Original Article
Food Science and Technology

ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

DOI: https://doi.org/10.1590/fst.78121

Optimization of raw material properties of natural starch by food glue based on dry heat method

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Abstract

Starch is a versatile food component with several industrial applications. Its chemically altered derivatives provide a wide range of high-tech applications in the food and non-food markets. Altered starches are intended to compensate for one or more of native starches' drawbacks, such as decrease of viscosity and hardening ability during processing and preservation, especially at low pH, syneresis, retrogradation properties, and so on. Some of the most frequent modifications used to create starch derivatives include cross-linking, dextrinization, hydroxy alkylation, esterification, and oxidation. The effect of the amount of modified starch, the amount of glycerol, and the drying temperature on the film performance of dry heat modified lotus seed starch were studied. The film performance indexes were optimized by response surface analysis and weighted evaluation function method. The optimal film forming conditions for dry heat modified lotus seed starch was obtained as follows: the amount of modified lotus seed starch was 3.53%, the amount of glycerol was 1.51%, and the drying temperature was 80 °C. This research result will provide theoretical guidance for the industrial production of dry heat-modified lotus seed starch edible film.

Keywords: chemically altered; low pH; starch derivatives; dietary fibre; food market.

Practical Application: Food packaging by dry heat-modified lotus seed starch edible films.

1 Introduction

Modified starches have been studied and produced for more than a century, and they are used in a variety of sectors including food, paper, and textiles (Rapaille & Vanhemelrijck, 1997; Reddy & Seib, 1999). To improve the quality of different prepared meals such as snack foods, breads, and cakes, chemically modified starches such as esterified, etherified, and/or cross-linked starches have recently been widely employed (Miller et al., 1991; Miyazaki et al., 2006). After being chemically, physically, or enzymatically modified, starch, a major component of cereal grains and roots, is utilized in the food, paper, and textile industries. Using amylolytic enzymes, some portions of starch are hydrolyzed into maltodextrin or dextrin, a low molecular weight starch. In the food and pharmaceutical sectors, enzymatically modified starches are often utilized. Pregelatinization and heat-treatment of starch are examples of physical modifications (Abbas et al., 2010; Guzmán-Maldonado & Paredes-López, 1995). According to a study, physically changed starches may now be made via a dry heating technique (DHT) (Gou et al., 2019; Maniglia et al., 2020). Thermally treated starches were shown to be practically comparable to chemically cross-linked starches, according to the researchers. Different forms of gums (hydrocolloids) combined with starch have been found to improve the properties of starch products and heat treatment (Chi et al., 2019). The research on starch modification by dry heating with ionic gums was originally published in a paper (Lim et al., 2002). Dry heating with sodium alginate or carboxymethylcellulose (CMC) increased the viscosity of waxy maize starch paste but decreased the viscosity of potato starch paste. Dry heat treatment with xanthan generated the most significant increases in paste viscosity of the starches when compared to sodium alginate and CMC (Lim et al., 2003; Sun et al., 2013). The waxy rice starch was discovered to have a continual rise in pasting viscosity when heated with a combination of phosphate salts and xanthan. (Li et al., 2008). More research is needed to understand the chemical process between starch and ionic gum when heated dry.

Currently, the use of new edible film packaging materials to replace plastic packaging has become a new trend in the development of food packaging (Pilli, 2020; Iamareerat et al.,

Received 17 May, 2021
Accepted 06 Oct., 2021
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2018). The type and nature of starch, preparation conditions, and additives are the main factors affecting the performance of edible starch films (Campo et al., 2016). The effects of starch content, film-forming temperature, type, the content of plasticizers, and reinforcing components on the properties of starch films have been studied (Abral et al., 2021). However, the edible starch films, especially those made of straight-chain, high-straight-chain, and hydroxypropyl high-straight-chain starches, are limited in their application due to plasticization moisture barrier effect, and poor mechanical properties.

In this study, the effects of different amounts of modified starch, glycerol, and drying temperature on the film-forming characteristics of dry heat modified lotus seed starch were investigated. Response surface analysis and the weighted evaluation function method were investigated used to optimize the film-forming parameters. The optimal process parameters were determined by response surface analysis and the weighted evaluation function method.

