Study on Preparation and Storage Stability of Camellia Oil-based Gel Oil

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Abstract [**Objectives**] This study was conducted to investigate the optimal preparation conditions and storage stability of camellia oil-based gel oil. [**Methods**] With camellia oil as the base oil, rice bran wax and monoglyceride as gelling agents, a kind of composite gel oil was prepared by the direct gel method. The effects of different mass ratios of rice bran wax to monoglyceride, amounts of gelling agent, heating time and temperatures on the oil precipitation rate and hardness of gel oil were investigated. The optimal preparation conditions were determined by a response surface optimization experiment, and the storage stability of the prepared gel oil was studied with peroxide value and acid value as evaluation indexes. [**Results**] The results showed that the optimal preparation process of gel oil was as follows: mass ratio of rice bran wax to monoglyceride 2 : 8, addition amount of rice bran wax and monoglyceride 10%, heating temperature 95 ℃, and heating time 49 min. The peroxide value and acid value of composite gel oil stored at 4 ℃ and room temperature for 5 weeks both showed an upward trend, and the acid value of gel oil differed significantly at different storage temperatures, which showed that the cold storage environment was more suitable for the gel oil. Compared with the gel oil prepared by single gelling agent, the camellia oil-based gel oil prepared by compounding rice bran wax and monoglyceride had lower oil precipitation rate and moderate hardness. [**Conclusions**] This study lays a theoretical foundation for developing new gel oil and expanding the application scope of camellia oil.

Key words Rice bran wax; Monoglyceride; Camellia oil; Response surface; Storage stability
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At present, traditional baking oils such as vegetable butter, shortening and fresh cream contain a lot of trans fatty acids. Trans fatty acids can lead to obesity, increased blood lipid and increased risk of cardiovascular diseases, and are harmful to human body[1]. Gel oil is a kind of solid oil with supramolecular network structure formed by adding some gelling agents to liquid oil under certain processing conditions. It has the advantages of "zero trans" and low saturated fatty acid[2–4]. A large number of research results show that gel oil, as a new type of special oil for food, can be used to replace commonly used solid fat in the market and is widely used in pharmaceutical, food and cosmetic industries[5]. Gelling agents for preparing gel oil mainly include biological wax, ethyl cellulose, fatty acids and fatty alcohols, phytochemicals (sitosterol and oryzanol), and monoglyceride[6–9]. Camellia oil is a unique traditional edible vegetable oil in China[10]. It is rich in many natural endogenous antioxidant components, such as tocopherol, phytosterol, squalene, fat-soluble vitamins and camellioside[11]. It has the functions of reducing blood lipid, cholesterol, antioxidation and preventing cardiovascular diseases, and has broad application prospects in food, medicine and other industries. However, for the product development of camellia oil, at present, it only stays in edible vegetable oil, and there is little research on camellia oil gel oil.

In this study, rice bran wax and monoglyceride were added to camellia oil in proportion to prepare gel oil, and response surface methodology was applied to optimize the process, and the storage stability was evaluated by peroxide value and acid value, aiming to broaden the application of camellia oil and provide theoretical support for the preparation of new functional oils.

Materials and Methods

Materials and reagents

Camellia oil (Jiangxi Tiancheng Oil and Fat Co., Ltd.); rice bran wax (Shengtao Biotechnology Co., Ltd.); monoglyceride (Jialishi Additive Co., Ltd.).

Instrumentation and equipment

Constant temperature magnetic stirrer (Changzhou Guohua Electric Appliance Co., Ltd.); texture analyzer CT3 (Brookfield Company, USA); centrifuge H/T18MM (Hunan Herexi Instrument & Equipment Co., Ltd.).

Experimental methods

Preparation of gel oil A certain amount of monoglyceride and rice bran wax in different proportions was added to 30 g of camellia oil. The obtained mixture was placed in a constant-temperature magnetic stirring pot, stirred at a constant speed and a certain temperature, and heated for a certain period of time until the added materials were completely dissolved. It was then placed in a refrigerator at 4 ℃ for 24 h to form gel oil.

