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Optimization of Ultrasound-assisted Extraction of *PhysalisalkekengiL.var.francheti* Seed Oil

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Ultrasound-assisted extraction was performed using*PhysalisalkekengiL.var.francheti* (*Physalisalkekengi L.*) seeds as the raw material. The influence factors including material-liquid ratio, extraction temperature, extraction duration and ultrasonic power were chosen through single factor experiment. Using extraction rate as the response value, Box-Behnken design involving 4 factors (3 levels for each factor) was adopted. The results showed that the optimal parameters for ultrasound-assisted extraction of *Physalisalkekengi L.* seed oil were as follows: material-liquid ratio 1:1843 (g/ml), temperature 44.06℃, duration 39.45min, and ultrasonic power 92.14W. Under the optimal conditions, the extraction rate was (23.10±0.02) %.

1. Introduction

PhysalisalkekengiL.var.francheti (*Physalisalkekengi L.*), also named franchetgroundcherrycalyxor, is an herbaceous perennial of the family Solanaceae. This plant is widely planted in China, especially in Northeast China and Northwest China. Having a rich content of Vitamin C, carotenoid and minerals(Ramadan, 2011),*Physalisalkekengi L.* can be used for medical purposes due to its detoxifying, blood pressure lowering, hypoglycemic, antibacterial activity and antitumour activity(Bastosa, et al.,2008). Therefore, *Physalisalkekengi L.* was regarded as a healthcare herbaceous fruit with edible and medicinal value (Puente, etal. 2011).
Each fruit of *Physalisalkekengi L.* contains about 210-320 seeds which are shaped like kidneys, have light yellow color and the seeds account for about 60% of the total weight of fruits(Yu, et al.,2012). Much work has been done in the research and development of *Physalisalkekengi L.* products, such as fruit wine and beverage. And meanwhile, fruit processing usually resulted in the production of a large number of seeds. Because of the rich nutrients contained in the seeds such as fat, proteins and polysaccharides(Yu, et al., 2012), it is beneficial to study the utilization of *Physalisalkekengi L.* seeds to enhance the comprehensive exploitation of *Physalisalkekengi L.*
The industrial methods for seed oil extraction include pressing, Soxhlet extraction, aqueous enzymatic extraction and ultrasound-assisted extraction. Among them, ultrasound-assisted extraction has the features of low extraction temperature and high extraction efficiency. It is ideal for seed oil extraction, and many studies have been reported on ultrasound-assisted extraction of plant soil (Clodoveo, et al., 2013; Pingret, et al., 2014).
Although some studies have been published on the extraction process of *Physalisalkekengi L.*seed oil (Liu et al., 2011), parameter optimization based on response surface method is rarely discussed. Response surface method (RSM) is a statistical approach used to investigate the optimal conditions in a multifactorial system. It allows the modeling and analysis of response affected by multiple variables with higher accuracy and better optimization effect (Khuri and Mukhopadhyay, 2010). In the present study, the seeds of *Physalisalkekengi L.* were subjected to ultrasound-assisted extraction with optimization done through response surface analysis. The findings provide theoretical basis for further exploitation of *Physalisalkekengi L.* resources.

2. Materials and methods

2.1 Materials and reagents

Seeds of *Physalisalkekengi L.* were provided by Jilin Jiteshan Grape Wine Factory. Petroleum ether (60-90℃, analytically pure) was purchased from Tianjin Hengxing Chemical Reagent Co., Ltd.

