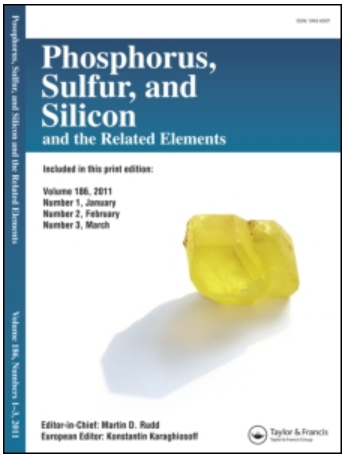

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

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To cite this Article Zhongdong, Liu , Yan, Yang , Xinlou, Gao and Li, Gao(2008) 'Study on the Preparation of Wheat Starch Phosphate with Low Degree of Substitution', Phosphorus, Sulfur, and Silicon and the Related Elements, 183: 2, 547 — 554
To link to this Article: DOI: 10.1080/10426500701764916
URL: http://dx.doi.org/10.1080/10426500701764916

Phosphorus, Sulfur, and Silicon, 183:547–554, 2008

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500701764916

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Study on the Preparation of Wheat Starch Phosphate with Low Degree of Substitution

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In this article, we used the response surface analysis to optimize the response condition of the starch phosphate. The sodium pyrophosphate was used as the catalyst in esterification to prepare wheat starch phosphate, and we explored the influence of pH, temperature, operation time, and the amount of phosphate vs. the viscosity characteristic of the products. The best technological conditions were determined by means of response surface design, and the perfect result was obtained after the nature research of all the products. This research serves as reference to find out how to raise the economic benefits of wheat further processing and how to handle its bulk by-products, especially for industry, the most important wheat producing area.

Keywords Response surface; wheat starch; starch phosphate

INTRODUCTION

Starch is a kind of important biological polysaccharide, which is synthesized in the cytoplasm of plant cells, supplying energy for the growth of life.[1] Meanwhile, it is widely used in many areas, such as food, medicine, paper manufacturing and textile industry, and so on.

Starch cannot be dissolved in cool water. Because of the physicochemical property limitation of original starch, it cannot meet the

This work was supported by the Natural Science Foundation of China under Contract No. 20672029/3020762, National 863 program of the Ministry of Science and Technology of P. R. China under Contract No. 2007AA100401. International Cooperation Program of the Ministry of Science and Technology of P. R. China under Contract No. [1999] 0251/[2001] 0103/[2001] 277. The authors thank Professor Zhao Yufen and Doctor Yan Liu and Gang Li (College of Chemistry and Engineering, Xiamen University, Xiamen 361005, P. R. China) for their kind help and valuable advice.

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requirement of industry development. Therefore, it is the modified starch which is usually applied in the industry.

The starch phosphate is one kind of modified starch products, which is also a kind of anionic starch. Compared to the original ones, the aleurone of starch phosphate is not liable to the degradation of bacteria, having a higher viscosity, transparency and glueyness. During the food processing, the starch phosphate is used as the emulsifier, thickener or stabilizer. For its good stability of freezing-melting, the starch phosphate is especially suitable to be applied in the frozen foods.[2] The starch phosphate is used for the paper sizing in the paper manufacturing industry and in the textile industry, it is used for the fabric sizing, dressing, printing and dying, both of which has a better result than the original starch. In addition, the starch phosphate can be used as adhesive, sedimentation agent, drug bulking agent and detergent etc.[3]

After proper experimental design, Response Surface Methodology (RSM) is applied to fit the function relationship between factors and response value using multiple quadratic regression equations and to find out the best technological parameter to solve the multivariate problem by the analysis of the regression equations.[4] At the present time, it is one of the effective methods to reduce cost and optimize processing condition in the chemistry, food and biology industry etc.[5–9]

In order to improve the performance of wheat starch and to broaden its application range, the response surface analysis is used in this article to optimize the response condition of the starch phosphate.

MATERIALS AND METHODS

Materials

Wheat starch was obtained from Shangqiu Limited Liability Company of Flour, China.

