

# Optimization of Alkaline Hydrogen Peroxide conditions for preparing High Soluble Apple Pomace Dietary Fiber with response Surface Methodology

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## Article Info

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## Abstract

The optimization of alkaline hydrogen peroxide (AHP) conditions for preparing high soluble apple pomace dietary fiber (APDF) was studied using a Box-Behnken design. The effects of pH, H<sub>2</sub>O<sub>2</sub> concentration, temperature and time on the APDF yield and soluble dietary fiber (SDF) contents were systematically investigated by response surface methodology. Analysis of variance showed that a second-order polynomial equation could be used to predict the experimental data ( $R^2 > 0.94$ ). The APDF yield and SDF content were significantly affected by pH and temperature. The optimum conditions maximizing the APDF yield and SDF contents were: pH=11.30, H<sub>2</sub>O<sub>2</sub> concentration=1 g·100 mL<sup>-1</sup>, temperature=80°C and time=1 h. Under these conditions, the recorded response values for APDF yield was 760.0 g·kg<sup>-1</sup> and for SDF content was 302.0 g·kg<sup>-1</sup>, respectively. The swelling capacity and color of prepared APDF were significantly improved.

**Keywords:** Optimization; Apple pomace; Soluble dietary fiber; Alkaline hydrogen peroxide; Response surface methodology.

## Introduction

Apple pomace (AP) is one of the main by-products generated during apple juice processing. A vast amount of AP (~1.8 million tons) is produced every year in China. However, most of the AP is either used as animal feed and fertilizer or discarded as industry waste that is not fully utilized and poses potential environmental hazards [1,2]. AP contains abundant nutritional substance, such as pectin, poly phenol and minerals [3]. In particular, AP is an excellent source of dietary fiber, constituting over 60% of the dry AP [4].

Dietary fiber is generally classified as soluble dietary fiber (SDF) and insoluble dietary fiber (IDF). SDF appears to be superior to IDF in improving physiological outcomes and technological performance [5]. Evidence suggests that SDF has the capability to increase viscosity, improve gut health, reduce the glycemic response and plasma cholesterol, and prevent cardiovascular disease and cancer [6,7]. Additionally, SDF has improved technological applications over IDF for gel formation and emulsification in food industry. However, the content of SDF in AP is very low. Thus, to improve the usefulness of AP, it is essential to produce high soluble apple pomace dietary fiber (APDF).

Several physical and enzyme approaches, such as twin-screw extrusion technology, micronization technology and xylanase hydrolysis, have been applied to improve the solubility of dietary fiber in different agricultural by-products [8-11]. However, these techniques are expensive and require special equipment. Alkaline hydrogen peroxide

(AHP) treatment is a simple and low cost method, which has been used to prepare dietary fiber powders from cereal and fruit by-products [12,13]. It exhibits better effect on removing lignin and improving the physical properties of the byproducts compared to other methods [12]. Moreover, hydrogen peroxide has been removed completely by degrading into oxygen and water during the treatment [14]. The solubility of hemicellulose in cell wall has been promoted by AHP treatment [15]. However, little research has been done to increase the SDF content of AP by AHP treatment.

The purpose of this study was to optimize the preparation conditions of APDF by AHP treatment to reach the maximum yield of APDF. The independent variables include inlet pH, H<sub>2</sub>O<sub>2</sub> concentration, treatment temperature, and treatment time. The APDF yield and SDF content, which are important parameters of APDF and can be affected significantly by the independent variables, were chosen as evaluation parameters. The chemical composition and physicochemical properties of prepared APDF by AHP treatment were also evaluated.

## Materials and Methods

### Materials

Dried AP (Gala, Fuji, and Starking apples were mixed) was obtained from China Haisheng Fresh Fruit Juice Co. Ltd. The samples were crushed into a powder using a sample mill (Tecator Cyclotec 1093, FOSS, Sweden) and passed through a 60-mesh sieve. The 30% hydrogen peroxide, sodium hydroxide (NaOH), hydrochloric acid (HCl), ethanol and acetone were obtained from Sinopharm Chemical Reagent Beijing Co., Ltd (Beijing, China).

### APDF preparation by AHP treatment

AHP solution was prepared as described previously by the method of Pourfarzad et al. [5]. Different concentrations of hydrogen peroxide solutions were prepared and the pH was adjusted with 4 mol.L<sup>-1</sup> NaOH. Then, AP was treated with AHP solution at a ratio of 1:20 (w/v, g.mL<sup>-1</sup>). The sample was then incubated in a water bath at a certain temperature for a period of time. After neutralization with 6 mol. L<sup>-1</sup> HCl, 95% ethanol was added to the solution to precipitate APDF for 2 h, where the ratio of ethanol volume to sample volume was 4:1. The precipitation was collected with a filter, and dried in an air-oven at 60°C for 12 h. The APDF was then crushed into powder and passed through a 60-mesh sieve.

