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Optimisation of heat-moisture treatment conditions for producing high amounts of resistant starches from purple sweet potato and yam starches using response surface methodology

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Abstract:

Heat-moisture treatment, a physical modification method of starch, causes changes in the internal structure of starch and thereby produces resistant starch (RS). In this study, the heat-moisture treatment conditions (moisture content, heating temperature, and incubation time) was optimized to maximise the RS contents of the treated sweet potato and yam starches, using the Box-Behnken design and response surface analysis. The predicted maximised RS content of the treated sweet potato starch (43.9%) was obtained under optimal conditions of 34.76% moisture content, heating temperature of 100.11°C, and incubation time of 6.01h; the predicted maximised RS content of the treated yam starch (36.8%) was obtained under optimal conditions of 30.06% moisture content, heating temperature of 109.68°C, and incubation time of 6.59h using a quadratic model within the range of various process variables. The experimental RS contents of the treated sweet potato and yam starches obtained under optimal treatment conditions were 42.4% and 35.4%, respectively; this confirms that the models were valid and adequate because the predicted data and experiment data did not differ significantly. The results also indicate that the RS contents of the treated sweet potato starch were significantly higher than that of the treated yam starch. As a result, both starch structure and treatment conditions were determined to significantly affect the formation of RS in the heat-moisture treated starches.

Keywords: **heat-moisture treatment, resistant starch, response surface methodology, sweet potato starch, yam starch.**

Classification number: **2.2**

Introduction

In modern times, the number of patients who are overweight or suffer from diabetes is rapidly increasing both among children and adults because of their high intake of saturated fatty acids, high total fat intake, and inadequate consumption of dietary fibre [1-4]. Many studies have reported that diabetes-related diseases such as obesity, cardiovascular disease, and type 2 diabetes have been prevented or controlled by increasing amounts and varieties of fibre-containing foods [5]. However, the increase in dietary fibre content in food products significantly affects sensory and textural properties of these foods, such as negative effects on the final bread quality, which results in reduced volume and altered texture and consistency of the bakery product [6]. Recently, the RS from various starch sources has been widely used as a 'low-carbohydrate' ingredient in food formulations [7] because its health benefits resemble those of dietary fiber [8-9]. Englyst, et al. [10] used the term 'RS' to describe a small fraction of starch that resisted to hydrolysis by exhaustive α-amylase and pullulanase treatment *in vitro*. Currently, RS is defined as the fraction of dietary starch which escapes digestion in the small intestine and does not contribute to the blood glucose levels of healthy individuals [11]. Therefore, WHO recommends consuming 27-40 g of RS per day to prevent colon diseases [9].

Recently, physical, chemical, and enzymatic modifications have been developed to produce RS from various starch sources. Among these methods, the heat-moisture treatment (MHT) is a well-known hydrothermal method of increasing levels of SDS and RS in starches without destroying their granular structures [12]. The starch is treated at low moisture levels (<35% moisture, w/w) and at high temperatures (84-120°C) for 15 min to 16h [13]. Hung, et al. [14] reported that the RS contents

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of sweet potato and yam starches increased from 14.7% and 21.6% in native form to 27.2% and 31.0% in treated starches, respectively, after heat-moisture treatment at a moisture content of 30% and at a heating temperature of 110°C for 8h. Conversely, Huang, et al. [15] report that the RS content of the heat-moisture-treated sweet potato starch at a moisture content of 30% and at a heating temperature of 100°C for 2h (one cycle) decreased from 25.94% to 13.73% during the first cycle and then increased to the maximum amount of 20.99% after five treating cycles. The increase in RS content was also observed for corn, pea, and lentil starches after heat-moisture treatment under conditions of 30% moisture content, a heating temperature of 100°C, and treatment time of 2h [16]. In addition, Chung, et al. [16] also demonstrated that the amounts of RS of corn, pea, and lentil starches treated at 120°C were higher than those treated at 100°C. Therefore, the differing treatment conditions of the hydrothermal method resulted in different degrees of RS formation. Although many heat-moisture treatment conditions have been applied to investigate changes in physicochemical properties and digestibility of starches [17], the results of RS production after heat-moisture treatment of sweet potato starch remained variable because of the differing treatment conditions used in these studies [14, 15, 18]. In addition, the amylose content and starch characteristics, such as crystallinity and chain-length distribution, also affect the RS formation of starch after heat-moisture treatment [17]. Therefore, the objective of this study is to optimise heat-moisture treatment conditions (moisture content, heating temperature, and incubation time) to obtain the highest RS contents of sweet potato and yam starches using Box-Behnken designs and response surface analysis. Sweet potato and yam starches were selected in this study because these tuber starches possess different starch characteristics such as amylose content, chain-length distribution, and crystallinity [14]. In addition, limited information concerning RS formation of sweet potato and yam starches has been previously discovered.

