Optimization of ultrasonic-assisted extraction of inulin from *Jerusalem artichoke*

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Abstract. An efficient ultrasonic-assisted extraction of inulin from *Jerusalem artichoke* was investigated by Response Surface Methodology (RSM). The results showed that the yield of ultrasonic-assisted extraction was 62.07 ± 0.39% over the microwave (40.85 ± 0.28%) and hot water extraction (27.42 ± 0.42%). The Box-Behnken Design (BBD) was used to optimize the effects of three parameters (temperature-X1, ultrasonic power-X2 and time-X3) on inulin yield. Analysis of variance showed that the contributions of X1, X3, X12, X13, X22 were significant. The optimal yield of inulin was 82.93 ± 1.03% at 82 ℃, 120 W and 18 min.

1 Introduction

Inulin, a non-digestible carbohydrate, is natural functional dietary fibers for improving bowel health [1]. A significant work had been reported on determination of degree of polymerization and prebiotic effect evaluation of inulin from *Jerusalem artichoke* [2].

At present, hot water extraction mostly was used for the industrial production of inulin, but common problems were low extraction rate, excessive loss and quality instability [3]. Ultrasound-assisted extraction has a fast and efficient advantages and recognizes as an alternative approach to traditional extraction methods due to high-density, high-frequency sound waves and the role of promoting the dissolution of active ingredients [4]. Compared with conventional extraction, it not only accelerated the extraction rate, saved the extraction time and reduced the organic solvent waste, but also enhanced the efficiency and the quality [5].

Therefore, the significant variables (temperature, ultrasonic power and time) were investigated by Response Surface Methodology (RSM). By optimizing the inulin extraction, it could provide theoretical parameters for practical production.

2 Materials and Methods

2.1 Materials

Jerusalem artichoke was purchased from Jinzhou farm produce market, then crushed to pass through 40 mesh screen and stored at -4 ℃ until used for further analysis. Other chemicals were all of analytical grade.

2.2 Extraction of inulin

The *Jerusalem artichoke* powder (5.00 g) was put into a 500 mL beaker and added the water (the liquid ratio of 1:20). The inulin was extracted in the ultrasonic cell disintegrator (SJIA-1200W, Ningbo Shuangjia Instrument Co., Ltd., China) with different parameter: temperature (50-90 ℃), ultrasonic power (50-200 W) and time (5-25 min). Then, the extract was centrifuged at 3000 rpm for 20 min and the yield of inulin was determined. The total sugar was measured by the phenol-sulfuric acid method, and the reducing sugar content using the DNS method. The yield of total inulin was calculated as follows (1):

$$y = \frac{x_1 - x_2}{w} \times 100\% \tag{1}$$

Where x_1 is the content of total sugar content, x_2 is the content of reducing sugar content, and w represent dried sample weight.

2.3 Comparison of three different extraction methods

The yield of inulin from experimental group (ultrasonic-assisted extraction) and control group (hot water and microwave extraction) were compared with same parameter: 70℃ for 30 min. Moreover, the ultrasonic power 140W and the microwave power 320W.

2.4 Single-factor experiments

The yield of inulin was evaluated through determining the effect of temperature, ultrasonic power and time during the ultrasonic-assisted extraction procedure by a single-factor design. One factor was changed while the other factors were kept constant in each experiment.

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2.5 Box-Behnken Design (BBD)

Experiments were established based on BBD with three factors at three levels. Three parameters including temperature (℃), ultrasonic power (W) and time (min) were chosen as variables based on the results of single-factor experiments and named as X1, X2, and X3, respectively. Extraction yield (Y) was taken as the response of the design experiments. Table 1 lists the ranges of independent variables and their levels.

Table 1. Independent variables and their levels used for BBD

Independent variables	Levels		
	-1	0	1
ultrasonic temperature (X1)	70	80	90
ultrasonic power (X2)	80	110	140
ultrasonic time (X3)	10	15	20

2.6 Statistical analysis

The data were presented as the mean ± SD and evaluated by one-way analysis of variance (ANOVA). Difference was considered to be statistically significant if $p < 0.05$. All statistical analyses were carried out by IBM SPSS Statistics 20.

3 Results and discussions

3.1 Effects of different extraction methods on inulin yield

It can be seen from Table 2 that the yield of ultrasonic-assisted extraction was 62.07%, followed by 40.85% microwave and 27.42% hot water extraction. Compared with the hot water extraction, ultrasonic-assisted extraction significantly increased 34.65%, and it was non-destructive for inulin and benefited from a shorter extraction time [6].

Ultrasonic wave created and collapsed more bubble when it passed through the liquid medium, thus leading to microjet formation and acoustic streaming. Due to the swelling of the materials and enlargement of the pores, it enhanced the extraction yield of inulin [7].

