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Optimization of the Technology for Preparing Soluble Dietary Fiber from Extruded Soybean Residue

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1. Introduction

It is well known that dietary fiber plays an important role in many physiological processes and in the prevention of diseases of different origin (Rodriguez et al., 2006; Champ et al., 2000). According to the solubility in water, total dietary fiber (TDF) can be categorized into two groups, namely soluble (SDF) and insoluble (IDF) dietary fiber. SDF and IDF have been known to play different physiological roles in human health (Vasanthan et al., 2002; Burlcitt et al., 1974). SDF appears to be more effective than IDF in many healthy aspects. Therefore, preparation of SDF is especially important.
China is origin of soybean, and soybean production has been highest in the world. China's consumption of soybean products is considerable. However, a large number of by-products that is soybean residue would be produced in soybean processing. Previously, most of soybean residue only was used as feed or fertilizer. Its practical value was not fully utilized. Soybean residue is a good dietary fiber resource (Bourquin et al., 1996; Tharanathan et al., 2003). The content of total dietary fiber in soybean residue is about 60% (Zheng et al., 2005; Jiang et al., 2001). It has a broad development prospects. There are many methods that can be used to prepare SDF, such as acid (Wang et al., 2004), alkaline (Wang et al., 2004; Zheng et al., 2008), enzymatic (Liu et al., 2008), fermentation (Tu et al., 2007), mechanical method (Tu et al., 2007; Jin et al., 1996) and the combined method (Xu et al., 2005; Tu et al., 2008) and so on, but the raw materials were processed without extrusion residue. The extruded soybean residue as raw material was treated with alkali, which has not been previously reported. The present research is to optimize the conditions of SDF preparation. The high yield of SDF will be prepared under the optimal conditions.

2. Materials and methods

2.1 Materials

Soybean residue was supplied by bean products factory of Northeast Agricultural University (Harbin, China). Heat stable α-amylase, Protease and amyloglucosidase were obtained from Sigma Chemical Company. All the other reagents were of analytical grade.

2.2 Sample preparation

Fresh soybean residue was dried overnight in an oven at temperature of 60°C. Then the dried soybean residue was ground using an electric grinder and sieved through 0.425mm mesh. The ground soybean residue was extruded by a single-screw extruder. The conditions of extrusion were the following: feed moisture approximately 20%, mass temperature approximately 160°C, screw speed approximately 175rpm and a diameter of 10 mm. The extruded soybean residue was ground and sieved through 0.425mm mesh. The extruded soybean residue was used for alkali treatment.

2.3 Alkali treatment

Twenty grams of soybean residue or extruded soybean residue were mixed with 400 ~ 560mL concentration of 0.40 ~ 1.20% (W/V) NaOH solution. The slurries containing NaOH were incubated in a water bath for a selected period of time (40 ~ 80min) at different temperature (70 ~ 90°C). The alkali treatment variables are presented in table 1. After neutralization with 6mol/L HCL, the resultant suspensions were centrifugated at 3000g for 20min, and the supernatants were added four times the volume of 95% ethanol. Let precipitate form at room temperature 1h. The SDF was obtained after centrifugation (3000g, 10min), and was dried at 60°C. Soybean residue or extruded soybean residue was treated with alkali in the same conditions, the yields of SDF were compared.

Independent variable	Symbol		Levels				
	Uncodified	Codified	-2	-1	0	+1	+2
Ratio of liquid to solid	X_1	x_1	20 : 1	22 : 1	24 : 1	26 : 1	28 : 1
Temperature (°C)	X_2	x_2	70	75	80	85	90
Time (h)	X_3	x_3	40	50	60	70	80
Alkali concentration (%)	X_4	x_4	0.40	0.60	0.80	1.00	1.20

Table 1. Independent variables and their levels used for the central composite design and optimization of alkali treatment conditions

2.4 The yield of soluble dietary fiber determination

The SDF content of the precipitate was determined using the Enzymatic-Gravimetric method (Lee et al., 1992). The yield of SDF was expressed as:

$$\text{Yield of SDF }(\%) = \frac{\text{dry weight of precipitate(g)} \times \text{content of SDF}(\%)}{\text{weight of soybean residue or extruded soybean residue(g)}} \times 100\% \quad (1)$$

2.5 Experimental design

A statistical tool utilizing five levels, four variables and central composite design, with 31 individual points, was employed to study the effects of alkali treatment on SDF preparation from extruded soybean residue. The independent variables and their levels were selected, based on the preliminary experiments in our laboratory (data not shown). The independent variables X_i were coded as x_i, which are defined as dimensionless, according to the Eq.(2):

$$x_i = (X_i - X_0) / \Delta X_i \quad (2)$$

Where x_i is the coded value of an independent variable, X_i is the real value of an independent variable, X_0 is the real value of an independent variable at the centre point, and ΔX_i is the step change value. The independent variables and their levels are presented in Table 2. The 31 runs were performed in a totally random order to minimize bias. Each experiment had two replications and the average prepared SDF was taken as the response, Y. The responses generated from the experiment are presented in Table 3.