Single factor experiments

Effect of mass ratio of rice bran wax to monoglyceride on the quality of gel oil Rice bran wax and monoglyceride were added at a certain mass ratio to 30 g of camellia oil (the total amount of

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rice bran wax and monoglyceride accounted for 10% of the mass of camellia oil). The mass ratios of rice bran wax to monoglyceride were set as follows: 0 : 10, 2 : 8, 4 : 6, 6 : 4, 8 : 2 and 10 : 0, respectively. Each part of obtained oil was added in a constant-temperature magnetic stirring pot, stirred at 80 ℃, and heated until the added materials were completely dissolved. After cooling to room temperature, the oil was stored in a refrigerator at 4 ℃ for 24 h. Next, hardness and oil precipitation rate of the oil were measured to determine the appropriate mass ratio of rice bran wax to monoglyceride.

Effect of the amount of rice bran wax and monoglyceride on the quality of gel oil Into 30 g of camellia oil, the compound gelling agent was added at 7%, 8%, 9%, 10% and 11%, respectively. The operation was the same as that in "Effect of mass ratio of rice bran wax to monoglyceride on the quality of gel oil".

Effects of heating temperature on the quality of gel oil Into 30 g of camellia oil, 10% of compound gelling agent was added, and the obtained oil mixture was heated at 80, 85, 90, 95, and 100 ℃, respectively. The operation was the same as that in "Effect of mass ratio of rice bran wax to monoglyceride on the quality of gel oil".

Effects of heating time on the quality of gel oil Into 30 g of camellia oil, 10% of compound gelling agent was added, and the obtained oil mixture was heated for 20, 30, 40, 50, and 60 min, respectively. The operation was the same as that in "Effect of mass ratio of rice bran wax to monoglyceride on the quality of gel oil".

Response surface experiment

The factors and levels set in the response surface experiment are shown in Table 1.

Table 1 Factor level coding table

Level	Factor		
	Addition amount of gelling agent A//%	Heating time B//min	Heating temperature C//℃
-1	9	40	90
0	10	50	95
1	11	60	100

Study on storage stability The gel oil was placed at room temperature and 4 ℃, respectively. Its acid value and peroxide value were measured at regular intervals, so as to study the degree of oxidative deterioration of gel oil at different storage temperatures and evaluate its storage stability.

Determination of gel oil quality

Determination of oil precipitation rate The determination of oil precipitation rate referred to the method of Si *et al.* [13]. Three parallel tests were done for each group to take an average value.

Determination of hardness The hardness of prepared gel oil was determined according to the parameters of texture analyzer in Dai's study[14]. Three parallel tests were done for each group to take an average value.

Determination of peroxide value The determination of peroxide value referred to the first method "Titration" in GB 5009.227—2016 *Determination of Peroxide Value in Food.*

Determination of acid value The acid value of prepared gel oil was determined according to the first method "Cold solvent indicator titration" in GB 5009.227-2016 *Determination of Acid Value in Food.*

Data analysis SPSS 25.0 was employed for correlation analysis, and Origin 8.0 was employed for drawing. All the data were the averages of three repeated tests.

