



# Optimization and application of spray-drying process on oyster cooking soup byproduct

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## Abstract

Oyster drying processes have produced a large amount of cooking soup byproducts. In this study, oyster cooking soup byproduct was concentrated and spray-dried after enzymatic hydrolysis to produce seasoning powder. Response surface methodology (RSM) was performed on the basis of single-factor studies to optimize the feeding temperature, hot air temperature, atomization pressure, and total solid content of oyster drying. Results revealed the following optimized parameters of this process: feeding temperature of 60 °C, total solid content of 30%, hot air temperature of 197 °C, and atomization pressure of 92 MPa. Under these conditions, the oyster powder yield was 63.7% ± 0.7% and the moisture content was 4.1% ± 0.1%. Our pilot trial also obtained 63.1% yield and 4.0% moisture content. The enzyme hydrolysis of cooking soup byproduct further enhanced the antioxidant activity of the produced oyster seasoning powder to some extent. Spray drying process optimized by RSM can provide a reference for high-valued applications of oyster cooking soup byproducts.

**Keywords:** oyster cooking soup byproduct; seasoning powder; spray drying; response surface methodology; parameters optimization.

**Practical Application:** Hydrolytic oyster byproduct enhanced antioxidant activity was spray-dried for seasoning powder.

## 1 Introduction

Oyster aquaculture is mainly found in China, and its annual yield of approximately 3.89 million tons accounts for more than half of oyster production worldwide (Chen et al., 2014). Oyster, called “sea milk” in Western countries (Wang et al., 2008a), consists of up to 52.6% and 12% (dried weight, DW) proteins and fats, respectively (Cruz-Romero et al., 2007). Its proteins are composed of various amino acids and a high taurine content (Je et al., 2005). Oyster is also rich in  $\omega$ -3 unsaturated fatty acids and polyunsaturated fatty acids, which constitute approximately 50% of its total fatty acids (Cruz-Romero et al., 2008). Oyster extract performs many functions, including anti-bacterial (Defer et al., 2013; Liu et al., 2008), antihypertensive (Qian et al., 2008), anti-oxidation (Umayaparvathi et al., 2014; Wang et al., 2014), and anti-cancer activities (Umayaparvathi et al., 2014), ACE inhibition (Wang et al., 2008b), and DNA damage repair (Qian et al., 2008). Therefore, oyster is globally considered as valuable seafood with high nutritional value.

In addition to fresh oysters, half of these oysters are processed into traditional dried or canned oysters, smoke oysters, traditional oyster sauce, and fermented oysters every year (Chen et al., 2008). During processing, a large amount of cooking soup byproducts are produced and directly disposed or traditionally concentrated for low-value seasonings. Oyster cooking soup byproducts containing high amounts of protein can cause environmental pollution. Therefore, technology upgrading and process optimization should be achieved to process oyster soup

byproducts, and to develop and utilize aquatic byproducts and wastes comprehensively.

Spray drying technology is widely used in the food industry to produce powder continuously (Fazaeli et al., 2012; Gharsallaoui et al., 2007). It is also applied to dry liquid foods (Anekella & Orsat, 2013) and food natural extracts (Jiménez-Martín et al., 2015; Silva et al., 2013). However, spray drying technology has not yet to be comprehensively investigated to obtain powder from oyster byproducts after enzymatic hydrolysis.

Response surface methodology (RSM) is regarded as an effective method to optimize processing parameters for functional food components (Ke & Chen, 2016; Li et al., 2016; Samaram et al., 2015). In the present study, RSM was further applied on the basis of single-factor experiments to optimize process parameters of spray drying, including total solid content, hot air temperature, and atomization pressure. Our study could provide experimental data for the high-value utilization of oyster byproducts and for further development and utilization of aquatic shellfish byproducts.