Run	**Independent variables** [b]				**Response(Y)** [c]
	x_1	x_2	x_3	x_4	
1	**-1**	**-1**	**-1**	**-1**	**22.05**
2	**1**	**-1**	**-1**	**-1**	**23.47**
3	**-1**	**1**	**-1**	**-1**	**22.86**
4	**1**	**1**	**-1**	**-1**	**24.59**
5	**-1**	**-1**	**1**	**-1**	**23.91**
6	**1**	**-1**	**1**	**-1**	**24.55**
7	**-1**	**1**	**1**	**-1**	**25.46**
8	**1**	**1**	**1**	**-1**	**26.58**
9	**-1**	**-1**	**-1**	**1**	**23.62**
10	**1**	**-1**	**-1**	**1**	**25.63**
11	**-1**	**1**	**-1**	**1**	**25.03**
12	**1**	**1**	**-1**	**1**	**27.62**
13	**-1**	**-1**	**1**	**1**	**26.24**
14	**1**	**-1**	**1**	**1**	**27.51**
15	**-1**	**1**	**1**	**1**	**30.27**
16	**1**	**1**	**1**	**1**	**32.16**
17	**-2**	**0**	**0**	**0**	**23.85**
18	**2**	**0**	**0**	**0**	**27.07**
19	**0**	**-2**	**0**	**0**	**23.55**
20	**0**	**2**	**0**	**0**	**28.56**
21	**0**	**0**	**-2**	**0**	**22.32**
22	**0**	**0**	**2**	**0**	**28.23**
23	**0**	**0**	**0**	**-2**	**21.34**
24	**0**	**0**	**0**	**2**	**27.93**
25	**0**	**0**	**0**	**0**	**26.17**
26	**0**	**0**	**0**	**0**	**26.65**
27	**0**	**0**	**0**	**0**	**26.77**
28	**0**	**0**	**0**	**0**	**26.68**
29	**0**	**0**	**0**	**0**	**25.73**
30	**0**	**0**	**0**	**0**	**26.94**
31	**0**	**0**	**0**	**0**	**26.09**

[a] Non-randomized.
[b] Coded symbols and levels of independent variables refer to Table 1.
[c] Averages of duplicated determination from different experiments.

Table 2. Central composite design and responses a

Source	Sum of squares	Degrees of freedom	Mean square	F-value	P-value	Significance
Model	169.2204	14	12.08717	65.10984	< 0.0001	significant
x_1	15.21634	1	15.21634	81.96568	< 0.0001	significant
x_2	31.763	1	31.763	171.0974	< 0.0001	significant
x_3	47.12404	1	47.12404	253.8426	< 0.0001	significant
x_4	59.5035	1	59.5035	320.5269	< 0.0001	significant
x_1x_2	0.247506	1	0.247506	1.333239	0.2652	nonsignificant
x_1x_3	0.500556	1	0.500556	2.696341	0.1201	nonsignificant
x_1x_4	0.507656	1	0.507656	2.734587	0.1177	nonsignificant
x_2x_3	3.001556	1	3.001556	16.16845	0.0010	significant
x_2x_4	2.697806	1	2.697806	14.53224	0.0015	significant
x_3x_4	2.847656	1	2.847656	15.33944	0.0012	significant
x_1^2	0.950089	1	0.950089	5.117832	0.0380	significant
x_2^2	0.032143	1	0.032143	0.173144	0.6829	nonsignificant
x_3^2	1.493398	1	1.493398	8.044472	0.0119	significant
x_4^2	4.316615	1	4.316615	23.25227	0.0002	significant
Residual	2.970285	16	0.185643			
Lack of Fit	1.810542	10	0.181054	0.936695	0.5591	nonsignificant
Pure Error	1.159743	6	0.19329			
Cor. Total	172.1907	30				

"Prob>F"<0.0500 significant; "Prob>F">0.0500 nonsignificant.

