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Optimization of production yield and functional properties of pectin extracted from sugar beet pulp

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ABSTRACT

A central composite design was employed to determine the optimum extraction condition to obtain higher yield, better color attribute as well as better rheological and emulsifying properties in pectin extracted from sugar beet pulp (SBP). A second-order polynomial model was developed for predicting the yield of sugar beet pulp pectin (SBPP) based on the composite design. Response surface methodology (RSM) was used to quantify the integral effect of three processing parameters (extraction temperature, time and pH) on yield, yield stress, color attribute (tint value) and emulsifying activity index (EAI). Through the frequency analysis it was found that the optimal temperature, time and pH value of the extraction were 93.7 °C, 3 h, and 1.21, respectively. The yield, yield stress and tint value of the SBPP extracted at the optimal condition were 24.45%, above 0.1 Pa and −6.0, respectively.

1. Introduction

Pectin belongs to the family of complex heteropolysaccharides presenting in cell walls of terrestrial plants and is commonly used in food industry as a stabilizer, a thickener and a gelling agent (Axelos, Thibault, & Lefebvre, 1989; Moe, Draget, Skjåk-Bræk, & Smidsrød, 1995). Commercially available pectin is primarily extracted from by-products of juice manufacturing, including citrus peels and apple pomace (May, 1990). A number of other agricultural by-products such as cacao pod husks (Vriesmann, Teófilo, & Petkowicz, 2011), lemon by-products (Masmoudi et al., 2008), peach pomace (Faravash & Ashtiani, 2008), sunflower heads (Sahari, Akbarian, & Hamedi, 2003), durian rind (Wai, Alkarkhi, & Easa, 2009), banana peels (Qiu et al., 2010), and soy hulls (Kalapathy & Proctor, 2001) can be used for pectin extraction. However, the pectin produced from these sources does not satisfy industrial requirements in terms of yield and functional properties.

Sugar beet pulp is a low value by-product of sugar refining industry. It contains 15–30% pectin on dry weight basis and is increasingly seen as a better alternative for pectin extraction (Yapo, Robert, Etienne, Wathelet, & Paquot, 2007a). The published literature suggests that SBPP is produced in small quantity for applications requiring better surface-active and emulsifying properties, metal ion removing and biosorption characteristics (Mata, Blázquez, Ballester, González, & Muñoz, 2009). SBPP possess these properties owing to the presence of a higher amount of protein in it (Leroux, Langendorff, Schick, Vaishnav, & Mazoyer, 2003). In addition SBPP contains abundant amount of soluble dietary fiber which is valued in functional and health foods (Ma, Wang, Yang, & Yu, 2012). SBPP contains galacturonic acid, rhamnose, arabinose and galactose as main sugar constituents which determine its application (Sun, Tomkinson, Sun, & Wang, 2000).

Most of the studies suggest that the acid extraction method is widely used to extract pectin (Buchholt, Christensen, Fallesen, Ralet, & Thibault, 2004; Harel, Mignot, Sauvage, & Junter; Micard & Thibault, 1999; Yapo et al., 2007a). In this extraction method, the pH and temperature of the medium and the extraction time are usually varied in the range of 0.6–2, 70–90 °C and 1–5 h, respectively. It is expected that the extraction factors such as the nature of acid used (Levigne, Ralet, & Thibault, 2002), time, temperature and the pH used during extraction (Yapo et al., 2007a) and solid–liquid ratio (Li, Jia, Wei, & Liu, 2012) affect the efficacy of the extraction. Among these factors, the effects of extraction temperature, time and pH are stronger than others (Levigne et al., 2002; Li et al., 2012; Yapo et al., 2007a), so they are chosen to be the independent variables in the research.

In addition, a finite stress (yield stress) is required to initiate a flow in gum solutions. This is one of the most useful properties of

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Table 1
Independent variables and their coded and actual values used for the RSM-based optimization.

Independent variable	Unit	Symbol	Coded levels				
			$-\alpha^a$	−1	0	1	α^a
Extraction temperature	°C	X_1	63.1821	70	80	90	96.8179
Extraction time	h	X_2	1.3182	2	3	4	4.6818
Extraction pH	–	X_3	0.6591	1	1.5	2	2.3409

[a] Star point α = 1.6818.

food gums because it helps to keep various components of the food formulation in place (Rao & Kenny, 1975). It has also been reported that the SBPP is more frequently used in food industry as an emulsifier rather than gelling or stabilizing agent (Funami et al., 2011). Furthermore, light colored pectin can be used in greater number of food products than the dark colored pectin. To the best of our knowledge, there are only two publications (Levigne et al., 2002; Li et al., 2012) which deal with optimization aspect of SBPP extraction. Even these two publications did not consider the functional properties (rheological, color and emulsifying properties) as optimization parameters. Hence, it is necessary to optimize the extraction process in order to improve the functional properties along with the production yield.

