Composition Optimization of Poly(vinyl alcohol)-/ Cornstarch-Blended Biodegradable Composite Using Response Surface Methodology

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ABSTRACT: Response surface methodology was used to analyze the effect of amylase level (X_1) and glycerol level (X_2) on the objective [water solubility index (WSI), water absorption index (WAI), and Max. loading] attributes of a poly(vinyl alcohol)-/cornstarch-blended composite. A rotable central-composite design (CCD) was used to develop models for the objective responses. The experiments were run at die temperature 100°C with a feed rate of 25 g/min and a screw speed of 35 rpm. Responses were most affected by changes in the amylase level (X_1) and to a lesser extent by glycerol level (X_2). Individual contour plots of the different responses were overlaid, and regions meeting the optimum WSI of 3.03 (%), WAI of 5.08 (g gel/g dry wt), and Max. loading of 29.36 (N) were identified at the amylase level of 2.8 (mL) and the glycerol level of 92.2 (mL), respectively. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 258–264, 2009

Key words: biodegradable; amylase; glycerol; composite; optimization

INTRODUCTION

Nonbiodegradable synthetic plastics, such as polystyrene, polypropylene, and polyethylene, are widely used in daily life, in food industry, medical field, and agriculture. A heavy environmental pollution accompanies their uses because of their nonbiodegradability. The disposal of waste plastics has become a serious problem.[1] Therefore, in the past two decades, biodegradable materials have been paid attention as alternatives to the petroleum-derived plastics.[1–9] Natural biopolymers including starch, cellulose, and chitosan were tested, alone or combined with synthetic polymers, for the possibility to form a fully or partially biodegradable film.[1] Of these materials, starch is the most attractive candidate because of its low cost, easy availability, and high production from annually renewable resources.[7]

In fact, the low water resistance and high brittleness of starch films limited their extensive application.[10] Therefore, many attempts have been made to overcome these problems by blending starch with synthetic polymers. However, the biodegradability of starch film decreased with addition of the nondegradable synthetic polymers. Therefore, much interest lies in blending starch with biodegradable synthetic polymers.[11–15]

Poly(vinyl alcohol) (PVA) is a hydrophilic biodegradable polymer, which is mainly composed of C—C bonds.[6] However, its water solubility is related to its degree of hydrolysis, molecular weight, and the modification while blending with other processing additives,[16] with a maximum value at the degree of hydrolysis of 88%.[17] From the cost and practicality points, it is preferable that the blend contains much starch. However, the properties of the blend films deteriorated as the starch proportion in the film formulation increased. This might result from a poor compatibility between starch and PVA[18] and phase separation during film preparation.[19] Moreover, the water resistance of the native starch/PVA film deteriorated when compared with that of the PVA film.

The effectiveness of response surface methodology (RSM) in the development and optimization of cereal products has been highlighted by different authors.[20–24] The basic principle of RSM is to relate product properties of regression equations, which describe interrelations between input parameters and product properties. Some of the good examples of appropriate applications of this technique in food extrusion are the optimization of complex products or properties or of many process variables.[25]

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In this study, investigations were undertaken to formulate an amylase level (X_1) and glycerol level (X_2) additive-based PVA-/cornstarch-blended composite by extrusion technology. The well-established functional property of amylase act as a chain attacker of the alpha-1,4-link of starch, whereas the glycerol act as a good destructuring-plasticizing agent.[26,27] The objective of this study was to optimize the formulation of raw compositions, i.e., amylase level (X_1) and glycerol level (X_2) for the production of a PVA-/cornstarch-blended composite by RSM.

MATERIALS AND METHODS

Materials

Edible corn starch (a white fine powder with 10 wt % moisture content containing 30 wt % amylose and 70 wt % amylopectin) was obtained from the Hong Chi Company Limited, Taiwan, Republic of China. PVA [product name code BF-17, complete alkalization PVA, degree of polymerization (DP) average as 1700–1800, molecular weight (MW) for 75,000–80000] was purchased from the Chang Chun Petroleum Chemistry Incorporated Company, Taiwan, Republic of China. Amylase was used at a concentration similar to the one usually found in human blood plasma (50 U/L). The enzyme solution had an activity of 0.35 mg/(U min) at pH 6.9 and 20°C/g of soluble starch. To stabilize an amylase, 1 mL calcium chloride was employed and was obtained from human saliva, Sigma reference A0521. Glycerol level (AR grade) was obtained from Sigma Chemical, St. Louis, MO.