2 Material and methods

2.1 Material

Material and reagents

Frozen fresh lotus**,** provided by Greenfield Fujian Grievance Food Co.; Glycerol and calcium chloride are of analytical purity; sodium alginate food grade.

Instruments

UV-2002, UV-Visible spectrophotometer; SPX-150C constant temperature and humidity incubator; DJG-9053A electric blast drying box; SE202F electronic balance; HH-6 digital display thermostatic water bath; Z-123 digital display micrometre; EZ-TEST mass spectrometer.

2.2 Methods

Extraction of lotus seed starch

Place the thawed frozen lotus seeds in a high-speed tissue masher, add a little water and pulp them, and pass them through a 100-mesh sieve; The filtrate precipitation was allowed to stand for 6 h. The upper slurry water was discarded and the lower precipitate was dissolved in distilled water. The lower layer of precipitated powder was dried in an oven at 45 °C for 13 h. The powder was then allowed to precipitate for 4 h and repeated 3 times (moisture content 10% or so), instantly obtain lotus seed starch (Zeng et al., 2009).

Preparation of dry heat denaturing lotus seed starch

Take 40 mL of distilled water and add 0.2 g of sodium alginate, after it is dissolved, add 19.8 g of lotus seed starch and stir for 30 m to make it evenly mixed, adjust the pH of the solution to 7; pour into a flat tray and dry in an oven at 45 °C for 13 h (moisture content 10% or less). The powder was ground and crushed through 80 mesh sieves, and then heated at 130 °C for 3 h.

Production process of dry heat-denatured lotus seed starch film

Take a certain amount of dry heat-denatured lotus seed starch and dissolve it in distilled water to form starch milk and add a certain amount of glycerol and put it in a boiling water bath to paste for 30 min; Then, the film was placed in a water bath at 50 °C for 10 min and then dried in a blast oven for 72 h (temperature: 25%, relative humidity: 58%.); final measurement of the membrane indexes.

Membrane performance measurement

(1) Measurement of film thickness. The average value was measured by a micrometre with a precision of 0.001 mm on the film to be measured.

(2) Determination of tensile strength and elongation at break of membranes. The tensile strength and elongation at break of the film to be measured were determined using a mass spectrometer, and the film to be measured was cut into strips of 12 cm long and 1.5 cm wide, and the effective tensile length was controlled to be 10 cm.

(3) Determination of water vapor transmission coefficient of membrane. The cup method of GB/T 1037-1988 was adopted and slightly improved. The $CaCl_2$ was crushed and made uniform in size and dried in an oven at 200 °C for 2 h. After it cooled, it was added to the dry weighing bottle at room temperature; Seal the mouth of the weighing bottle with a uniform film without holes and wrinkles, then double seal the film with adhesive tape and paper tape, and weigh the bottle in a constant temperature and humidity chamber (maintain relative humidity at 58% and temperature at 25 °C); The water vapor permeability coefficient (WVP) of the membrane to be measured is calculated by measuring the weight gain of the weighing bottle in a certain period of time.

(4) Determination of membrane solubility. The denatured starch film to be tested was cut into 2 cm and 3 cm pieces and placed in a desiccator with anhydrous silica gel for 7 days. The sample was weighed and recorded as W_0 (approximated to 0.000 1 g). Place in a conical flask, add 80 mL of deionized water and shake at 25 °C for 1 h in an oscillating water bath with a frequency of 50 Hz. The undissolved membrane was then filtered and dried in an oven at 60 °C to a constant weight and weighed as W_1 (Romero-Bastida et al., 2005).

(5) Measurement of film transparency. The measured film was cut into a rectangle of 1 cm and 2 cm and attached to the surface of a cuvette with a side length of 1 cm, and its transmittance (%) was measured at 650 nm (Malumba et al., 2010).

(6) Determination of membrane puncture strength. The method of Mali et al. (2005) is used. The film to be tested is cut into a 4.1 cm diameter circle and placed on the mass

spectrometer plate. A 3 mm diameter puncture needle is selected, and the travel speed of the mass spectrometer is set to 0.8 mm/s until the specimen is punctured. The force-deformation curve was obtained until the specimen was punctured, and the force of the specimen at the puncture point was recorded in F, expressed in N, and measured three times in parallel.