Results and Analysis

Single factor experiments

Effect of mass ratio of rice bran wax to monoglyceride on gel oil With the mass ratio of rice bran wax to monoglyceride varying, the oil precipitation rate and hardness of gel oil changed, as shown in Fig. 1 and Fig. 2. It can be seen from Fig. 1 that with the the mass of monoglyceride increasing, the oil precipitation rate increased and reached a peak of 10.05% when the mass ratio of rice bran wax to monoglyceride was 6 : 4. The value decreased when the mass ratio of rice bran wax to monoglyceride changing from 6 : 4 to 2 : 8, and reached a minimum of 1.99% when the mass ratio of rice bran wax to monoglyceride was 2 : 8. However, when the mass ratio of rice bran wax to monoglyceride was 0 : 10, the oil precipitation rate increased again to 7.83%. In Fig. 2, when the mass ratio of rice bran wax to monoglyceride was 10 : 0, the hardness was slightly higher because rice bran wax was added alone, but with the mass ratio of rice bran wax to monoglyceride keeps increasing from 8 : 2 to 2 : 8, the hardness increased continuously and reached a peak of 225 g when the mass ratio of rice bran wax to monoglyceride was 2 : 8. When the mass ratio of rice bran wax to monoglyceride was 0 : 10, the hardness decreased slightly, which might be due to that the gelling agent was single monoglyceride. It could be seen that compared with monoglyceride, rice bran wax had stronger ability to bind oil. To sum up, the mass ratio of rice bran wax to monoglyceride with the highest hardness and the lowest oil precipitation rate was 2 : 8. This result is consistent with the suitable ratio of monoglyceride to rice bran wax in the preparation process of rapeseed oil-based gel oil by combining rice bran wax with monoglyceride studied by Niu *et al.* [15].

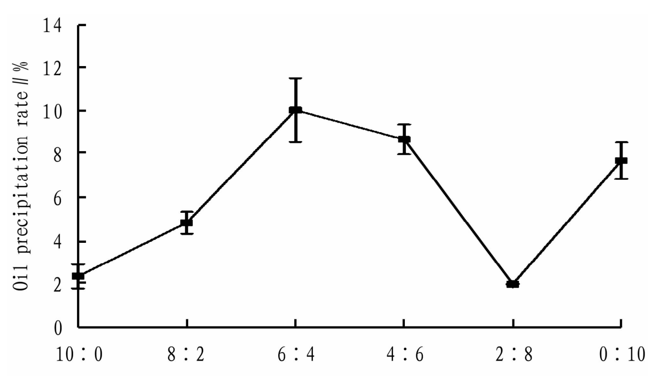

Fig. 1 Variation of oil precipitation rate of gel oil with different mass ratios of rice bran wax to monoglyceride

Effect of addition amount of rice bran wax and monoglyceride on gel oil With the addition amount of gelling agent changing, the oil precipitation rate and hardness of gel oil changed, as shown in Fig. 3 and Fig. 4. As can be seen from Fig. 3, with the increase of the addition amount of gelling agent (from 7% to 11%), the oil precipitation rate gradually decreased, from 5.73% to 1.93%, but when the addition amount increased from 10% to 11%, the change of oil precipitation rate was not significant, which might be due to that the increase of the addition amount of gelling agent made the gel network more compact and improved the solidification effect of the liquid. As can be seen from Fig. 4, with the increase of the addition amount (from 7% to 11%), the hardness of gel oil also showed an upward trend, from 210 to 655 g. Because the difference of oil precipitation rate was small between the gelling agent addition amounts of 10% and 11%, considering the cost, the addition amount of rice bran wax and monoglyceride was selected as 10%.

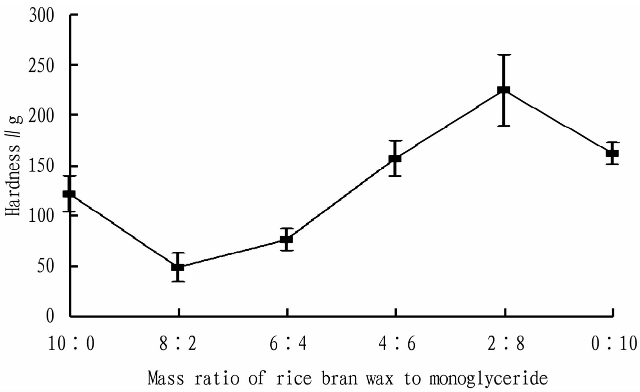

Fig. 2 Changes in hardness of gel oil with different mass ratios of rice bran wax to monoglyceride