Extruder

A single-screw extruder made by Yea Jing Machinery (Taiwan, Republic of China) with screw compression ratio 2.8, $\Psi = 2.8$, $L/D = 32$ was used. Electrical resistive heater (220 V, 1700 W) heated the zone at die temperature. A single-screw volumetric feeder fed the formulas (Table I). A 19.1 mm × 2.93 mm rectangular-shaped single hole die was used to give continuous extrudates. The experiments were run at die temperature 100°C with a feed rate of 25 g/min and a screw speed of 35 rpm.

Experimental design

This study is based on the hypothesis that water solubility index (WSI), water absorption index (WAI), and Max. loading are functionally related to specific composition and attempts to fit multiple-regression equations describing quality composition responses.[28,29] Table I lists the levels and levels of the RSM design with different amylase and glycerol levels.

TABLE I
The Levels of the RSM Design with Different Operating Conditions

	Coded		Uncoded	
Treatments	X_1	X_2	Amylase level (mL)	Glycerol level (mL)
1	1	1	2	70
2	−1	1	8	70
3	−1	−1	2	130
4	1	−1	5	130
5	0	1.414	5	142.43
6	0	−1.414	5	57.57
7	1.414	0	9.24	100
8	−1.414	0	0.76	100
9	0	0	5	100
10	0	0	5	100
11	0	0	5	100
12	0	0	5	100
13	0	0	5	100

WAI and WSI were measured using a technique developed for cereals.[13] The ground extrudate was suspended in water at room temperature for 30 min, gently stirred during this period, and then centrifuged at 3000 × g for 15 min. The supernatant was decanted into an evaporating dish of known weight. The WSI is the weight of dry solids in the supernatant expressed as a percentage of the original weight of sample. The WAI is the weight of gel obtained after the removal of the supernatant per unit weight of original dry solids. Determinations were made in triplicate.

Max. loading were carried out on the tension test specimens. About 5 mm/min was used for the biodegradable plastics, and the tests were carried out in conformity with ISO 294, ISO 527 (TS 1396), and TS 720.[30–32] The tests were carried out at least five times for each specimen, and the results were averaged arithmetically.

The design depends upon the symmetrical selection of variation increments about the center composition. These levels of variation were chosen to be within the range of reasonable formulations, and the increments were carefully selected, since interpretation of the results was valid only within the experimental limits.[28,29] The levels selected were also based on the conclusions of a previous study,[2,4,7,8,10] which are important for PVA-/cornstarch-blended composite. However, the optimum processing variables for each response did not fall exactly in the same region in the two-dimensional space formed by the compositions levels. Moreover, those constraints were set such that all responses (WSI, WAI, and Max. loading) met their optimum acceptable region with the same composition levels. Therefore,

TABLE II
The Box-Behnken Arrangement and Responses

Treatments	Responses		
	Water solubility index Y_1 (%)	Water absorption index Y_2 (%)	Max. loading Y_3 (N)
1	2.91 ± 0.27	5.23 ± 0.26	34.11 ± 0.25
2	3.79 ± 0.20	4.64 ± 0.21	28.03 ± 0.46
3	1.96 ± 0.15	5.25 ± 0.32	21.18 ± 0.51
4	2.80 ± 0.19	5.09 ± 0.29	18.15 ± 0.34
5	2.67 ± 0.17	5.11 ± 0.33	20.67 ± 0.37
6	4.65 ± 0.38	4.63 ± 0.31	29.71 ± 0.38
7	3.67 ± 0.21	4.79 ± 0.27	23.77 ± 0.23
8	1.99 ± 0.10	5.48 ± 0.19	30.75 ± 0.41
9	3.34 ± 0.25	4.99 ± 0.30	26.01 ± 0.44
10	3.77 ± 0.23	4.75 ± 0.30	26.87 ± 0.31
11	3.09 ± 0.19	4.93 ± 0.23	26.69 ± 0.35
12	3.69 ± 0.16	4.82 ± 0.24	26.64 ± 0.38
13	3.56 ± 0.29	4.77 ± 0.28	26.34 ± 0.56

it was assumed that a WSI should be more than 3.03 but less than 3.12; WAI should be more than 5.03 but less than 5.08; and Max. loading should be more than 29 but less than 29.5 N.