Table 3. Analysis of variance (ANOVA) of the regression parameters for the response surface model

2.6 Statistical analysis

The response surface regression (RSREG) procedure of the Design Expert (Version 7.1.3, Stat-Ease Inc., Minneapolis, Minnesota, USA) was used to fit the experimental data to the second-order polynomial equation to obtain coefficients of the Eq. (3).

$$Y = \beta_0 + \sum_{i=1}^{4} \beta_i x_i + \sum_{i=1}^{4} \beta_{ii} x_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{ij} x_i x_j \quad (3)$$

Where Y is the response variable, x_i and x_j are the coded independent variables, and β_0, β_i, β_{ii} and β_{ij} are the regression coefficients of variables for intercept, linear, quadratic and interaction regression terms, respectively. The analysis of variance (ANOVA) tables were generated, and the effect and regression coefficients of individual linear, quadratic and interaction terms were determined. The significance of each coefficient in the polynomial was tested using an F-test. The regression coefficients were used for statistical calculations to generate response surfaces and contour plots.

2.7 Verification of model

The optimal conditions of alkali treatment depended on ratio of liquid to solid, temperature, incubation time and alkali concentration, and were obtained using RSM. For verification of the model, the SDF was prepared under optimal conditions and the prepared SDF was determined. The experimental and predicted values were compared in order to determine the validity of the model.

3. Results and discussion

3.1 Comparison of the yields of SDF

Soybean residue or extruded soybean residue was treated with alkali in the same conditions (a ratio of liquid to solid 24 : 1, a temperature of 80°C, a processing time of 60min and an alkali concentration of 0.80%), the yields of SDF were compared in Table 4.

Raw materials	Yield of SDF [a] / %
Soybean residue	10.16±0.09
Extruded soybean residue	26.36±0.55

[a] Values are means±SD (n=4)

Table 4. Comparison of the yields of SDF

The results indicated that the yield of SDF prepared from extruded soybean residue was significantly higher than the yield of SDF prepared from soybean residue. The structure of dietary fiber was changed while the raw materials were extruded. Therefore, the conditions of SDF prepared from extruded soybean residue should be optimized in order to increase the yield of SDF.

3.2 Fitting the models

The study utilized RSM to develop a prediction model for optimizing the alkali treatment conditions of SDF prepared from extruded soybean residue. The experimental conditions and the corresponding response values from the experimental design are presented in Table 2. The independent and dependent variables were analyzed to obtain a regression equation that could predict the responses within the given range. The regression equation for SDF preparation (Y) is as follows:

$$\begin{aligned} Y = {} & 32.17125 + 1.76482x_1 - 0.70210x_2 - 0.23514x_3 - 32.77976x_4 + 0.012438x_1x_2 - \\ & 0.00884375x_1x_3 + 0.44531x_1x_4 + 0.0086625x_2x_3 + 0.41063x_2x_4 + 0.21094x_3x_4 - \\ & 0.045569x_1^2 - 0.00134107x_2^2 - 0.00228527x_3^2 - 9.71317x_4^2 \end{aligned} \quad (4)$$

The plot of experimental values of prepared SDF (%) versus those calculated from Eq. (4) indicated a good fit, as presented in Fig. 1.

The results of analysis of variance (ANOVA) for the CCD are shown in Table 3. For the model fitted, the coefficient of determination (R^2), which is a measure of degree of fit. Joglekar and May (1987) suggested that, for a good fit of a model, R^2 should be at least 0.80. The coefficient of determination (R^2) was 0.983. This implies that 98.3% of the variations could be explained by the fitted model. The probability (P) value of the regression model significance was less than 0.001. Therefore, the developed model could adequately represent the real relationship among the parameters chosen. A regression analysis was carried out to fit mathematical models to the experimental data aiming at an optimal region for the responses studied. Some nonsignificant terms were neglected, and the predicted model was not refitted. The predicted model can be described by the following equation in terms of coded values:

$$Y = 21.72394 + 2.54265x_1 - 0.61817x_2 - 0.45167x_3 - 22.23493x_4 + 0.0086625x_2x_3 + 0.41063x_2x_4 + 0.21094x_3x_4 - 0.044678x_1^2 - 0.0022496x_3^2 - 9.62400x_4^2 \quad (5)$$