## 2 Materials and methods

### 2.1 Materials

Oyster cooking soup byproduct (solid mass fraction of approximately 3.5%) was provided by Xiamen Yangjiang Food Co., Ltd., China. This byproduct was produced during dried

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oyster pretreatment. In this process, fresh oyster was cooked in steam-jacketed pot for 5 min at 100 °C-102 °C. Flavourzyme and benase (Novozymes Chinese investment Co., Ltd., China) were added to hydrolyze proteins and thus obtain peptides and amino acids and enhance the flavor. The soup byproduct from the oyster cooking pre-treatment was concentrated in vacuum equipment (Xiamen Yangjiang Food Co., Ltd., China) at a temperature of 75 °C and a pressure of 0.08 MPa until the total solid content of 16%-36% was reached. The different total solid contents of the soup byproduct were blended with the designed components. The mixture was sprayed using a laboratory scale spray dryer (SY6000, Shanghai World Biotechnology Equipment Engineering Co., Ltd., China). After optimization in laboratory scale, 1-ton hydrolysis pilot was designed and commissioning in Xiamen Yangjiang Food Co., Ltd, China.

## 2.2 Total solid content measurement

The total solid content of the concentrated oyster byproduct was determined using a previously described drying method (Chen et al., 2012). Total solid content / % =  $\frac{m_2}{m_1} \times 100$ , where  $m_1$  is the weight of the exact amount of concentrated oyster soup in g, and  $m_2$  is the constant weight of the solid content in g after drying at 105 °C.

## 2.3 Moisture content determination

The moisture content of the drying powder from oyster soup byproduct was measured with a reduced-pressure drying method (Chen et al., 2012). Moisture content / % =  $\frac{M_w - M_d}{M_d} \times 100$ , where  $M_d$  is the dried weight of the oyster powder in g and  $M_w$  is the wet weight of the oyster powdering.

## 2.4 Powder yield rate determination

The yield rate of the oyster byproduct powder is calculated as follows: Yield of oyster byproduct powder / % =  $\frac{M(1-R)}{M_0} \times 100$ , where  $M$  is the weight of oyster powder collected after spray drying, g;  $R$  is the moisture content of the oyster powder after spray drying, %; and  $M_0$  is the total solid content of the concentrated oyster cooking soup, g.

## 2.5 Antioxidant capacity

The antioxidant capacity of oyster byproduct powder was determined through 1,1-diphenyl-2-picrylhydrazyl (DPPH) free radical scavenging assay as described previously (Wang et al., 2014) with few modifications. The concentrated cooking soup and the byproduct hydrolysate before and after spray drying were obtained. The byproduct sample (2 mL) with various concentrations (5-100 mg/mL) was mixed with 0.2 mM DPPH methanolic solution. The mixture was vortexed for 1 min and kept in a dark place at room temperature (25 °C) for 30 min. Its absorbance was then read at 517 nm by a UV spectrophotometer. The scavenging activity of the sample on DPPH was calculated using the following equation: Scavenging activity (%) =  $[1 - (A_s - A_b) / A_c] \times 100$ ,

where  $A_s$  is the absorbance of the reaction mixture,  $A_b$  is the absorbance of the blank (reaction solution without DPPH), and  $A_c$  is the absorbance of the control (reaction solution without sample). The assay was carried out in triplicate, and results were presented as mean  $\pm$  SD.

## 2.6 Preliminary experiments

Various factors affect spray drying. In our experiment, four single factors were examined to observe the effects of each factor on the oyster powder yield and moisture content, with a feeding speed of 20 mL/min. The experimental conditions were set as follows:

The total solid contents of oyster soup at 16%, 20%, 24%, 28%, 32%, and 36% respectively, were examined at a hot air temperature of 190 °C, a feeding temperature of 60 °C, and an atomization pressure of 90 MPa. Oyster byproduct was dried at hot air temperatures of 170 °C, 180 °C, 190 °C, 200 °C, and 210 °C, a feeding temperature of 60 °C, an atomization pressure of 90 MPa, and a total solid content of 32% total solid content.