Methods

Preparation of Wheat Starch Phosphate

Sodium pyrophosphate was chosen as catalyst to prepare the wheat starch phosphate. Sodium pyrophosphate and urea were dissolved in water, added wheat starch, mixed intensively, regulated pH, stirred, dried, ground and sieved, reacted at 150°C for 75 minutes.

Response Surface Experiment Design

Through research, we found that pH, temperature, reaction time, and the amount of phosphate are the key four factors in esterification. Therefore, we adopted Central Composite Model and orthogonal experimental design (four factors and three levels, 21 experiments in total). The model is:

$$\begin{aligned} Y = {} & b0 + b1A + b2B + b3C + b4D + b11A^2 + b22B^2 \\ & + b32C^2 + b42D^2 + b12A^*B + b13A^*C + b14A^*D \\ & + b23B^*C + b24B^*D + b34C^*D. \end{aligned} \quad (1)$$

We analyzed the response surface and optimized equation make sure forecast response value which whether is most spherical surface or least spherical surface or saddle surface. Then we analyzed and ensured how to obtain better experiment result.

RESULTS AND DISCUSSION

Experiment Design and Experiment Results

According to basic research results, the amount of phosphate (A) we used was 0.02 g–0.04 g, pH(B) 5.1–5.9, temperature (C) 140°C–160°C, and time(D) 45 min–105 min in the experiment. Experiment design and results are shown in Tables I and II. Variable analysis is shown in Table III.

When $P < 0.05$, the items of A, B, A^2, C^2, AB, and AC are marked in the model. By the analysis of the model, we can obtain the following data:

$$R^2 = 0.9040, \quad \text{Adj}\, R^2 = 0.9800, \quad \text{Adeq}\, \text{Precision} = 6.058. \quad (2)$$

Adeq Precision is the ratio of noise to signal, which is usually greater than four. In our experiment, the value of Adeq Precision is 6.058,

TABLE I Experiment Design

Factors	(A) Amount of phosphate/g	(B) pH/mL	(c) Temperature/°C	(D) Time/min
Levels	0.02	5.1	140	45
	0.03	5.5	150	75
	0.04	5.9	160	105

TABLE II Experiment Design and Results of Four Factors

	A (g)	B	C (°C)	D (min)	V
1	0.04	5.5	150	75	3800
2	0.04	5.1	140	105	3000
3	0.03	5.5	160	75	2800
4	0.02	5.1	140	45	2000
5	0.02	5.9	140	105	2200
6	0.03	5.5	150	75	4030
7	0.03	5.9	150	75	3700
8	0.04	5.9	160	45	2890
9	0.03	5.5	150	75	4030
10	0.03	5.5	150	75	4030
11	0.02	5.9	160	105	3000
12	0.03	5.5	150	75	4030
13	0.02	5.5	150	75	3680
14	0.03	5.5	140	75	3700
15	0.03	5.1	150	75	3800
16	0.04	5.1	160	105	3000
17	0.03	5.5	150	105	3200
18	0.03	5.5	150	45	3700
19	0.02	5.1	160	45	2700
20	0.04	5.9	140	45	2800
21	0.03	5.5	150	75	4030

therefore, the model building can suitably reflect the experiment results. Coefficient (R^2) is 0.9040, which indicates that the equation is in conformity with the fact, providing better estimation.