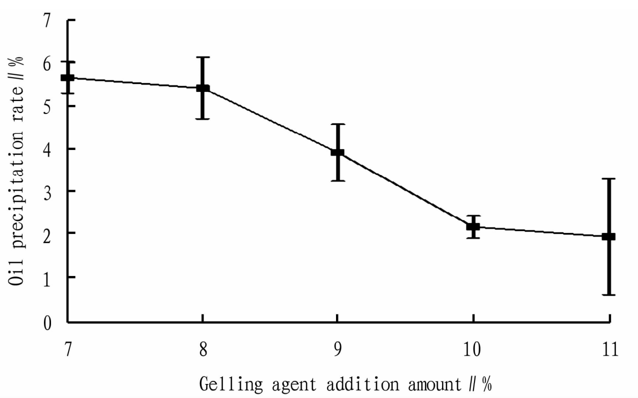

Fig. 3 Variation of oil precipitation rate of gel oil with different gelling agent addition amounts

Effect of heating temperature on gel oil With the change of heating temperature, the oil precipitation rate and hardness of gel oil changed, as shown in Fig. 5 and Fig. 6. It can be seen from Fig. 5 that with the heating temperature increasing (from 80 to 95 ℃), the oil precipitation rate of gel oil gradually decreased, reaching the lowest at 95 ℃, from 3.47% to 1.5%. The oil precipitation rate increased again to 3.5% at 100 ℃, which might be due to the fact that rice bran wax and monoglyceride were more fully dissolved in camellia oil with the increase of temperature, but the structure of rice bran wax and monoglyceride might be destroyed at too high temperature, and the oil precipitation rate increased. As can be seen from Fig. 6, the hardness of gel oil also increased with the temperature increasing (from 80 to 95℃) and reached a peak of 745 g at 95 ℃, but it decreased to 370 g at 100 ℃, probably because the molecular structure of monoglyceride and rice bran wax was destroyed by high temperature. To sum up, the heating temperature was selected as 95 ℃.

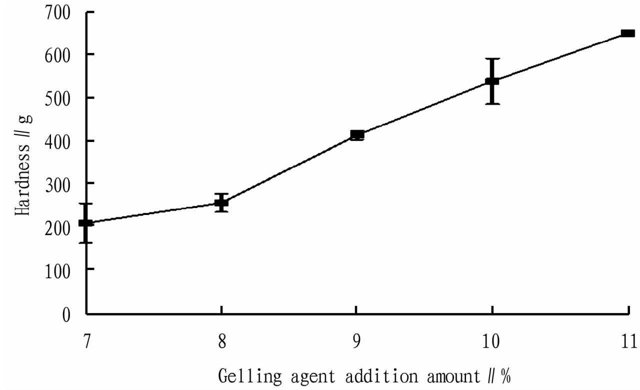

Fig. 4 Changes in hardness of gel oil with different gelling agent addition amounts

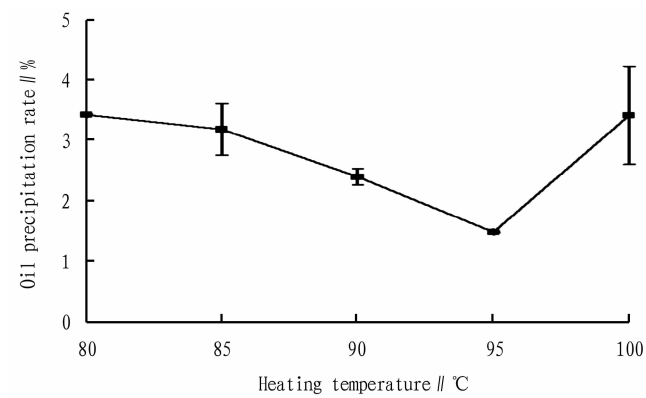

Fig. 5 Variation of oil precipitation rate of gel oil at different heating temperatures