### Determination of APDF yield

2 g of AP was added to 40 mL of AHP solution. The APDF was prepared according to the above stated method. The APDF yield was calculated using the equation:

$$\text{APDF yield (g kg}^{-1}\text{)} = \frac{W_2}{W_1} \times 1000 \quad (1)$$

Where W<sub>1</sub> is AP mass and W<sub>2</sub> is APDF mass.

### Determination of SDF

0.5 g of APDF sample was suspended in 20 mL of water and incubated for 2 h at 60°C. To separate the SDF and IDF, the suspension liquid was then centrifuged using a high speed refrigerated bench-top centrifuge (Neofuge 15R, Heal Force,

Hong Kong, China) for 15 min at 9000 rpm. To obtain SDF, the supernatant was precipitated with four volumes of 95% ethanol for 2 h, and filtered through a filtering crucible (G<sub>2</sub>). The SDF residue was then washed twice with 15 mL 78% ethanol, 95% ethanol and acetone. The SDF was dried, weighed and the SDF content was calculated as weight of SDF residue minus the protein and ash, using the following equation:

$$\text{SDF (g kg}^{-1}\text{)} = \frac{R_1 + R_2 + R_3 - P - A}{S_1 + S_2 + S_3} \times 1000 \quad (2)$$

Where R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub> are SDF residue masses; S<sub>1</sub>, S<sub>2</sub> and S<sub>3</sub> are APDF masses; P is protein mass and A is ash mass.

### Experimental design and statistical analysis

The pH (X<sub>1</sub>: 10.5, 11 and 12), H<sub>2</sub>O<sub>2</sub> concentration (X<sub>2</sub>: 1g 100 mL<sup>-1</sup>, 1.67 g . 100 mL<sup>-1</sup> and 2.33 g . 100 mL<sup>-1</sup>), treatment temperature (X<sub>3</sub>: 60°C, 70°C and 80°C) and treatment time (X<sub>4</sub>: 1 h, 2 h and 3 h) were selected as the independent variables. Response surface methodology (RSM) was used for modeling and analyzing of the optimization process. The experimental design employed in the experiment was a Box- Benhnken design which consisted of 24 runs and four replicates of the central point for the estimation of pure error (Table 1). The response variables were APDF yield (Y<sub>1</sub>) and SDF content (Y<sub>2</sub>). All the experiments were carried out in a random order to minimize the effect of unexplained variability in the observed responses due to systematic errors [16]. The experimental results were fit to the following second-order polynomial model:

Table 1. Design approach and experimental result of response surface methodology.

Code	X <sub>1</sub> pH	X <sub>2</sub> H <sub>2</sub> O <sub>2</sub> concentration (g·100 mL <sup>-1</sup> )	X <sub>3</sub> Treatment temperature (°C)	X <sub>4</sub> Treatment time (h)	Y <sub>1</sub> APDF yield (g·kg <sup>-1</sup> )	Y <sub>2</sub> SDF content (g·kg <sup>-1</sup> )
1	10.5 (-1)	1 (-1)	70 (0)	2 (0)	752.2±6.0	190.1±6.4
2	11.5 (1)	1 (-1)	70 (0)	2 (0)	743.5±3.6	272.5±3.7
3	10.5 (-1)	2.33 (1)	70 (0)	2 (0)	696.0±7.5	238.2±1.9
4	11.5 (1)	2.33 (1)	70 (0)	2 (0)	748.5±3.3	283.0±2.3
5	11 (0)	1.67 (0)	60 (-1)	1 (-1)	784.7±3.0	244.1±3.7
6	11 (0)	1.67 (0)	80 (1)	1 (-1)	746.8±11.0	272.6±9.5
7	11 (0)	1.67 (0)	60 (-1)	3 (1)	747.3±9.2	260.8±1.3
8	11 (0)	1.67 (0)	80 (1)	3 (1)	702.4±11.8	278.8±0.3
9	10.5 (-1)	1.67 (0)	70 (0)	1 (-1)	764.8±10.8	206.2±3.6
10	11.5 (1)	1.67 (0)	70 (0)	1 (-1)	756.7±8.5	290.6±4.5
11	10.5 (-1)	1.67 (0)	70 (0)	3 (1)	665.3±5.9	241.4±8.5
12	11.5 (1)	1.67 (0)	70 (0)	3 (1)	746.9±5.6	260.9±6.2
13	11 (0)	1 (-1)	60 (-1)	2 (0)	774.7±6.1	239.5±2.3
14	11 (0)	2.33 (1)	60 (-1)	2 (0)	774.0±3.3	260.7±1.3
15	11 (0)	1 (-1)	80 (1)	2 (0)	731.3±9.3	274.9±7.7
16	11 (0)	2.33 (1)	80 (1)	2 (0)	717.9±2.2	270.0±2.1
17	10.5 (-1)	1.67 (0)	60 (-1)	2 (0)	765.0±6.8	204.5±2.9
18	11.5 (1)	1.67 (0)	60 (-1)	2 (0)	766.5±9.0	268.0±5.7
19	10.5 (-1)	1.67 (0)	80 (1)	2 (0)	663.6±9.6	216.9±1.5
20	11.5 (1)	1.67 (0)	80 (1)	2 (0)	735.7±11.8	292.6±3.8
21	11 (0)	1 (-1)	70 (0)	1 (-1)	775.1±14.8	251.3±2.0
22	11 (0)	2.33 (1)	70 (0)	1 (-1)	753.5±8.2	268.2±7.7
23	11 (0)	1 (-1)	70 (0)	3 (1)	724.4±14.2	259.8±5.7
24	11 (0)	2.33 (1)	70 (0)	3 (1)	747.5±15.9	263.1±5.1
25	11 (0)	1.67 (0)	70 (0)	2 (0)	736.1±17.2	271.5±5.9
26	11 (0)	1.67 (0)	70 (0)	2 (0)	746.8±0.3	267.9±1.7
27	11 (0)	1.67 (0)	70 (0)	2 (0)	735.0±20.0	278.0±4.0
28	11 (0)	1.67 (0)	70 (0)	2 (0)	726.1±11.2	279.2±5.7