Materials and methods

Materials

Starches used in this study were directly isolated from fresh purple sweet potatos (*Ipomoea batatas)* and yams (*Dioscoreaceae atatas*) in the laboratory, as previously reported by Hung, et al. [14]. The isolated sweet potato contained 1.1% protein, 0.9% lipid, 0.1% ash, and 97.9% total carbohydrate, while the yam starch contained 0.8% protein, 1.3% lipid, 0.1% ash, and 97.8% total carbohydrate [14]. Amylose contents of sweet potato and yam starches were 18.7 and 22.3%, respectively [14].

Alpha-amylase from *A. oryzae* (~30 U/mg, product # 10065) and amyloglucosidase from *A. niger* (≥300 U/ml, product # A7095), which were purchased from Sigma-Aldrich Co. (St. Louis, MO, US), were used in this study. Other chemicals were purchased from Merck Co. (Darmstadt, Germany).

Starch characteristics

Thermal characteristics of starches were determined using a differential scanning calorimeter (DSC-60, Shimadzu Co., Kyoto, Japan) [19]. An aluminum vessel which contained 3.0±0.1 mg of starch and 10 µl of distilled water was sealed and remained at room temperature for over 30 min for equilibration. The vessel was then heated from 30 to 120°C at a rate of 10°C/min by a DSC-60 heater. An empty vessel was used as a reference. The initial, peak, and recovery temperatures and transition enthalpy were automatically calculated using a TA-60WS program (Shimadzu Co.).

Crystalline characteristics of starches were determined using an X-ray diffractometer (Rigaku Co., Ltd, Rint-2000 type, Tokyo, Japan). The XRD system was operated at 40 kV and 80 mA, and diffractograms of the starches were recorded from 2° 2θ to 35° 2θ, with a scanning speed of 8°/min and a scanning step of 0.02° [19].

Box-Behnken designs for heat-moisture treatments of starches

The heat-moisture treatment of starches was performed based on the method of Hung, et al. [14]. The starches (100 g) were directly weighed and mixed with water at a desired moisture content level. The sample was well-dispersed and equilibrated at room temperature for 24h before being heated in a forced air oven at a specific temperature for a controlled duration. After heat-moisture treatment, the starch samples were cooled and then dried at 45ºC for 24h to a moisture content of approximately 10%.

Table 1. Coded levels of variables selected for the experiments.

Variable	Coded	Range and level		
		-1	*0*	*+1*
Moisture content (%)	X_1	25	30	35
Heating temperature (°C)	X_2	100	110	120
Incubation time (h)	X_3	6	7	8

A three-factor Box–Behnken design and optimisation [20] was used to optimise the heat-moisture treatment conditions for all variable factors to obtain the highest RS content. Three important factors, including moisture content (X_1), heating temperature (X_2), and incubation time (X_3)

were selected as the independent variables, and RS content (Y) was selected as the dependent response variable. Three different levels of each independent variable, including moisture contents (25, 30, and 35%), heating temperatures (100, 110 and 120°C), and incubation times (6, 7 and 8h) were used and coded as -1, 0, and +1 for low, middle, and high levels, respectively, as presented in Table 1. A total of 15 experiments were conducted for three independent variables based on the Box-Behnken design.

The mathematical relationship between response (Y) and independent variables (X) was demonstrated by the following regression equation.

Where Y is the quadratic response, β_0, β_i, β_j and β_{ij} are the regression coefficients for intercept, linear, quadratic, and interaction terms, respectively. X_i and X_j are the coded values of the i[th] and j[th] independent variables. The variables X_iX_j represent the first order interaction between X_i and X_j for (i<j).