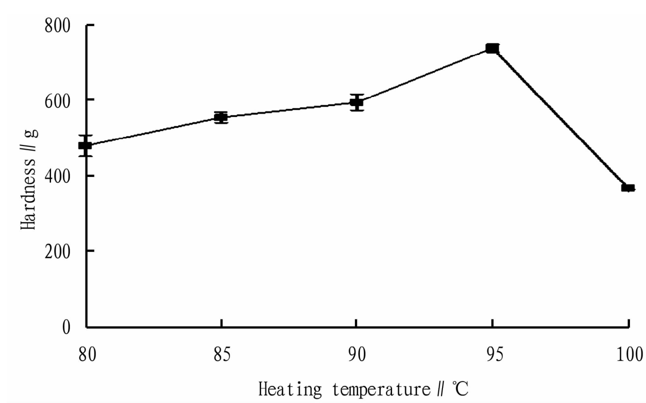

Fig. 6 Variation of hardness of gel oil at different heating temperatures

Effect of heating time on gel oil With the change of heating time, the oil precipitation rate and hardness of gel oil changed, as shown in Fig. 6 and Fig. 7. As can be seen from Fig. 6, with the heating time increasing (from 20 to 60 min), the oil precipitation rate of gel oil gradually decreased from 6.4% to 2%, which might be due to the full dissolution of rice bran wax and monoglyceride with the increase of heating time, making the structure between gelling agent and gel oil more compact. As can be seen from Fig. 8, with the heating time increasing, the hardness of gel oil first increased and then decreased, and it reached a peak of 675 g when the heating time was 40 min. To sum up, considering the heating time comprehensively, 50 min was selected.

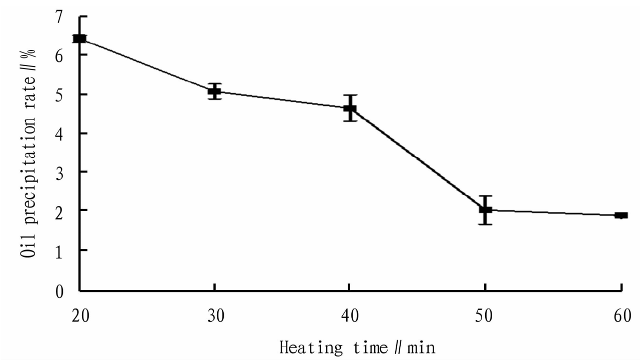

Fig. 7 Changes of oil precipitation rate of gel oil under different heating time

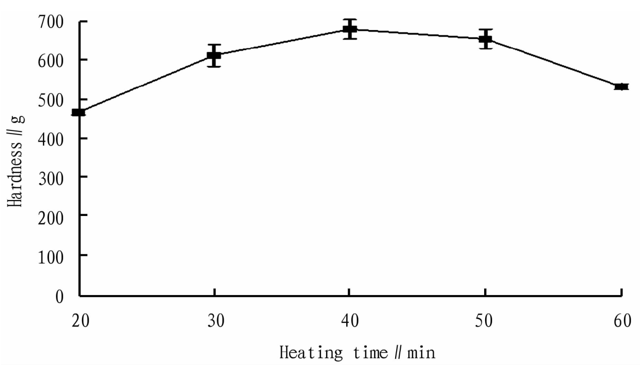

Fig. 8 Changes in hardness of gel oil under different heating time

Analysis of response surface results

On the basis of single factor experiments, the addition amount of gelling agent (A), heating temperature (B) and heating time (C) were selected as independent variables, and the oil precipitation rate of gel oil (y) was taken as response value. A three-factor three-level experimental scheme was designed to optimize the preparation process of composite gel oil. The response surface results are shown in Table 2.

Software Design-Expert10 was employed to perform regression on the experimental results in the table, and the multivariate quadratic equation of the response value y and three factors was obtained as follows: Oil precipitation rate $= 2.184 - 0.893\,75A + 0.331\,25B - 0.127\,5C - 0.537\,5AB + 0.46AC + 0.035BC + 1.914\,25A^2 + 0.929\,25B^2 + 0.971\,75C^2$.