Mean± SD (n=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,  $\beta_0, \beta_i, \beta_{ii}$  and  $\beta_{ij}$  are the regression coefficients of variables for constant, linear, quadratic, and interaction regression terms, respectively, and  $X_i$  and  $X_j$  are the independent variables. The analysis of variance (ANOVA) was applied to validate the significance of the models, the lack of fit, and each regression coefficient. The  $R^2$  value, adjusted  $R^2$  value and coefficient of variation (C.V.) were also calculated.

### Analysis of chemical composition

The moisture, lipid, protein, ash, total dietary fiber (TDF), SDF and IDF contents in AP and prepared APDF were analyzed. To determine the contribution of vapor content, a moisture test apparatus (MA 100, Sartorius, Germany) was used. The lipid was determined gravimetrically by extraction with petroleum ether using a Soxhlet apparatus (SER 148, VelpScientifica, Italy) (AOAC method 920.39). The protein was measured using a KjeldahlAzotometer (Kjeltec 2300, FOSS, Denmark), where the factor used for converting nitrogen content to crude protein content was 6.25 (AOAC method 955.04). The ash was analyzed by igniting the sample in a muffle furnace set (Lindberg/ Blue, Thermo Fisher, America) at 550°C for 5 h (AOAC method 923.03). TDF, IDF and SDF were determined by the non-enzymatic-gravimetric method (AOAC method 993.21). The total starch content of AP had been determined to be less than 2%.

### Analysis of physicochemical properties

The physicochemical properties of APDF including water holding capacity (WHC), oil holding capacity (OHC), swelling capacity (SC) and color were measured and evaluated.

WHC and OHC were measured by the method described by Mei et al. [17] with slight modifications. 0.5 g samples were weighed and added into 50 mL centrifuge tubes. Then 30 mL of distilled water was added to each sample, and the tubes were vortexed to disperse the samples. After standing for 18 h at room temperature, the samples were centrifuged at 9000 rpm for 15 min. The supernatant was removed and the samples were weighed again. The WHC was calculated as the amount of water held by 1 g of sample. Similarly, 0.2 g samples were incubated with 1.5 g of corn oil in a 10 mL centrifuge tube. Other steps were in accordance with standard methods of determining WHC. The OHC was calculated as the amount of oil held by 1 g of sample.

SC was determined using the bed volume technique as described previously [18]. The samples were placed in graduated conical tubes, and the bottom of the tubes was gently tapped until the volume of the samples was stabilized at 0.2 mL. Approximately 10 mL of distilled water was added to each sample and mixed thoroughly. After 18 h, the swelling volume was recorded. The SC was calculated as the volume swelled by 1 g of sample.

Color analysis was done according to the method of El-Kadiri et al. [19]. using a colorimeter (Chroma Meter CR-300, Minolta Co., Japan). The CIE  $L^*a^*b^*$  color model was used for

determination of the color of AP and APDF. The three parameters of interest were:  $L^*$ , which represents the lightness of color (0=black; 100=white);  $a^*$ , which represents red ( $a^*>0$ ) and green ( $a^*<0$ ); and  $b^*$ , which represents yellow ( $b^*>0$ ) and blue ( $b^*<0$ ).

## Results and Discussion

### Construction of the model

In the present study, the effects of independent factors ( $X_1, X_2, X_3$ , and  $X_4$ ) on the responses of prepared APDF by AHP treatment were analyzed using RSM. The design and responses of each experiment were shown in table 1. Yield is an important index for evaluation of the method. The yield of the prepared APDF varied from 663.6 g.kg<sup>-1</sup> to 784.7g.kg<sup>-1</sup>, with the corresponding SDF content in the prepared APDF ranging from 190.1 g.kg<sup>-1</sup> to 290.6 g.kg<sup>-1</sup>. To examine the effects of the conditions on the APDF yield and SDF content, a second-order polynomial model (4) and (5) were obtained by multiple regression analyses as follows:

$$Y_1 = 736.0 + 15.9X_1 - 5.3X_2 - 26.2X_3 - 20.6X_4 + 15.3X_1X_2 + 17.7X_1X_3 + 22.4X_1X_4 - 3.2X_2X_3 + 11.2X_2X_4 - 1.7X_3X_4 - 8.4X_1^2 + 8.3X_2^2 + 4.7X_3^2 + 5.4X_4^2 \quad (4)$$

$$Y_2 = 274.1 + 30.9X_1 + 7.9X_2 + 10.7X_3 + 2.7X_4 - 9.4X_1X_2 + 3.1X_1X_3 - 16.2X_1X_4 - 6.5X_2X_3 - 3.4X_2X_4 - 2.6X_3X_4 - 21.0X_1^2 - 7.7X_2^2 - 6.2X_3^2 - 4.4X_4^2 \quad (5)$$

