Influence of controlled freezing-point temperature on the freshness and taste of pickled and dried grass carp

LiYan Cai [a], JinQing Wan[b]

[1]College of Food Science and Technology, Shanghai Ocean University, Shanghai 201306, China;

[a]liyan-cai@outlook.com,*Corresponding author Email: [b]jqwan@shou.edu.cn

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Abstract. Pickling and drying can help aquatic products with their flavor which is highly related to temperature. Taking grass carp fillets as the research objects, quality changes were studied during pickling, drying and storage under controlled freezing-point temperature (CFT:-0.5±0.5℃) and 12 ±0.5℃. Results showed that total volatile basic nitrogen (TVB-N) value of grass carp fillets under CFT were significantly lower than that in 12℃. TVB-N value of fillets was only 29.84 mg N/100g under CFT after 75 days storage and reached 30.88mg N/100g under 12℃ only after 35 days storage. Inosine 5-monophosphate (IMP) of the fillets both increased firstly and then decreased smoothly and they all increased to the highest level on first day with the value of 169.30 mg/100g under CFT and 156.20 mg/100g under 12℃ respectively. During the storage, IMP of the fillets decreased much slower and maintained a higher level. Compared to fillets under 12℃, free amino acids and equivalent umami concentration (EUC) of fillets under CFT had significant increase ($P<0.05$). At the end of the storage, EUC of fillets was 1.568 MSG/100g under CFT and 0.898 MSG/100g under 12℃. Therefore, fillets under CFT had higher freshness and flavor and longer storage time.

1. Introduction

Grass carp (Ctenopharyngodon idella) is one of the four domestic fish species in China which is widely developed in food industry, but it is weak in fishery product amount and category. Most of fish was added in excess salt to extend shelf life but were not effectively dried to maintain quality after pickling, leading to sharply decrease on the safety of products and potential harm to consumers' health [1]. And lower salt content was proved to be effective on the stability of storage and sensory attributes [2].

Temperature has significant influence on the pickling, drying and preserving process of fish products, higher temperature resulted in higher loss of FAA, and lower IMP increase and worse flavor [3, 4]. Freezing-point is the temperature range between 0℃ and the limit at which food freeze. Generation of compounds containing nitrogen which are related to marine product decay could be reduced under CFT. It was reported that CFT can help fish with microorganism reproduction and quality changes [5]. Freezing-point technology could inhibit decay and increase concentration of amino acid related to taste [6] and help free amino acid increase [7].

Traditional pickling drying not only modifies flavor but also decreases water activity of products, inhibits decay and strengthens taste [8]. While there is much research on this subject [9, 10], study about freezing-point pickle drying on the attributes of fishery products was rare. Comparisons were made on the freshness and taste change of pickle drying grass carp fillets between the condition of 12℃ and freezing-point. The influence on pickling, drying and preserving of grass carp fillets via freezing-point tech was explored and the results provide basic theory for the application of freezing-point technology.

2. Material and Methods

2.1 Preparation of fish and Experiment design

Carps purchased from an aquatic product market in shanghai were immediately delivered to the laboratory alive. Fishes were scaled, gutted, beheaded, washed, backbone meat was taken and cut into fillets of 0.5-0.8 cm in thickness.

Prepared seasoning of 6% salt, 1% sugar, 6% ginger and 0.5% pepper was added to carp fillets, sealed in aluminum packages. They were separately pickled under 0.5±0.5℃ and 12±0.5℃ in two constant temperature humidity chamber with 70% humidity. The drying process was taken place in vacuum drying chamber which is designed by our laboratory. Specifically, carp fillets of group 1 were pickled for 1.5 days, drying for 1 day and stored for 35days under 12℃; group 2 were pickled for 14 days, drying for 1 day and stored for 75days under CFT.

2.2 Determination of the freezing point

Temperature was collected and recorded by temperature acquisition instrument. Put the platinum resistance near the tail, then stored in the -18˚C refrigerator. After 6 hours, draw the frozen curve to determine the freezing point and the range

2.3 Determination of the moisture content

Refer to GB / T 5009.3-2010, the determination of moisture content in food: samples were weighed accurately, placed in glass container, put in a 105 ˚C oven, and weighed again after 6 hours.

