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Influence of extrusion-saccharification parameters to filtration of syrup made of enzymatic extruded degermed corn

Yuyan Fan[1], Guihai Shi[3], Shuhua Wu[1], Zhehao Zhang[1],
Hongjun Li[1,2] ,Chengye Ma[1,2*]

School of Agricultural and Food Engineering, Shandong University of Technology, Zibo, China
1. School ofAgricultural Engineering and Food science, Shandong University of Technology, Zibo, 255000, China; 2. Key Laboratory of Shandong Provincial Universities for Technologies in FunctionalAgricultural Products, Zibo,25500, China; 3. Xiwang Meat Food co. LTD, Yantai, 264001,China
Corresponding author: Chengye Ma

Abstract: *The paper studied the influence of the extrusion saccharification parameters on filtration speed of hydrolyzed extrudates of degermed corn with amylase. Experimental data was analyzed using SAS 9.1 and obtained the optimized parameters. The parameters were shown below: the additive amount of thermostable α-amylase during extrusion was 0.80L/t, the screw rotation speed was 140r/min, the additive amount of thermostable α-amylase during liquefying was 0.60L/t, liquefaction time was 20.0min, additive amount of glucoamylase during saccharifying was1.50L/t. The process of extruded degermed corn added thermostable α-amylase to make glucose syrup was feasible which provide theoretical basis for industrialization in glucose syrup production.*

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I. Introduction

Starch is the major component of degermed corn which has less content of lipids, protein and ash[1]. The lipids can reduce the flavor of food, and could complex with amylose which reduce the expansion degree and solubility[2]. The starch forms unsoluble particles with lipids during hydrolyzation[3]. The protein can make the starch change color, and produce foam. All these impurities interfere with the filtration of syrup during the starch was hydrolyzing by enzymatic extrusion.

The filtration speed of high DE value(>95%) glucose syrup made of extruded degermed corn added thermostable α-amylase was fast because of the high hydrolyzation degree and low viscosity.But the filtration was influenced by the extrusion saccharification parameters[4]. The objective of the study was to determine the effect of extrusion saccharification parameters on filtration of glucose syrup made of enzymatic extruded degegmed corn.

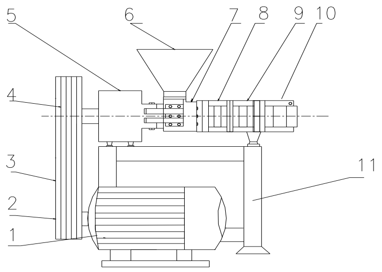
II. Materials And Methods

2.1 Equipment

The equipment was the home-made single-screw extruder. The structure diagram was shown in Figure 1. Extruder consisted of modular barrel (three pieces) and screw (four pieces), with the productivity of 100kg/h. The screw rotation speed was 0~1200r/min, stepless adjustable. The barrel was at temperature of 0~300℃, continuously adjustable and was equipped with an automatically controlled closed-loop digital instrumentation system. The clearance between the templates and screw top was adjustable. Die diameter of the extruder was adjustable.

2.2 Materials

The degermed corn was purchased from Baodi Corn Processing Factory. According to our measurement, the moisture content was 12.77% (w/w), starch content was 75.36%, protein content was 8.25%, crude fat was 1.04%. The thermostable α-amylase was purchased from Shandong Longda Bio-technology Co.. Ltd (the bioactivity was 20000u/ml). The glucoamylase was purchased from Yangshao Biochemical Engineering Co.. Ltd (the bioactivity was 100000u/ml).



1. Motor 2. Small pulleys 3. Belt 4. Large pulleys 5. bearing block 6. feed hopper 7. Barrel piece 1st 8. Barrel piece 2nd 9. Barrel piece 3rd 10. Barrel piece 4th 11. Rack

Figure 1. Single screw cooking extruder

2.3 Determination of filtration speed of syrup

The filtration speed [L/(m^2·h)] was representation by volume of glucose syrup per unit area(m2) and unit time(h). The Buchner funnel (the diameter was 9 cm) was used to filtrate the extrudate slurry to collect 100ml syrup. The filtration speed (for short 'FS') was calculated with the following equation:

$$FS=\frac{v}{\frac{1}{4}\pi d^2\times t}=\frac{100\times10^{-3}}{\frac{1}{4}\pi(9\times10^{-2})^2\times t}=15.7190t^{-1}\,[L/(m^2\cdot h)] \quad (1)$$

The solid of syrup was measured by the method of refractive index. The procedure has been described in article of National Standard GB/T 20885-2007.