3 Experimental design

(1) Single-factor experimental design. The fixed glycerol mass fraction of 1.5% and drying temperature of 80 °C were used to compare the effects of different denatured lotus seed starch mass fractions of 3.0%~5.0% on the edible film properties. The effect of different glycerol mass fractions (1.0%~3.0%) on the performance of edible films was compared with that of modified lotus seed starch at 3.5% mass fraction and drying temperature of 80 °C. Fixed denatured lotus seed starch mass fraction of 3.5% and glycerol mass fraction of 1.5% to compare the effects of different drying temperatures of 50~90% on the edible film properties.

(2) Response surface experimental design. Based on the single-factor test, the amount of modified lotus seed starch, the amount of glycerol and the drying temperature were selected as the test factors. The tensile strength, elongation at break, water vapor permeability coefficient and transparency were used as indicators. A 3-factor, 3-level Box-Benhnken experimental design was used to optimize the film-forming process parameters by using a weighted evaluation function method to combine and standardize the four indicators (Ferreira et al., 2007).

4 Data statistics and analysis

The results of the one-way test were analysed using DPS7.05. The Box-Benhnken model in Design-Expert 8.05 was used to perform linear regression and ANOVA on the response surface test results.

5 Results and discussion

5.1 Effect of modified starch dosage on the performance of dry heat denaturing lotus seed starch film

As shown in Table 1, the force between sodium alginate and starch molecules increases as the content of sodium alginate increases, thus increasing the continuity and denseness of the film. As the content of modified starch increases, the film's water content decreases, and the excess starch molecules form a rigid structure, making the film stiffer and more brittle, and prone to breakage. When the amount of dry heat-denatured lotus seed starch in the film solution is 3.5%, the performance of the starch film is relatively optimal.

5.2 Effect of glycerol dosage on the properties of dry thermally denatured lotus seed starch film

It can be seen from Table 2 that the addition of glycerol increases the number of hydroxyl groups per unit volume and increases the number of bound water molecules. When the content of glycerol is low, the intermolecular bonding between starch molecules is strong, but the film formed is thin and dry due to the few water molecules bonded to it, and it is less flexible and easier to break.

5.3 Effect of drying temperature on the properties of dry thermally denatured lotus seed starch film

From Table 3, it can be seen that the mechanical properties of the starch film are best when the drying temperature is 80%. This is because the suitable drying temperature helps the bonding and orientation of the molecules and the film structure formed is orderly and dense. When the temperature is low, the drying time is long, and the glycerol binds too much water, the starch content in the membrane skeleton is relatively reduced, the membrane density is reduced, the structure is loose, the membrane is thicker, the water vapor permeability coefficient is high, the solubility is high, and the transparency is low. When the temperature is too high, the water evaporates too fast, and

Table 1. The effect of modified starch on the qualities of the film.

Amount of modified starch/ %	Tensile strength /Mpa	Elongation at break /%	*WVP*	Puncture strength /N	Transparency /%	Solubility /%
3.0	3.68	48.64	2.36	5.65	77.99	62.89
3.5	5.75	42.90	1.93	8.88	85.15	60.15
4.0	4.84	34.36	2.22	8.97	79.93	52.62
4.5	5.09	28.05	2.04	8.73	74.33	51.49
5.0	5.46	26.53	2.81	10.41	75.23	51.60

Table 2. The effect of adding glycerol on a film's qualities.

Glycerin /%	Tensile strength /Mpa	Elongation at break /%	*WVP*	Puncture strength /N	Transparency /%	Solubility /%
1.0	2.08	20.60	1.36	5.76	73.90	59.49
1.5	4.84	34.36	2.22	8.97	79.93	52.62
2.0	2.24	33.42	5.25	3.94	77.20	62.14
2.5	1.15	29.88	7.24	2.76	76.40	65.56
3.0	0.97	27.69	8.42	1.83	73.90	63.80

Table 3. The effect of drying temperature on the qualities of the film.

Drying temperature /°C	Tensile strength /Mpa	Elongation at break /%	*WVP*	Puncture strength /N	Transparency /%	Solubility /%
50	2.79	51.70	3.16	5.39	73.70	53.03
60	2.21	35.96	2.96	4.42	73.10	55.12
70	2.77	30.73	2.66	4.48	74.30	52.53
80	4.84	34.36	2.20	8.97	79.90	52.62
90	3.54	45.25	3.20	5.02	81.30	48.85

the molecular movement increases and the hydrogen bonds between glycerol and water molecules are not sufficiently formed, and the molecules are deposited without oriented arrangement. The brittle and hard texture of the film is not easy to tear, the tensile strength and puncture strength are low, and the water molecules can easily pass through the film, further reducing the solubility and transparency.