2.4 PH value

Referring to GB/T 9695.5-1988, 10g samples and 90ml pure water were fully mixed in conical flask and were determined by acidometer after 30 minutes.

2.5 Total volatile basic nitrogen

Using semi-micro Kjeldahl method for the determination of the total volatile basic nitrogen content. Samples about 5g were weighed accurately, smashed and put directly in FOSS alimentary canal, added light magnesium oxide about 0.6g.

2.6 ATP-related compounds

ATP-related compounds were determined by HPLC based on the method of Yokoyama [11].

2.7 Free amino acids

Referring to YAMAGUCHI12, free amino acids were determined by HPLC.

2.8 Equivalent umami concentration

A synergistic effect of amino acids and nucleotides MSG equivalents are synergistic effects of amino acids and nucleotides [14]. Calculated as follows:

$$EUC = \sum a_i b_i + 1218\,(\sum a_i b_i)\,(\sum a_j b_j).$$

EUC is monosodium glutamate in units of g MSG / 100g; a_i is the amount of flavored amino acids (Asp and Glu); b_i is the relative freshness coefficient of flavored amino acids relative to MSG (Glu is 1, Asp is 0.077); a_j is the amount of 5'-nucleotides; b_j is the relative freshness coefficient of 5'-nucleotide relative to IMP (5'-IMP is 1, 5'-AMP = 0.18); 1218 is the synergistic constant.

2.9 Data analysis

Using Excel and Origin 9.1 Pro on the data analysis. The indicators take parallel samples 3, significant differences were analyzed by one-dimensional ANOVA, with a significance level of $P < 0.05$.

3. Results and Discussion

3.1 Moisture content

Dry end, calculated at 12℃ dry fish fillet moisture content of 17.95%, ice dry filament moisture content of 18.24%.

3.2 Results of freezing point

The freezing point was -1.0 degree, namely its ice temperate range is from -1.0˚C to 0℃. The

temperate of vacuum devices was strictly controlled (0~-1.0) range.

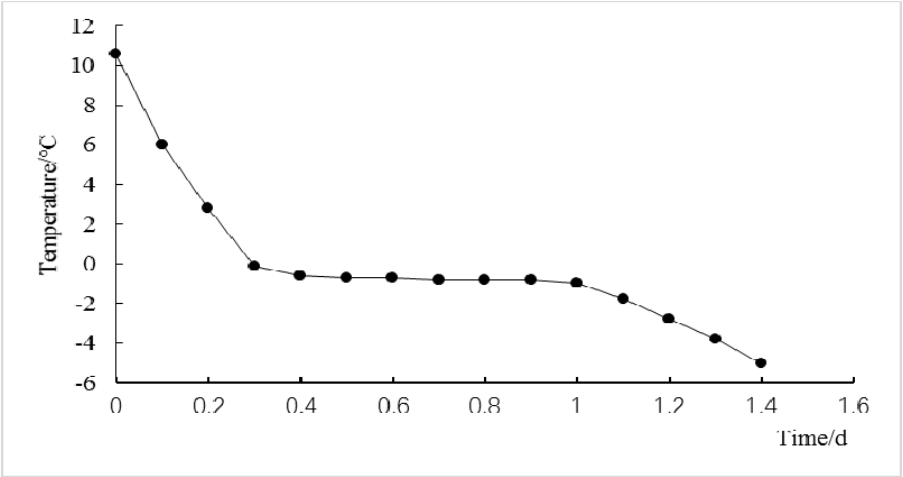

Figure 1 Freezing curve of Grass Carp

3.3 pH changes

Table 1 shows the change in fish pH at 12℃ and ice temperature. The pH value of the fresh fish was 6.24. At the end of the drying, the pH value of 12℃ and CFT was 6.28 and 6.27, respectively. There was no significant difference in the fillets at two temperatures during pickling and drying. With the increase of storage time, the alkaline substances in fish are increasing and the pH value gradually increases. Therefore, the ice temperature is more conducive to maintaining fish pH.