2.4 Process flow syrup made of extrudates

Enzymatic extruded degermed corn → ground(20 mesh fineness) → weighging (50g) → mixing (the ratio of material to water was 1:2, pH 6.0~6.2, ambient temperature) → liquefying (adding thermostable α-amylase, incubation at 90℃) → cooling to 60℃ → adjusting acidity to pH4.3~4.4 used 10%H2SO4 → saccharifying (adding glucoamylase, incubation at 90℃ for 12h) →inactivation of enzyme (95℃ 10min) → vacuum filtration → glucose syrup → determination.

2.5 Experiment arrangement and results

In order to assess the effect of operating parameters on filtration speed of starch slurry of saccharified extrudates, a central composite rotatable response surface experimental design based on a five-variable five-levelsystem was carried out (Table 1). The five factors were chosen as parameters of extrusion process: additive amount of thermostable α-amylase during extrusion (x1), screw rotation speed (x2), additive amount of thermostable α-amylase during liquefying (x3), liquefaction time (x4), additive amount of glucoamylase during saccharifying (x5). The index was filtration speed of slurry (y). Meanwhile, the method of the quadratic orthogonal rotating combination design of five factors and five levels was applied[5]. Factor's level coding is shown in Table 1. The experiments arrangement and results are shown in Table 2.

Experimental data was analyzed using Statistical Analysis System 9.1 to fit second order polynomial equations to response variables[6]. Surface plots were drawn using SAS 9.1 computer software to show the effect of two independent variables while the other were held constant [7].

Table 1 Levels of independent variables

Levles	Factors				
	x1	x2	x3	x4	x5
-2	0.2	80	0.1	9	0.5
-1	0.5	110	0.3	17	1.0
0	0.8	140	0.5	25	1.5
1	1.1	170	0.7	33	2.0
2	1.4	200	0.9	41	2.5

Table 2 Experimental arrangements and results

No.	Factors Arrangement					Filtration speed
	x_1	x_2	x_3	x_4	x_5	/L/($m^2 \cdot h$)
1	1	1	1	1	1	388.22
2	1	1	1	-1	-1	694.62
3	1	1	-1	1	-1	192.45
4	1	1	-1	-1	1	440.74
5	1	-1	1	1	-1	381.97
6	1	-1	1	-1	1	572.49
7	1	-1	-1	1	1	301.98
8	1	-1	-1	-1	-1	532.56
9	-1	1	1	1	-1	322.79
10	-1	1	1	-1	1	572.71
11	-1	1	-1	1	1	222.51
12	-1	1	-1	-1	-1	487.59
13	-1	-1	1	1	1	254.96
14	-1	-1	1	-1	-1	327.40
15	-1	-1	-1	1	-1	199.29
16	-1	-1	-1	-1	1	257.65
17	2	0	0	0	0	375.96
18	-2	0	0	0	0	301.56
19	0	2	0	0	0	290.10
20	0	-2	0	0	0	332.15
21	0	0	2	0	0	416.70
22	0	0	-2	0	0	286.25
23	0	0	0	2	0	358.37
24	0	0	0	-2	0	387.51
25	0	0	0	0	2	381.97
26	0	0	0	0	-2	242.06
27	0	0	0	0	0	282.94
28	0	0	0	0	0	347.48
29	0	0	0	0	0	324.69
30	0	0	0	0	0	254.65
31	0	0	0	0	0	347.25
32	0	0	0	0	0	305.58
33	0	0	0	0	0	349.11
34	0	0	0	0	0	276.12
35	0	0	0	0	0	509.29
36	0	0	0	0	0	379.49

III. Results And Discussion

3.1 Establishment and inspection of regression model of filtration speed

The regression models fitted to experiment results shown good correlation coefficients(R^2=0.7609). The equation (2) was significant (p=0.0453) and the lackfit of the regression model was insignificant (p=0.0685>0.05) which shown that the second order polynomial equations could fit the experimental results (Table 4).

$$\begin{aligned} y_1 &= 331.5815 + 42.0387x_1 + 17.0513x_2 + 47.55537x_3 \\ &- 69.9946x_4 - 11.9829x_5 + 4.3928x_1^2 - 39.9500x_2x_1 \\ &- 2.5159x_2^2 + 16.1719x_3x_1 + 24.3569x_3x_2 + 7.5716x_3^2 \\ &- 20.6244x_4x_1 - 32.3619x_4x_2 - 1.0606x_4x_3 + 12.9378x_4^2 \\ &- 4.0381x_5x_1 - 1.1956x_5x_2 + 15.6631x_5x_3 + 16.8594x_5x_4 \\ &+ 25.2066x_5^2 \end{aligned} \tag{2}$$