Table 2. Comparison of three extraction methods

Extraction methods	Inulin yield (%)
Hot water	27.42 ± 0.42
Microwave	40.85 ± 0.28
Ultrasonic-assisted	62.07 ± 0.39

3.2 Effect of three parameters on the yield of inulin

The effect of temperature on the yield of inulin was shown in Fig.1(A). Ultrasonic parameters were as follows: ultrasonic power 110 W and 15 min. The yield of inulin increased with the increasing extraction temperature and reached the critical value (74.7±1.6%) when extraction temperature was 80℃, and then the curve began to decrease due to the destroyed of the inulin structure.

The effect of different ultrasonic power on the yield of inulin was shown in Fig.1(B). The results indicated that the maximum yield of inulin (69.8 ± 2.2%) was reached when ultrasonic power was 110 W, and then it began to decrease, as generated a large number of bubbles and weakened the scattering chemical likely caused the local solution to warm up thereby partly degradation of the inulin structural [8].

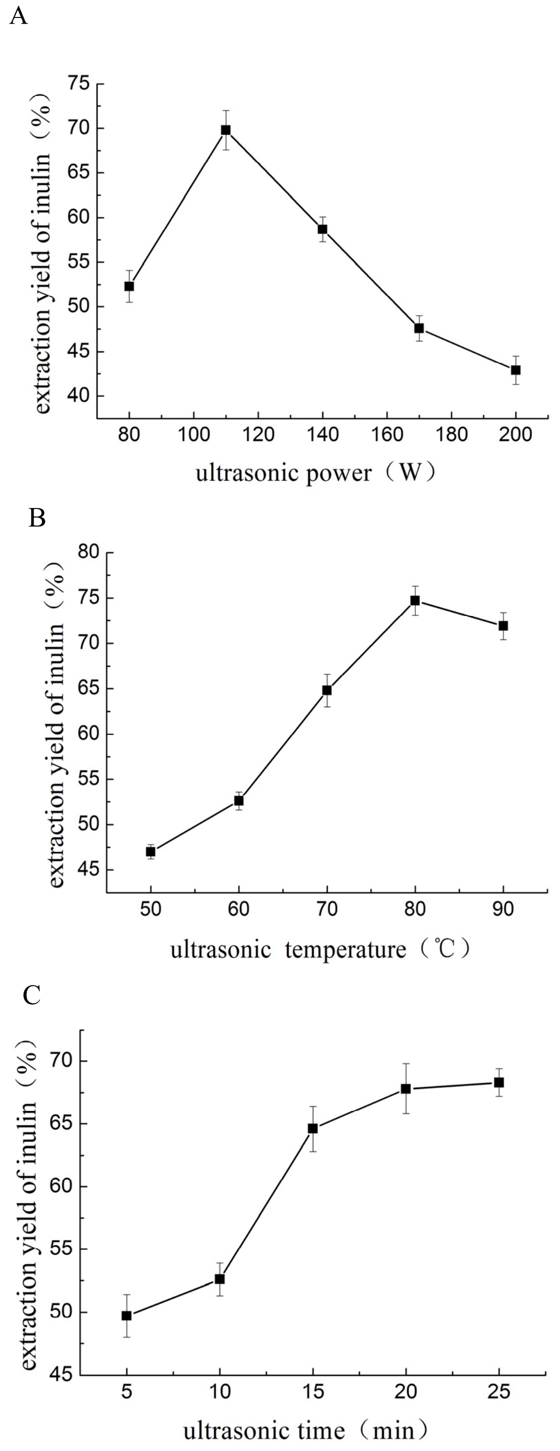

Fig. 1. Effect of temperature (A), ultrasonic power (B) and time (C) on the yield of inulin

The effect of time on extraction yield of inulin at 70 ℃

and 110 W was shown in Fig.1(C). The yield increased with the increasing time and reached the value (67.8 ± 2%) at 20 min, and then the upward trend was obviously reduced and tended to be steady. Time was a critical factor that would affect the extraction efficiency and selectivity of the fluid. It was reported that a long extraction time presents a positive effect on the production of polysaccharides, but excessive extraction could waste time and energy [9].

Table 3. BBD and response values of the yield of inulin

Number	X_1 (°C)	X_2 (W)	X_3 (min)	Yield (%)
1	0 (80)	0 (110)	0 (15)	82.21
2	-1 (70)	0 (110)	1 (20)	63.51
3	1 (90)	1 (140)	0 (15)	65.93
4	0 (80)	0 (110)	0 (15)	82.76
5	0 (80)	-1 (80)	1 (20)	77.37
6	-1 (70)	1 (140)	0 (15)	49.72
7	-1 (70)	-1 (80)	0 (15)	53.25
8	1 (90)	0 (110)	1 (20)	67.11
9	0 (80)	0 (110)	0 (15)	84.25
10	1 (90)	0 (110)	-1 (10)	74.83
11	0 (80)	1 (140)	1 (20)	83.18
12	0 (80)	-1 (80)	-1 (10)	73.36
13	-1 (70)	0 (110)	-1 (10)	55.45
14	0 (80)	0 (110)	0 (15)	81.55
15	0 (80)	0 (110)	0 (15)	83.69
16	0 (80)	1 (140)	-1 (10)	67.24
17	1 (90)	-1 (80)	0 (15)	59.31

