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Effects of freeze-thawing pretreatment on heat pump drying quality of tilapia fillets

Li Min*, Guan Zhi-Qiang, Wu Bao-Chuan
College of Engineering, Guangdong Ocean University,Guangdong, 524088, (CHINA)
E-mail: yuefeilimin@126.com

ABSTRACT

Freeze-thaw is conducive to water exudation and removal, and it may lead to the mutant in organization structure. In order to understand the regular rule of how freeze-thawing pretreatment affects the heat pump drying quality of tilapia fillets, comprehensive experiments of tilapia fillets dried by heat pump were operated with 4 variable factors of freeze-thawing pretreatment consisting of freezing temperature, melting temperature, freezing time and melting time. Moreover, optimal matching was measured by response surface. The result illustrates that compared samples without freeze-thawing pretreatment, those under appropriate freeze-thawing pretreatment would obtain effective improvements of diverse index. Under repeated orthogonal optimization of diverse index, the best condition of freeze-thawing pretreatment that could be obtained is under -32℃ as the freezing temperature, 20℃ as the melting temperature, 1.0h as the freezing time and 87min as the melting time. And under such condition, if the tilapia fillets were dried with 0.30±0.02 moisture content of dry basis for 10 hours, the Ca^{2+}-ATPase activity of dry products of tilapia fillets was 2.35μmolPi/mgprot/h, and the rehydration rate of that was 37.22%, the rigidity was 9.7N, and the comprehensive grade was 91.191, as a result of which, compared with those samples without freeze-thawing pretreatment, the comprehensive grade ascended by 22.5%. Therefore, the conclusion can provide reference for updating the freeze-thawing pretreatment technology of aquatic products like tilapia fillets.

KEYWORDS

Tilapia fillets; Freeze-thawing pretreatment; Heat pump drying; Drying quality; Comprehensive score.

INTRODUCTION

Drying as a kind of way for originally processing can play an indispensable role in decreasing the cost of storage of food, increasing its shelf life and change the taste of food, which is also a common processing way of tilapia fillets. Hot air drying, a common way, may lead to some changes such as the denaturation of the color, taste, aroma and protein and fat oxidation because of its overlong drying time and over high drying temperature, so that the drying method cannot be controlled easily under an appropriate drying condition for reducing the drying time of tilapia fillets and improving the product quality at the same time. Freeze-thawing, working as the pretreatment technology, has already got primary application. Guo Ting[1] used freeze-thawing pretreatment to work on sweet potatoes, which made them have decreasing hot air drying time with increasing times of freeze-thawing. Ramírez C[2] used freeze-thawing circulation during the pretreatment of apple pieces, which discovered that freeze-thawing can make huge damage to the structure of apple cells by enlarging the cell cavity, so that the drying speed could be accelerated. Urrutia Benet G[3] compared the fresh tomatoes with tomatoes after freeze-thawing circulation and then concluded that, after freeze-thawing circulation, the appearance of tomatoes got changes and the extent of enzymatic browning was larger due to the big loss of water inside. Long Wu[4] made an experiment that eggplant was frozen-dried after freeze-thawing pretreatment, and he found that the quality of the product of absorbing water again after freeze-drying was better than that of traditional product. Ando H[5] let carrots experience osmotic dehydration during freeze-thawing circulating pretreatment for protecting their soft character and succeeded to get the expectant results. Grimi N[6] compared two kinds of sugar beet under pulsed electric field(PEF) and freeze-thawing pretreatment respectively and he discovered that only under the latter circumstance, was the fracture pressure value of the sugar beet's tissue after pretreatment the lowest and was the degeneration degree of fracture organization the highest. Oszmianski J[7] indicated that freeze-thawing process made the best protection for L - ascorbic acid、anthocyanidin and procyanidine by exploring the protective agent of polyphenol compounds during the strawberries' freeze-thawing process. And the results mentioned above illustrate that it is easier and better for biological tissues of fruit and vegetables or active porous medium to get dehydrated and dried and maintain a quite high level of processing quality. In the meantime, results by experimenting on the meat products like cold working aquatic products with freeze-thawing pretreatment demonstrate that, freeze-thawing process would make some extent damage to food's tissue and structure[8], but it is not common in the reports that freeze-thawing process can be applied into the dry working meat products.