The increments of variation for each variable spaced around the center point levels, along with the responses are presented in Table II. Feed compositions were coded for solutions of the multiple regression (prediction) equations.[28,29]

A central composite design (CCD) (Table II) was adopted.[28,29] In this design, for two variables, the size of the experiment was reduced by using the 2^k, factorial (2^2), thus making the total number of experiments equal to 13 instead of 50 with full factorial design.[28,29] Experiments were randomized to minimize the effects of unexplained variability in the observed responses because of the external factors. The function was assumed to be approximated by a second-degree polynomial equation:

$$Y_k = b_{k0} + \sum_{i=1}^{2} b_{ki}X_i + \sum_{i=1}^{2} b_{ii}X_i^2 + \sum_{i\neq j=1}^{2} b_{kij}X_iX_j \qquad (1)$$

where b_{k0} is the value of the fitted response at the center point of the design, i.e., point (0, 0), b_{ki}, b_{ii}, and b_{kij} are the linear, quadratic, and cross-product regression terms, respectively.

Analysis of data

The regression analysis was conducted using the "stepwise variable selection backward elimination" procedure,[28,29] for fitting the model represented by eq. (1) to the experimental data. Optimization of the polynomial thus fitted was performed by numerical techniques, using the mathematical optimizer procedure of the Minitab 14.2 software package that deals with constraints. The mapping of the fitted response surfaces was achieved using the internal microprogram of the Minitab 14.2. The response surfaces and contour plots for these models are plotted as a function of two variables. The overlapping of the contour plots was done to take into account the three responses for their optimum values corresponding to two variables at a time.

RESULTS AND DISCUSSION

Diagnostic checking of the fitted model

Regression analyses for different models indicated that the fitted quadratic models accounted for more than 90% of the variations in the experimental data, which were highly significant. Multiple regression equations were generated relating WSI, WAI, and Max. loading to coded levels of the variables.[28] The developed models were indicated as given, whereas terms in those equations are based on the evaluation of P-values in Tables III–VIII.

$$\text{Water solubility index } (Y_1) = 3.4775 + 0.7906X_1 - 0.7934X_2 - 0.7827X_1^2$$

$(R^2 = 0.829)$

$$\text{Water absorption index } (Y_2) = 4.85835 - 0.31228X_1 + 0.20712X_2 + 0.29262X_1^2$$

$(R^2 = 0.865)$

$$\text{Max. Loading } (Y_3) = 25.464 - 2.3969X_1 - 6.2915X_2 + 0.8354X_1^2 + 3.8224X_1X_2$$

$(R^2 = 0.992)$

TABLE III
The Regression Model of Variance of Process Variables to Extrudate's Water Solubility Index

Term	Coefficient	SE Coefficient	T	P
Constant	3.4775	0.1378	25.2360	0.001
X_1	0.7906	0.1797	4.4000	0.003
X_2	−0.7934	0.1666	−4.7620	0.002
$X_1 \times X_1$	−0.7827	0.2451	−3.1940	0.015
$X_2 \times X_2$	0.0161	0.2432	0.0660	0.949
$X_1 \times X_2$	0.1682	0.3997	0.4210	0.687
$S = 0.3139$				
$R^2 = 90.0\%$				
R^2(adj) = 82.9%				

X_1, amylase level; X_2, glycerol level; S, the coefficient of variation.

TABLE IV
The Analysis of Variance of Process Variables to Extrudate's Water Solubility Index

Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
Regression	5	6.2281	6.2281	1.24562	12.64	0.002
Linear	2	5.07556	5.29217	2.64608	26.85	0.001
Square	2	1.13509	1.062	0.531	5.39	0.038
Interaction	1	0.01745	0.01745	0.01745	0.18	0.687
Residual error	7	0.68993	0.68993	0.09856		
Lack-of-fit	3	0.38413	0.38413	0.12804	1.67	0.308
Pure error	4	0.3058	0.3058	0.07645		
Total	12	6.91803				

TABLE V
The Regression Model of Variance of Process Variables to Extrudate's Water Absorption Index