Where  $X_1, X_2, X_3$  and  $X_4$  are the pH, H<sub>2</sub>O<sub>2</sub> concentration (g.100 mL<sup>-1</sup>), treatment temperature (°C), and treatment time (h), respectively, while  $Y_1$  is the yield of APDF (g.kg<sup>-1</sup>), and  $Y_2$  is the content of SDF (g.kg<sup>-1</sup>).

### Analysis of variance (ANOVA)

ANOVA was used to analyze the significance and accuracy of the quadratic models. From table 2, the ANOVA results showed that the obtained second-degree polynomial model (4) was extremely significant ( $P<0.0001$ ) for APDF yield with a satisfactory determination coefficient ( $R^2=0.9423$ ), suggesting that this model can accurately explain the relationship between the independent variables and the response. The adjusted determination coefficient (Adj  $R^2=0.8802$ ) was also high, indicating the high accuracy of this model. Meanwhile, the lack of fit ( $P=0.3748>0.05$ ) of this model was not significant. The value of the coefficient of variation (C.V.) was 1.40%, less than 5%, indicating that the quadratic polynomial model was reproducible. Thus, the model was accurate for predicting the APDF yield by AHP treatment. The  $P$ -values were used to reflect whether a regression coefficient is significant. The smaller the  $P$ -value, the more significant the coefficient is [9]. The regression coefficient analysis indicated that the linear coefficients ( $X_1, X_3, X_4$ ) were very significant ( $P<0.01$ ). Treatment temperature had the most significant effect on APDF yield, followed by treatment time and pH. The interaction terms  $X_1X_3$  and  $X_1X_4$  were extremely significant ( $P<0.01$ ), and the interaction term  $X_1X_2$  was significant ( $P<0.05$ ). However, the H<sub>2</sub>O<sub>2</sub> concentration, quadratic terms and other terms had no significant effects on APDF yield ( $P>0.05$ ).

**Table 2.** Analysis of variance (ANOVA) for APDF yield of alkaline hydrogen peroxide treatment

Source	Sum of Squares	Degree of freedom	Mean Square	F-Value	P-value	Significant
Model	22884.2	14	1634.6	15.18	< 0.0001	**
X <sub>1</sub>	3035.4	1	3035.4	28.18	0.0001	**
X <sub>2</sub>	338.1	1	338.1	3.14	0.0999	NS
X <sub>3</sub>	8232.9	1	8232.9	76.44	< 0.0001	**
X <sub>4</sub>	5108.8	1	5108.8	47.43	< 0.0001	**
X <sub>1</sub> X <sub>2</sub>	934.9	1	934.9	8.68	0.0114	*
X <sub>1</sub> X <sub>3</sub>	1247.8	1	1247.8	11.58	0.0047	**
X <sub>1</sub> X <sub>4</sub>	2011.9	1	2011.9	18.68	0.0008	**
X <sub>2</sub> X <sub>3</sub>	40.5	1	40.5	0.38	0.5502	NS
X <sub>2</sub> X <sub>4</sub>	499.4	1	499.4	4.64	0.0506	NS
X <sub>3</sub> X <sub>4</sub>	12.1	1	12.1	0.11	0.7428	NS
X <sub>1</sub> <sup>2</sup>	428.0	1	428.0	3.97	0.0676	NS
X <sub>2</sub> <sup>2</sup>	414.9	1	414.9	3.85	0.0715	NS
X <sub>3</sub> <sup>2</sup>	133.2	1	133.2	1.24	0.2862	NS
X <sub>4</sub> <sup>2</sup>	176.5	1	176.5	1.64	0.2229	NS
Residual	1400.2	13	107.7			
Lack of Fit	1184.4	10	118.4	1.65	0.3748	NS
Pure Error	215.8	3	71.9			
Cor Total	24284.4	27				
R <sup>2</sup>	0.9423					
Adj R <sup>2</sup>	0.8802					
C.V.%	1.40					

NS: no significant effect at level < 0.05; \*P < 0.05; \*\* P < 0.01

X<sub>1</sub>: pH; X<sub>2</sub>: H<sub>2</sub>O<sub>2</sub> concentration; X<sub>3</sub>: treatment temperature; X<sub>4</sub>: treatment time

C.V: coefficient of variation

The ANOVA results for the SDF content of prepared APDF are shown in table 3. The regression model (5) was highly significant (P < 0.0001), and the lack of fit (P = 0.3331 > 0.05) was not significant. The R<sup>2</sup> value of the model indicated that 96.69% of the behavior of the SDF content could be explained by this model. The Adj.R<sup>2</sup> was 0.9313 and the C.V. was 2.68%, both confirming that the model could be used to accurately predict the SDF content of prepared APDF by AHP treatment.