Table 1 pH value of Grass Carp fillets

Stage	Time	12℃ pH	Time	CFTpH
Pickled	0d	6.24±0.02a	0d	6.24±0.02 a
	1.5d	6.26±0.07	7d	6.32±0.15
			14d	6.28±0.06
Dry	1d	6.28±0.05 a	1d	6.27±0.08 a
Storage	10d	6.49±0.13 a	10d	6.29±0.05 a
	20d	6.68±0.05 a	35d	6.37±0.02 a
	35d	6.86±0.03 b	65d	6.46±0.03
			75d	6.50±0.05

3.4 Changes in fish TVB-N

GB2733-2015 provides fresh water fish and shrimp TVB-N≤20mg N / 100g as a qualified product, however, referring to SCHERER et al [26], pickled fish products TVB-N ≤ 15 mg N / 100g was taken for the first grade, TVB-N ≤ 20 mg N / 100g was taken for the second grade, more than 30 mg / 100g for corruption.

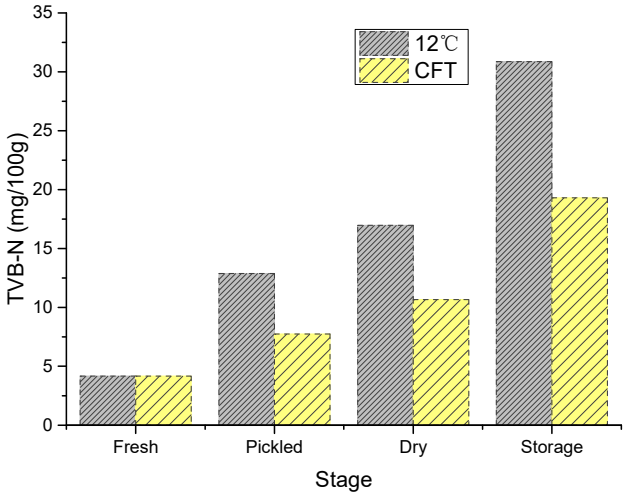

Figure 2 TVB-N value of Grass Carp fillets

The results of the changes in fish TVB-N at ice temperature and 12℃ were shown in Picture 1. It can be seen that different temperatures have a significant effect on TVB-N. Ice cream fish pickled 14 days better than 12℃ pickled 1.5 days, dry ice fish fillet is still a grade, 12℃ fish for the second grade. In the later storage, the TVB-N growth was also slower in the ice storage, and the TVB-N was 17.99 mg N / 100g at the 35th day of the storage of the ice fish, and the TVB-N For 30.88mg / 100g has been corrupt, and ice-fish fillet storage on the 75th day when approaching corruption. Therefore, the ice temperature to maintain fish freshness, delay the corruption has a significant advantage.

3.5 Changes of ATP and IMP in Fish

ATP and its associated species are the main flavor substances of fish, ATP degradation under the action of endogenous enzymes, the degradation pathway is ATP → ADP → AMP → IMP → HxR → Hx, where IMP is the most dominant in the nucleotides Material, can greatly enhance the fish flavor.

ATP and IMP of fillets changes shown in Figure 3. In 1 day, the ATP content of the fish was rapidly decomposed under both two temperatures, IMP was significantly increased, reached the highest value, the IMP was 169.30 mg / 100g under CFT and 156.20 mg / 100g under 12℃, the difference was significant. With the increase of time, the ATP of the fish was stable at the two temperatures, and the ATP decomposition of the fish was slower in the ice temperature environment, and the IMP was also higher than that of the 12℃ fish fillet. The difference was significant at the 35th day of storage at the 35th day, and the difference was significant at the end of the ice storage (ie, 75 days), the IMP dropped to 134.74 mg / 100g , Still higher than 12℃ storage 35 days IMP. The results showed that the temperature had a significant effect on the IMP of the dried fish fillet, and the ice temperature could keep the dried fish fillets better.