The main objectives of this work were to quantify the effects of extraction temperature, time and pH on the yield, color, rheological and emulsifying properties of SBPP. A second-order polynomial model was developed to predict the yield of SBPP as a function of extraction conditions (temperature, time and pH) using response surface methodology (RSM). We have also investigated the relationships between the variables (temperature, time and pH) and their responses by means of response surface analysis. The optimal condition for the extraction was obtained by considering both the yield and the functional properties of SBPP.

2. Materials and methods

2.1. Materials and chemicals

Sugar beet pulp (SBP) was obtained from COFCO Tunhe Co., Ltd. (Xinjiang, China). This SBP was dried and pulverized into powder to go through 60-mesh sieve using a high speed multi-function mill (Bingdu Electrical Appliances Co., Ltd., Shanghai, China) and stored at ambient temperature. Food grade corn oil was used to prepare emulsions and it was purchased from local supermarket (Jinlongyu, Qinhuangdao, China). Analytical grade anhydrous ethanol and hydrochloric acid (HCl) were purchased from Beijing Beihua Chemical Company (Beijing, China). Analytical grade 25% (w/w) ammonia solution ($NH_3{\cdot}H_2O$) was provided by Tianjin Tianda Chemical Company (Tianjin, China).

2.2. Experimental design

Response surface methodology (RSM) was used to determine the optimum condition and the integral effects of three independent variables: extraction temperature (Z_1), extraction time (Z_2) and extraction pH (Z_3) on the acid extraction of SBPP. A central composite design consisting of 20 experimental runs was used. This design included six replicates (trial 15–20) at the center point. The level of factors and their coding are presented in Table 1. The design matrix and the responses are presented in Tables 2 and 3, respectively. The experiments were performed at all design points in randomized order.

2.3. Pectin extraction

The extraction of pectin from the dried sugar beet pulp (SBP) with 3.33% of moisture content was carried out according to the experimental design shown in Table 2. The suspension samples were prepared in a 3000 ml beaker and the solid–liquid ratio was maintained at 1:20 (w/v). These suspension samples were gently stirred at 200 rpm with a digital mechanical agitator (RW 20; IKA, Staufen, Germany). The pH of the suspensions was adjusted to the required value with 12 M HCL. The temperature of suspensions was regulated using a temperature controlled water bath. After the reaction was completed, the resulting slurries were cooled down, adjusted to pH 4.5 with 25% (w/w) $NH_3{\cdot}H_2O$ and filtered using a Buchner funnel under vacuum condition. The supernatant was subsequently recovered and its volume was measured. The pectin was precipitated and recovered from the supernatant using two volumes of 100% ethanol for 1 h at ambient temperature, then hand-squeezed in a nylon cloth to remove the ethanol. Finally, the sugar beet pulp pectin (SBPP) was washed twice with 100% ethanol, hand-squeezed in a nylon cloth to remove the remaining ethanol and dried in a hot air oven at 50 °C for 5 h.

2.4. Pectin yield

The yield of SBPP was calculated using Eq. (1):

$$\text{Yield of SBPP}(\%) = \frac{m_0}{m} \times 100\% \quad (1)$$

where m_0 (g) is the weight of dried SBPP, m (g) is the weight of dried SBP powder.

2.5. Functional properties

2.5.1. Rheological properties

For rheological tests, the SBPP samples extracted under different conditions were dissolved in deionized water (pH 6.4–7.0) using a magnetic stirrer for 1 h at 25 °C. The concentration of SBPP was maintained at 2% (w/w) in all the solutions used for rheological tests.

Rheological measurements were carried out using a strain-controlled rheometer (AR2000ex; TA Instruments Ltd., New Castle, DE, USA). The temperature of the samples was maintained at 25 °C during these tests using a water bath connected to a Peltier system (±0.1 °C). The apparent viscosity was measured over a shear rate range of 100–0.01 s^{-1} using a cone-and-plate geometry (40 mm diameter, 1° angle, 27 μm gap). A thin layer of silicone oil was applied on the surface of the samples in order to prevent evaporation. Before each measurement, the sample was allowed to settle down (to reach equilibration) for which 2 min long equilibrium time was applied.