Therefore, if freeze-thawing pretreatment can be used into the dry machining process of aquatic products, can reasonable match of freeze-thawing conditions play an effective role in quickening dehydration and improving the quality of the dry products simultaneously? And in this article, the original materials are tilapia fillets, which helped us find out the rules between the variation of freeze-thawing parameters and the variation of the quality of the tilapia dry products by changing and optimally matching the freeze-thawing parameters. And, finally, a brand-new method of drying pretreatment is discussed, in order to make examples for enhancing the dehydrating and drying pretreatment process of meat products like aquatic products.

MATERIAL AND METHODS

Experimental materials and equipment

Fresh tilapia, bought in Zhanjiang Dongfeng Market, whose weight was about 1kg each. Reagents: Adenosine triphosphatase assay kits and Coomassie brilliant blue G250 protein fast stain kits from Nanjing Jiancheng Bioengineering Institute.

Self-built heat pump drying device[9]:3P power, temperature -20-80℃, humidity 20%-80%;HHS-type electric heated water bath, Shanghai Industrial Co.,Ltd. Boxun medical equipment factory;DZF-6050 vacuum oven, Shanghai Jing Hong Laboratory Instrument Co.,Ltd.; BD-730LT-86L-I ultra-low temperature freezer, Qingdao Haier Group;FYL-YS-50L incubator, Beijing Fuyi Electric Co.;T-18 homogenizer, Germany IKA group;GL-10LMD refrigerated centrifuge, Hunan Xingke Scientific Instrument Co.,Ltd.; Sigma 1-14 high-speed desktop centrifuge, Germany Sigmal Company;UV-8000 UV-visible spectrophotometer, Shanghai Yuanxi Analysis Instrument Co.,Ltd.;CR-10 handheld colorimeter, Japan Konica Minolta Holdings,Inc.;TMS-PRO texture analyzer, U.S.FTC Company;AY120 analytical balance, Shimadzu Corporation.

Experimental methods

Process flow **:**Raw fish — section — weighing — freezing — thawing — weighing —heat pump drying — determining index

Points in operation**:**

Section：A commercially available fresh tilapia（weight was about 1kg）was killed quickly， the meats slices were taken after removing scales,head and tail, innards[10]. And then it was required to control the standard of the fish fillet is 100mm×50mm×5mm each（weight was about 30g）. After that, external water of fillets was fully absorbed by absorbent paper after cleaning,before being weighed standby.

Fish fillets freezing and thawing: According to the experimental conditions, the cryostat's and the incubator's temperature were set to a predetermined value, and the fish fillets will be loaded into the polyethylene bags sent to the cryostat later for being frozen for a period of time. Afterwards, the fish fillets would be moved into the incubator for thawing for a period of time.

Heat pump drying：For tilapia fillets with 5mm thickness, because there would be a better drying quality under 45 ℃ as the heat pump temperature, 2.5m / s as wind speed, and 30% humidity conditions around，heat pump drying tested the tilapia fillets under this circumstance[10]. Firstly, the parameters of heat pump drying device was reqired to be adjusted to predetermined value of the test requirements. Secondly, after removing the frozen-thawed fish fillets from polyethylene plastic bags, absorbent paper gently and repeatedly suck the seeping water on the surface and fillet weight would be measured accordingly. Finally, move the fish fillets into the the clean barbed wire tray of heat pump oven afterwards. Then, the fish fillets would receive drying treatment.