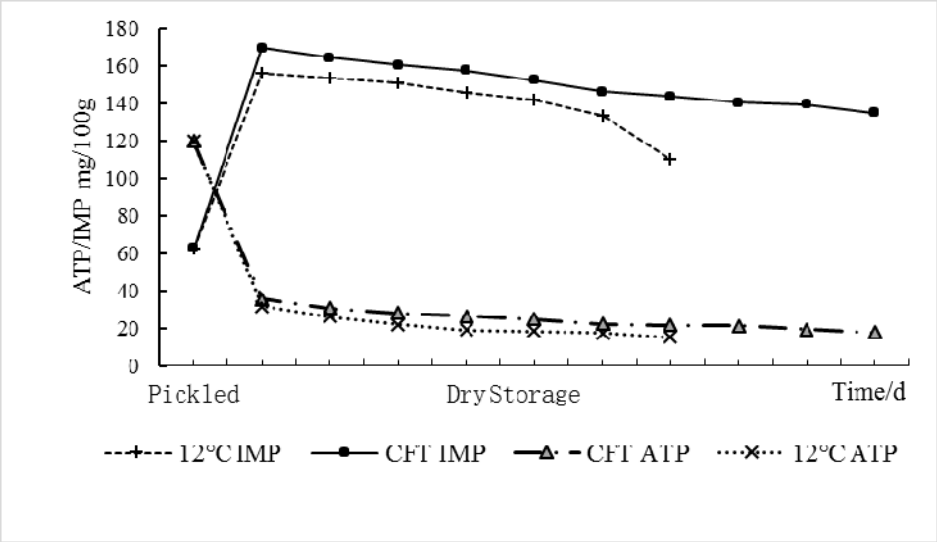

Figure 3 ATP/IMP of Grass Carp fillets

3.6 Changes in free amino acids

Table 3 lists the changes in the total flavored free amino acid (FAA) content and the total amount of 18 free amino acids, and the aspartic acid (Asp), glutamic acid (Glu) and glycine (Gly) are sweet, Fresh taste, sweet taste, isoleucine (Ile), leucine (Leu) and histidine (His) slightly bitter taste, but can increase the complexity of aquatic products taste, histidine will form some Meaty features.

Aspartic acid and glutamic acid is the main flavor of aquatic products, and other flavor substances have synergistic effect. Asp population showed a decreasing trend, and the difference between pickling and storage was significant. The Glu had the largest increase among six main FAA, after 35 days storage, Glu under CFT had the growth of 168% and that under 12℃ had the growth of ongly 107%. Due to longer storage under CFT, the fillets had the maximum growth of 203% which enhanced the taste of fish significantly. The effect of temperature on Gly was slightly different, and the effect of pickling and drying on Gly was the same, but the difference was gradually observed at storage time.

The contents of the three bitter amino acids were increased at 2.25 and 3.19 times, respectively, and the Ile and Leu were well below the threshold and the His amplitude was more significant At the end of storage, the thresholds were 20.60% and 54.90%, respectively. It can be seen that the ice temperature environment can help to suppress the bitterness of fish fillets.

The average increase of FAA was 12.37%, 12.01% at 12℃ and 1.67% and 2.29%, respectively, and the effect of dry on FAA was not significant The At the 35th day of storage, the total amount of FAA increased by 30.44%, 12℃ increased by 25.17%, and the increase of FAA was 41.70% on the 75th day. The freezing temperature was favorable for the formation of fish flavor.

Table 2 Free amino acid of Grass Carp fillets (wet basis)

Unit: mg/100g

T	Phase	time	Asp	Glu	Gly	Ile	Leu	His	FAA	Increase (%)
12℃	Pickled	0d	1.36	3.01	96.59	16.09	4.06	8.85	400.18	/
		1.5d	1.01	3.36	120.16	29.98	8.47	16.02	451.44	12.01
	Dry	1d	0.89	4.63	124.11	33.86	12.04	18.28	457.41	14.30a
	Storage	10d	0.87	4.71	121.92	38.47	14.01	23.38	475.50	18.82b
		20d	0.75	5.81	129.86	42.17	16.11	22.66	489.11	22.23b
		35d	0.56	6.26	130.11	45.54	24.73	24.12	500.98	25.17b
CFT		0d	1.36	3.01	96.59	16.09	4.36	8.85	400.18	/
	Pickled	7d	1.31	5.44	112.55	25.8	10.80	17.45	467.83	16.92
		14d	1.31	6.01	120.03	26.80	11.09	17.64	481.66	20.37
	Dry	1d	1.33	6.77	120.44	30.06	13.74	18.17	488.35	22.04b
		10d	1.13	7.00	129.04	32.06	15.42	20.56	500.05	24.96a
	Storage	35d	0.92	8.07	140.11	39.13	24.82	20.54	522.01	30.44a
		60d	0.79	10.11	146.33	42.18	28.01	26.25	530.04	32.25
		75d	0.70	9.12	149.22	40.43	29.21	30.98	567.06	41.70