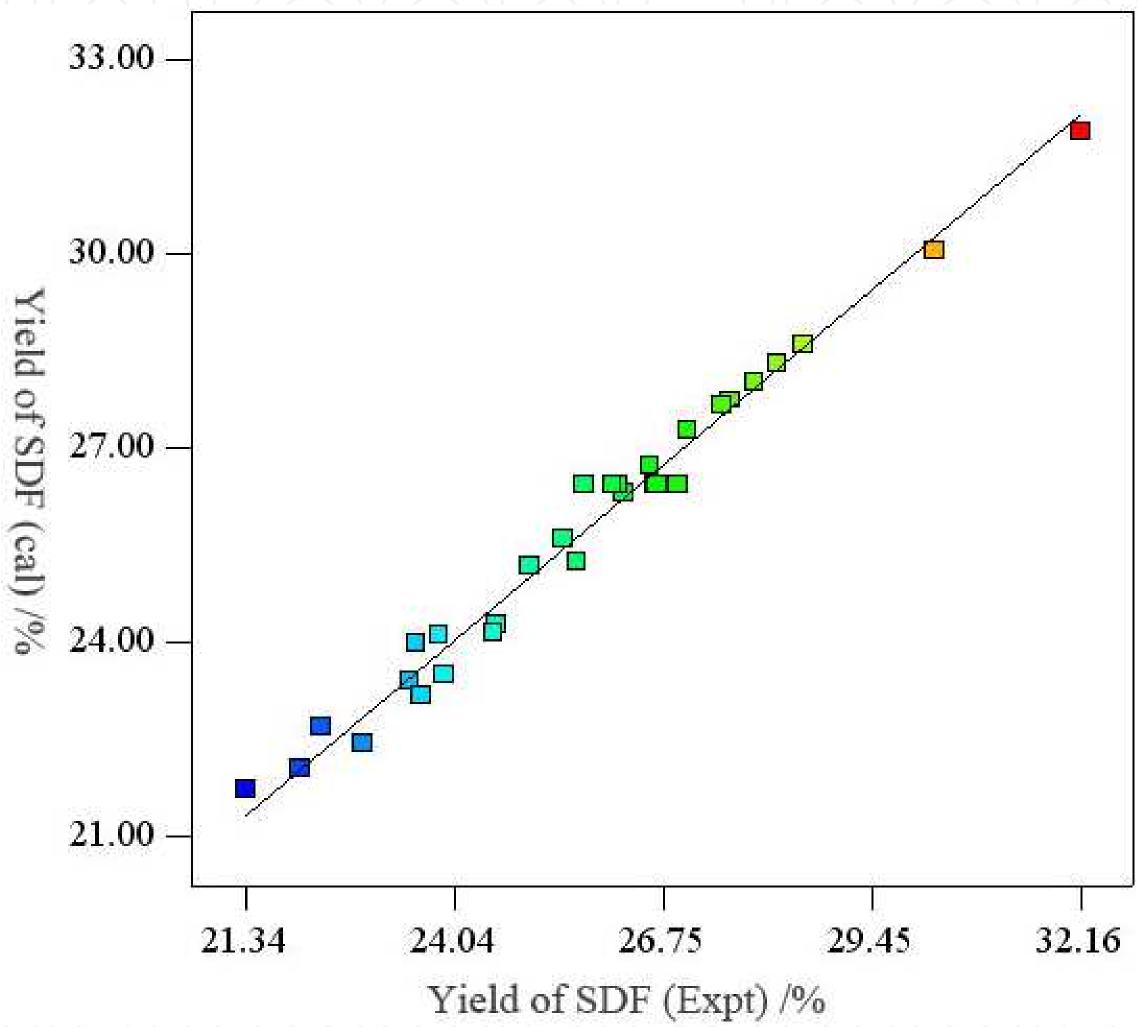

Fig. 1. Correlation of predicted SDF with experimental prepared SDF

3.3 Effects of independent variables on responses

The significance of each coefficient was determined using the F-test and p-value. The corresponding variables would be more significant if the absolute F-value becomes greater and the p-value becomes smaller (Atkinson et al., 1992). It can be seen that the variables with the largest effect were the linear terms of x_1, x_2, x_3, x_4 and the quadratic terms of x_1^2, x_3^2, x_4^2, followed by the interaction effects of x_2x_3, x_2x_4, x_3x_4. The results indicated that the effects order of the linear terms on the yield of SDF were as follows: alkali concentration (x_4), ratio of liquid to solid (x_1), temperature (x_2) and time (x_3). To aid visualization, the response surfaces and contour plots of alkali teatment conditions are shown in Figs. 2–7.