Herschel–Bulkley model (Wang, Li, Wang, & Adhikari, 2011) given by Eq. (2) was used to evaluate the flow behavior of SBPP solutions,

$$\tau = \tau_0 + k\gamma^n \quad (2)$$

where τ is the shear stress (Pa), τ_0 is the yield stress (Pa), k is the consistency index (Pa·s^n), γ is the shear rate (s^{-1}) and n is the flow behavior index (dimensionless).

Table 2
The arrangement of the central composite design.

Trial	Coded variables			Actual variables		
	X_1	X_2	X_3	Z_1 (°C)	Z_2 (h)	Z_3
1	1	1	1	90	4	2
2	1	1	−1	90	4	1
3	1	−1	1	90	2	2
4	1	−1	−1	90	2	1
5	−1	1	1	70	4	2
6	−1	1	−1	70	4	1
7	−1	−1	1	70	2	2
8	−1	−1	−1	70	2	1
9	−1.6818	0	0	63.1821	3	1.5
10	1.6818	0	0	96.8179	3	1.5
11	0	−1.6818	0	80	1.3182	1.5
12	0	1.6818	0	80	4.6818	1.5
13	0	0	−1.6818	80	3	0.6591
14	0	0	1.6818	80	3	2.3409
15	0	0	0	80	3	1.5
16	0	0	0	80	3	1.5
17	0	0	0	80	3	1.5
18	0	0	0	80	3	1.5
19	0	0	0	80	3	1.5
20	0	0	0	80	3	1.5

2.5.2. Emulsifying properties

Emulsions with oil volume fraction (ϕ) of 0.056 were prepared by mixing the 100 ml of 2% (w/w) SBPP solution and 5 g corn oil (density of corn oil was 0.84 g/cm^3). Primary emulsions were produced using a high-speed emulsifier (Ultra-Turrax T 25; IKA, Staufen, Germany) at 12,000 rpm for 3 min. Subsequently, the final emulsions were prepared by passing these primary emulsions through a homogenizer (AH-100D, ATS Engineering Inc., Shanghai, China) at 300 bar pressure in three passes.

The emulsion activity of SBPP can be expressed in terms of turbidity T (Pearce & Kinsella, 1978). The emulsions were serially diluted to 900 dilution (30 dilution carried out twice serially) with 0.1% (w/v) sodium dodecyl sulphate (SDS). The absorbance was determined immediately after emulsification using an ultraviolet spectrophotometer (TU-1810; Beijing Purkinje General Instrument Co., Ltd., Beijing, China) at 500 nm with 0.1% (w/v) SDS solution as a blank. The turbidity T of emulsions was calculated using Eq. (3).

$$T = \frac{2.303AV}{I} \tag{3}$$

where T is turbidity of emulsions (1 m^{-1}), V is the dilution factor (dimensionless), A is the absorbance at 500 nm (dimensionless), and I is path length which is 0.01 m.

The emulsion activity index (EAI) was calculated using Eq. (4) (Pearce & Kinsella, 1978) as follows:

$$EAI = \frac{2T}{\phi c} \tag{4}$$

where ϕ is the oil volume fraction of the dispersed phase (dimensionless) and c is the concentration of SBPP in emulsion (w/w).

2.5.3. Color measurements

The color of SBPP powder was measured by a colorimeter (Hunter Lab UltraScan VIS) and expressed in terms of tint value (TV). Tint values are positive for probes with greenish tint and negative for probes with reddish tint, and the tint value is zero for a perfectly reflecting diffuser (Miljković, Purenović, Novaković, & Ranđelović, 2011).

Table 3
The responses of the parameters used in central composite design.

Trial	Responses					
	Yield	τ_0	K	n	TV	EAI
1	13.20	0.087	0.036	0.932	−7.12333	101.297
2	24.42	0.137	0.031	0.829	−6.09667	112.856
3	11.08	0.081	0.038	0.935	−7.64667	94.674
4	22.74	0.113	0.030	0.908	−4.87333	103.375
5	9.84	0.054	0.051	0.930	−8.90333	95.973
6	16.68	0.074	0.042	0.953	−5.78333	98.960
7	8.24	0.037	0.055	0.935	−7.95000	98.960
8	18.28	0.037	0.041	0.960	−5.99333	100.258
9	10.10	0.032	0.054	0.945	−7.75000	83.765
10	24.96	0.149	0.027	0.880	−4.66333	102.726
11	12.76	0.051	0.045	0.957	−8.68000	95.713
12	17.20	0.084	0.044	0.926	−6.47667	106.752
13	24.86	0.031	0.125	0.773	−7.09333	94.674
14	6.16	0.037	0.037	0.959	−9.12667	86.492
15	14.81	0.077	0.042	0.944	−7.45000	95.973
16	14.88	0.084	0.039	0.942	−8.34667	99.739
17	14.28	0.079	0.040	0.942	−7.53667	119.089
18	14.36	0.073	0.043	0.944	−7.70333	101.557
19	14.14	0.086	0.040	0.933	−7.55000	94.674
20	14.74	0.072	0.045	0.941	−6.80333	103.895