Oyster soup was heated to 40 °C, 50 °C, 60 °C, 70 °C, and 80 °C and placed into the spray drying machine at a hot air temperature of 190 °C, an atomization pressure of 90 MPa, and a total solid content of 32%. Oyster byproducts were sprayed drying at atomization pressures of 60, 70, 80, 90, and 100 MPa, a hot air temperature of 190 °C, a feeding temperature of 60 °C, and a total solid content of 32%.

## 2.7 RSM experimental design

RSM is an empirical modeling technique applied to evaluate the relationship among variables and results (Ke & Chen, 2016) and to optimize the factors for the target range. Box-Behnken center combination design principle in Design-Expert (Version 8.0) was used to design our experimental trials and to optimize the corresponding experimental factors. On the basis of the preliminary experiment results, we assigned the total solid content ( $X_1$ ), hot air temperature ( $X_2$ ), and atomization pressure ( $X_3$ ) as the main parameters. For the modeling requirement, three levels of each factor were designed at the same distance of -1, 0, and 1 (Table 1). All of the trials were performed in triplicate and the mean value was used in RSM analysis.

## 2.8 Statistical analysis

Single-factor test data were analyzed by LSD method in DPS. RSM data, modeling, equation significance  $R$ , and fitting coefficient were analyzed by using Design-Expert 8.0.  $P < 0.05$  indicated significant differences.

## 3 Results and discussion

### 3.1 Preliminary experiments

In Table 2, the oyster powder yield increased significantly ( $P < 0.05$ ) as total solid content increased from 16% to 32%, and the highest powder yield was 60.80%. Total solid content ranging from 28% to 36% was chosen to evaluate the RSM factor with low moisture content and a high oyster powder yield. Hot air

**Table 1.** Box-Behnken experiment and results.

Run	Variable level			Variable conditions			Yield / %
	X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Total solid content %	Hot air temperature °C	Atomization pressure MPa	
1	0	1	1	32	200	100	62.1 ± 1.0
2	-1	0	1	28	190	100	61.0 ± 1.7
3	0	0	0	32	190	90	61.5 ± 1.1
4	-1	-1	0	28	180	90	52.1 ± 1.4
5	-1	0	-1	28	190	80	53.0 ± 1.3
6	0	1	-1	32	200	80	43.4 ± 0.7
7	1	0	1	36	190	100	60.6 ± 1.9
8	-1	1	0	28	200	90	56.6 ± 0.9
9	0	-1	1	32	180	100	47.1 ± 1.2
10	0	0	0	32	190	90	63.0 ± 0.8
11	1	1	0	36	200	90	58.0 ± 1.5
12	1	0	-1	36	190	80	51.7 ± 1.9
13	0	0	0	32	190	90	63.1 ± 0.4
14	0	0	0	32	190	90	62.8 ± 0.9
15	0	0	0	32	190	90	61.0 ± 1.2
16	1	-1	0	36	180	90	51.3 ± 0.9
17	0	-1	-1	32	180	80	52.4 ± 0.6

**Table 2.** Effects of total solid content and hot air temperature on spray-dried oyster powder.

Total solid content /%	Yield /%	Moisture Content /%	Hot air temperature /°C	Yield /%	Moisture Content /%
16	52.1 ± 0.3 <sup>c</sup>	5.1 ± 0.1 <sup>a</sup>	170	49.8 ± 1.07 <sup>d</sup>	4.8 ± 0.1 <sup>a</sup>
20	54.4 ± 0.8 <sup>d</sup>	4.6 ± 0.1 <sup>b</sup>	180	57.7 ± 1.3 <sup>b</sup>	4.5 ± 0.1 <sup>a</sup>
24	58.2 ± 0.4 <sup>c</sup>	4.2 ± 0.1 <sup>c</sup>	190	62.0 ± 1.5 <sup>a</sup>	4.2 ± 0.08 <sup>b</sup>
28	59.8 ± 0.4 <sup>b</sup>	4.2 ± 0.1 <sup>d</sup>	200	61.7 ± 0.9 <sup>a</sup>	4.1 ± 0.04 <sup>bc</sup>
32	60.8 ± 0.5 <sup>a</sup>	4.4 ± 0.1 <sup>c</sup>	210	53.2 ± 1.3 <sup>c</sup>	3.8 ± 0.1 <sup>c</sup>
36	59.8 ± 0.6 <sup>b</sup>	4.6 ± 0.1 <sup>b</sup>			