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2.2 Method

2.2.1 Process workflow of ultrasound-assisted extraction

↓Petroleum ether

Seeds of *Physalisalkekengi L.*→Crushing→Sieving→Weighing→Ultrasound-assistedextraction→Vacuumfiltration→Concentration with rotary evaporator→Drying →Weighing

2.2.2 Key points of the extraction process

Fruits of *Physalisalkekengi L.* were cleaned, dried and then crushed. The crushed fruits were passed through the 20-mesh sieve to obtain the seed powder, and 5g of such seed powder was weighed and placed into a 250ml conical flask. Then an appropriate amount of petroleum ether (boiling range 60-90℃) was added. The conical flask was placed on the ultrasonic cleaner, and ultrasound-assisted extraction was carried out under different temperature, duration and power. The mixture of material and liquid was subjected to vacuum filtration, and the filter cake was washed three times. Three filtrates were combined and concentrated using a rotary evaporator under the temperature of 50℃.At the same time, petroleum ether was recovered. Finally, the resulting oil was dried to constant weight to obtain the crude seed oil.

2.2.3 Determination of extraction rate

Extraction rate (%) = (m1-m2)/m×100%

Where m1 is the total mass (g) of the receiving bottle and the seed oil; m2 is the mass (g) of the receiving bottle; m is the mass of the seed.

2.2.4 Single factor experiment

The main influence factors of seed oil extraction were material-liquid ratio, temperature, duration and ultrasonic power. The same experimental conditions as in solvent selection were used: material-liquid ratio 1:18 (g/mL), temperature 45℃, duration 40min, power 80W. Other 3 factors being constant, the effects of material-liquid ratio (1:10, 1:14, 1:18, 1:22, 1:26g/mL), duration of ultrasound-assisted extraction (20, 30, 40, 50, 60min), temperature (35, 40, 45, 50, 55℃) and ultrasonic power (60, 70, 80, 90 and 100W) on the extraction rate was determined. The optimal single factors were then determined based on the extraction rate.

2.2.5 Response surface design

According to the principle of central composite design, 4 factors, namely, material-liquid ratio, temperature of ultrasound-assisted extraction, duration of ultrasound-assisted extraction and ultrasonic power, were screened. Three levels were designed for each factor in response surface analysis so as to determine the optimal parameters.

3. Results and analysis

3.1 Single factor experiment

3.1.1 Effect of material-liquid ratio on extraction rate

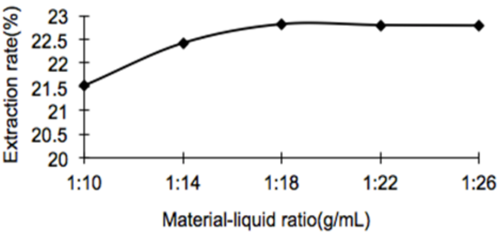

Figure1: Effect of material-liquid ratio on the extraction rate

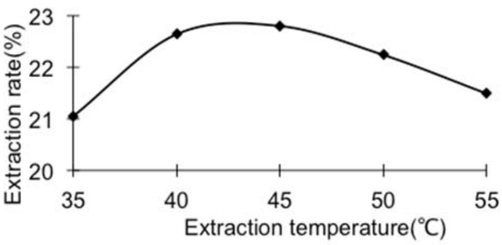

Figure 2: Effect of extraction temperature on extraction rate

As seen from Fig. 1, when the material-liquid ratio was 1:10-1:18g/mL, the extraction rate increased with the increase of the material-liquid ratio, from 21.52% to 22.82%. It was due to the concentration of the plant oil was decreased with the increasing content of solvent and the concentration difference between the raw material and solvent increased, Therefore, Afterwards, the extraction rate showed a trend of slight decline. Thus 1:1.8(g/mL) was determined as the optimal material-liquid ratio.

3.1.2 Effect of extraction temperature on extraction rate

It can be seen from Fig. 2 that temperature had a considerable impact on the extraction rate and the extraction rate first increased and then decreased. When temperature increased from 30℃ to 45℃, the extraction rate increased from 21.05% to 22.8%. However, the extraction rate declined with further temperature rise. The extraction rate dropped to 21.49% at the temperature of 55℃. This is because temperature rise facilitated oil diffusion, but an excessively high temperature caused the evaporation of solvent. Therefore, the optimal temperature was determined as 45℃.