The equation of regression was obtained by the analysis of software Design expert 6.0.10:

$$\begin{aligned} V(\text{viscosity}) = {} & 3938.47 + 60.00A - 50.00B + 69.00C - 250.00D \\ & - 122.20A^2 - 12.20B^2 - 612.20C^2 - 412.20D^2 \\ & - 351.25A^*B - 176.25A^*C - 73.75A^*D + 23.75^*C \\ & - 163.75B^*D + 1.25C^*D. \end{aligned} \quad (3)$$

Analysis of Influence Factors of Viscosity

The analysis of variance shows that each of the four factors makes a contribution to the viscosity value, while the influence of phosphate consumption and the pH value is significant. In this article, owing to

TABLE III Variable Analysis Table

Source	Sum of Squares	DF	MS	F	Prob > F	Significance
Model	7.200E + 006	14	5.143E + 005	4.04	0.0478	
A	7200.00	1	7200.00	0.056	0.0200	*
B	5000.00	1	5000.00	0.039	0.0495	*
C	47610.00	1	47610.00	0.37	0.0635	
D	1.250E + 005	1	1.250E + 005	0.98	0.0603	
A2	38119.64	1	38119.64	0.30	0.0420	*
B2	32135.90	1	32135.90	0.25	0.0633	
C2	9.568E + 005	1	9.568E + 005	7.51	0.0337	*
D2	4.337E + 005	1	4.337E + 005	3.40	0.1146	
AB	1.974E + 005	1	1.974E + 005	1.55	0.0259	*
AC	2.485E + 005	1	2.485E + 005	1.95	0.0212	*
AD	8702.50	1	8702.50	0.068	0.0802	
BC	4512.50	1	4512.50	0.035	0.0857	
BD	42902.50	1	42902.50	0.34	0.0582	
CD	12.50	1	12.50	9.807E-005	0.0920	
Residual	7.647E + 005	6	1.275E + 005			
Lack of Fit	7.647E + 005	2	3.824E + 005			
Pure Error	0.000	4	0.000			
Cor Total	7.965E + 006	20				

the majority of researches on mono-factor, we emphasized on the inter-influence of two factors to the wheat starch phosphate.

Inter-Influence of the Phosphate Consumption and the pH Value

When urea consumption is 0.0460 g, temperature at 150°C and time for 75 minutes, the inter-influence to viscosity of the phosphate consumption and the pH value is shown in Figure 1.

As is indica ted in Figure 1, when pH is 5.1, with the increase of phosphate consumption, the viscosity of the starch phosphate increases significantly at first, while it gets to some extent, it maintains stable steadily. When pH is 5.9, the viscosity of the starch phosphate decreased gradually with the increasing of phosphate consumption. Therefore, high pH environment has a negative impact on viscosity. When the phosphate consumption is 0.02, the viscosity of the starch phosphate goes up significantly with the increase of pH. However, when the phosphate consumption is 0.04, there is no change at the beginning which is followed by the decline of viscosity when the pH value gets to a certain level. Otherwise, the analysis of variance table above also shows the

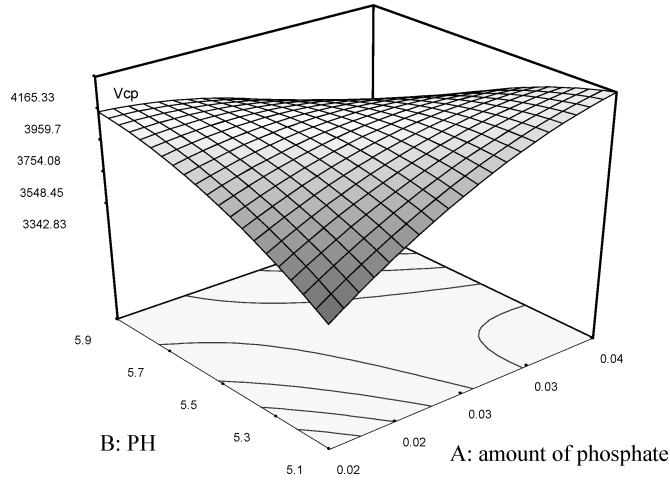

FIGURE 1 The inter-influence of the amount of phosphate and the pH to viscosity.

significant inter-influence of the phosphate consumption and the pH value.

Inter-Influence of the Phosphate Consumption and the Temperature

When pH is 5.5 and time for 75 minutes, the inter-influence to viscosity of the phosphate consumption and the temperature is shown in Figure 2.