Table 2 Response surface results

Experiment No.	A	B	C	Oil separation rate // %
1	10	50	95	1.98
2	10	40	90	4.30
3	10	50	95	1.47
4	10	40	100	3.98
5	11	50	90	3.50
6	11	50	100	4.16
7	11	40	95	4.30
8	10	50	95	2.10
9	9	40	95	4.32
10	10	50	95	2.43
11	9	50	100	5.72
12	11	60	95	4.66
13	9	60	95	6.83
14	9	50	90	6.90
15	10	60	90	4.12
16	10	50	95	2.94
17	10	60	100	3.94

Table 3 Regression analysis of variance

Source of variation	Sum of squares	Degree of freedom	Mean square	*F* value	*P* value	Significance
Model	34.72	9	3.86	8.08	0.005 8	* *
Addition amount of gelling agent A	6.39	1	6.39	13.38	0.008 1	
Heating time B	0.877 8	1	0.877 8	1.84	0.217 3	
Heating temperature C	0.130 1	1	0.130 1	0.272 3	0.617 9	
AB	1.16	1	1.16	2.42	0.163 8	
AC	0.846 4	1	0.846 4	1.77	0.224 8	
BC	0.004 9	1	0.004 9	0.010 3	0.922 2	
A^2	15.43	1	15.43	32.31	0.000 7	
B^2	3.64	1	3.64	7.61	0.028 1	
C^2	3.98	1	3.98	8.32	0.023 5	
Residual	3.34	7	0.477 6			
Lack of fit	2.15	3	0.717 6	2.41	0.207 3	
Pure error	1.19	4	0.297 6			
Total deviation	38.06	16				
R^2	0.912 2					
R^2_{Adj}	0.799 2					

" * * " represents an extremely significant difference ($P < 0.01$).

The variance analysis of response surface regression is shown in Table 5. From Table 5, it can be seen that the established regression model was significant, $P = 0.0058$, but the lack of fit item was not significant, $P = 0.2073 > 0.05$. R^2 was 0.912 2, which is close to 1, indicating that the linear relationship between the dependent variable and all the independent variables was significant, and the fitting degree of the selected quadratic polynomial model was good and the model was reliable.

The software Design-Expert13.0 was employed to analyze the regression model. The 3D diagrams of the effects of the interaction between two of such three factors as the amount of gelling agent, heating temperature and heating time on the oil precipitation rate of camellia oil-based gel oil were obtained.

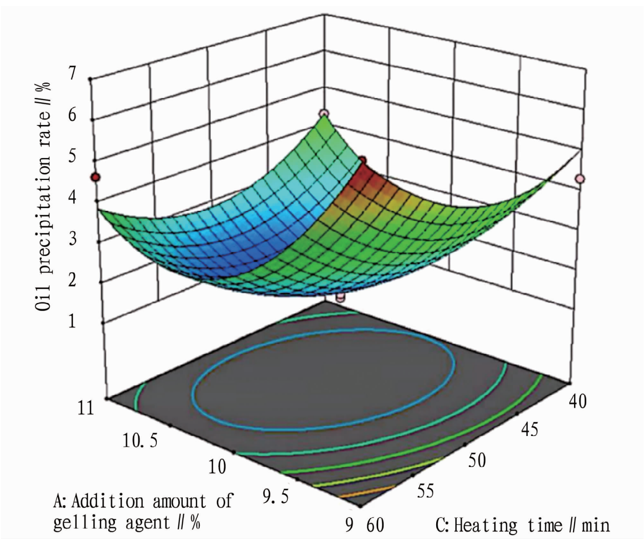

Fig. 9 Curved surface of the effect of heating time and gelling agent addition on oil precipitation rate