**Table 3.** Analysis of variance (ANOVA) for SDF content of alkaline hydrogen peroxide treatment.

Source	Sum of Squares	Df	Mean Square	F Value	p-value	significant
Model	18026.9	14	1287.6	27.15	< 0.0001	**
X <sub>1</sub>	11431.0	1	11431.0	241.03	< 0.0001	**
X <sub>2</sub>	754.0	1	754.0	15.90	0.0015	**
X <sub>3</sub>	1368.2	1	1368.2	28.85	0.0001	**
X <sub>4</sub>	84.3	1	84.3	1.78	0.2053	NS
X <sub>1</sub> X <sub>2</sub>	352.5	1	352.5	7.43	0.0173	*
X <sub>1</sub> X <sub>3</sub>	37.3	1	37.3	0.79	0.3914	NS
X <sub>1</sub> X <sub>4</sub>	1055.5	1	1055.5	22.26	0.0004	**
X <sub>2</sub> X <sub>3</sub>	171.3	1	171.3	3.61	0.0797	NS
X <sub>2</sub> X <sub>4</sub>	46.9	1	46.9	0.99	0.3381	NS
X <sub>3</sub> X <sub>4</sub>	27.5	1	27.5	0.58	0.4597	NS
X <sub>1</sub> <sup>2</sup>	2644.4	1	2644.4	55.76	< 0.0001	**
X <sub>2</sub> <sup>2</sup>	355.7	1	355.7	7.50	0.0169	*
X <sub>3</sub> <sup>2</sup>	227.2	1	227.2	4.79	0.0475	*
X <sub>4</sub> <sup>2</sup>	114.1	1	114.1	2.41	0.1449	NS
Residual	616.5	13	47.4			
Lack of Fit	530.9	10	53.1	1.86	0.3331	NS
Pure Error	85.7	3	28.6			
Cor Total	18643.5	27				
R <sup>2</sup>	0.9669					
Adj R <sup>2</sup>	0.9313					
C.V.%	2.68					

NS: no significant effect at level <0.05; \* P < 0.05; \*\* P < 0.01

X<sub>1</sub>: pH; X<sub>2</sub>: H<sub>2</sub>O<sub>2</sub> concentration; X<sub>3</sub>: treatment temperature; X<sub>4</sub>: treatment time

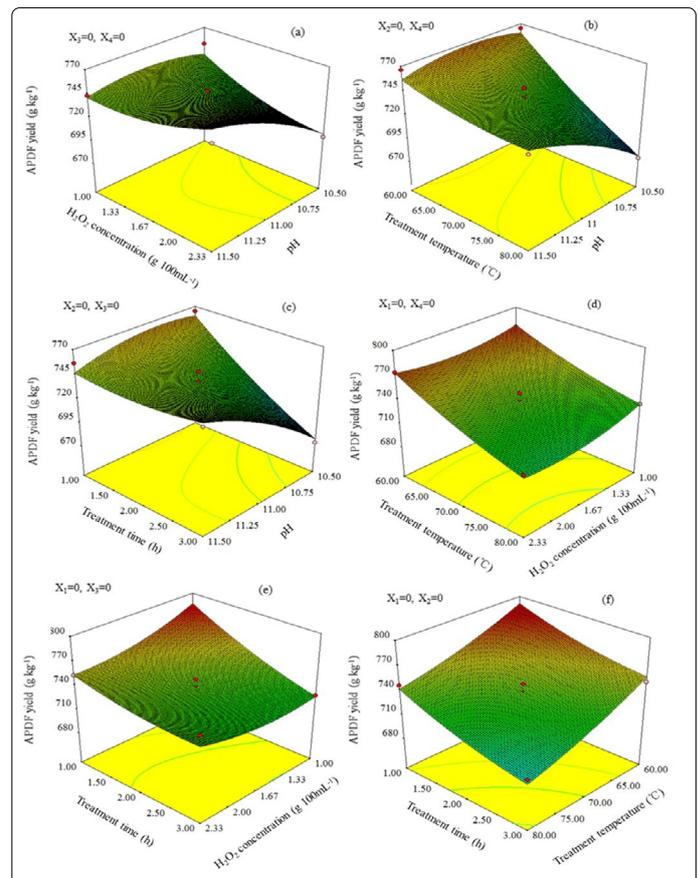
C.V: coefficient of variation

Table 3 also showed that coefficients significantly affected the SDF content. The model revealed that pH was the most significant factor affecting the SDF content, followed by treatment temperature and H<sub>2</sub>O<sub>2</sub> concentration. The treatment time was not significantly related to the SDF content. The

interaction term X<sub>1</sub>X<sub>4</sub> and the quadratic term X<sub>1</sub><sup>2</sup> were extremely significant (P < 0.01), and the X<sub>1</sub>X<sub>2</sub>, X<sub>2</sub><sup>2</sup> and X<sub>3</sub><sup>2</sup> were also significant (P < 0.05). However, the other terms were not significant (P > 0.05).