Table 3 Analysis of variance of the regression model for filtration speed

Source	Degree of Freedom	Sums of Squares	Mean Square	F Value	Pr > F
linear	5	224693	0.5267	6.61	0.0019
Quadratic	5	28343	0.0664	0.83	0.5459
Crossproduct	10	71592	0.1678	1.05	0.4498
Total Model	20	324627	0.7609	2.39	0.0453

Table 4 Analysis of lackfit of the regression model for filtration speed

Source	Degree of Freedom	Sums of Squares	Mean Square	F Value	Pr > F
Lack of Fit	6	67909	11318	2.99	0.0685
Pure Error	9	34105	3789.46		
Total Error	15	102015	6800.97		

3.2 Analysis of the effect of two independent variables on filtration speed

Figure 2 shown that the optimizing value of screw speed was 80r/minand the enzyme added during extrusion was 1.4L/t. The degradation degree was larger when the screw speed was slow. Because the resident time was longer, so the time of reaction of enzyme with starch was longer. The visicoty of saccharified degermed corn slurry was low, thus the filtration speed was quick.

The filtration speed was the fastest when the additive amount of thermostable α-amylase during extrusion and screw rotation speed were at level of '+2'. Although extrusion could lead to enzyme inactivation, the resident enzyme could degraded the starch which made the viscosity decrease. And the resident enzyme could hydrolyze the starch during the liquefaction and made the filter quick(Figure 3).

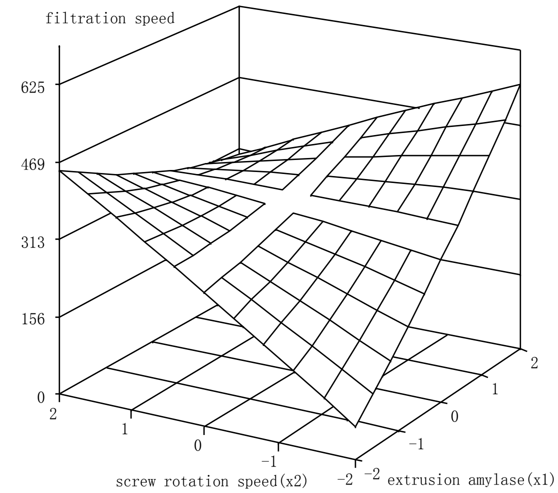


Figure 2 Response surface plots of filtration speed as a function of screw speed and amylase amount added in extrusion

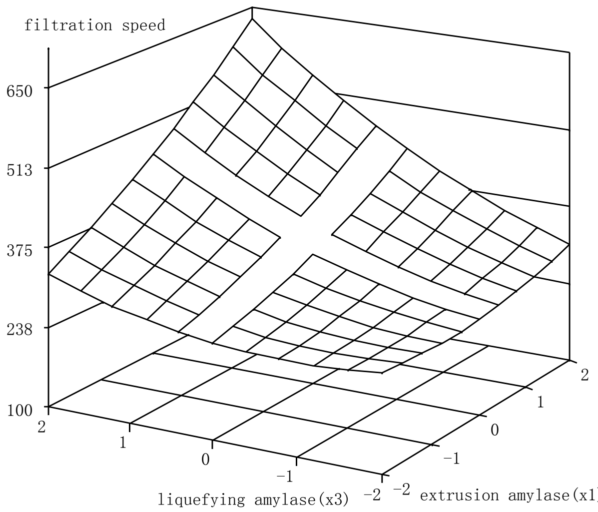


Figure 3 Response surface plots of filtration speed as a function of amylase amount added in liquefaction and amylase amount added in extrusion

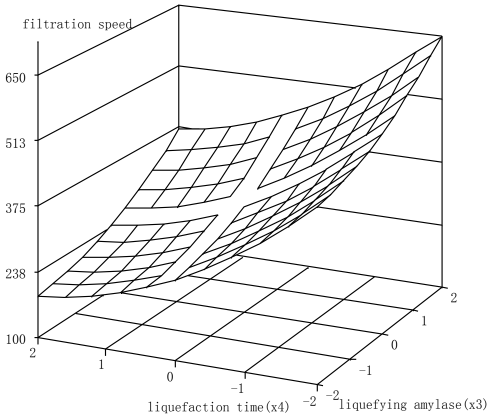


Figure 4 Response surface plots of filtration speed as a function of liquefaction time and amylase amount added in liquefaction