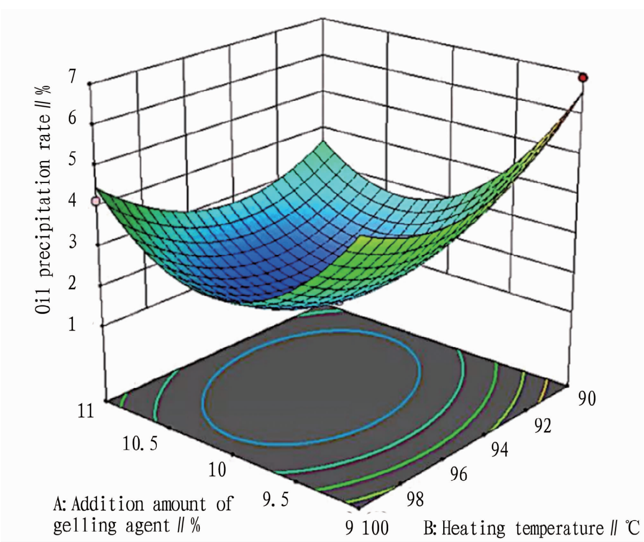

Fig. 10 Curved surface of the effect of heating temperature and gel addition on oil precipitation rate

The 3D diagrams of response surface could explain the relationship between preparation factors and the oil precipitation rate of camellia oil-based gel oil. The steeper the surface slope, the more significant the impact of influencing factors on the oil precipitation rate of gel oil. As shown in Fig. 9 and Fig. 10, the slopes of the two curved surface were steeper, indicating that the factors significantly affected the oil precipitation rate of gel oil. Fig. 11 shows that the surface inclination of heating temperature and heating time was low, so the effect of the factor on gel oil was not significant. Through the fitting analysis of the model, the optimal preparation conditions were predicted as follows: addition amount of composite gelling agent 10.215%, heating temperature 95.082℃, and heating time 48.8 min. Under these optimized preparation conditions, the predicted oil-holding capacity of composite camellia oil gel was 2.068%.

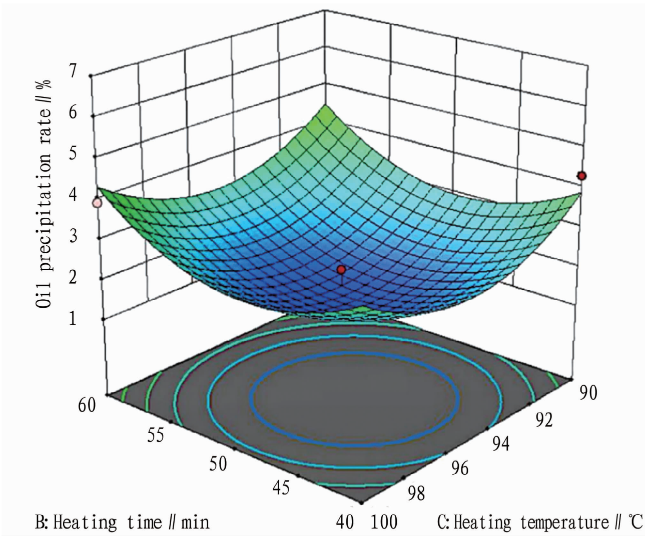

Fig. 11 Curved surface of the effect of heating temperature and heating time on oil precipitation rate

Verification test

According to practical operation, the optimal preparation conditions were adjusted as follows: addition amount 10%, heating temperature 95 ℃, and heating time 49 min. The test was repeated for three times. The average oil precipitation rate of composite gel oil was 1.72%, which was 0.348% different from the predicted value. It indicated that the model was accurate and reliable, and achieved the purpose of optimizing the preparation conditions.

Study on storage stability

Changes of peroxide value at different storage temperatures

As shown in Fig. 12, the peroxide value of gel oil increased after it was stored at room temperature and at 4 ℃ for five weeks, and the peroxide value of gel oil at 4 ℃ was lower than that stored at room temperature. The peroxide value increased from 0.115 to 0.148 mg/g at 4 ℃, while the peroxide value of gel oil stored at room temperature increased from 0.141 to 0.17 mg/g. The peroxide values measured at 4 ℃ were all less than those measured at room temperature, which showed that the properties of gel oil were more stable at 4 ℃. The measured data are in line with the stipulation that the peroxide value of camellia seed oil should be less than 0.25 mg/g in GB/T 11765-2018 *Oil-tea camellia seed oil*.