Experimental scheme design

The experimental single-factor test set-up

With the heat pump drying conditions and fish size unchanged, it was investigated that how is the impact of freezing time, freezing temperature, melting time, melting temperature of the certain freeze-thaw process conditions to the heat pump drying performance for tilapia fillets. The value range of each parameter is determined based on the results of pre-test, freezing time was set between 0.5h and 2.5h,freezing temperatures was set between-10℃and-50℃,melting time was set from 1.0h to 3.0h,melting temperature was set from 5℃to25℃. Additionally, fish fillets quality would be measured at intervals of 1 hour during the drying process, moisture content of the dry basis was calculated. If the moisture content of dry basis reached 0.30 ± 0.02g/g, the drying process would be stopped. Moreover, the experimental group without freezing-thawing pretreatment worked as the contrast group.

Response surface optimization design

Combined with the experimental results of the single factor,chosen Box-Behnken methodology to design the models and select the. frozen time, frozen temperature, melting time, melting temperature as influencing factors. The response values were composite score values of rehydration rate,activity of Ca2+-ATPase and hardness for four factors and three levels of response surface experiments,which were analyzed by Design Export8.0 software for processing data and regression analysis. The design of the coding were shown in TABLE1.

TABLE 1 : Variables and levels in Box-Behnken central composite design

Factors	Level		
	-1	0	1
A(frozen temperature/**℃)**	-20	-30	-40
B(melting temperature **/℃)**	15	20	25
C(frozen time/**h)**	0.5	1.0	1.5
D(melting time/**h)**	1.0	1.5	2.0

Indicators and evaluation methods

Determination of moisture content

The direct drying method in Chinese Standard GB 5009.3-2010 is adopted: Tilapia fillets are put in a 105°C thermostatic oven for drying till constant mass and then weighed to yield the dry-matter mass of tilapia fillet. The wet-basis moisture content (W_0) is calculated as follows:

$$w_0 = \frac{m_0 - m_i}{m_0} \qquad (1)$$

where m_0 is the mass of tilapia fillet moisture and m_i is the mass of tilapia fillet dry matter in grams.

Determination of product rehydration rate

The preliminary experiment indicates that product rehydration rate does not basically change after rehydrating for more than 40 min, so we chose 40 min as the rehydration time. Use filter paper to blot up the surface moisture of the rehydrated product repeatedly, and then weigh its mass. The determination procedure is as follows: Weigh dried tilapia fillet (M_q), put into 40°C thermostatic water bath boiler, rehydrate (40 min), take out and filter dry, remove surface moisture, and weigh the mass (M_h); the calculation formula for product rehydration rate (w_f) is

$$w_f = \frac{M_h - M_q}{M_q} \times 100\% \qquad (2)$$

Determination of myofibrillar protein activity of Ca^{2+}-ATPase

The determination of dried tilapia myofibrillar protein Ca2+-ATPase activity using the enzyme ATP test kit. The method was agreed with that of literature[11].

Determination of hardness

Hardness, representing the force that can deform the fillets to a certain extent, would be larger if the fillets can be ruminated more difficultly. And texture analyzer was used to determine the tilapia fillets' hardness after rehydration and cylindrical probe with 5mm diameter was used. Moreover, the determining speed was 1mm/s, and 50% of the samples were deformed. The tilpia fillets with different treatments would be used in later test under the same condition of rrhydration.

Comprehensive scoring methods

Comprehensive weighted scoring methods were used for dealing with 3 indexes that consist of rehydration, myofibrillar protein (Ca^{2+}-ATPase) and dry products' hardness after rehydration and the results were served as response values for response surfaced analysis. For rehydration rate and activity of Ca^{2+}-ATPase, the greater these values, the better quality of dried products. The hardness of rehydrated fillets was greater than that of fresh (7.4 ± 0.55 N). Generally, it's better for dried products the property of them after rehydration are close to that of fresh. So the smaller of hardness, the better. Out of 100 points in total, the scores of rehydration rate and activity of Ca^{2+}-ATPase both set at a = 35 points, and hardness at a = 30 points.