Term	Coefficient	SE Coefficient	*T*	*P*
Constant	4.85835	0.04185	116.095	0.001
X_1	−0.31228	0.05457	−5.722	0.001
X_2	0.20712	0.0506	4.093	0.005
$X_1 \times X_1$	0.29262	0.07442	3.932	0.006
$X_2 \times X_2$	0.04349	0.07386	0.589	0.575
$X_1 \times X_2$	0.19471	0.12139	1.604	0.153
$S = 0.09534$				
$R^2 = 92.1\%$				
$R^2(\text{adj}) = 86.5\%$				

X_1, amylase level; X_2, glycerol level; S, the coefficient of variation.

TABLE VI
The Analysis of Variance of Process Variables to Extrudate's Water Absorption Index

Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
Regression	5	0.74497	0.74497	0.148994	16.39	0.001
Linear	2	0.59622	0.5696	0.284799	31.33	0.001
Square	2	0.12537	0.14107	0.070537	7.76	0.017
Interaction	1	0.02339	0.02339	0.023388	2.57	0.153
Residual error	7	0.06363	0.06363	0.00909		
Lack-of-fit	3	0.02035	0.02035	0.006783	0.63	0.635
Pure error	4	0.04328	0.04328	0.01082		
Total	12	0.8086				

TABLE VII
The Regression Model of Variance of Process Variables to Extrudate's Max. Loading

Term	Coefficient	SE Coefficient	*T*	*P*
Constant	25.464	0.1637	155.528	0.001
X_1	−2.3969	0.2135	−11.227	0.001
X_2	−6.2915	0.198	−31.778	0.001
$X_1 \times X_1$	0.8354	0.2912	2.869	0.024
$X_2 \times X_2$	0.1903	0.289	0.658	0.531
$X_1 \times X_2$	3.8224	0.4749	8.049	0.001
$S = 0.3730$				
$R^2 = 99.5\%$				
$R^2(\text{adj}) = 99.2\%$				

X_1, amylase level; X_2, glycerol level; S, the coefficient of variation.

TABLE VIII
The Analysis of Variance of Process Variables to Extrudate's Max. Loading

Source	DF	Seq SS	Adj SS	Adj MS	*F*	*P*
Regression	5	219.823	219.823	43.9646	210.12	0
Linear	2	206.957	149.087	74.5434	356.26	0
Square	2	0.434	1.685	0.8427	4.03	0.069
Interaction	1	12.432	12.432	12.432	59.42	0
Residual error	7	1.465	1.465	0.2092		
Lack-of-fit	3	1.007	1.007	0.3356	2.93	0.163
Pure error	4	0.458	0.458	0.1144		
Total	12	221.287				

All the main effects including linear and quadratic and the interaction of effects were calculated for each model. The regression coefficients are shown in Tables III, V, and VII, and the analysis of variances obtained for all the models are shown in Tables IV, VI, and VIII. The correlation coefficient for WSI, WAI, and Max. loading ($R^2 = 0.829$, $R^2 = 0.865$, and $R^2 = 0.992$, respectively) are very high for a response surface.

First, Table III indicates that the amylase level has positive linear but a negative quadratic effect on WSI. The glycerol level has a negative linear effect on WSI. Table IV shows that the multiple regression analysis of the WSI model was significant and the interaction of variables of the model was not significant.

Second, Table V shows that the amylase level has a negative linear but positive quadratic effect on WAI. The glycerol level has a positive linear effect on WAI. Table VI shows that the multiple regression analysis of the WAI model was significant and the interaction of variables of the model was not significant.

Finally, Table VII illustrates that the amylase level has negative linear and quadratic effects on Max. loading. The glycerol level has great negative linear effect on Max. loading. The interaction of amylase level and glycerol level has a great positive effect on Max. loading. Table VIII shows that the multiple regression analysis of the Max. loading model was significant and the interaction of variables of the model was not significant.