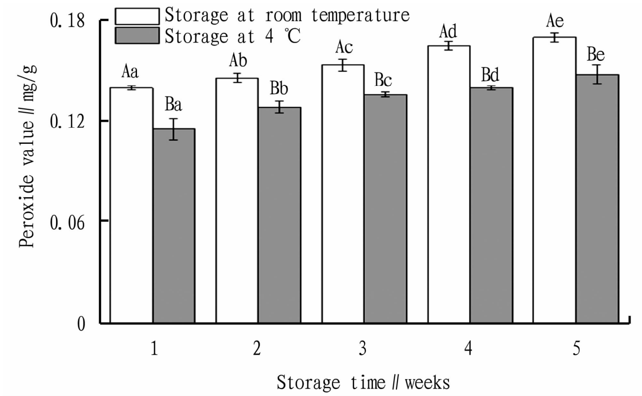

Fig. 12 Changes of peroxide value of gel oil at different storage temperatures

Changes of acid value at different storage temperatures It can be seen from Fig. 13 that the acid value of gel oil increased with time after being stored at 4 ℃ and room temperature for a period of time. The acid value increased from 0.64 to 1.66 mg/g at room temperature and from 0.6 to 1.32 mg/g at 4 ℃. The acid value of gel oil stored at 4 ℃ was lower than that of gel oil stored at room temperature, but the difference in acid value between gel oil stored at 4 ℃ and at room temperature for 1 – 2 weeks was small, which might be due to that the change of gel oil was not significant in a short storage time. It could be seen that cold storage environment was more conducive to the storage of gel oil. The measured data are all in line with the stipulation that the acid value of camellia seed oil is less than 2 mg/g in GB/T 11765-2018 *Oil-tea Camellia Seed Oil*.

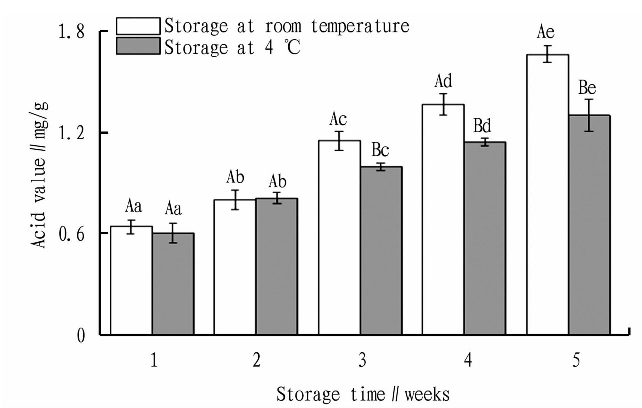

Fig. 13 Changes of acid value of gel oil under different storage conditions

Conclusions and Discussion

In this study, the optimal technical conditions for preparing camellia oil-based gel oil by compounding rice bran wax and monoglyceride were determined as follows: mass ratio of rice bran wax to monoglyceride 2 : 8, addition amount of rice bran wax and monoglyceride 10%, heating temperature 95 ℃, and heating time 49 min. The peroxide value and acid value of composite gel oil stored at 4 ℃ and room temperature for 5 weeks both showed an upward trend, and the acid value of gel oil differed significantly under different storage conditions, which showed that the cold storage environment was more suitable for the storage of gel oil. To sum up, the preparation of camellia oil-based gel oil by compounding rice bran wax and monoglyceride provides a new idea for the development of new gel oil, and it can be used as a substitute for solid fat. This study provides a certain theoretical basis for the development of healthy oil. Meanwhile, it expands the application scope of camellia oil, changes the inherent form of camellia oil and provides a theoretical basis for the development of camellia oil-based gel oil products.

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