Note: Asp, Glu, Gly, Lle, Leu, His thresholds are 100, 5, 130, 90, 190, 20 mg / 100g, respectively.

3.7 Fish EUC changes

The fish gourmet equivalent at different temperatures is shown in Table 5. From the table, it was found that the EUC of fish fillet was increased at 239.31%, the EUC of the 12℃ was 194.27%, and the EUC was 383.59% at the end of the pickling. To ensure the freshness of fish under the premise of the ice on the taste of fish has a significant role in upgrading. The EUC of the fillet was increased by 0.129 g MSG/100g and 0.217g MSG/100g, respectively. The EUC was 1.498g MSG/100g, and the EUC was 1.567g MSG/100g at the end of storage, the growth of EUC was 0.168 g MSG/100g before and after storage, and the change of EUC was not obvious at 12℃, and 0.001g MSG/100g was decreased before and after storage. Therefore, the ice temperature to enhance the taste of fish has a significant effect, which pickled the most significant effect. In addition, the EUC value of the table over time, sometimes high and low fluctuations in the phenomenon, because the IMP and free amino acid changes caused by the synchronization.

Table 3 Changes in EUC of Grass Carp fillets (wet basis)

AMP、IMP、ASP、GLU unit: g/100g EUC unit: g MSG/100g

Phase	12℃						CFT					
	T	AMP	IMP	ASP	GLU	EUC	T	AMP	IMP	ASP	GLU	EUC
Pickled	0d	0.0321	0.0624	0.0013	0.0030	0.262	0h	0.0321	0.062	0.0014	0.0030	0.262
	1d	0.1621	0.1562	0.0010	0.0033	0.771	1d	0.1652	0.1693	0.0014	0.0035	0.889
	1.5d	0.0603	0.1511	0.0010	0.00336	0.682	7d	0.0567	0.1643	0.0013	0.0054	1.183
							14d	0.0502	0.1604	0.0013	0.0060	1.267
Dry	1d	0.0588	0.1457	0.0008	0.0046	0.899	1d	0.0473	0.1574	0.0013	0.0068	1.396
Storage	10d	0.0522	0.1420	0.0009	0.0047	0.886	10d	0.0502	0.1522	0.0011	0.007	1.399
	20d	0.0406	0.1331	0.0008	0.0058	1.009	20d	0.0379	0.1462	0.0010	0.0074	1.397
	35d	0.0322	0.1101	0.0006	0.0006	0.898	35d	0.0363	0.1437	0.00092	0.0081	1.498
							50d	0.0300	0.1403	0.00082	0.0088	1.576
							60d	0.0287	0.1393	0.0008	0.0101	1.800
							75d	0.0260	0.1348	0.00007	0.0091	1.567

4. Conclusion

Compared with the change of quality of grass carp in two different temperatures, the ice temperature is beneficial to maintain the acidic environment of fish, which is beneficial to maintain the freshness of fish fillets, curb corruption and prolong the storage period. IMP is the main flavor material of fish, all reached the highest value in the first day of pickling, in which the ice pickling and storage is conducive to the promotion and maintenance of fish IMP. Compared with the free amino acid content, the fillet grew significantly in the ice temperature environment, and the difference of Glu was the most obvious, and the Glu was 3 times of the threshold at the end of ice storage, which could significantly enhance the taste of fish. The increase of free amino acid storage at the end of the ice field was 41.70%, while that at 12 ℃ was only 25.17%. Compared with the change of MSG, the fish EUC was significantly higher than that of 12 ℃. In summary, the cold pickled dry temperature is conducive to delay the fish and the fish can make fresh fish delicious.

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