5.4 Optimization of dry thermal denaturing lotus seed starch film preparation process

Based on the results of the single-factor test, the amount of dry heat-denatured lotus seed starch, the amount of glycerol, and the drying temperature were selected as the test factors, and the tensile strength, elongation at break, water vapor permeability coefficient, and transparency were used as the indicators to conduct the response surface analysis of the dry heat-denatured lotus seed starch film formation process. The Box-Benhnken model in the Design-Expert version 8.0.6 software was used to carry out the 3-factor, 3-level experimental design, and the results are shown in Tables 4 and 5.

Regression modelling and significance of dry heat-denatured lotus seed starch film

Since the effect of each factor level on the response value of each index is not a simple linear relationship, the coefficients in the quadratic multiple regression equation between each factor and each response value were tested according to the experimental results in Table 5, and the regression equation was obtained after excluding the insignificant term at the level of significance of 0.5.

Tensile strength regression model: $R1=5.56+0.35A -0.60A^2-1.50B^2-2.02C^2$

Regression model for elongation at break: $R2 =36.19 - 1.26A + 2.43B - 1.60C - 1.05AB - 4.54A^2 - 1.92B^2 - 6.63C^2$

Water vapor permeability coefficient regression model: $R3 = 7.53 -0.87A + 2.23B-0.93C-1.19BC+0.97A^2+1.86B^2+6.74C^2$

Transparency regression model: $R4=91.59+3.10C+3.37AC-4.12A^2-4.71B^2-2.57C^2$

In order to find an optimal process parameter for each index of dry heat-denaturing lotus seed starch film, we need to find the best process parameter to meet the requirements of high tensile strength (R1), high elongation at break (R2), low water vapor transmission coefficient (R3), and high transparency (R4), and we use the weighted evaluation function method to combine the four indexes into one set of factors. The weighted evaluation function method is used to standardize the 4 indexes into one set of factors, which is expressed by Y = {tensile strength, elongation at break, water vapor permeability coefficient, transparency}.

Table 4. Factors and levels coding of Box-Benhnken experiment.

code	Factors		
	A (Amount of modified starch) %	B (Glycerin dosage) %	C (Drying temperature) °C
-1	3.0	1.0	70
0	3.5	1.5	80
1	4.0	2.0	90

6 Response surface interaction

(1) Interaction of modified starch dosage and glycerol dosage. From Figure 1, we can see that the slope of the response surface is relatively gentle, indicating that the interaction between the amount of modified starch and glycerol can be tolerated by the composite index of the response value. When the amount of modified starch is fixed, the value of Y increases and then decreases as the amount of glycerol increases, and when the amount of glycerol is fixed, the value of Y increases and then decreases as the amount of modified starch increases. The contour shape is nearly circular, it shows that the interaction effect between the amount of modified starch and the amount of glycerin is not significant, this is consistent with the ANOVA results.

(2) Interaction of modified starch dosage and drying temperature. It can be seen from Figure 2 that the slope of the response surface is steeper, indicating that the combined index of the response value is more sensitive to the change of the amount of modified starch and drying temperature. When the amount of modified starch is fixed, the value of Y is increased and then decreased rapidly with the increase of drying temperature. When the drying temperature is kept constant, the Y value increases and then decreases as the amount of modified starch increases, and the drying temperature has a more significant effect on the overall index than the amount of modified starch. The elliptical shape of the contour line indicated that the interaction between the amount of modified starch and the drying temperature was significant, which was consistent with the ANOVA results.

Table 5. Experiment design and response surface results.