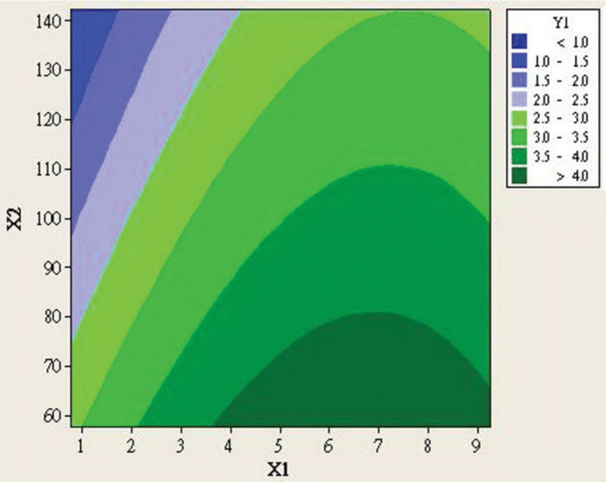

Figure 1 The contour and response surface plots of extrudate's water solubility index (WSI). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Analysis of variance

Once a model was selected, an analysis of variance was calculated to assess how well the model represents the data. The analyses of variances for different responses are presented in Tables IV, VI, and VIII. To evaluate the goodness of the model, the coefficient of variation (the level of the standard error of estimate to the mean value expressed as a percentage) and *F*-value tests were conducted. As a general regulation, the coefficient of variation should be not greater than 10%.[28,29]

In this study, the coefficients of variation for WSI, WAI, and Max. loading were 0.3139, 0.09534, and 0.3730%, respectively. Also, the *F*-value for both responses was significant at the 95% level, as shown in Tables IV, VI, and VIII. The contour and response surface plots for both responses are shown in Figures 1–3. From the analyses of residuals (data not

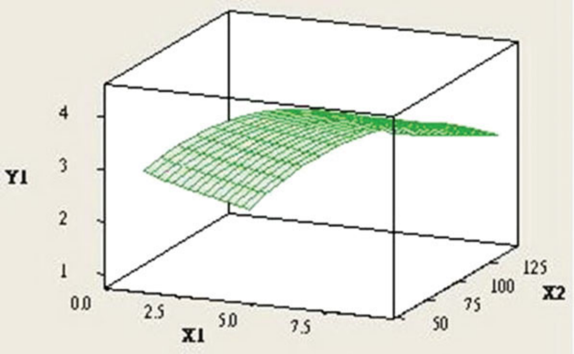

Figure 2 The contour and response surface plots of extrudate's water absorption index (WAI). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

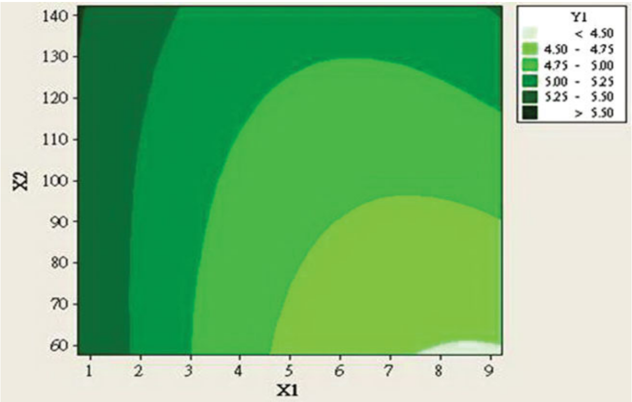

Figure 3 The contour and response surface plots of extrudate's Max. loading. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

shown), it is possible to conclude that they were randomly distributed around zero and there is no evidence of outliers (no point lying away from the mean more than four times the means).[28,29]

Conditions for optimum responses

The direction in which to change variables to optimize WAI, WSI, Max. loading were usefully indicated by the models. The multiple regression equation Y_1 was solved for the optimum WSI [3.03 (%)], Y_2 was solved for the optimum WAI [5.08 (g gel/g dry wt)], and Y_3 was solved for the optimum Max. loading [29.36 (N)]. The optimum conditions to achieve the above responses are presented in Figure 5. Optimum values of WSI, WAI, and Max. loading for all the variables lie close to the middle of the experimental range, indicating the validity of the selection of the variables range, and the models was accepted because of their significance at $P < 0.01$. The response surface models were obtained by selecting three variables, and the one remaining have the value that leads to the optimum response in the equations of Y_1, Y_2, and Y_3. Some selected surfaces are presented in Figures 1–3.