Note: Then different superscript letters represent the significance level of 0.05.

temperature also significantly affected the oyster powder yield ( $P < 0.05$ ; Table 2). The highest oyster powder yield was observed at 190 °C. The highest oyster powder yield was observed at 190 °C. In our experiment, a low temperature of 190 °C of inlet hot air caused the oyster powder to adhere strongly to the inner wall of drying machine. Conversely, the yield decreased at temperatures higher than 190 °C possibly because of the conversion of oyster powder from a glassy state to a rubbery state during spray drying (Chen et al., 2012). The moisture content of oyster powder decreased as hot air temperature increased. High feeding temperature accelerated oyster soup drying (Table 3). The oyster powder yield did not significantly increase at feeding temperatures between 50 °C to 70 °C. A feeding temperature of 60 °C was set in the subsequent spray drying to enhance the energy-saving property and quality of the product. Atomization pressure also influences the oyster powder yield, and the obtained yields were significantly different ( $P < 0.05$ ). Atomization pressure from 80 Mpa to 100 Mpa was chosen for further RSM optimization.

### 3.2 Optimization of oyster powder yield by RSM

The RSM test was performed 17 combinations according to Box-Behnken design with the randomized order (Table 1). A feeding temperature of 60 °C and a feeding speed of 20 mL per min were applied to all spray drying processes. The regression

quadratic model of the variable and yield was expressed as follow Equation 1:

$$Y(\%) = 62.27 - 0.15X_1 + 2.15X_2 + 3.78X_3 + 0.55X_1X_2 + 0.22X_1X_3 + 6.01X_2X_3 - 1.25X_1^2 - 6.65X_2^2 - 4.46X_3^2 \quad (1)$$

where Y represents the yield (%) of the oyster powder and X<sub>1</sub>, X<sub>2</sub>, and X<sub>3</sub> represent the total solid content, hot air temperature, and atomization pressure, respectively.

Analysis of variance was performed to evaluate the significance of the model. Variable, model, and coefficient analyses were conducted (Table 4). The surface response of the regression quadratic model with F value of 48.64 indicated that the regression equation was highly significant ( $P < 0.01$ ), and the lack of fit of P-value of 0.2411 > 0.05 denoted that the model was well fit for the experimental data of spray drying for oyster cooking soup. R<sup>2</sup> of 0.98 and the adjusted R<sup>2</sup> of 0.96 suggested that the regression quadratic model was significant and satisfactory to express the relationship between the oyster powder yield and variable factors.

ANOVA also revealed that total solid content did not significantly affect oyster powder yield ( $P > 0.05$ ), whereas hot air temperature and atomization pressure significantly affected the yield ( $P < 0.01$ ). The effects of the factors on oyster powder yield

**Table 3.** Effects of feeding temperature and atomization pressure on spray-dried oyster powder.

Feeding temperature /°C	Yield /%	Moisture Content /%	Atomization pressure /MPa	Yield /%	Moisture Content /%
40	60.1 ± 1.6 <sup>bc</sup>	4.8 ± 0.1 <sup>a</sup>	60	43.8 ± 1.2 <sup>a</sup>	5.4 ± 0.1 <sup>a</sup>
50	62.6 ± 0.9 <sup>a</sup>	4.4 ± 0.07 <sup>b</sup>	70	53.3 ± 1.1 <sup>b</sup>	5.0 ± 0.2 <sup>b</sup>
60	62.8 ± 1.1 <sup>a</sup>	4.2 ± 0.1 <sup>c</sup>	80	56.7 ± 0.7 <sup>c</sup>	4.5 ± 0.2 <sup>c</sup>
70	61.3 ± 0.8 <sup>ab</sup>	3.9 ± 0.1 <sup>d</sup>	90	62.6 ± 0.7 <sup>d</sup>	4.1 ± 0.2 <sup>d</sup>
80	58.5 ± 1.3 <sup>c</sup>	3.4 ± 0.1 <sup>e</sup>	100	63.5 ± 0.4 <sup>e</sup>	3.7 ± 0.2 <sup>e</sup>

Note: Then different superscript letters represent the significance level of 0.05.