The optimal values of the selected variables were calculated by solving the regression equation and also by analysng the response surface contour plots using a design expert software (version 7.0.0, STAT-EASE Inc., Minneapolis, MN, USA). The validity and adequacy of the predictive models were determined through experimental analysis at optimal conditions suggested by the design expert [20].

Determination of starch fractions (RDS, SDS, RS)

After heat-moisture treatment, the native and treated starches were then measured for rapid digestible starch (%RDS), slowly digestible starch (%SDS), and RS (%RS) based on the methods of Englyst, et al. [21], as previously described by Hung, et al. [14]. Starch (0.3 g, db) was mixed with 20 ml of sodium acetate buffer (pH 6.0) and boiled for 30 min in a water bath. The sample was then equilibrated at 37°C for 15 min prior to adding an enzyme solution (5 ml) of α-amylase (1,400 U/ml) and amyloglucosidase (13 AGU/ml). The starch solution was incubated with shaking at 37°C for 120 min, and the total glucose concentrations of the 20 min-digested and 120 min-digested hydrolysates (G_{20} and G_{120}, respectively) were determined using the phenol-sulfuric acid method. The remaining residue was intensively hydrolysed with amyloglucosidase (50 AGU/ml) after hydrolysing by 7M KOH. The final hydrolysate was then determined for total glucose concentration (TG). The total glucose levels at different digestive times (G_{20}, G_{120} and TG) were used to calculate for RDS, SDS, and RS as follows [21]:

$$RDS = G_{20} \times 0.9$$

$$SDS = (G_{120} - G_{20}) \times 0.9$$

$$RS = (TG - G_{120}) \times 0.9$$

Statistical analysis

The statistical analysis was performed through an analysis of variance (one-way ANOVA) with Duncan's multiple-range test to compare treatment means at $p<0.05$, using SPSS software version 16 (SPSS Inc., USA).

The regression and graphical analysis of the data was conducted using a design Expert software (version 7.0.0, STAT-EASE Inc., Minneapolis, MN, USA).

Results and discussion

Starch characteristics

Table 2. Amylose content and thermal characteristics of sweet potato and yam starches*.

Starch	Amylose content (%)	Thermal characteristic			
		Onset (°C)	*Peak (°C)*	*Completion (°C)*	*Enthalpy (J/g)*
Sweet potato	18.7[a]	71.2[a]	75.3[a]	80.2[a]	17.8[a]
Yam	22.3[b]	75.5[b]	79.3[b]	86.0[b]	35.8[b]

*Data followed by the same letter in the same column are not significantly different (p≤0.05).

Thermal characteristics of starches isolated from sweet potato and yam tubers are indicated in Table 2. The gelatinisation temperature of the sweet potato starch was between 71.2 and 80.2°C, which was significantly lower than that of the yam starch (75.5 to 86.0°C). Transition enthalpy of the sweet potato starch was also significantly low relative to the yam starch. The higher transition enthalpy of yam starch relative to that of the sweet potato starch was due to the higher amylose content of yam starch relative to sweet potato starch (Table 2). This result aligns with the previous reports which stated that the root starches containing higher amylose contents had a larger transition enthalpy relative to those containing lower amylose contents [19, 22, 23].

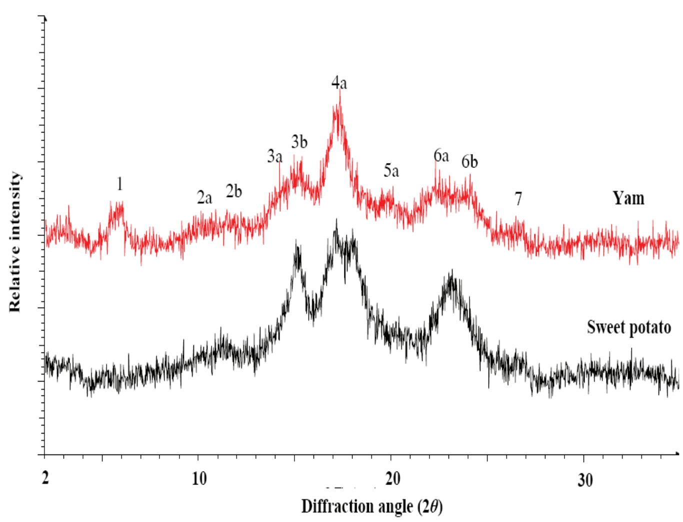

Fig. 1. X-ray diffraction patterns of sweet potato and yam starches.

