

Study on the preparation process of cross-linked porous cassava starch

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Abstract. Using cassava starch as raw material, preparation process of porous cross-linked cassava starch was studied. Using TSTP as cross-linking agents, Orthogonal design was applied for the optimization of cross-linked porous starch preparation process. The results showed that the optimal conditions of cross-linked porous cassava starch were as follows: reaction temperature 45°C, reaction time 20 h, 1% of the amount of the enzyme, the enzyme ratio of 1:5, pH5.50, substrate concentration of 40%.

1. Experimental

1.1. Materials and chemicals

Cassava starch, Glucoamylase and Soybean oil were food grades. Sodium trimetaphosphate, Sodium hydroxide, α -amylase and other reagents were all analytical degree.

1.2. Preparation of Cross-linked starch

1.2.1. Determination of cross-linking degree

It is difficult to directly determine the cross-linking degree of the cross-linked starch for the cross-linking degree is very low[1-8]. In the research, the sedimentation method was used to measure the cross-linking degree.

0.5g cross-linked starch with 25mL distilled water was put into a 100mL beaker. The beaker was put into a water bath of 85°C and was stirred slightly for 2 min. Then the beaker was taken out of the water bath and was cooled to room temperature. The reaction products were put into some centrifuge tubes and were centrifuged at a speed of 4000r/min for 10min. The volume of sedimentation was calculated through the following formula:

$$S=10-V \quad (1)$$

S-sedimentation, mL;

V-supernatant volume, mL.

1.3. Preparation of porous starch

1.3.1. Optimization of preparation process

Using reaction time (A), starch concentration (B), pH (C), enzyme dosage (D) and reaction temperature (E) as five factors, L16 (4⁵) orthogonal test design was conducted (Tab.1). The optimum technological parameters for preparation of cassava porous starch by enzymatic hydrolysis were



determined by the index of oil absorption. 2g cross-linked starch was put into the reaction flask with a certain concentration, α -amylase and glucoamylase were dissolved in buffer to certain concentration, then transferred the enzyme liquid to the starch suspension, stirring for a certain time. After the reaction, adding 0.2mL 4%NaOH to stop the reaction, filtrating. The samples are placed in a drying oven at 45°C to a constant weight[9-10].

Tab.1 Factors and levels of orthogonal experiment design

levles	factors				
	A	B	C	D	E
	Time/h	Starch breast concentration /%	pH	Enzyme dosage /%	Temperature /°C
1	20	25	4.00	1.0	40
2	24	30	4.50	1.5	45
3	28	35	5.00	2.0	50
4	32	40	5.50	2.5	55

The oil absorption rate was calculated according the following equation:

$$\text{Oil absorption rate } S = \frac{M_2 - M_1}{M_1} * 100\%$$

(2)

Where M1 was the dried weight of the starch, M2 was the weight of starch that has absorbed oil.

2. Results and discussions

2.1. Enzymatic hydrolysis

2.1.1. Orthogonal test results

Tab.2 The results of orthogonal test

NO.	factors					Oil absorption/%
	A	B	C	D	E	
1	1	1	1	1	1	71.76
2	1	2	2	2	2	68.06
3	1	3	3	3	3	72.5
4	1	4	4	4	4	64.55
5	2	1	2	3	4	52.86
6	2	2	1	4	3	54.19
7	2	3	4	1	2	90
8	2	4	3	2	1	52.42
9	3	1	3	4	2	68.62

10	3	2	4	3	1	52.86
11	3	3	1	2	4	66.76
12	3	4	2	1	3	83.17
13	4	1	4	2	3	69.26
14	4	2	3	1	4	55.65
15	4	3	2	4	1	33.63
16	4	4	1	3	2	75.71
K1	69.218	65.625	67.105	75.145	52.668	
K2	62.368	57.69	59.43	64.125	75.597	
K3	67.852	65.722	62.297	63.483	69.78	
K4	58.563	68.962	69.168	55.247	59.955	
R	10.655	11.272	9.738	19.898	22.929	

By orthogonal test, we can choose the optimal preparation process for cross-linked cassava porous starch preparation, the order of influencing factors for cross-linked porous starch absorption rate was reaction temperature > enzyme dosage > starch emulsion concentration > reaction time > pH value. Considering the factors that affect the preparation of cross-linked porous starch, the optimum technological conditions were A₁B₄C₄D₁E₂, those are temperature 45°C, the enzyme content 1%, the concentration of starch 40%, the reaction time 20h, pH5.50.

3. Conclusions

In this study, the preparation process and the optimum conditions of cassava starch were discussed. After cross-linking, enzymatic hydrolysis and other conditions, the oil absorption rate of cassava starch was increased, which indicated that the degree of resistance to paste was improved, and the adsorption ability was improved. The best conditions for cross-linking: reaction temperature 50°C, reaction time 3 h, pH10.00, cross-linking agent dosage of 0.5% cassava starch concentration 40%; the optimal preparation conditions for cross-linked cassava porous starch, reaction temperature 45 °C, reaction time 20 h, enzyme dosage 1%, pH5.50, substrate concentration 40%.

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References

- [1] Wu,Y.,Du,X.,Ge,H.,et al. Preparation of microporous starch by glu-coamylase and ultrasound. *Starch (Stärke)*, 2011(63): 217-225.
- [2] Zhang, B., Cui,D., Liu,M., et al.Corn porous starch: Preparation, characterization and adsorption property. *International Journal of Biological Macromolecules*, 2012(50):250-

256.

- [3] Mahsa M., Sara H., Asgar F.. Functional properties of microporous wheat starch produced by α -amylase and sonication. *FoodBioscience*, 2015 (11):79-84
- [4] Bai Y., Shi Y.C.. Structure and preparation of octenyl succinic esters of granular starch, microporous starch and soluble maltodextrin. *Carbohydrate Polymers*, 2011 (83) :520-527
- [5] Xu, A., Seib, P. A.. Determination of the level and position of substitution in hydroxypropylated starch by high-resolution ^1H -NMR spectroscopy of alphas limit dextrans. *Journal of Cereal Science*, 1997 (25):17-26.
- [6] Bao, J. S., Xing, J., Phillips, D. L., et al.. Physical properties of octenyl succinic anhydride modified rice, wheat, and potato starches. *Journal of Agricultural and Food Chemistry*, 2003 (51):2283-2287.
- [7] Bhosale, R., & Singhal, R. Process optimization for the synthesis of octenylsuccinyl derivative of waxy corn and amaranth starches. *Carbohydrate Polymers*, 2006 (66): 521-527.
- [8] Segura-Campos, M., Chel-Guerrero, L., Betancur-Ancona, D.. Synthesis and partial characterization of octenylsuccinic starch from *Phaseolus lunatus*. *Food Hydrocolloids*, 2008 (22):1467-1474.
- [9] Song, X. Y., He, G. Q., Ruan, H., et al. Preparation and properties of octenyl succinic anhydride modified early indica rice starch. *Starch/Stärke*, 2006 (58): 109-117.
- [10] Hung, P. V., Huong, N. T. M., Phi, N. T. L., et aln. Physicochemical characteristics and in vitro digestibility of potato and cassava starches under organic acid and heat-moisture treatments. *International Journal of Biological Macromolecules*, 2017 (95) 299-305