2.6. Statistical analysis

The generalized second-order polynomial model used in the response surface analysis is given by Eq. (5) (Wang et al., 2007).

$$Y = \beta_0 + \sum_{i=1}^{4}\beta_i X_i + \sum_{i=1}^{4}\beta_{ii} X_i^2 + \sum\sum_{i<j=1}^{4}\beta_{ij} X_i X_j \quad (5)$$

where β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively, and X_i and X_j are the independent variables.

The second-order polynomial given by Eq. (5) was fitted to the experimental data to obtain the regression equations, and the analysis of data was performed using the Design Expert software (Version 8.05b, Stat-Ease Inc., Minneapolis, MN). The significant effect was separated according to analysis of variance (ANOVA) as non-significant ($p > 0.05$) or significant ($p < 0.05$). The 3D response surface plots were generated using the Matlab software (Version R2009A, The MathWorks Inc., MA, USA). The optimum conditions were obtained using a DPS data processing system (Qiyi Tang, China).

3. Results and discussion

3.1. Yield

3.1.1. Fitting the model

In order to obtain an optimal response region for the parameters studied, a regression analysis (Table 4) was performed to fit mathematical model to the experimental data. Predicted response *Y* for the yield of SBPP was found to be represented by the second-order polynomial equation (Eq. (6)) in terms of coded values.

$$\begin{aligned} Y = {} & 14.55230 + 3.17726X_1 + 0.82502X_2 - 5.21420X_3 + 0.94580X_1^2 \\ & + 0.04424X_2^2 + 0.23162X_3^2 + 0.47500X_1X_2 - 0.75000X_1X_3 \\ & + 0.45500X_2X_3 \end{aligned} \quad (6)$$

where *Y* is the yield of SBPP (%); and X_1, X_2, and X_3 are the coded variables representing extraction temperature, time and pH, respectively.

Table 4 lists the analysis of variance (ANOVA) for the fitted quadratic polynomial model representing the SBPP yield. The ANOVA shows that the quadratic regression model (Eq. (6)) is highly significant as the Fisher *F*-test (*F*, mean square regression/mean square residual = 28.91) yields a very low probability value ($p < 0.0001$). This probability value means that there is only a 0.01% chance that a “Model F-Value” of this magnitude could occur due to noise. However, the “Lack of Fit F-value” of 41.47 implies the “Lack of Fit” is also significant. Therefore, the model requires further analysis.

The goodness of fit of the model is further checked by the coefficient of determination (R^2) which was found to be 0.963. This R^2 value indicates that only 3.7% of the total variation is not explained by the model. The value of the adjusted coefficient of determination (adjusted $R^2 = 0.930$) is also high which indicates the appropriateness of the model in predicting the experimental data. The low coefficient of variation (CV = 9.36%) among the replicate experimental data indicates that the repeatability or precision of the experimental data was very good. The “adequate precision” is an index of the signal to noise ratio and the value 18.975 indicates a good fit for the model. From the above analysis, it is concluded that the model represented by Eq. (6) can be confidently used to navigate the design space.

3.1.2. Analysis of response surfaces

The 3D response surfaces provide a method to visualize the relationship between responses and independent variables. In this work, 3D response surfaces were obtained by keeping one of the variables constant at zero level while varying the other two variables.

The response surfaces showing the effect of extraction temperature (Z_1) and extraction time (Z_2) on the yield of SBPP as represented by Eq. (6) are shown in Fig. 1a. The effect of extraction pH on the yield has been assumed to be zero in this instance. As can be seen from this figure, the yield of SBPP mainly depends on the extraction temperature. This figure further shows that the increase in extraction yield is quadratic as a function of temperature for all the extraction times tested corresponding to Eq. (6). However, the yield of SBPP is linearly related to the extraction time as the quadratic term was found to be non-significant ($p > 0.05$). The linear relationship between extraction yield and extraction time holds at all the temperature tested. These results agree with the reported relationships between yield and temperature, yield and time presented in the case of SBPP (Yapo et al., 2007a).