**Effect of interaction on APDF yield**

According to the results of the ANOVA shown in Table 2, the interactions of X<sub>1</sub>X<sub>2</sub>, X<sub>1</sub>X<sub>3</sub>, and X<sub>1</sub>X<sub>4</sub> were all significant (P < 0.05). Previous study showed that the best way of visually expressing the effect of variables on the responses within the experimental space was to generate response surface plots of the model [16]. The response surfaces for the effects of variables on the APDF yield are shown in figure 1. The APDF yield decreased with the increase of H<sub>2</sub>O<sub>2</sub> concentration, treatment temperature and treatment time at pH of 10.5 (Figure 1a). This reduction was attributed to the changes of the reaction conditions resulting in the decomposition of the dry matter [20]. When the H<sub>2</sub>O<sub>2</sub> concentration, pH, treatment temperature, treatment time were at higher levels, the APDF yield increased with the increase in pH (Figures 1a-1c). That is probably because the increases in pH would increase the salt content in the AHP system, offsetting the dry matter decomposition and increasing the yield. Interestingly, no significant effects of H<sub>2</sub>O<sub>2</sub> concentration, treatment temperature and treatment time on the APDF yield were observed at pH of 11.5.



**Figure 1.** Response surface showing the effects of preparation variables of alkaline hydrogen peroxide on apple pomace dietary fibre (APDF) yield. (a) pH and H<sub>2</sub>O<sub>2</sub> concentration, (b) pH and treatment temperature, (c) pH and treatment time, (d) H<sub>2</sub>O<sub>2</sub> concentration and treatment temperature, (e) H<sub>2</sub>O<sub>2</sub> concentration and treatment time, (f) treatment temperature and treatment time. X<sub>1</sub>: pH; X<sub>2</sub>: H<sub>2</sub>O<sub>2</sub> concentration (g·100mL<sup>-1</sup>); X<sub>3</sub>: treatment temperature (°C); X<sub>4</sub>: treatment time (h).

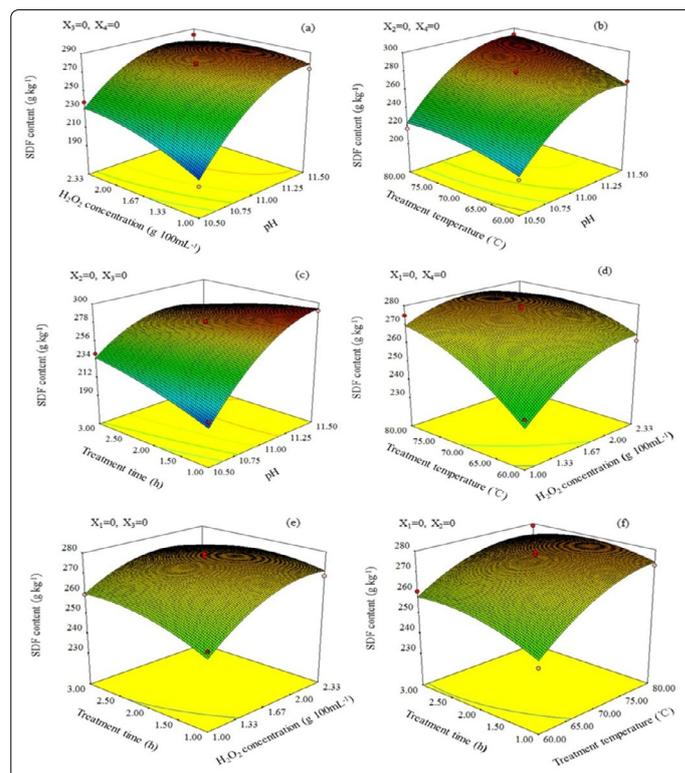
The interactions of  $X_2X_3$ ,  $X_2X_4$ , and  $X_3X_4$  are shown in figures 1d-1f at pH of 11. In each of the response surface plots, the maximum yield of APDF was obtained when both of the variables were at -1. This is related to the weakest reaction conditions. The results further confirmed that APDF yield decreased with the gradual increase of  $H_2O_2$  concentration, treatment temperature and treatment time, and no significant interactions were observed between them. The optimum condition for the maximum yield of APDF was found to be at a pH of 10.83, a  $H_2O_2$  concentration of 1.23 g·100 mL<sup>-1</sup>, a treatment temperature of 60.16°C and a treatment time of 1.04 h. Under this condition, the APDF yield was 811.0 g·kg<sup>-1</sup>.