Test number	Factor level			Indicators			
	A	B	C	R1	R2	R3	R4
	Amount of modified starch/%	Glycerin dosage/%	Drying temperature/°C	Tensile strength/MPa	Elongation at break/%	WVP	Transparency/%
1	-1	-1	0	2.95	27.27	9.6	82.85
2	1	-1	0	3.66	26.56	7.26	82.71
3	-1	1	0	3.28	35.02	13.69	83.13
4	1	1	0	3.95	30.09	10.88	82.35
5	-1	0	-1	2.18	28.47	16.53	84.74
6	1	0	-1	3.19	24.73	15.73	78.62
7	-1	0	1	3.01	23.79	14.85	84.43
8	1	0	1	3.4	23.09	13.85	91.78
9	0	-1	-1	2.01	26.94	13.39	81.14
10	0	1	-1	2.3	31.57	20.82	81.49
11	0	-1	1	1.88	24.25	13.82	85.66
12	0	1	1	1.98	27.82	16.5	88.93
13	0	0	0	5.31	36.16	7.9	91.68
14	0	0	0	5.82	36.8	7.05	91.45
15	0	0	0	4.95	35.18	8	91.93
16	0	0	0	6.08	35.99	6.85	90.74
17	0	0	0	5.63	36.83	7.83	92.13

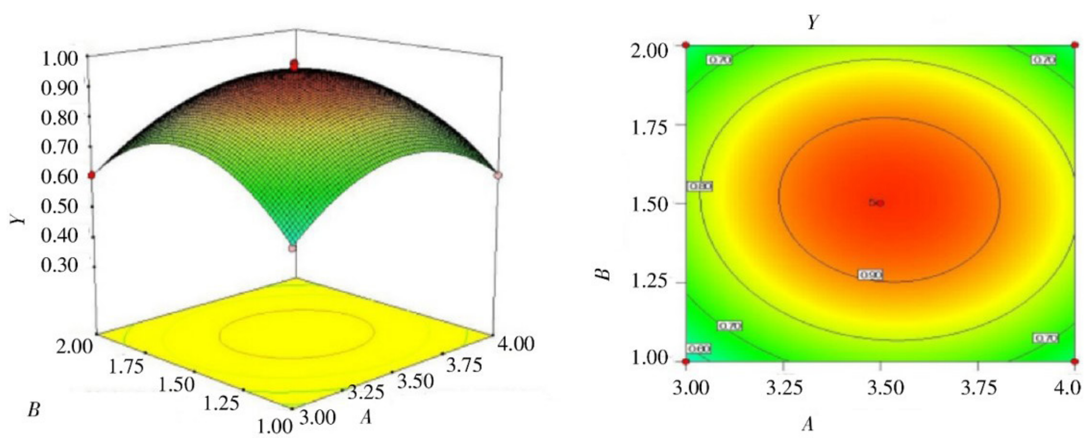

Figure 1. Response surface plots for the effect of addition of modified starch and glycero on comprehensive index (Y).

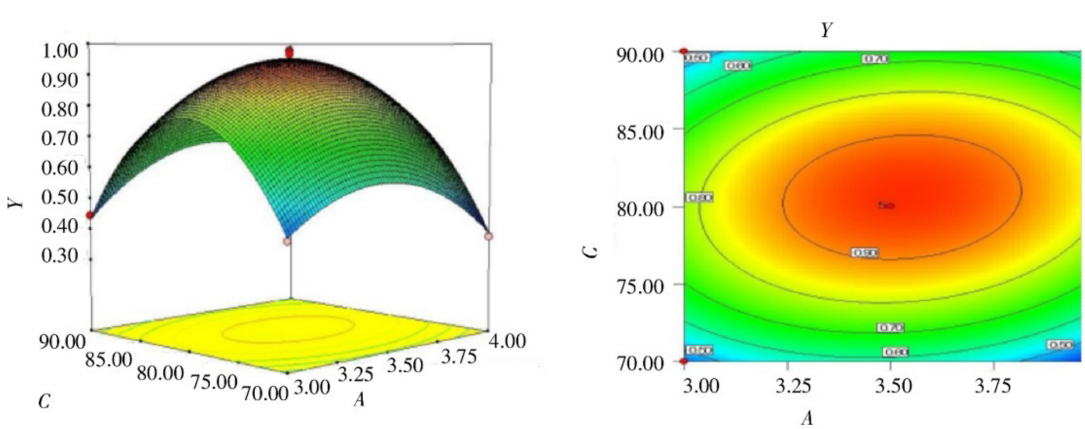

Figure 2. Response surface plots for the effect of modified starch addition and drying temperature on comprehensive index (Y).