As is indicated in Figure 2, when the temperature is 140°C, the viscosity of the starch phosphate increases slowly with the increase of phosphate consumption. When the temperature is 160°C, the viscosity of the starch phosphate almost maintains stable with the increase of phosphate consumption. When the phosphate consumption is fixed, there is a rapid raise of the viscosity which is followed by the decline with the increase of temperature. When the value of viscosity reaches the summit at 152°C, the starch phosphate preparation is the best. Otherwise, the analysis of variance table above also shows the significant inter-influence of the phosphate consumption and the temperature.

Optimization of the Response Condition of the Starch Phosphate

If the preparation technique of starch phosphate with low degree of substitution is applied to the food industry, according to the principle of

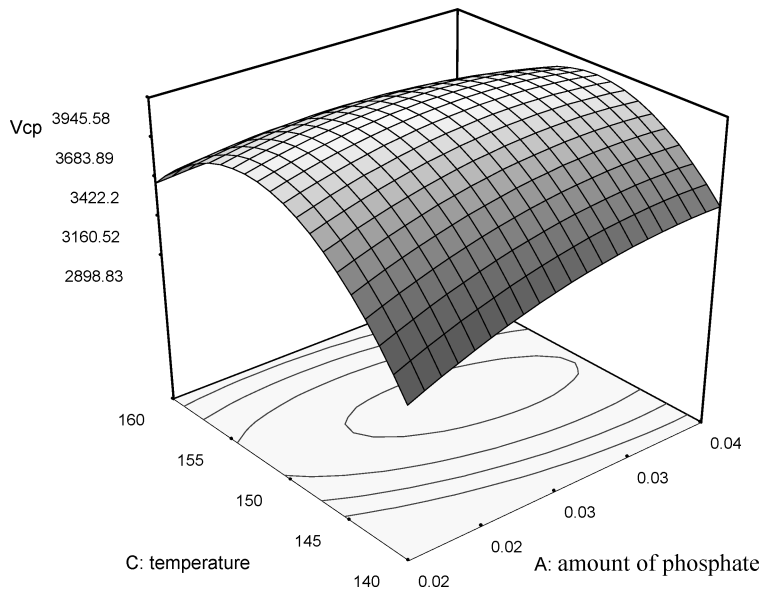

FIGURE 2 The inter-influence of the amount of phosphate and the temperature to viscosity.

least cost and most benefit, the optimal condition is built by regression model and analyzed by the statistic software Design Expert. The results are shown in Table IV.

As is indicated in Table IV, the preparation condition of starch phosphate is: phosphate consumption 0.02 g–0.03 g, pH 5.1–5.8, temperature at 142°C–160°C and time for 51 min–104 min, totally ten groups of optimization condition.

TABLE IV The Best Experiment Group in Computer Program

Number	Amount of phosphate(A)/g	pH(B)	Temperature(C)/°C	Time (D)/min	Probability
1	0.02	5.3	154	66	1
2	0.03	5.4	145	56	1
3	0.02	5.4	158	54	1
4	0.03	5.3	158	104	1
5	0.03	5.3	152	75	1
6	0.02	5.6	152	49	1
7	0.02	5.1	142	51	1
8	0.02	5.6	148	52	1
9	0.03	5.8	150	65	1
10	0.03	5.6	160	73	1

Experiment Verification

According to the ten groups of parameter in Table IV, we selected the ninth group to verify its result. When the phosphate consumption is 0.03 g, pH 5.8, temperature at 150°C and time for 65 min, the viscosity is up to 4500, which is 11% higher than the maximal result obtained before, proving that it is feasible to apply the method of response surface to optimize the preparation condition of starch phosphate.

CONCLUSION

We made use of Response Surface Methodology to optimize the reaction condition of the starch phosphate and achieved good result. It is indicated that the parameter which was reasonable given by the software and the method which was feasible utilized Response Surface Methodology to optimize reaction condition of the starch phosphate.

This report might provide a method to find out how to raise the economic benefits of wheat advanced processing and to handle the bulk wheat products.

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