3.1.3 Effect of extraction duration on extraction rate

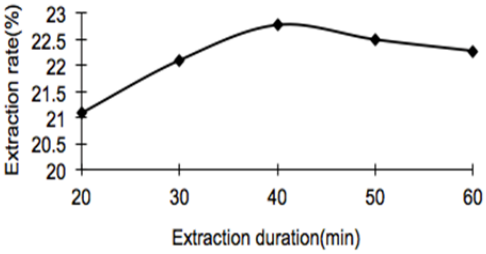

Figure 3: Effect of extraction duration on extraction rate

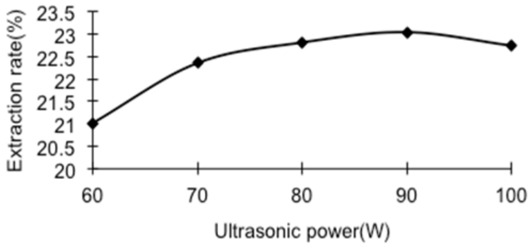

Figure 4: Effect of ultrasonic power on the extraction rate

As shown in Fig. 3, when the extraction duration was extended from 20min to 40min, the extraction rate increased from 21.09% to 22.78%, which was the maximum value. Beyond this, the extraction rate declined. The reason is that ultrasonic treatment promoted the outward diffusion of seed oil from inside the cells and the seed oil was basically dissolved at 40min. However, long-term ultrasonic treatment may lead to the degradation of some seed oil. Therefore, the optimal extraction duration was determined as 40min.

3.1.4 Effect of ultrasonic power on extraction rate

It can be seen from Fig. 4 that as the ultrasonic power increased from 60W to 90W, the extraction rate increased from 21.02% to 23.05%. As the ultrasonic power further increased, the extraction rate began to decline. This is because higher ultrasonic power increased the oscillation rate of solvent and oil and therefore facilitated the dissolution of seed oil. When the ultrasonic power was larger than 90W, the cavitation produced by the ultrasonic waves not only disrupted the cell wall, but also damaged the molecular structure of the seed oil, causing the extraction rate to decline. Therefore, the optimal ultrasonic power was determined as 90W.

3.2 Response surface design

3.2.1 Response surface design and analysis of variance

Box-Behnken design was adopted using the extraction rate of seed oil as the response value. After the screening by single factor experiment, material-liquid ratio (A), extraction temperature (B), extraction duration (C) and ultrasonic power (D) were chosen as the independent variables (3 parallels for each group). The response surface design of 4 factors × 3 levels was adopted. Each level for each factor was encoded as shown in Table 1. The response surface design and the experimental results are shown in Table 2.

Table 1: Factors and levels in response surface design

Factor	Level		
	-1	0	1
A Material-liquid ratio (g/mL)	1:14	1:18	1:22
B Extraction temperature (℃)	40	45	50
C Extraction duration (min)	30	40	50
D Ultrasonic power (w)	80	90	100

Fitting of quadratic polynomial was performed to the experimental data in Table 2 using Design Expert software, and a quadratic multiple regression equation was obtained.

$Y=23.05+0.15A-0.12B+0.13C+0.11D+0.03AB+0.08AC-0.05AD+0.69BC+0.09BD-0.09CD-0.22A^2-0.37B^2-0.53C^2-0.13D^2$.