For rehydration rate, and activity of Ca^{2+}-ATPase indexes, calculated as follows:

$$Y_i = a \cdot \frac{W_i}{W_0} \tag{3}$$

For hardness, calculated as follows:

$$Y_i = a \cdot \frac{W_0}{W_i} \tag{4}$$

Where, Y_i is the weighted score for the index; *a* is weighting of scores for the indicators; W_0 represents the optimum value in this set of experiments; W_i is the actual measured value for each test index.

RESULTS AND DISCUSSION

Preliminary experiment

Effects of different freezing time

Fixed melting time was 2.0h、freezing temperature was at -40℃and melting temperature was at 25℃,and under this condition, freezing-thawing pretreatment of fish fillets were using different freeze time ---0.5h、1.0h、1.5h、2.0h and 2.5h respectively,to investigate the effects of different freezing time on the drying quality of fish fillets. The results are shown in Figure 1.

As can be seen in Figure 1,Along with the increase in freezing time, the rehydration rate and activity of Ca2+-ATPase of dried tilapia fillets showed a trend of first increase and then decrease. When freezing time was 1.0h,the rehydration rate had a maximum of 30.07%, Ca^{2+}-ATPase activity had a maximum value of 2.26μmolPi/mgprot/hour, and with the control group had significant difference ($p<0.05$). Rehydration properties depended on the product shrinkage, porosity and degree of cell damage[12]. Possiblely under the conditions of freeze time 1.0h,The fish fillets had the highest porosity, were in favor of drying and rehydration process reabsorption of water. The test also found that, heat pump drying time was the shortest under 1.0h freezing time condition, so the protein thermal denaturation was the minimum, It was better to keep the Ca^{2+}-ATPase activity than the others. The degree of metamorphism of fish itself and microbial breeding probability may be much more serious under the shorter time freezing conditions, so degree of the protein denaturation of the 0.5h frozen pretreatment drying samples was slightly higher than the 1h'. Along with the extension of the freezing time the hardness showed a tendency the first drop than increasing. 1.0h Freezing time had minimum hardness, and the control group were significantly different ($p <0.05$).

Effects of different melting time

Fixed freezing time was 1.5h、freezing temperature was -40℃and melting temperature was 25℃,and under this condition, freezing-thawing pretreatment of fish fillets were carried out in different melting time--- 1.0h、1.5h、2.0h、2.5h and 3.0h respectively,to investigate the effects of different melting time on the drying quality of fish fillets，the results were shown in Figure 2.

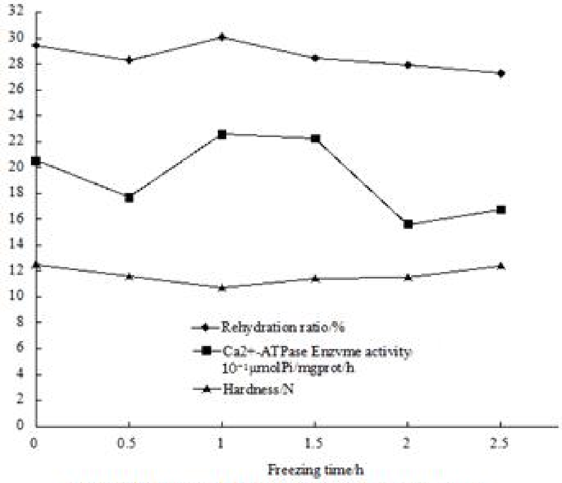

Figure 1 Effect of freezing time drying quality (0h-Contrast)

