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Biogenic amine and quality changes in lightly salt- and sugar-salted black carp (*Mylopharyngodon piceus*) fillets stored at 4 °C

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ABSTRACT

The effects of low salt and sugar dry-curing on the quality changes of black carp (*Mylopharyngodon piceus*) fillets stored at 4 °C were evaluated by sensory, physical, chemical, and microbiological methods. Fish samples were left untreated (control), or were dry-cured with 1.5% salt (T1) or 1.5% salt + 1.2% sugar (T2). Curing treatments reduced chemical changes reflected in HxR, Hx, pH, and total volatile base nitrogen (TVB-N); decreased cooking loss; and increased overall sensory quality of fish ($p < 0.05$) compared to untreated samples. Significantly lower values of cadaverine and putrescine were observed in T1 and T2 compared to the control after the 2nd and 4th day, respectively ($p < 0.05$). There were significant differences ($p < 0.05$) between T1 and T2 for pH, TVB-N, total aerobic counts (TAC), and sensory characteristics. Sensory characteristics were significantly correlated with TAC, TVB-N, putrescine, and cadaverine in all samples ($p < 0.01$).

1. Introduction

Black carp (*Mylopharyngodon piceus*) is one of the main freshwater fish species aquacultured in China and is mostly distributed in the middle and lower reaches of the Yangtze River and its affiliated lakes. The yield of black carp in China in 2012 was 494,908 tonnes. Owing to its rapid growth and relatively high market price, as well as the healthcare potential for biological control of nuisance aquatic mollusks, black carp has a higher economic value than other species of farmed fish. Hitherto, studies on black carp have mainly related to artificial breeding (Rothbard et al., 1997), aquaculture (Sun, Ye, Chen, Wang, & Chen, 2011), and biocontrol of mollusks (Venable, Gaudé, & Klerks, 2000). However, reports are scarce on the postmortem quality changes of black carp muscle.

High moisture levels, rich nutrient content, and microbial activity render fish an easily perishable food product. The spoilage of fish, accompanied with physical and chemical changes and microbial growth, is a very complex process. Numerous measures have been taken to improve fish quality and extend shelf life, such as high hydrostatic pressure (Erkan & Üretener, 2010), vacuum packaging (Lyhs et al., 2001), edible films coatings (Song, Liu, Shen, You, & Luo, 2011), and salting (Yanar, Çelik, & Akamca, 2006), among others. Salting is among the most popular one and its preservative effect, which also enhances the flavour, can extend the shelf life of fish by reducing water activity, decreasing opportunity for microbial attack, and enhancing functional properties of fish protein (Yanar et al., 2006). However, at high salt concentration the proteins may denature, resulting in stronger protein-protein bonds, muscle shrinkage, and dehydration (Thorarinsdottir, Arason, Geirsdottir, Bogason, & Kristbergsson, 2002). As consumer preference for prepared foods is increasing, the demand for lightly salted fish fillets is growing. It is thus meaningful to investigate how low levels of salt influence fish quality.

Sugar-salted fish products, termed *gravad*, are very popular dishes traditionally manufactured in Nordic countries (Lyhs et al., 2001). Sugar is also widely used in Chinese dishes, especially those in China's southeastern coastal cities. Sugar has been used to stabilize frozen surimi as a cryoprotectant (Sultanbawa & Li-Chan, 1998), protects fish myofibrillar proteins (Lee, 1984), and decreases biogenic amine accumulation in slightly fermented sausages (Bover-Cid, Izquierdo-Pulido, & Vidal-Carou, 2001), among others. Hong, Luo, Zhou, and Shen (2012) investigated the effects of brining with low concentration of sucrose combined with salt on the quality parameters of bighead carp fillets stored at 4 °C. However, limited information is available on the effect of sugar on the formation of biogenic amines (BAs) in fish.

The aim of this work was to evaluate the effects of low salt and sugar on the quality of black carp fillets by evaluating the sensory

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attributes, physicochemical quality, and microbial changes, as well as by determining biogenic amines of fillets during storage at 4 °C.

2. Materials and methods

2.1. Sampling, preparation and storage conditions

Twelve black carps (weight of 6750 ± 225 g, length of 80.08 ± 2.75 cm) were purchased from an aquatic product wholesale market in Beijing, China, in January 2013, and were immediately delivered to the laboratory alive. Fish were euthanized by concussion, scaled, gutted, deheaded, washed, and cut into small pieces (about 7–8 cm in length). Each piece was filleted into two sides, after which the fillets were left to drain on sterile stainless steel wire mesh for 3 min and then prepared for dry-curing.

Prior to performing this experiment, different curing concentrations were prepared and tested for consumer acceptability. Two acceptable curing concentrations were obtained (1.5% salt and 1.5% salt + 1.2% sugar) during this testing.