**Table 4.** Variance analysis of regression model.

Source	Sum Squares	df	Mean Squares	F-value	P-value
Model	590.28	9	65.59	48.64	< 0.001
X <sub>1</sub>	0.17	1	0.17	0.13	0.7336
X <sub>2</sub>	37.08	1	37.08	27.50	0.0012
X <sub>3</sub>	114.21	1	114.21	84.69	< 0.001
X <sub>1</sub> X <sub>2</sub>	1.20	1	1.20	0.89	0.3764
X <sub>1</sub> X <sub>3</sub>	0.19	1	0.19	0.14	0.7190
X <sub>2</sub> X <sub>3</sub>	144.36	1	144.36	107.06	< 0.001
X <sub>1</sub> <sup>2</sup>	6.53	1	6.53	4.85	0.0636
X <sub>2</sub> <sup>2</sup>	181.05	1	181.05	134.26	< 0.001
X <sub>3</sub> <sup>2</sup>	83.69	1	83.69	62.06	0.001
Residual	9.44	7	1.35		
Lack of Fit	5.79	3	1.93	2.11	0.2411
Pure Error	3.65	4	0.91		
Corrected Total	599.72	16			

Note:  $P < 0.05$  meant significant, and  $P < 0.01$  meant highly significant.

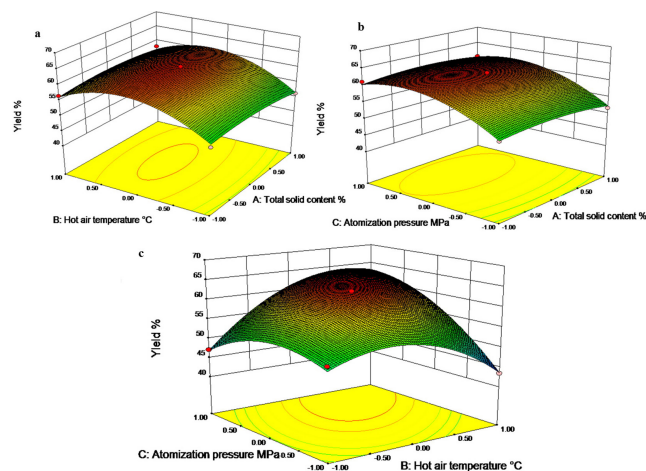
exhibited the following trend:  $X_3 > X_2 > X_1$ , that is, atomization pressure > hot air temperature > total solid content.

### 3.3 Interaction of spray drying variables and optimum conditions

The interaction of spray drying factors on powder yield was depicted by response surface. Response surfaces and contour plots are shown in Figure 1a, b, c. The interactions between total solid content and hot air temperature and between total solid content and atomization pressure were not significant, whereas hot air temperature and atomization pressure strongly affected each other ( $P < 0.01$ ). Considering the effect of the interaction of atomization pressure and hot air temperature, we found that the optimal conditions of response value for oyster powder yield were  $X_1 = -0.38$ ,  $X_2 = 0.71$ , and  $X_3 = 0.20$ , which corresponded to total solid content of 29.5%, hot air temperature of 197.1 °C, and atomization pressure of 92 MPa, respectively. The predicted yield of oyster powder spray-dried under these optimal conditions was 63.5%.