A positive relation was found between the ratio of liquid to solid and the yield of SDF (Figs. 2, 3 and 4). The yield of SDF was increased with the increase of ratio of liquid to solid, especially when the ratio of liquid to solid was within the range of 20-26. Though a further increase was shown when the ratio of liquid to solid was more than 26, it was very slight. The effect of temperature on the yield of SDF is shown in Figs. 2, 5 and 6. The effect of temperature on the yield of SDF was similar to the ratio of liquid to solid. The response surface and contour plots of the effects of temperature was presented in Figs. 2, 5 and 6. The yield of SDF was increased with the increase of temperature in this study. The yield of SDF was increased with the extension of time, especially when the time was within the range of 40-65min. Though a further improvement was shown when the time was longer than 65min, it was very slight. The effect of alkali concentration on the yield of SDF is shown in Figs. 4, 6 and 7. The yield of SDF was increased with the increase of alkali concentration, especially when the alkali concentration was within the range of 0.40-1.00%. Though a further increase was shown when the alkali concentration was more than 1.00%, it was very slight.

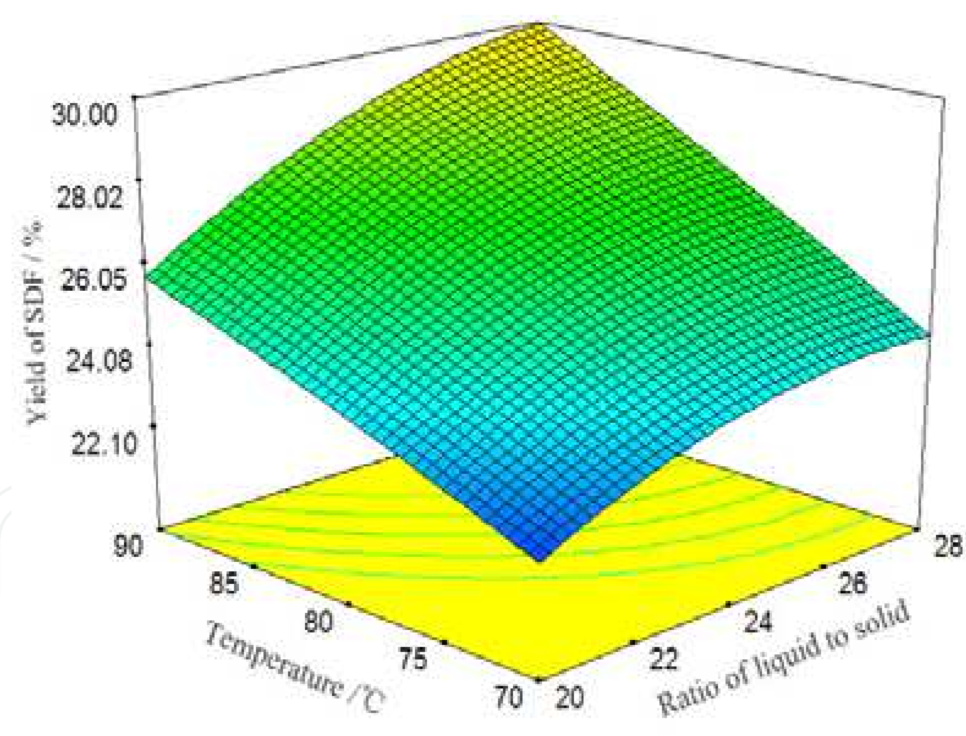

Fig. 2. Response surface for effects of ration of liquid to solid and temperature on the yield of SDF