3.3 Statistical analysis and the model fitting

There were a total of 17 runs for optimizing each parameter in the BBD. The corresponding results and the design matrix of RSM experiments to determine the effects of the three independent variables were shown in Table 3. The mathematical model describing the extraction yield of inulin (Y) as a function of the test independent variables over their selected ranges was given by Eq. (2):

$$\begin{aligned} y &= 82.89 + 5.66X1 + 0.35X2 + 2.54X3 \\ &+ 2.54X1X2 - 3.95X1X3 + 2.98X2X3 \\ &- 17.95X1^2 - 7.89X2^2 + 0.28X3^2 \end{aligned} \tag{2}$$

The regression coefficients of Eq. (2) calculated and tested for their significance using variance (ANOVA), and the analysis results of fit statistics of yield (Y) for predictive model were shown in Table 3. In our research, the F-value (F = 30.4) and P-value ($P < 0.001$) implied that the model was extremely significant and the lack-of-fit F-value of 16.53 was also significant which meant that the model was sufficiently accurate for predicting the relevant response. The quadratic regression model showed that R^2 and adj-R^2 were 0.9751 and 0.9430 which indicated that 97.51% of the variation could be represented by the fitted model and 94.30% of the variation were explained by the model. It could be seen from Table 4 that X1, X3, X13, X12, X22 were important factors in the inulin yield with very small P values ($P < 0.05$).

3.4 Interpretation and optimization of response surface model

The three-dimensional response surface could show a visual interaction between two tested variables and the relationships between each variable levels and related responses in the experiment, which was also used to determine the optimum conditions (Fig.2).

3.5 Verification of predictive model

The suitability of the model equation for predicting the optimum response values was tested using the optimal conditions. Because the optimal values were difficult to operate in the actual experiments, they were carried out with slight modifications: 82°C, 120 W and 18 min. Under these conditions, the experiment yield of inulin was 82.93 ± 1.03%, which was not significantly different ($P> 0.05$) from the predicted value of 84.29%. This result proved that the model designed in this study was valid.

4 Conclusions

Ultrasound-assisted extraction, which was an efficient and time-saving extraction technique, could be used to improve the extraction yield of inulin. The ultrasonic-assisted extraction (62.07 ± 0.39%) was superior to the microwave (40.85 ± 0.28%) and hot water extraction (27.42 ± 0.42%). By optimized variables by RSM, the optimal yield of inulin was 82.93 ± 1.03% at 82°C, 120 W and 18 min, which was coincided with the predicted value of 84.29% closely.

Table 4. Results of ANOVA of regression model for the yield of inulin (* $p < 0.05$ significant; ** $p < 0.01$ extremely significant).

Source	Sum of Squares	df	Mean Square	F Valve	P-valve Prob>F
Model	2123.82	9	235.98	30.4	<0.001**
A	255.95	1	255.95	32.97	<0.001**
B	0.97	1	0.97	0.12	0.735
C	51.46	1	51.46	6.63	0.037*
AB	25.76	1	25.76	3.32	0.111
AC	62.25	1	62.25	8.02	0.025*
BC	2.79	1	2.79	4.58	0.070

A2	1356.79	1	1356.79	174.78	<0.001**
B2	262.01	1	262.01	33.75	<0.001**
C2	0.34	1	0.34	0.044	0.840
Residual	54.34	7	7.76		
Lack of fit	49.58	3	16.53	13.87	0.014*
Pure error	4.76	4	1.1		
Cor total	2178.16	16			
C.V.%	3.93				
R^2	0.9751				
Adj-R^2	0.9430				

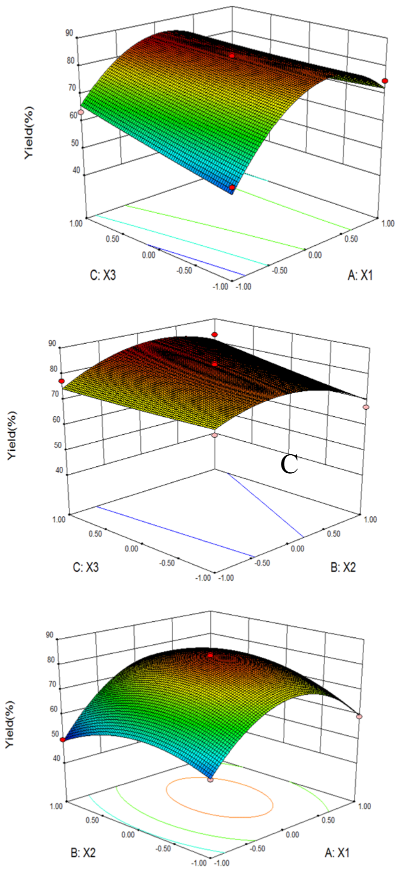

Fig. 2. The 3-D plots of variables (X1: temperature; X2: ultrasonic power; X3: time) on the yield of inulin.

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