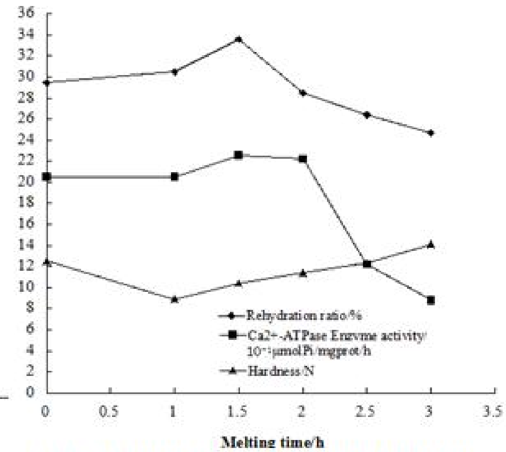

Figure2 Effect of melting time to drying quality (0h-Contrast)

As can be seen in Figure 2,The rehydration ratio showed a trend of first increasing and then decreasing, and reached the maximum value in the melting time of 1.5h. The activity of Ca^{2+}-ATPase also showed a similar tendency. While the hardness was displayed along with prolonging the melting time and gradually increasing trend. This may be due to along with the increasing of melting time,backward stiff phenomenon of fish fillets is exacerbation[13],Resulting in a more compact structure, dry products rehydration rate reduction and increase hardness. At the temperature of 25℃ melting condition, too long or too short melting time would lead to an increase in drying time. At the same time, prolong the melting time also increases the possibility of microbial growth and oxidation denaturation of proteins and other biological macromolecules, so that the Ca^{2+} -ATPase activity was lost.

Effects of different freezing temperature

Fixed freezing time was 1.5h、thawing time was 2.0h and melting temperature was 25℃. Under this condition, freezing-thawing pretreatment of fish fillets were carried out in different freezing temperature (-50℃、-40℃、-30℃、-20℃ and -10℃ respectively),to investigate the effects of different freezing temperature on the drying quality of fish fillets, the results were shown in Figure 3.

As can be seen in Fig3,the rehydration rate and Ca^{2+}-ATPase activity of stem increased first and then decreased with the decrease of frozen temperature, at the temperature of -10℃ and the temperature of -50℃,the Ca^{2+}-ATPase activity decreased by 36.6% and 31.3% compared with the control group. Hardness presents the first decrease and then increase phenomenon.-10℃freezing temperature may be frozen insufficiency, resulting in melted organization limp collapse and aggravate the protein denaturation. While -50℃freezing temperature weakened stripping bound water, resulting in longer drying time and bigger thermal denaturation of protein.

Effects of different melting temperature

Fixed freezing time was 1.5h、thawing time was 2.0h and freezing temperature was -40℃, under this condition, freezing-thawing pretreatment of fish fillets were carried out in different melting temperature of 5℃、10℃、15℃、20℃ and 25℃ respectively,to investigate the effects of different melting temperature on the drying time of fish fillets, the results were shown in Figure 4

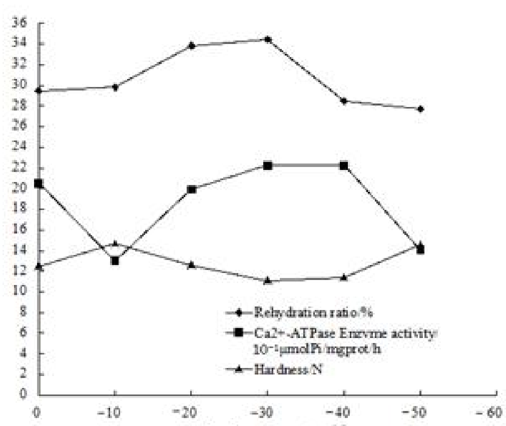

Figure3 Effect of freezing temperature to drying quality (0℃ – contrast)

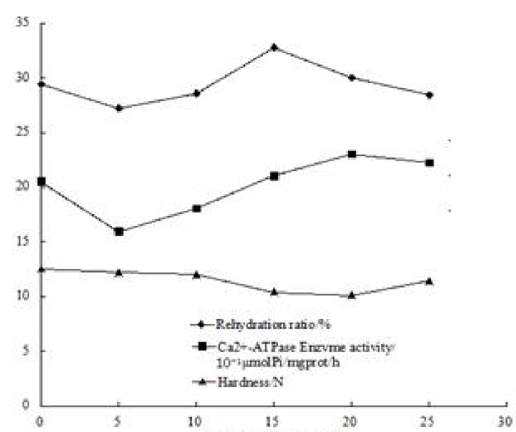

Figure4 Effect of thawing temperature todrying quality (0℃ – contrast)