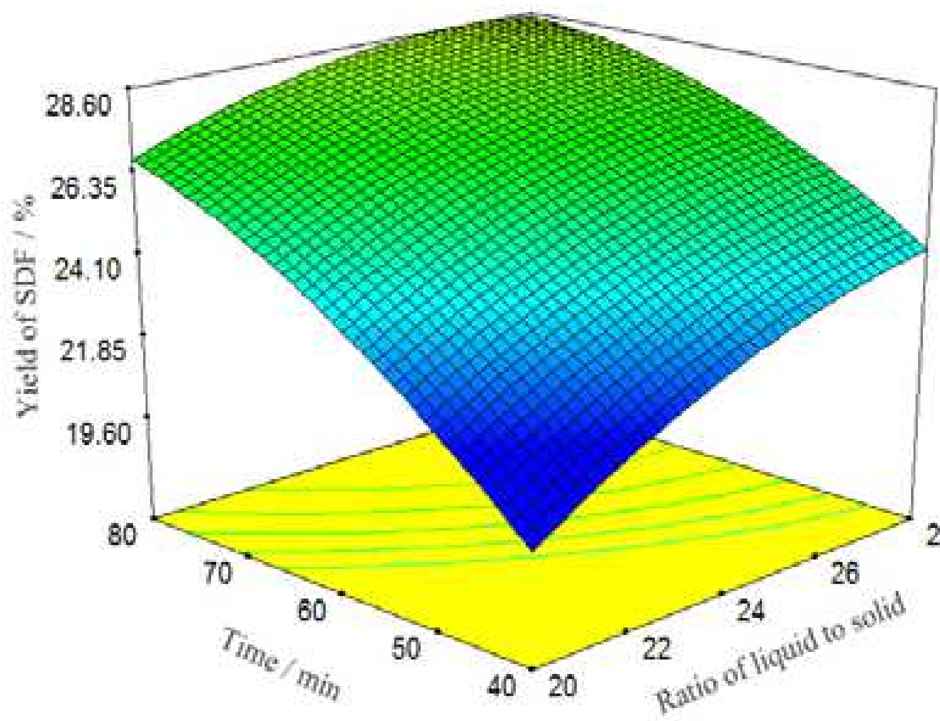

Fig. 3. Response surface for effects of ration of liquid to solid and time on the yield of SDF

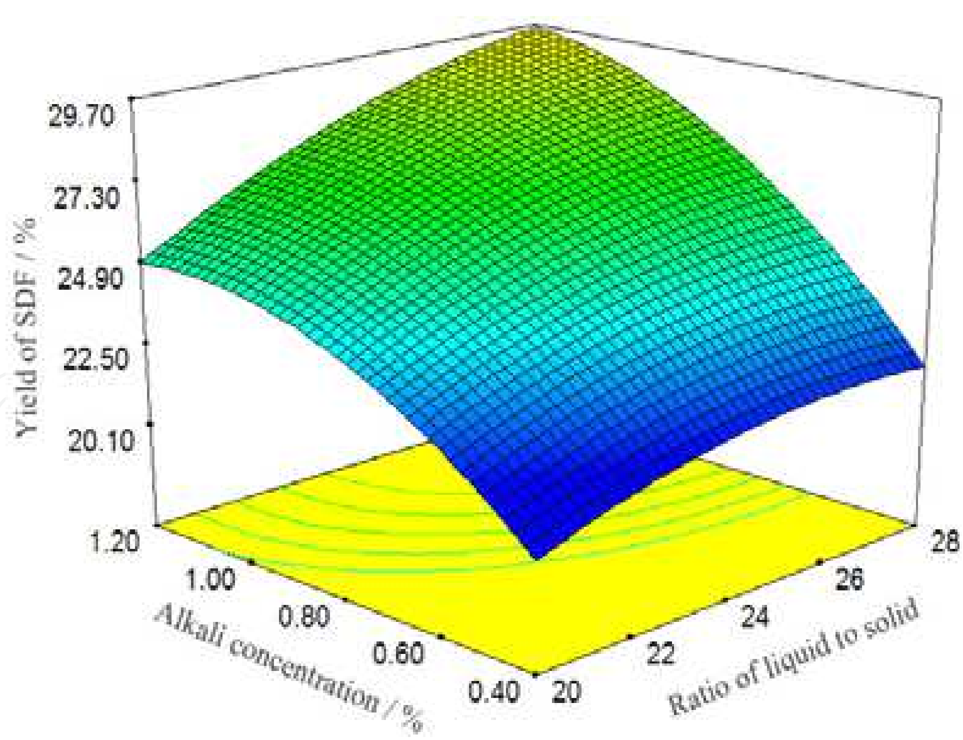

Fig. 4. Response surface for effects of ration of liquid to solid and alkali concentration on the yield of SDF