The X-ray diffraction patterns demonstrated that native sweet potato and yam starches had different crystalline structures (Fig. 1). The native sweet potato starch had major peaks around d-spacings 5.8 Å (line 3b), 5.2, and 4.8 Å (line 4a, 4b) and 3.8 Å (line 6a), while yam starch peaks at 15.8 Å (line 1), about 5.9 Å (line 3a, 3b), 5.2 Å (line 4a), and 4.0 and 3.7 Å (lines 6a, 6b). As a result, the native sweet potato starch exhibited the A-type crystal, whereas the native yam starch displayed the B-type crystal, as classified by Zobel [24]. These results are consistent with those previously reported by Hoover [25].

Therefore, the differences in amylose contents, thermal characteristics, and crystalline structures of the native sweet potato and yam starch may affect RS formation under heat-moisture treatment.

Optimisation of RS content through response surface methodology

Significant factors used in heat-moisture treatment, including moisture content, heating temperature, and incubation time, were optimised using Box-Behnken design to maximise RS contents of sweet potato and yam starches. Table 3 illustrates the design matrix with three independent variables (coded values) and experimental responses (RS contents (% w/w, db) of treated sweet potato and yam starches). Based on the treatment conditions formulated by the Box-Behnken design, the highest RS content of the treated sweet potato starch was 41.64% after treating the native starch under the conditions of 30% moisture content at a heating temperature of 100°C for 6h, while the lowest RS content of this type of starch was 24.96%; this was obtained under the conditions of 25% moisture content and a heating temperature of 120°C for 7h. The highest RS content of the treated yam starch was 34.57%, which was obtained under treatment conditions of 35% moisture content and a heating temperature of 120°C for 7h, while the lowest RS content of this type of starch was 15.57% under the conditions of 35% moisture content and a heating temperature of 110°C for 8h. The data were then analysed through multiple regression analysis, and the regression coefficients for the equation concerning the relationship between three variables and a response were determined and presented in Table 4. Moreover, the results of the analysis of variance (ANOVA) with the Fisher's statistical test using Design Expert software (Design Expert 7.0.0) are indicated in Table 5. The coefficient of determination (R^2), which can be defined as the ratio of the explained variation to the total variation, was used to evaluate the fitness and adequacy of the model. The empirical model fits the actual values if the R^2 value is near unity. The R^2 values of the regressed models which predicted the RS contents of the treated sweet potato and yam starches were 0.9763 and 0.9565, respectively; this suggests a high dependence and correlation between the measured and predicted values of the responses.

Table 3. Full factorial Box-Behnken design matrix with three independent variables in coded units and experimental responses.

Trial run	Coded variable			Experimental response (RS content, %)	
	Moisture content (%)	*Heating temperature (°C)*	*Incubation time (h)*	*Sweet potato*	*Yam*
	X_1	X_2	X_3	Y_1	Y_2
1	+1	0	+1	31.96	15.57
2	0	-1	+1	37.65	19.64
3	+1	-1	0	35.15	20.08
4	0	-1	-1	41.64	32.40
5	-1	0	-1	34.08	32.28
6	0	+1	-1	32.44	34.12
7	+1	0	-1	38.34	31.45
8	-1	-1	0	32.93	32.20
9	0	+1	+1	27.48	25.19
10	0	0	0	26.90	30.67
11	0	0	0	27.24	31.35
12	-1	+1	0	24.96	34.46
13	+1	+1	0	29.66	34.57
14	0	0	0	27.10	32.79
15	0	-1	+1	33.95	19.62

Table 4. Coefficients of the response function to predict resistant starch content of sweet potato and yam starches through regression analysis.

Factor	Coefficient estimate	
	Sweet potato	*Yam*
Intercept	27.08	31.63
X_1	1.15	- 2.11
X_2	- 4.10	3.00
X_3	- 1.93	- 6.28
X_1X_2	0.62	3.06
X_1X_3	- 1.56	-0.80
X_2X_3	- 0.24	0.96
X_1^2	1.69	- 2.19
X_2^2	1.91	0.92
X_3^2	5.81	- 4.68

X_1: moisture content (%); X_2: heating temperature (°C); X_3: incubation time.