Table 2: Results of response surface design

No.	Factor				Extraction rate/%	
	A	B	C	D	Measured value	Predicted value
1	0	1	0	-1	22.22	22.23
2	-1	0	1	0	22.19	22.2
3	0	-1	0	-1	22.63	22.64
4	0	-1	1	0	21.68	21.7
5	1	1	0	0	22.5	22.52
6	0	0	-1	-1	22.07	22.06
7	1	0	-1	0	22.23	22.24
8	0	0	0	0	23.06	23.05
9	-1	0	-1	0	22.05	22.1
10	0	1	1	0	22.84	22.84
11	0	0	1	-1	22.48	22.49
12	1	-1	0	0	22.71	22.7
13	0	0	0	0	23.04	23.05
14	0	1	-1	0	21.22	21.21
15	1	0	0	-1	22.8	22.79
16	0	0	-1	1	22.5	22.46
17	-1	-1	0	0	22.49	22.45
18	0	0	0	0	23.05	23.05
19	0	-1	-1	0	22.82	22.82
20	0	1	0	1	22.63	22.63
21	-1	1	0	0	22.18	22.16
22	0	0	1	1	22.55	22.54
23	0	0	0	0	23.06	23.05
24	0	-1	0	1	22.68	22.69
25	0	0	0	0	23.02	23.05
26	-1	0	0	-1	22.4	22.39
27	1	0	0	1	22.9	22.92
28	1	0	1	0	22.69	22.66
29	-1	0	0	1	22.7	22.72

Multiple regression analysis of the extraction data was performed, and the results of analysis of variance and significance test for each term in the regression equation are shown in Table 3. It can be seen from Table 3 that $p<0.0001$ for the model, indicating extreme statistical significance. The p value was 0.131>0.05 in the lack-of-fit test indicating statistical insignificance, and the regression model fit the experimental data well. R^2=99.8%>0.9 for the regression equation, indicating good correlation between the experimental values and the predicted values. R^2_{Adj}=99.6%, indicating that the model explained 99.6% of the variation of response values. (R^2_{Adj}- R^2_{Pred})<0.2 indicated that the model was accurate (Erbay and Icier, 2009). For the monomial term and quadratic term $p<0.001$, indicating extremely significant difference. Therefore, the fitted regression equation was suitable for the analysis of the experimental data and prediction. The interaction terms AC, AD, BC, BD and CD all reached the level of extremely significant difference. As shown by p values and F values, material-liquid ratio had the greatest impact on the extraction rate, followed by extraction duration, extraction temperature and ultrasonic power successively.

Table 3: Analysis of variance for response surface design

Source of variation	Sum of squares	Degree of freedom	Mean square	F value	Prob>F	Significance
Model	5.169	14	0.369	500.917	< 0.0001	**
A-Material-liquid ratio	0.276	1	0.276	374.464	< 0.0001	**
B-Extraction temperature	0.168	1	0.168	227.952	< 0.0001	**
C-Extraction duration	0.198	1	0.198	268.107	< 0.0001	**
D-Ultrasonic power	0.154	1	0.154	209.096	< 0.0001	**
AB	2.50E-03	1	2.50E-03	3.391	0.0868	
AC	0.026	1	0.026	34.729	< 0.0001	**
AD	0.01	1	0.010	13.566	0.0025	**
BC	1.904	1	1.904	2583.488	< 0.0001	**
BD	0.032	1	0.032	43.953	< 0.0001	**
CD	0.032	1	0.032	43.953	< 0.0001	**
A^2	0.304	1	0.304	411.817	< 0.0001	**
B^2	0.894	1	0.894	1213.349	< 0.0001	**
C^2	1.831	1	1.831	2484.231	< 0.0001	**
D^2	0.104	1	0.104	140.441	< 0.0001	**
Residual	0.010	14	7.37E-04			
Lack of fit	9.20E-03	10	9.20E-04	3.286	0.131	Not significant
Pure error	1.12E-03	4	2.80E-04			
Total	5.180	28				
R^2=99.8% R^2_{Adj}=99.6% R^2_{Pred}=98.9% (C.V.)=0.12%						

Note: *P<0.05, indicating significant difference; ** P<0.01, indicating extremely significant difference.

3.2.2 Response surface analysis

The response surfaces obtained from the regression equations are shown in Fig. 5-10, from which we can see the effects of combination of two factors on the extraction rate of seed oil from *Physalisalkekengi L.* when zero level was taken for the remaining two factors. The 3D surface plots of the response in Fig. 5 to 10 are sharp or relatively sharp and the response values vary with the change of the factors. Maximum response value is found within the value range of all 4 factors, indicating that the values of all 4 factors are reasonable. As shown by the contour lines in Fig. 5 to 10, the interaction term AB is represented by circular lines, while other interaction terms are represented by elliptical lines. Thus the interaction term AB reached the level of insignificant difference, whereas the interaction terms AC, AD, BC, BD and CD reached the level of statistically significant difference (Zhao, et al. 2012). This agreed with the analysis of variance.