**Effect of interaction on SDF content**

As shown in figure 2a, the content of SDF increased with the increase of pH and  $H_2O_2$  concentration. When the PH remained constant at 10.5, the content of SDF increased from 190.1g·kg<sup>-1</sup> to 272.5g·kg<sup>-1</sup> when  $H_2O_2$  concentration increased from 1% to 2.33%. Li et al [21]. showed that the solubilization of hemicelluloses increased with increase in  $H_2O_2$  concentration in alkali-treated rice straw. The improvement of SDF content with increasing  $H_2O_2$  concentration may be attributed to the generation of reactive hydroxyl radicals such as ·OH and  $O_2^-$  [22]. The hydroxyl radicals can attack the molecular chain, thus destruct the structure of cellulose, resulting in the degradation of hemicellulose. When the  $H_2O_2$  concentration remained constant at 1 g·100 mL<sup>-1</sup>, the content of SDF increased about 43.34% at the pH ranged from 10.5 (190.1 g·kg<sup>-1</sup>) to 11.5 (272.5 g·kg<sup>-1</sup>). However, the SDF content declined after the pH and  $H_2O_2$  concentration increased to a certain level. This is likely related to the increased levels of free radicals, which accelerated the breakage of polysaccharides' glucosidic bonds to degrade into low molecular weight sugars.

Figure 2b shows that the content of SDF increased with the increase in treatment temperature, similar to the results of Doner and Hicks [23]. Who reported that increased temperatures contributed to the extraction of hemicellulose B by AHP. These results indicated that higher temperatures may accelerate the decomposition of  $H_2O_2$  to further increase the solubility and diffusion of SDF. However, the effect of treatment temperature on the content of SDF was less significant than that of pH. The content of SDF in the prepared APDF increased with extended treatment time at lower level of pH (Figure 2c). However, the content of SDF at pH of 11.5 decreased with the increase of treatment time (Figure 2d). The content of SDF increased with the increase of  $H_2O_2$  concentration when the treatment temperature (Figure 2d) and time (Figure 2e) were at lower levels. However, the content of SDF increased, and then decreased with the treatment temperature (Figure 2d) and time (Figure 2e) when  $H_2O_2$  concentration was at higher level. The effect of treatment temperature on the content of SDF was more significant than that of treatment time (Figure 2f). These result indicated that a stronger reaction condition had negative influence on the increase of SDF content in APDF. In the present study, the optimum condition for the maximum of SDF content was found as: pH of 10.51,  $H_2O_2$  concentration of 1.19 g·100 mL<sup>-1</sup>,

treatment temperature of 69.48°C and treatment time of 1.13 h. Under these conditions, the SDF content was 185.14 g·kg<sup>-1</sup>.



**Figure 2.** Response surface showing the effects of preparation variables of alkaline hydrogen peroxide on soluble dietary fibre (SDF) content. (a) pH and  $H_2O_2$  concentration, (b) pH and treatment temperature, (c) pH and treatment time, (d)  $H_2O_2$  concentration and treatment temperature, (e)  $H_2O_2$  concentration and treatment time, (f) treatment temperature and treatment time.  $X_1$ : pH;  $X_2$ :  $H_2O_2$  concentration (g·100mL);  $X_3$ : treatment temperature (°C);  $X_4$ : treatment time (h).

**Optimization of conditions and verification of results**

pH,  $H_2O_2$  concentration, treatment temperature and treatment time were optimized simultaneously through a desirability function which would satisfy all the responses with requirements to obtain optimum preparation conditions. The ultimate aim was to obtain the highest APDF yield and SDF content. In the present study, the predicted optimal conditions were obtained at pH of 11.34,  $H_2O_2$  concentration of 1 g·100 mL<sup>-1</sup>, treatment temperature of 79.96°C and treatment time of 1h. An experiment was carried out to confirm the adequacy of the models at PH of 11.30,  $H_2O_2$  concentration of 1g·100 mL<sup>-1</sup>, treatment temperature of 80°C and treatment time of 1h. The results showed that the experimental values of APDF yield and SDF content were  $760.0 \pm 3.3$  g·kg<sup>-1</sup> and  $302.0 \pm 7.2$  g·kg<sup>-1</sup>, respectively, while the predicted values of them were 764.8 g·kg<sup>-1</sup> and 289.7 g·kg<sup>-1</sup>, respectively. Although the APDF yield slightly decreased, using multi-objective optimization approach compared to that of single objective optimization, yet SDF content improved about 56.5%. In the present study, the optimal value of pH is 11.3, close to the value reported by Gould [22], who showed that pH 11.5 was the optimum pH for the dissociation reaction of  $H_2O_2$ . It was indicated that the optimum pH in our study for preparation APDF had the lowest residual of  $H_2O_2$ .

The experimental values for multi-objective optimization were in perfect agreement with the predicted values with less than 1% error, indicating that the RSM model was valid and adequate for describing the preparation process. Hence, the optimum conditions for preparation of high soluble APDF by AHP treatment were as follows: pH was 11.30, H<sub>2</sub>O<sub>2</sub> concentration was 1 g·100 mL<sup>-1</sup>, treatment temperature was 80°C and treatment time was 1 h. Under these conditions, the response values were APDF yield of 760.0 ± 3.3 g·kg<sup>-1</sup>, and SDF content of 302.0 ± 7.2 g·kg<sup>-1</sup>, respectively.