Table 5. Analysis of variance for the response surface quadratic model.

Source	df	Sweet potato		Yam	
		F-value	*p-value*	*F-value*	*p-value*
Model	9	22.89	0.0015*	12.22	0.0066*
X_1	1	6.71	0.0489*	6.91	0.0466*
X_2	1	85.58	0.0002*	13.98	0.0134*
X_3	1	18.98	0.0073*	61.13	0.0005*
X_1X_2	1	0.98	0.3684	7.25	0.0432*
X_1X_3	1	6.20	0.0551	0.50	0.5101
X_2X_3	1	0.15	0.7150	0.71	0.4376
X_1^2	1	6.68	0.0492*	3.44	0.1229
X_2^2	1	8.53	0.0330*	0.60	0.4735
X_3^2	1	79.31	0.0003*	15.69	0.0107*
Residual	5				
Lack of fit	3	89.19	0.0111	6.67	0.1331
R^2		0.9763		0.9565	

*Significant (p-value <0.05); X_1: moisture content (%); X_2: heating temperature (°C); X_3: incubation time.

The F-test and *p*-value obtained through the analysis of variance (ANOVA) were used to determine the significance of each coefficient. The *p*-value denotes the probability value. The *p*-values of the adjusted models were 0.0015 and 0.0066 for both sweet potato and yam starches, respectively; these were lower than 0.05, which indicates that both models were significant. For the model of sweet potato starch, the X_1, X_2, X_3, X_1^2, X_2^2, and X_3^2 factors were the most significant among the factors which influenced the response because the *p*-values of these variables were all lower than 0.05. For the model of yam starch, X_1, X_2, X_3, and X_3^2 were also the significant factors which influenced the response, while the *p*-value of X_1X_2 was 0.0432 (lower than 0.05) which indicates that the interaction between moisture content and heating temperature affected the response. As a result, the equations which illustrated the relationship between three variables including moisture content, heating temperature, and incubation time, and the responses are formed in a reduction form as follows:

$$Y_1 = 27.08 + 1.15X_1 - 4.10X_2 - 1.93X_3 + 1.69X_1^2 + 1.91X_2^2 + 5.81X_3^2$$

$$Y_2 = 31.63 - 2.11X_1 + 3.00X_2 - 6.28X_3 + 3.06X_1X_2 - 4.68X_3^2$$

where: Y_1 and Y_2 are the predicted responses of the RS contents of the sweet potato and yam starches, respectively, X_1, X_2, and X_3 are coded variables for moisture content, heating temperature, and incubation time, respectively.

Table 6. Optimal conditions for producing high amounts of resistant starches.

Variable	Sweet potato		Yam	
	Calculation	*Confirmation*	*Calculation*	*Confirmation*
Moisture content (%)	34.76	35	30.06	30
Heating temperature (°C)	100.11	100	119.68	120
Incubation time (h)	6.01	6	6.59	6.5
RS (%)	43.9	42.4	36.8	35.4

The optimisation of the process variables to maximise RS contents of the heat-moisture treated sweet potato and yam starches was performed by solving the quadratic models using the studied experimental range of various variables. Table 6 presents the predicted values of the responses under optimal conditions (in the range constraint) for the models. For the sweet potato starch, the optimal conditions included a moisture content of 34.76%, a heating temperature of 100.11°C, and an incubation time of 6.01h to achieve the highest RS content of 43.9%. For the yam starch, the highest RS content was 36.8%, which was achieved under the optimised conditions of a moisture content of 30.06%, a heating temperature of 119.68°C, and an incubation time of 6.59h. These models were experimentally assessed to confirm the RS contents of the treated sweet potato and yam starches. However, it is difficult to maintain the recommended conditions during processing. Therefore, optimal conditions were targeted using the rounded numbers of all factors, as displayed in Table 6. As a result, the experimental RS content of the treated sweet potato starch under the experimental conditions of heat-moisture treatment, including moisture content of 35%, heating temperature of 100°C, and incubation time of 6h, was 42.4%; this did not significantly differ from the calculated data (43.9%). Likewise, the experimental RS content of the treated yam starch (35.4%), which was obtained under the experimental conditions of heat-moisture treatment, including a moisture content of 30%, a heating temperature of 120°, and an incubation time of 6.5h, did not significantly differ from the calculated data (36.8%). Therefore, the model conditions were targeted to be optimal for the development of RS contents of the heat-moisture-treated sweet potato and yam starches, and the data obtained confirmed the validity and adequacy of the models.