### 3.4 Optimization of spray drying and confirmation

The optimum of a total solid content of 30%, a hot air temperature of 197 °C, and an atomization pressure of 92 MPa were carried out to obtain practical operations. The feeding temperature of 60 °C and feeding speed of 20 mL per min were used for spray drying. The oyster powder yield obtained experimentally was confirmed in triplicate of the three groups, and the yield result was 63.7% ± 0.7%, which was not significantly

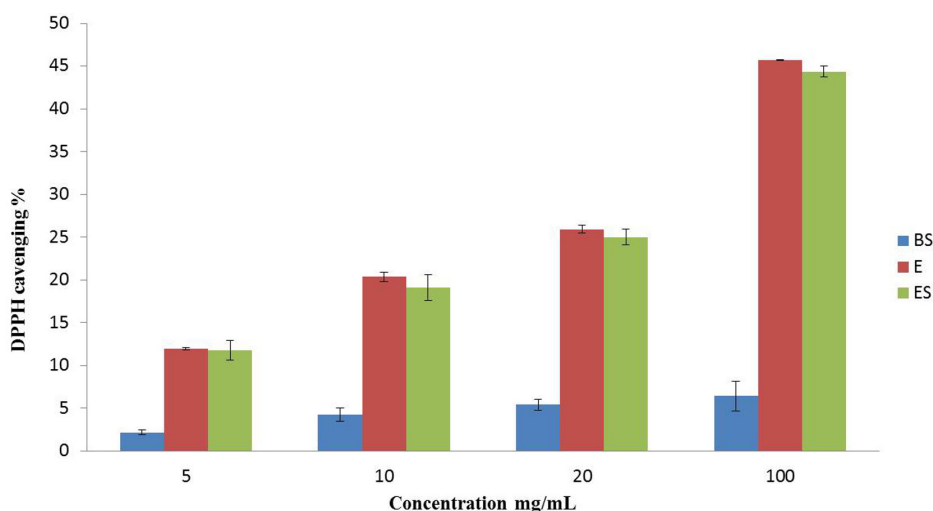


**Figure 1.** Response surface of spray drying on the oyster powder yield. (a) Hot air temperature and Total solid content; (b) Total solid content and atomization pressure; (c) Hot air temperature and atomization pressure.

different from the predicted value. The moisture content was determined as 4.1% ± 0.1% and the organoleptic quality of oyster powder was quite acceptable. The pilot trial revealed 63.1% yield and 4.0% moisture content. These results verified that the RSM regression model was adequate to optimize the spray drying conditions.

### 3.5 DPPH free radical scavenging capacity

Antioxidant activity was determined in the concentrated oyster cooking soup, oyster byproduct hydrolysate before and



**Figure 2.** DPPH free radical scavenging of oyster byproducts. BS, E, and ES represent the concentrated oyster cooking soup, oyster byproduct hydrolysate before drying, and oyster byproduct hydrolysate after spray drying, respectively.

after spray drying (Figure 2). After hydrolysis was performed using flavourzyme and benase, the antioxidant activity of the oyster byproduct hydrolysate increased, but spray drying did not significantly affect the antioxidant activity ( $P > 0.05$ ). The antioxidant peptides separated from the enzyme hydrolysate of the oyster protein has been described in previous studies (Umayaparvathi et al., 2014; Wang et al., 2014). In our study, enzyme hydrolysis enhanced the flavor and increased the antioxidant activity of the seasoning powder to some extent.

#### 4 Conclusion

RSM could effectively describe the effects of various factors on oyster powder yield. The following optimum conditions for the spray drying of oyster powder were obtained by optimizing our mathematical prediction model: total solid content of 30%, hot air temperature of 197 °C, and atomization pressure of 92 MPa. Under these conditions, the yield of oyster powder was  $63.7\% \pm 0.7\%$  and the moisture content was  $4.1\% \pm 0.1\%$ . Our pilot trial revealed 63.1% yield and 4.0% moisture content. Enzyme hydrolysis enhanced the flavor and increased the antioxidant activity of the seasoning powder to some extent. Therefore, spray drying technology could be used to produce powder from oyster cooking soup byproduct.

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