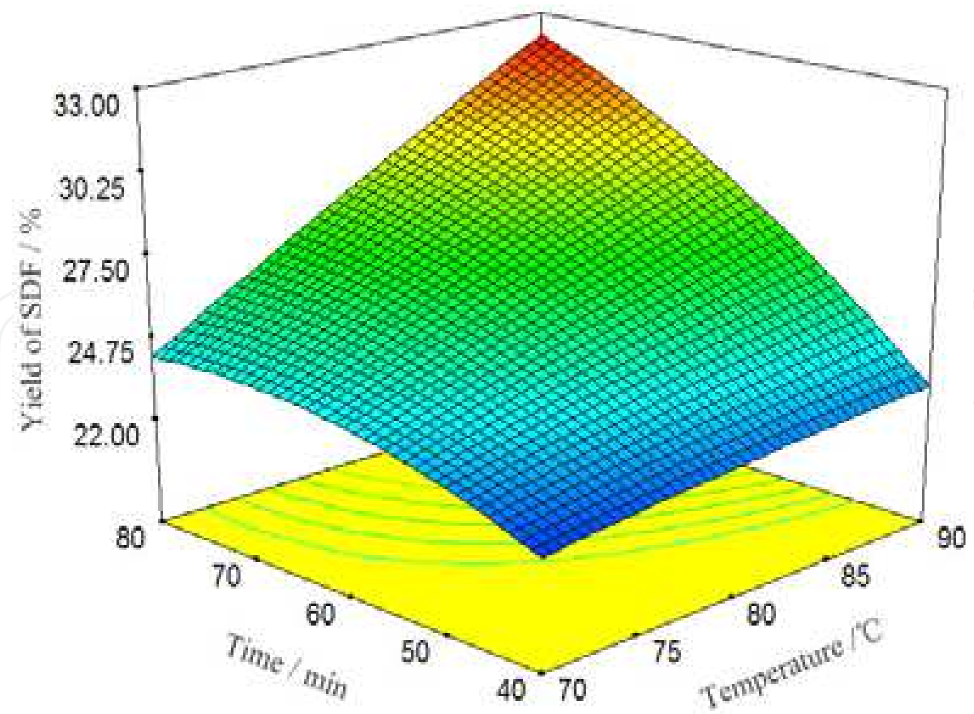

Fig. 5. Response surface for effects of temperature and time on the yield of SDF

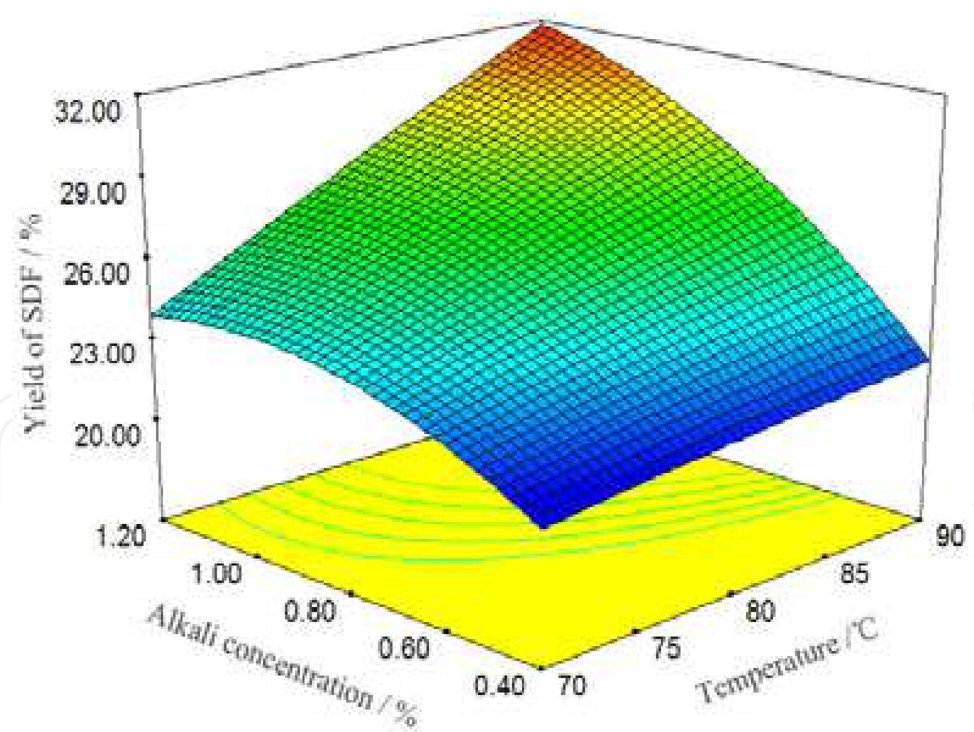

Fig. 6. Response surface for effects of temperature and alkali concentration on the yield of SDF