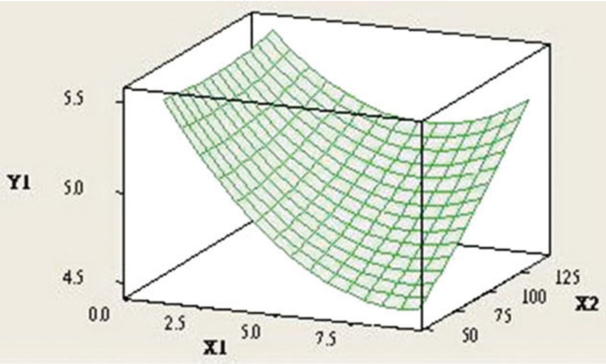

Figure 4 The optimum operating condition from the contour plots. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

In moving along the horizontal axis in Figure 1, it can be seen that with the increase of amylase level, it had a parabolic effect on WSI. However, the glycerol level has a negative linear effect on WSI. The optimum value of WSI lies very close to the middle of experimental region.

Figure 2 shows that with the increase of amylase level, it had a negative parabolic effect on WSI. However, the glycerol level has a positive linear effect on WSI. The optimum value of WAI lies very near to the bottom of the experimental region. Gomez and Aguilera[33] reported that the increase of the starch level will increase with the amount of the —OH functional group, which resulted in the increase of water absorption.

In Figure 3, the amylase level has a negative linear effect on Max. loading and the optimum lies near the middle value of the level. However, the glycerol level also has a negative linear effect on Max. loading.

Superimposition of contour plots of responses

Areas of optimum performance were located by superimposing contour graphs for WSI, WAI, and Max. loading for compositions levels, which established limits of acceptable quality for each factor. Since the optimum processing variables for each response did not fall exactly in the same region in the two-dimensional space formed by the compositions levels, constraints were set such that all responses (WSI, WAI, and Max. loading) met their optimum acceptable region with the same composition levels. It was assumed that a WSI should be more than 3.03 but less than 3.12, WAI more than 5.03 but less than 5.08, and Max. loading should be more than 29 but less than 29.5 N.[10,17,19,11]

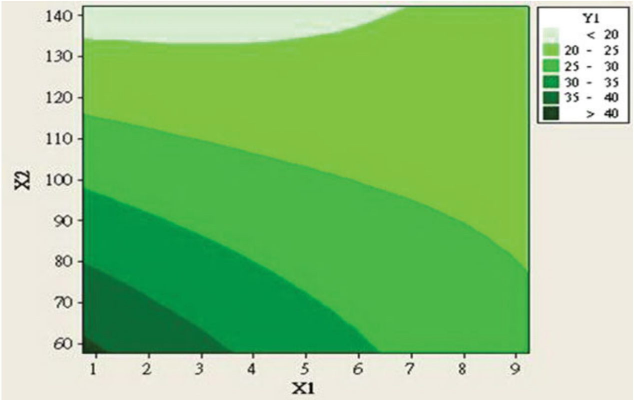

Figure 5 The predicted responses values by the optimum formula. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Superimposing the individual contour plots for the response variables resulted in the identification of a region (shown by the white-colored area), which satisfied all constraints and runs at die temperature 100°C with a feed rate of 25 g/min and a screw speed of 35 rpm as shown in Figure 4. However, it may not be advisable to set the experimental conditions very rigid and, therefore, a moderation level has been given to each process variable and response as shown in Figure 5. Hence, the final optimum conditions, for WSI of 3.03 (%), WAI of 5.08 (g gel/g dry wt), and Max. loading of 29.36 (N) were identified at the amylase level of 2.8 (mL) and the glycerol level of 92.2 (mL), respectively, and they were computed as shown in Figure 5. The optimum conditions were experimentally tested, obtaining a WSI of 3.123 (%), WAI of 5.157 (g gel/g dry wt), and Max. loading of 30.23 (N).

CONCLUSION

This could be concluded that the system of WSI, WAI, and Max. loading of PVA-/cornstarch-blended composite can be effectively optimized using RSM and with a minimum number of experiments. Also, computerized computations, model building, and generation of three-dimensional graphs and contours will be effective in simplifying the complexity of the preparation of PVA-/cornstarch-blended composites with different variables used. According to the optimum conditions given for the variables, the process can be scaled up for industrial production, and the PVA-/cornstarch-blended composite are suitable in the application of biodegradable plastics.

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