The formation of RS during heat-moisture treatment was caused by the formation of some interactions during heat-moisture treatment that have survived after gelatinisation and partly resisted the accessibility of starch chains through the hydrolysing enzymes [16]. Therefore, both the treatment conditions, including moisture content, heating temperature, and time, and starch characteristics, such as amylose-lipid interactions and amylose-amylose or amylose-amylopectin interactions, exerted a significant influence on the digestibility of starches [26]. The results of this study indicate that the heat-moisture treatment exerted a greater impact on the sweet potato starch than the yam starch under optimal treatment conditions. As a result, the higher RS content of the treated sweet potato at optimal treatment conditions was obtained relative to that of the treated yam starch. These results were caused by the fact that the formation and lateral association of double helices involving amylopectin chains in the heat-moisture treated B-type starches would be significantly slower, more difficult, and less strong relative to the heat-moisture-treated A-type starch [22]. In addition, the sweet potato starch required a low temperature (100°C) but a high moisture content (35%) to form the highest RS content, while the yam starch required a high temperature (120°C) but a low moisture content (30%) to maximise RS content. Therefore, the optimal condition to maximise the RS content of starch differed based on the nature of the starch.

RS contents of native and treated starches

Amounts of rapid digestible starch (RDS), slowly digestible starch (SDS), and the RS of native and treated sweet potato and yam starches are indicated in Table 7. Amounts of RDS and SDS in native sweet potato starch were higher than those in native yam starch. However, the RS content of the native sweet potato starch was lower relative to the native yam potato. Under optimal heat-moisture treatment conditions, the amounts of SDS and RS of the treated sweet potato and yam starches significantly increased relative to those of the native starches. The SDS and RS contents of the treated sweet potato under optimal treatment conditions were significantly higher than those of the treated yam starch, although the amount of RS of the treated yam starch was higher relative to the treated sweet potato starch when these starches were heat-moisture-treated under the same conditions of 30% moisture content and a heating temperature of 110°C for 8h, as reported by Hung, et al. [14]. In addition, the RS contents of the treated sweet potato and yam starch obtained in this study under optimal treatment conditions were significantly higher than those obtained by Hung, et al. [14]. Therefore, the formation of RS in the starch through heat-moisture treatment was not only affected by the internal structures and amylose contents of the starches but was also affected by the heat-moisture treatment conditions. The highest amount of RS is obtained if the starch is treated under optimal conditions specific to each starch based on the type and structure of the starch.

Table 7. RDS, SDS, and RS of native and heat-moisture-treated sweet potato and yam starches*.

Sample	RDS (%)	SDS (%)	RS (%)
Sweet potato starch			
Native	78.7±2.0^{d}	6.6±0.5^{a}	14.7±1.5^{a}
Heat-moisture	43.2±2.3^{a}	14.4±2.9^{c}	42.4±0.6^{d}
Yam starch			
Native	73.8±2.2^{c}	4.7±1.0^{a}	21.6±1.8^{b}
Heat-moisture	55.7±0.3^{b}	8.9±1.8^{b}	35.4±1.5^{c}

*Data followed by the same letter in the same column are not significantly different (p≤0.05).

Conclusions

The RS contents of the heat-moisture-treated sweet potato and yam starches were maximised using the Box-Behnken design and the response surface analysis. The results indicate that moisture content, heating temperature, and incubation time were the most pivotal factors which affected the RS formation of the heat-moisture-treated starch. The quadratic models within the studied experimental range of various process variables were used to maximise the RS contents of the treated starches. As a result, the experimental RS contents of the treated starches obtained using the optimal conditions of heat-moisture treatment did not significantly differ from the data calculated using the quadratic models, meaning that the models were valid and adequate. Under optimal treatment conditions, the RS content of the treated sweet potato starch was higher relative to the treated yam starch because of the differences in the internal structures and amylose contents of these starches. Therefore, the heat-moisture treatment condition must be optimised for each starch to obtain the highest RS content of starch.

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