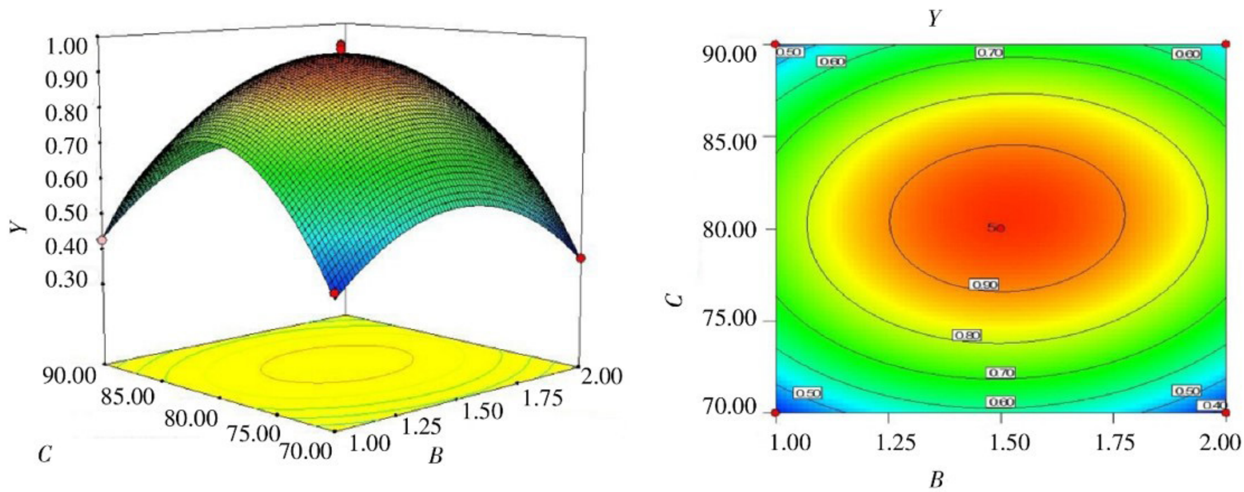

Figure 3. Response surface plots for the effect of glycero addition and drying temperature on comprehensive index (Y).

(3) Interaction of glycerol dosage and drying temperature. It can be seen from Figure 3 that the slope of the response surface is steeper, indicating that the response value is more sensitive to the change of glycerol dosage and drying temperature. When the amount of glycerin is fixed, the Y value of the composite index increases rapidly with the increase of drying temperature and then decreases rapidly, and when the drying temperature is fixed, the Y value also increases and then decreases with the increase of glycerin, and the drying temperature has a slightly more significant effect on the composite index than the amount of glycerin. The shape of the contour line is flat and circular, indicating that there is an interaction between the amount of modified starch and drying temperature, but it is not significant.

7 Conclusion

In recent years, the use of biodegradable starch materials to produce edible packaging films has received increasing attention and has become a hot topic of research in countries around the world. It was shown that the performance of edible starch films made from various types of corn starch, potato starch, and glutinous rice starch mixed with various types of ionic gels was improved. Currently, dry heat-modified starch is used extensively in the preparation of edible films. Research treated purple potato starch with xanthan gum to increase the crystallinity of starch, and the molecular arrangement and structure were tighter and stronger, indicating that the addition of water-soluble polysaccharides to starch or the modification of starch by physical and chemical means can improve the properties of the starch film (Zeng et al., 2015). Another research mixed rice starch and sodium carboxymethyl cellulose abstraction (CMC) to prepare edible starch film after dry heating (Mandal & Chakrabarty, 2019).

Research has studied the pasting characteristics of lotus starch blended with hydrophilic gum and showed that the addition of hydrophilic gum had a significant effect on the pasting characteristics of lotus starch (Liu et al., 2018). The gelatinization characteristics of the blend system of lotus seed starch and hydrophilic glue were studied. In this study, sodium alginate was added to lotus seed starch for dry thermal denaturation, and the effects of the addition of denaturing powder, glycerol, and drying temperature on the properties of the starch film were investigated to obtain the best parameters for the preparation of dry thermal denaturing lotus seed starch film. The mass fraction of modified lotus seed starch was 3.53%, the mass fraction of glycerol was 1.51%, and the drying temperature was 80.61 °C. The results of this study will provide theoretical guidance for the industrial production of edible films from dry heat-modified lotus seed starch.

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