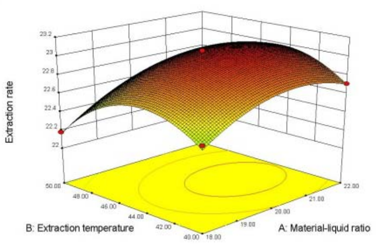

Figure5: Response surface for the combination of material-liquid ratio and extraction temperature

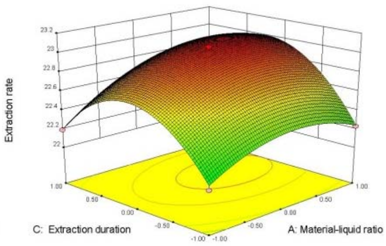

Figure6: Response surface for the combination of material-liquid ratio and extraction duration

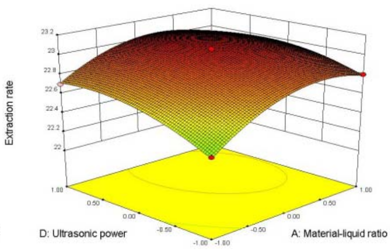

Figure7: Response surface for the combination ofmaterial-liquid ratio and ultrasonic power

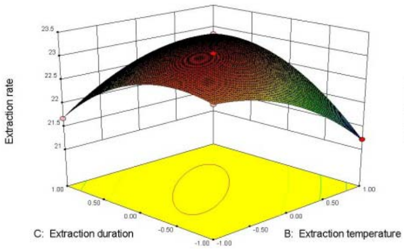

Figure8: Response surface for the combination ofextraction temperature and extraction duration

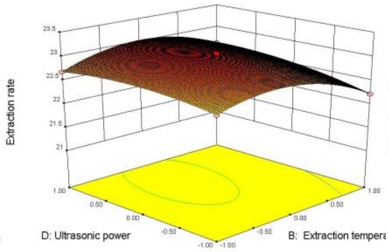

Figure9: Response surface for the combination ofextraction temperature and ultrasonic power

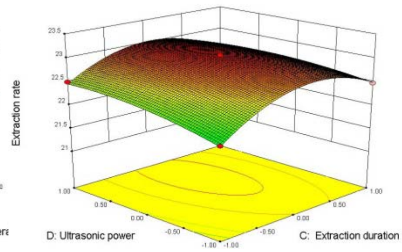

Figure 10: Response surface for the combination of extraction duration and ultrasonic power

3.2.3 Determination of optimal extraction conditions

Using Design Expert software 8.0.6, the optimal process parameters for the extraction of seed oil from *Physalisalkekengi L.* were determined as follows: material-liquid ratio 1:18.43g/mL, extraction temperature 44.06℃, extraction duration 39.45min, and ultrasonic power 92.14W. Under these parameters, the extraction rate of seed oil from *Physalisalkekengi L.* was (23.10±0.02) %.

4. Conclusions

Through single factor experiment, petroleum ether was chosen as the solvent for ultrasound-assisted extraction of seed oil from *Physalisalkekengi L.* Then the process parameters were optimized by using response surface methodology. The four influence factors of extraction efficiency were arranged as follows in the descending order of influence degree: material-liquid ratio, extraction duration, extraction temperature and ultrasonic power. By the fitting of quadratic regression equation, the optimal conditions were finally determined as follows: material-liquid ratio 1:18.43 (g/mL), extraction temperature 44.06℃, extraction duration 39.45min and ultrasonic power 92.14W. Under these parameters, the extraction rate of seed oil from *Physalisalkekengi L.* was (23.10±0.02) %.

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