As can be seen in Fig4,rehydration ratio has a maximum value at 15℃ melting temperature,the Ca2+-ATPase activity of samples had a higher value between 15 to 25 ℃ melting temperature. The activity of Ca^{2+}-ATPase value was lower than that of contrast when the melting temperatures below 15℃. Hardness of tilapia dried goods was closer to fresh under moderate melting temperature condition. With the increase of the thawing temperature,thawing rate increased, micro structural damage of meat products was bigger also, could lead to muscle fiber pore greaten, caused damage more on morphology[14].

Response surface optimization test

According to the results of single factor experiments, four factors of frozen time, melting time, freezing temperature and melting temperature response surface optimization design, response surface experimental design and results were shown in TABLE 2. Regression analysis was carried out on the response surface experiment databy using Design-Expert 8 software. Quadratic regression equation model was obtained between the comprehensive score and each factor,as follow:

$$Y = -303.88598 - 6.01561A + 9.75759B + 116.49362C + 190.01997D + 0.067657AB - 0.13479AC + 0.37208AD - 0.25406BC - 2.00766BD + 4.27045CD - 0.065652A^2 - 0.1089B^2 - 58.56299C^2 - 48.83584D^2$$

TABLE 2 : The arrangements and results of response surface analysis experiment

Test Number	A/ Freezing temperature（℃）	B/ Melting temperature（℃）	C/ Freezing time（h）	D/ Melting time（h）	Y_1/Ca^{2+}-ATPase Enzyme activity (μmol*Pi*/mg*prot*/h)	Y_2/ Rehydration rate (%)	Y_3/Hard-ness(N)	Y/Compre-hensive score
1	-30	20	1.0	1.5	2.43	39.56	11.1	91.18
2	-40	20	0.5	1.5	1.74	26.82	13.3	66.58
3	-30	20	0.5	1.0	1.87	25.02	13.2	66.99
4	-20	20	0.5	1.5	1.44	26.68	14.4	60.85
5	-20	20	1.5	1.5	1.86	28.24	14.2	68.35
6	-30	20	1.0	1.5	2.45	38.28	11.2	90.20
7	-40	25	1.0	1.5	2.09	31.89	11.1	79.64
8	-20	20	1.0	1.0	1.82	26.92	13.6	67.45
9	-20	25	1.0	1.5	1.99	29.23	9.0	81.05
10	-30	25	0.5	1.5	1.42	26.60	8.2	73.41
11	-40	20	1.0	2.0	1.96	27.09	12.0	71.91
12	-40	20	1.0	1.0	2.04	30.28	11.5	76.74
13	-30	15	0.5	1.5	1.87	27.91	13.8	68.70
14	-30	20	0.5	2.0	1.48	23.03	14.4	58.24
15	-30	20	1.5	1.0	1.58	29.22	13.3	66.42
16	-30	25	1.5	1.5	1.68	28.00	8.9	75.93
17	-30	20	1.0	1.5	2.34	36.43	10.9	87.49
18	-20	15	1.0	1.5	2.09	30.73	12.9	75.52
19	-30	20	1.0	1.5	2.36	36.62	10.2	89.56
20	-30	25	1.0	2.0	1.51	29.47	11.6	68.45
21	-30	15	1.5	1.5	1.69	34.02	12.3	73.76
22	-40	15	1.0	1.5	2.21	38.80	11.0	87.63
23	-30	15	1.0	2.0	2.18	29.38	11.7	77.51
24	-30	20	1.0	1.5	2.5	38.57	11.0	91.42
25	-30	15	1.0	1.0	1.51	31.34	11.5	70.34
26	-20	20.	1.0	2.0	2.05	26.60	13.8	70.06
27	-40	20	1.5	1.5	1.92	34.68	12.8	76.78
28	-30	25	1.0	1.0	2.22	29.73	10.3	81.36
29	-30	20	1.5	2.0	1.48	23.03	11.8	61.94