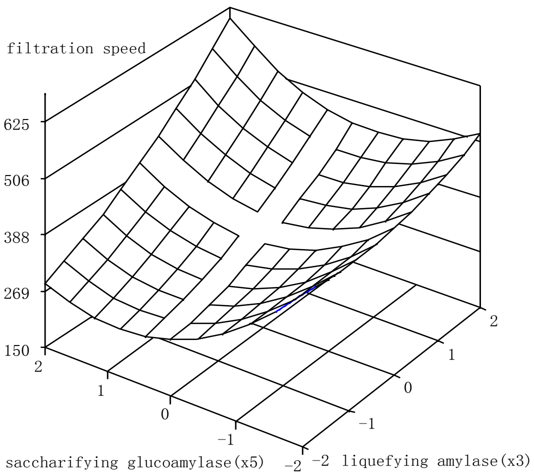


Figure 5 Response surface of filtration speed as a function of glucoamylase amount added in saccharifaction and amylase amount added in liquefaction

The additive amount of thermostable α-amylase during liquefying and liquefaction time were saccharifaction paremeters which greatly influence the filtration speed of slurry. The filtration speed was the fastest when the additive amount of thermostable α-amylase during liquefying and liquefaction time were at level of '+2'(Figure 4). The starch slurry viscosity was decreased great during liquefaction, and the flowability was increased. The aim of liquefaction set the stage for glucoamylase[8]. The starch was degraded with the amylase, thermo energy and shear .The reducing sugar was increased. The molecular weight was decreased, the viscosity was decreased, the flowability was increased when the enzymatic extruded corn was liquefied which offer the conditions for glucoamylase. If the liquefying was continued the hydrolyzed end product was glucose and maltose, the dextrose equivalent was low. The liquefaction was processed at the suitable temperature, when the liquefaction time was extended partly hydrolyzed starch was reintegrated which made the glucoamylase difficult to hydrolyzed the starch, and the yield was effected. So the degree of liquefaction must be controlled[9].

The filtration speed was increased as the additive amount of thermostable α-amylase during liquefying was increased. The reducing sugar was increased during saccharifaction with the additive amount of thermostable α-amylase during liquefying, and the viscosity was decreased which made the filtration speed quicker.

3.3 Optimization of the regression model

Experimental data was analyzed using SAS 9.1 to fit second order polynomial equations to filtration speed of slurry. The optimized extrusion-saccharification parameters was obtained using SAS 9.1 by frequency optimization. The parameters were shown below: the additive amount of thermostable α-amylase during extrusion was 0.73~1.00 L/t, the screw rotation speed was 130.00~154.10 r/min, the additive amount of thermostable α-amylase during liquefying was 0.48~0.66 L/t liquefaction time was 15.24~23.10 min, additive amount of glucoamylase during saccharifying was 1.39~1.62L/t.

3.4 Calidated and compared experiments

Three calidated tests were performed among the above optimized parameters. The controlled tests were performed also. The results were shown in Table 5. The indexs of extruded degermed corn added the thermostable α-amylase were superior to those of extruded degermed corn or native corn.

Table 5 Calidated and compared experiment arrangements

No.	Extrusion parameters			Liquefaction parameters		Saccharifaction parameters		Results		
	Moisture content (%)	Screw speed (r/min)	Amylase amount(L/t)	Amylase amount(L/t)	Time(min)	Glucomylase amount (L/t)	Time(h)	Filtration speed	Solid content	DE value(%)
1	30	130.0	0.70	0.50	15	1.40	12	582.38	32.57	95.79
2	30	140.0	0.80	0.60	20	1.50	12	726.67	33.12	98.71
3	30	150.0	1.00	0.70	25	1.60	12	686.23	32.88	96.66
c1	20	140	/	1.00	25	1.50	12	346.72	29.47	80.58
c2	/	/	/	1.00	20	1.50	12	227.26	/	75.44

Note: No.1 to 3 were calidated test; C1 was the test of extruded native degermed corn; C2 was the test of native degermed corn[10]. The extrusion conditions were shown below: the barrel temperature was60℃, the die diameter was12mm, the distance between screw top and template was 12mm.

IV. Conclusion

The paper studied the influence of the extrusion saccharification parameters on filtration speed of hydrolyzed extrudates of degermed corn with thermostable α-amylase. Three calidated tests were performed among the above optimized parameters. The controlled tests were performed also. Experimental data was analyzed using SAS 9.1 and obtained the optimized parameters. The parameters were shown below: the additive amount of thermostable α-amylase during extrusion was 0.80L/t, the screw rotation speed was 140r/min, the additive amount of thermostable α-amylase during liquefying was 0.60L/t, liquefaction time was 20.0min, additive amount of glucoamylase during saccharifying was 1.50L/t. The indexs of extruded degermed corn added the thermostable α-amylase were superior to those of extruded degermed corn or native corn. The process of extruded degermed corn added thermostable α-amylase to make glucose syrup was feasible which provide theoretical basis for industrialization in glucose syrup production.

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