Black carp fillets were randomly divided into three groups. The untreated group (control, $n = 39$) was used as control. The treated groups were dry cured with 1.5% salt (T1, $n = 42$) and 1.5% salt + 1.2% sugar (T2, $n = 42$), respectively. Dry salt or salt-sugar mixture was carefully and evenly sprinkled on the surface of the fish fillets. The addition of salt and sugar was expressed as percentage of initial fillet weight. Ordinary commercial food grade salt (CNSIC, Beijing, China) and granulated white sugar (COFCO Tunhe, Xinjiang, China) were used for dry-curing. The treated and untreated samples were packed in polyvinyl chloride bags and stored in refrigerators at 4 °C. Samples of white dorsal muscle from each fish fillet were taken randomly for analysis at selected time intervals (0, 2, 4, 8, 12, 24, 36, and 48 h for post-mortem storage and 4, 6, 8, 10, 12, 14, and 16 days for long-term storage).

2.2. Methods

2.2.1. Nucleotide degradation products

Preparation of ATP and its degradation products followed the procedure described by Zhu, Luo, Hong, Feng, and Shen (2013). The prepared supernatant sample was filtered through a 0.22 μm membrane filter and analyzed for nucleotide degradation products using reverse phase high-performance liquid chromatography (HPLC) (Shimadzu, LC-10AT series, Kyoto, Japan) equipped with SPD-10A (V) detector, COSMOSIL 5C18-PAQ column (4.6ID × 250 mm) (Nacalai Tesque, Inc., Kyoto, Japan). The mobile phase was 0.05 M phosphate buffer (pH 6.8); flow rate was 1 mL/min; injection volume was 20 μL; detection wavelength was 254 nm. ATP and its degradation products were identified and quantified based on the commercially obtained standard ATP, ADP, AMP, IMP, inosine (HxR), and hypoxanthine (Hx). *K* value was calculated as follows (Saito, Arai, & Matsuyoshi, 1959):

$$K \text{ value } \% = [(\mathrm{HxR} + \mathrm{Hx})/(\mathrm{ATP} + \mathrm{ADP} + \mathrm{AMP} + \mathrm{IMP} + \mathrm{HxR} + \mathrm{Hx})] \times 100$$

2.2.2. pH value, electrical conductivity (EC), and total volatile base nitrogen (TVB-N)

A sample (10 g) of fish flesh was dispersed in 100 mL of distilled water and stirred for 30 min by an electric blender, after which the mixture was filtered. The filtrate was collected at room temperature (about 20 °C) for further analysis. The pH value of the filtrate was measured using a digital pH meter (Mettler Toledo FE20/EL20, Shanghai, China). The TVB-N was determined according to the method of Song et al. (2011). The EC was measured as described by Hong et al. (2012).

2.2.3. Cooking loss

A 15 × 15 × 10 mm sample of fish flesh was packed in polyethylene bags and then immersed in a water bath at 85 °C for 15 min. Samples were weighed before (W_b) and after (W_a) cooking. Cooking loss was calculated as follows:

$$\text{Cooking loss } \% = \frac{W_b - W_a}{W_b} \times 100$$

2.2.4. Texture and colour

Texture profile analysis (TPA) was determined using the method described by Zhu et al. (2013). The colour of black carp fillets was measured following the method of Hong et al. (2012).

2.2.5. Sensory assessment (SA)

SA was performed according to the method described by Ojagh, Rezaei, Razavi, and Hosseini (2010) with some modifications. The attributes of raw fish fillets and cooked fish fillets (steamed for 5 min at 100 °C) were evaluated by a panel of 9 trained members. Each panel member was asked to rate the appearance, odour, texture, and morphology of raw fish muscle, as well as the flavour, taste, and broth turbidity of cooked fish muscle. A rating scale of 1–5 points was used for each attribute, with 5 equivalent to top quality fresh black carp and 1 indicative of borderline freshness. The total scores for each sample were calculated by adding the average score given by each panel member for each of the seven attributes.

2.2.6. Microbiological analysis

Total aerobic counts (TAC) indicated as aerobic plate counts were determined following Song et al. (2011). Twenty-five grams of fish flesh was weighed aseptically and then homogenized with 225 mL of sterilized 0.9% physiological saline for 1 min. A series of 1:10 (v/v) dilutions were made; 1 mL of serial dilution was plated onto plate count agar; and the plates were incubated at 30 °C for 72 h. All counts were expressed as log10 CFU/g and performed in duplicate.

2.2.7. Biogenic amines (BAs)

Preparation and derivatization of BAs of black carp fillets followed the method of Ikonić et al. (2013). Identification and quantification of BAs were performed by using HPLC (Shimadzu, LC-10AT series, Kyoto, Japan) equipped with SPD-10A (V) detector, COSMOSIL 5C18-PAQ column (4.6ID × 250 mm) (Nacalai Tesque, Inc., Kyoto, Japan). Chromatographic separation was carried out using a gradient elution of Ammonium acetate (0.1 M, solvent A) and acetonitrile (100%, solvent B) as follows: 0 min, 50% B; 25 min, 90% B; 35 min, 90% B; 45 min, 50% B. Flow rate was 0.8 mL/min; column temperature was 30 °C; injection volume was 20 μL; and peaks were detected at 254 nm.

2.2.8. Statistical analysis

All analyses were run in triplicate (except microbiological analyses, which were performed in duplicate). Data were expressed as mean values accompanied by the standard deviation of means. The least significant difference (LSD) procedure was used to test for difference between means using SAS software (2008). The significance level was set at 5%. Pearson's regression analysis was performed to determine the relationship between sensory, physical, chemical, and microbial quality.