TABLE 3 : Variance of regression model analysis

Sources of variance	Square sum	Freedom	Squared	*F values*	*P*	Significant
Model	2435.02	14	173.93	34.31	＜0.0001	* *
A	108.01	1	108.01	21.31	0.0004	* *
B	3.40	1	3.40	0.67	0.4263	
C	67.28	1	67.28	13.27	0.0027	* *
D	37.44	1	37.44	7.39	0.0167	*
AB	45.77	1	45.77	9.03	0.0095	* *
AC	1.82	1	1.82	0.36	0.5590	
AD	13.84	1	13.84	2.73	0.1206	
BC	1.61	1	1.61	0.32	0.5815	
BD	100.77	1	100.77	19.88	0.0005	* *
CD	4.56	1	4.56	0.90	0.3590	
A^2	279.58	1	279.58	55.16	＜0.0001	* *
B^2	48.07	1	48.07	9.48	0.0082	* *
C^2	1390.39	1	1390.39	274.31	＜0.0001	* *
D^2	966.87	1	966.87	190.75	＜0.0001	* *
Residual	70.96	14	5.07			
Loss of quasi	61.02	10	6.10	2.46	0.2005	No significant
Error	9.94	4	2.49			
Comprehensive	2505.99	28				

Note：* *mean p＜0.01，Extremely significant，*mean 0.01＜p＜0.05，significant

According to TABLE 3，, the use of the experimental model was significant（p<0.0001). The fitting equation for lack of fit was not significant (p=0.2005>0.05),and the coefficient of determination of the model wasR2 =0.9717, adjust to R2=0.9434,Indicates that the better fitting degree of regression equation, the relationship between factors and response value can be expressed by this model. From the table can also be obtained,the one item A, C, D, the two item A^2,B^2,C^2,D^2 and the interaction term AB, BD influenced on the results significantly. analysing by the F test, the effect order of each factor on the response value size were: freezing temperature (A) > freezing time (C) > melting time (D) >melting temperature (B).

According to the model can predict the optimum technological conditions:The freezing temperature -32.40℃, the melting temperature 20.12℃,the melting time1.04h, the freezing time 1.45h. Tilapia dried product quality theories comprehensive score was 90.5145 in this condition. To verify the accuracy of models, validated test. According to the actual operating situation, the experimental condition was set to: freezing temperature -32℃, the melting temperature 20℃,freezing time 1.0h, the melting time 87min, three times parallel test. The final index of drying tilapia fillets :The activity of Ca2+-ATPase was 2.35, the rehydration rate was 37.22%, the hardness was9.7N, comprehensive score was 91.191, the relative error is less than 7%compared with the theoretical value, proved that the model is reliable. Tilapia fillets drying time was 10h under the optimum process conditions. The corresponding parameters in the control group:The activity of Ca2+-ATPase was 2.056, the rehydration rate was 29.44%, the hardness was12.5N, comprehensive score was 74.45. Compared with the control group, the comprehensive score of the best freeze-thawing pretreatment process conditions drying sample increased by 22.5%.

CONCLUSIONS

With the freeze-thawing pretreatment process related to freezing time, freezing temperature, melting temperature and melting time affect the performance of the heat pump drying of tilapia fillets, the existence of proper parameters in improving its dry speed at the same time can effectively improve the drying quality of tilapia fillets. Based on the optimization of the freezing-thawing conditions response surface method, the two regression equation model and excellent freeze-thaw pretreatment condition parameters are obtained. Compared with the control group, the quality comprehensive score under the optimized conditions increased by 22.5%.

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