decreasing order: K > P > Na > Ca > Mg in raw common carp as well as K > P > Ca > Na > Mg in raw rainbow trout and pike. Łuczyńska et al. (2009) published concentrations of nine macroelements in muscles of fish from the Masurian Great Lakes in the order of: K > P > Na > Mg > Ca, and Polak-Juszczak (2016) demnstrated an order of macroelements similar to ours achieved for common carp. In turn, the microelement content in muscle tissues of the fish followed the decreasing sequence: Zn > Fe > Mn, whereas in the study of Łuczyńska et al. (2009) and Polak-Juszczak (2016) it was Zn > Fe > Cu > Mn.

The content of ash and minerals in processed fish

The ash content in processed fish was significantly dfferent (except for smoked pike and marinated trout) from tha in raw fish (Table 1). The enhancement of the ash content in smoked carp (31%) and rainbow trout (38%) could be caused by the presence of minerals in the spices (granulated onion, salt, pepper, granulated garlic, thyme and marjoram) used for pickling the fish fillets. A higher level of ash in marinated carp as well as in rainbow trout compared to the raw material (59% and 17%, respectively) could be the effect of the addition of spices, coating fish pieces in breadcrumbs and flour, deep frying in rapeseed oil, and also the adding of vinegar marinade (with sugar, spices and slices of vegetables). The statistical analysis of the results revealed significant differences in the content of almost all the determined minerals in smoked fish compared to raw material (Table 2). The content of most elements was higher in smoked than in raw carp. In turn, significantly enhanced amounts of Na (1566.4 mg kg^{-1}) and Zn (7.860 mg kg^{-1}) were found in smoked rainbow trout compared to the raw fish. The K content in smoked rainbow trout was 3242.8 mg kg^{-1} and it was comparable to the results obtained by Gokoglu et al. (2004). However, significantly more K (3209.6 mg kg^{-1}), Na (1600.8 mg kg^{-1}), Fe (4.150 mg kg^{-1}) and Zn (12.59 mg kg^{-1}) was determined in smoked pike than in raw material. The smoked products were characterized by different levels of Cu, from 0.050 mg kg^{-1} in trout and 0.060 mg kg^{-1} in pike to 0.480 mg kg^{-1} in carp samples. The high Cu content in smoked carp slices may be explained by the amount of this microelement in spices used for curing the fish (Sulisburska, Kaczmarek 2011). Significant changes in the mineral content of marinated fish compared to the raw material were observed. A significant increase in the level of Na (3914.7 mg kg^{-1}) and Mn (0.460 mg kg^{-1}) was found in carp. Marinated rainbow trout contained markedly more Ca (490.3 mg kg^{-1}), Na (4441.5 mg kg^{-1}), Fe (13.44 mg kg^{-1}), Zn (6.200 mg kg^{-1}) and Mn (1.680 mg kg^{-1}) – Table 2. In marinated products, the amount of Cu in common carp was 0.050 mg kg^{-1} and rainbow trout 0.490 mg kg^{-1}. According to Sulisburska and Kaczmarek (2011), the high content of Cu could be due to the presence of spices, breadcrumbs and oil used for deep frying.

A very high level and a considerable increase of Na in every type of fish products should be emphasized. It is known that a recommended daily dose of Na is 575 mg day^{-1} and a tolerable dose is 2350 mg day^{-1}, thus any higher level of this element is unhealthy (if eaten much and often), especially for consumers with cardiovascular problems, hypertension, etc. (Polak-Juszczak 2016). The amount of Na in marinated rainbow trout was 4441.8 mg kg^{-1} (Table 2). Much lower levels of Na (335.54-607.00 mg kg^{-1}, depending on a culinary process) were reported by Gokoglu et al. (2004) in rainbow trout subjected to various thermal treatments (frying, cooking, baking, grilling). According to Polak-Juszczak (2016), the high content of Na (as NaCl) ensures longer shelf-life of products, although it has been shown that the salt content in fish muscle causes an increase of the oxidation of highly unsaturated lipids (Aubourg, Ugliano 2002). From a nutritional point of view, the Ca, Fe and Zn levels, both in smoked and marinated fish products, are of particular importance. The sequence of macroelements was different in smoked products than in the raw material, except for the dominance of K. The smoked fish contained macroelements in the following order: K > Na > P> Ca > Mg (common carp), K > P > Na> Mg> Ca (rainbow trout), K > P > Na > Ca > Mg (northern pike). In marinated products, the amounts of macroelements decreased as follows: Na> P > K> Ca > Mg. The decreasing order of the microelements was Zn > Fe> Cu> Mn for each fish species after smoking, whereas marinated products were the richest in iron (Fe > Zn> Mn> Cu).

While monitoring fish products subjected to different processes, Polak-Juszczak (2016) showed that the decreasing sequence of macroelement tended to be similar in semi-processed and ready products, except for the dominance of Na, and that the level of microelements in fish products was similar to the amounts in fresh fish.

The literature lacks data concerning the mineral content of smoked or marinated common carp and smoked pike. Usydus et al. (2009) studied popular fish products (including smoked rainbow trout) and determined the content of calcium and phosphorus to be 46 ± 18 mg and 247 ± 16 mg 100 g^{-1}, respectively. Polak-Juszczak (2016) investigted the effect of fish salting, pickling and smoking on the content of selected minerals. Considerable changes in the levels of minerals in salted and pickled herring were observed, whereas a smaller loss of minerals was observed during smoking. The undesirable changes in the content of K, Mg and P occurring in products during fish meat processing could have resulted from water loss (Polak-Juszczak 2016).

CONCLUSIONS

Of the three fish species examined, common carp was characterized by high amounts of Mg, K, Na, P, Fe and Zn. Rainbow trout was the most abundant in K and P, and pike was the richest in P. The levels of most macro- and microelements in processed fish differed significantly from those in raw fish meat, especially the content of macroelements in carp products (higher amounts of nearly all the determined elements). Smoking as well as marinating resulted in an increase of the sodium level, with the highest values obtained for marinated rainbow trout samples.

The practical aim of this study was to expand consumers' knowledge about the mineral content of freshwater fish (including some popular species) after processing (smoking or marinating).

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