### The composition and physicochemical properties of prepared APDF

The main chemical composition and physicochemical properties of APDF are shown in table 4. The amount of moisture in prepared APDF decreased about 44.5% while the amount of ash increased about 373.2% compared to those in AP. The increase in ash may be due to the addition of sodium hydroxide, which confirmed the relation between APDF yield and pH. No significant changes were observed in protein content. The contents of TDF and SDF in prepared APDF by AHP treatment were 1.18 and 9.15 folds higher than those in AP, respectively. The increase in SDF content by AHP treatment is significantly higher than that by extrusion modification, microwave-assisted acid extraction and xylanase enzyme treatment [24,25], suggesting that the method of AHP treatment is effective for preparing APDF with a high content of SDF. The content of IDF decreased with the increase of SDF in prepared APDF, indicating that a part of IDF transformed into SDF during the AHP treatment.

The swelling capacity (SC) of AP has been improved by 94.4% in the prepared APDF. The increase in SC is likely related to the disruption of hydrogen bonding between cellulose chains by free radicals, and the exposed hydrophilic groups become easier to contact molecules of water [26]. The water holding capacity (WHC) and oil holding capacity (OHC) of the prepared APDF were slight higher than those of AP, consistent with the results of Sangnark and Noomhorm who found that AHP treatment improved WHC and OHC of rice straw [27]. The structure of APDF may be disrupted by AHP treatment, which increased the SDF content, and consequently reduced the porosity and capillary attraction of the fiber, thus the physical entrapment of water and oil was also reduced. The brightness L\* value of AP was improved from 68.08 to 70.16, while the a\* value and b\* value were reduced. It was indicated that the color of prepared APDF was bleached by AHP treatment compared to AP, which increased the application scope of bleached APDF in the food industry.

**Table 4.** Chemical composition and physicochemical properties of AP and APDF

	Moisture (g·kg <sup>-1</sup> )	Ash (g·kg <sup>-1</sup> )	Protein (g·kg <sup>-1</sup> )	Fat (g·kg <sup>-1</sup> )	TDF (g·kg <sup>-1</sup> )	IDF (g·kg <sup>-1</sup> )	SDF (g·kg <sup>-1</sup> )	WHC (g·g <sup>-1</sup> )	OHC (g·g <sup>-1</sup> )	SC (mL·g <sup>-1</sup> )	L*	a*	b*
AP	73.7±3.1 <sup>a</sup>	20.5±1.1 <sup>b</sup>	85.2±1.2 <sup>a</sup>	52.1±0.4 <sup>a</sup>	640.8±10.0 <sup>b</sup>	617.0±3.2 <sup>a</sup>	33.0±0.5 <sup>b</sup>	5.67±0.05 <sup>a</sup>	1.05±0.02 <sup>a</sup>	4.86±0.26 <sup>b</sup>	68.08±0.01 <sup>b</sup>	2.90±0.00 <sup>a</sup>	21.14±0.01 <sup>a</sup>
APDF	40.9±0.7 <sup>b</sup>	97.0±2.4 <sup>a</sup>	82.7±1.1 <sup>a</sup>	28.8±0.2 <sup>b</sup>	757.0±10.0 <sup>a</sup>	455.0±1.3 <sup>b</sup>	302.0±7.2 <sup>a</sup>	6.85±0.20 <sup>a</sup>	1.09±0.06 <sup>a</sup>	9.45±0.26 <sup>a</sup>	70.16±0.01 <sup>a</sup>	0.21±0.01 <sup>b</sup>	18.74±0.01 <sup>b</sup>

TDF: total dietary fibre; IDF: insoluble dietary fibre; SDF: soluble dietary fibre; WHC: water holding capacity; OHC: oil holding capacity; SC: swelling capacity; L\* a\* b\*: indicator of colour

Different letters superscripts in a column show values that differ significantly (P<0.05)

Mean ± SD (n=3)

## Conclusion

RSM was successfully applied for estimating the effect of pH, H<sub>2</sub>O<sub>2</sub> concentration, treatment temperature and treatment time on the APDF yield and SDF content of AP by AHP treatment. Results showed that PH and treatment temperature had an extremely significant effect on all the responses (P<0.01), and the effect of treatment temperature was the most significant on APDF yield and pH was the most significant on SDF content among the variables. The optimum conditions maximizing the APDF yield and SDF content were found as pH of 11.30, H<sub>2</sub>O<sub>2</sub> concentration of 1 g·100 mL<sup>-1</sup>, treatment temperature of 80°C and treatment time of 1 h. Under these conditions, the response values were APDF yield of 760.0 g·kg<sup>-1</sup> and SDF content of 302.0 g·kg<sup>-1</sup>, respectively. The chemical composition and physicochemical properties were changed in the prepared APDF, especially for SDF, whose content increased about 10 folds compared to that in AP. In addition, the SC and color of AP were also improved. These results demonstrated that AHP is an effective, clean, and affordable method for preparing APDF with a high content of SDF, posing potential applications in food industry.

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