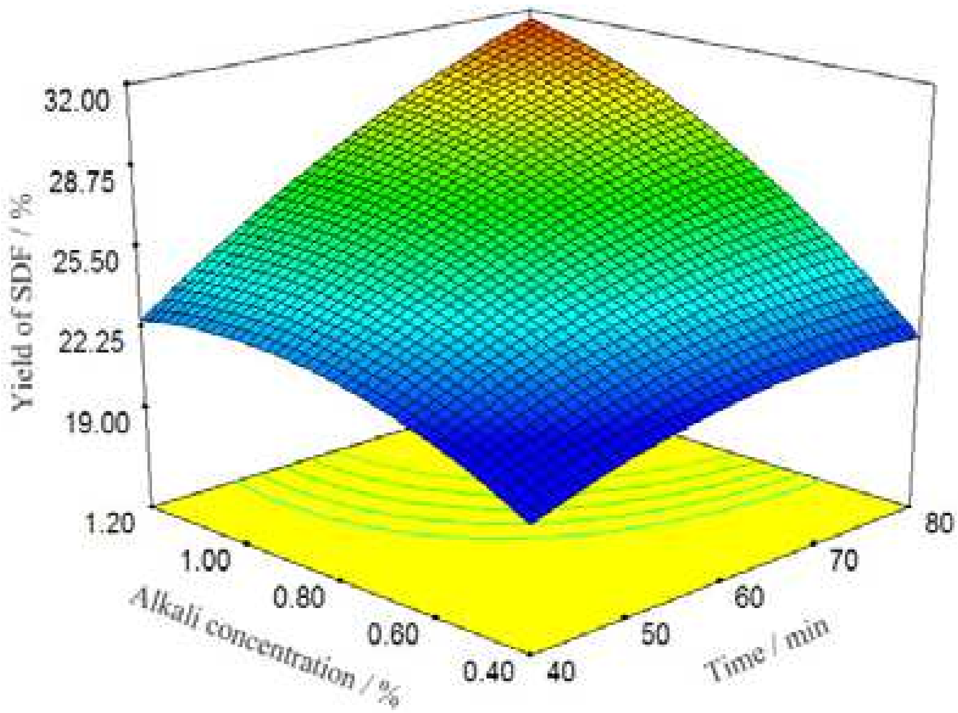

Fig. 7. Response surface for effects of time and alkali concentration on the yield of SDF

3.4 Optimization conditions and model verification

From the model, optimal conditions for alkali teatment of SDF preparation were obtained as follow: a ratio of liquid to solid 26 : 1, a temperature of 89℃, a processing time of 68min and an alkali concentration of 1.12%. Under optimal conditions, a maximum response of 33.96% SDF was predicted. The suitability of the model equation for predicting the optimum response value was tested by additional independent experiments under the optimal conditions. The results indicated that the experimental SDF value (34.12%) was not significantly different from the predicted SDF value (33.96%). The yield of SDF prepared from soybean residue was 13.51% under the optimal conditions. After alkali treatment, comparing the yield of SDF (34.12%) prepared from extruded soybean residue with the yield of SDF (13.51%) prepared from soybean residue, the yield of SDF prepared from extruded soybean residue was significantly more than the yield of SDF prepared from soybean residue under the optimal conditions.

4. Conclusions

The yield of SDF (26.36%) prepared from extruded soybean residue was higher than the yield of SDF (10.16%) prepared from soybean residue under the conditions (a ratio of liquid to solid 24 : 1, a temperature of 80°C, a processing time of 60min and an alkali concentration of 0.80%) . The effects order of the linear terms on the yield of SDF was as follows: alkali concentration, ratio of liquid to solid, temperature and time. The optimal conditions for the yield of soluble dietary fiber prepared from extruded soybean residue were: a ratio of liquid to solid 26 : 1, a temperature of 89°C, a processing time of 68min and an alkali concentration of 1.12%. Under optimal conditions, the yield of soluble dietary fiber prepared from extruded soybean residue was 34.12%, significantly higher than the yield of soluble dietary fiber prepared from soybean residue.

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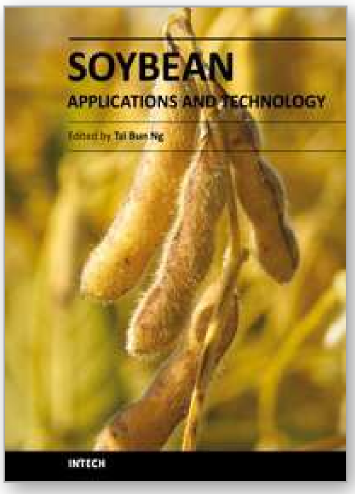

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Soybean is an agricultural crop of tremendous economic importance. Soybean and food items derived from it form dietary components of numerous people, especially those living in the Orient. The health benefits of soybean have attracted the attention of nutritionists as well as common people.