Fig. 1b presents the effects of two variables, namely extraction temperature (Z_1) and extraction pH (Z_3) on the yield of SBPP according to Eq. (6). The effect of extraction time is set at zero level in this instance. As shown in Fig. 1b, both the extraction temperature and pH have positive impact on the SBPP production. The extraction temperature exerts a quadratic effect on the yield response; hence, the SBPP yield increases quadratically with the increase in the extraction temperature within a range of 60–100 °C. As can be seen from Fig. 1b, the figure shows a linear increase on the yield response as the pH decreases. These observations are in agreement with the earlier report where it was reported that the yield of SBPP was higher at higher temperature and at lower pH (Masmoudi et al., 2008). Both the higher degree of hydrolysis and lower degree of depolymerization of protopectin under the above stated conditions contribute to this behavior of the newly liberated pectin (Vriesmann et al., 2011). The temperature range tested in this study does not lead to the thermal degradation of the pectin (Vriesmann et al., 2011). Similar results were reported by Pagán and Ibarz (1999) who found that the yield of pectin extracted in the hot dilute acid solution from fresh peach pomace increased with the increase of temperature and acidity.

Fig. 1c illustrates the effects of extraction time (Z_2) and pH (Z_3) on the yield of SBPP calculated by Eq. (6) at fixed extraction temperature of 80 °C. As can be seen from this figure, the SBPP yield has increased linearly with time while it has decreased linearly with increase in pH, but the SBPP yield has increased quite sharply with the decrease in pH. The highest yield of SBPP is obtained when the extraction time is at its longest and pH is at its lowest within the time and pH range tested. According to El-Nawawi and Shehata (1987), the acidic condition (low pH) facilitates the hydrolysis of insoluble pectin constituents into soluble pectin which increases the yield of pectin. The effect of time on the pectin yield is in accordance with the earlier research reported by Li et al. (2012).

3.2. Functional properties

3.2.1. Rheological properties

Fig. 2 presents the experimental shear viscosity data along with the best fit Herschel–Bulkley (H-B) curves. It can be seen from this figure the H-B model follows the experimental data well ($R^2 > 0.99$), indicating that the H-B model can predict the flow behavior of SBPP solution well. This rheological characteristics is quite similar to xanthan gum, which is also reported to follow the behavior of H-B fluids (Szczesniak, 1986; Urlacher & Noble, 1997).

The data presented in Table 3 show that under different extraction conditions both the yield stress and the flow behavior index (*n*)

Table 4
Analysis of variance (ANOVA) for response surface quadratic model for SBPP yield.

Source	Sum of squares	Degree of freedom	Mean square	*F*-value	Probability
Regression	539.6474	9	59.96	28.91	<0.0001
Residual	20.7395	10	2.07		
Lack of fit	20.2511	5	4.05	41.47	0.0001
Pure error	0.4884	5	0.098		
Total error	560.3869	19			
Std. dev.	1.44		R^2	0.963	
Mean	15.39		Adj. R^2	0.930	
C.V. %	9.36		Pred. R^2	0.722	
Press	156.00		Adeq. precision	18.975	

are quite significantly ($p < 0.05$) affected while the consistency coefficient k is not significantly ($p > 0.05$) affected. The large variation in n values indicates that the shear thinning behavior of extracted SBPP becomes more prominent with the increase in extraction temperature and acidity.

The ease or difficulty in inducing flow in SBPP can be judged on the basis of yield stress values of its solution because below this stress flow does not occur (Wei, Wang, & Wu, 2001). The yield stress values presented in Table 3 suggest that the SBPP solution has a coherent network structure which requires a certain amount of force to initiate the flow. In addition, Urlacher and Noble (1997) reported that yield stress in xanthan gum solution resulted from the interaction among xanthan macromolecules which formed a weak molecular network in the solution. Hence, the different yield stress values in SBPP (Table 3) indicate to some difference in composition of these SBPP samples obtained from various extraction regimes (Peinado, Rosa, Heredia, & Andrés, 2012).

Fig. 3 illustrates the interaction relationships of extraction temperature (Z_1) and time (Z_2), extraction temperature (Z_1) and pH (Z_3), extraction time (Z_2) and pH (Z_3) on the yield stress of SBPP. It can be observed from Fig. 3 that there is a quadratic effect of extraction temperature and pH, but a linear effect of extraction time on the yield stress response. Fig. 3a shows that the yield stress increases quadratically with the increase in the extraction temperature while it only increases linearly with the increase in the extraction time. Fig. 3b shows that the yield stress of SBPP increases quite rapidly (non-linearly) with the decrease in pH and reaches to its peak value at pH 1.5. The yield stress does not increase further when the pH is reduced <1.5. Fig. 3c further suggests that the extraction time also has a positive but relatively weak effect on the yield stress. Overall,

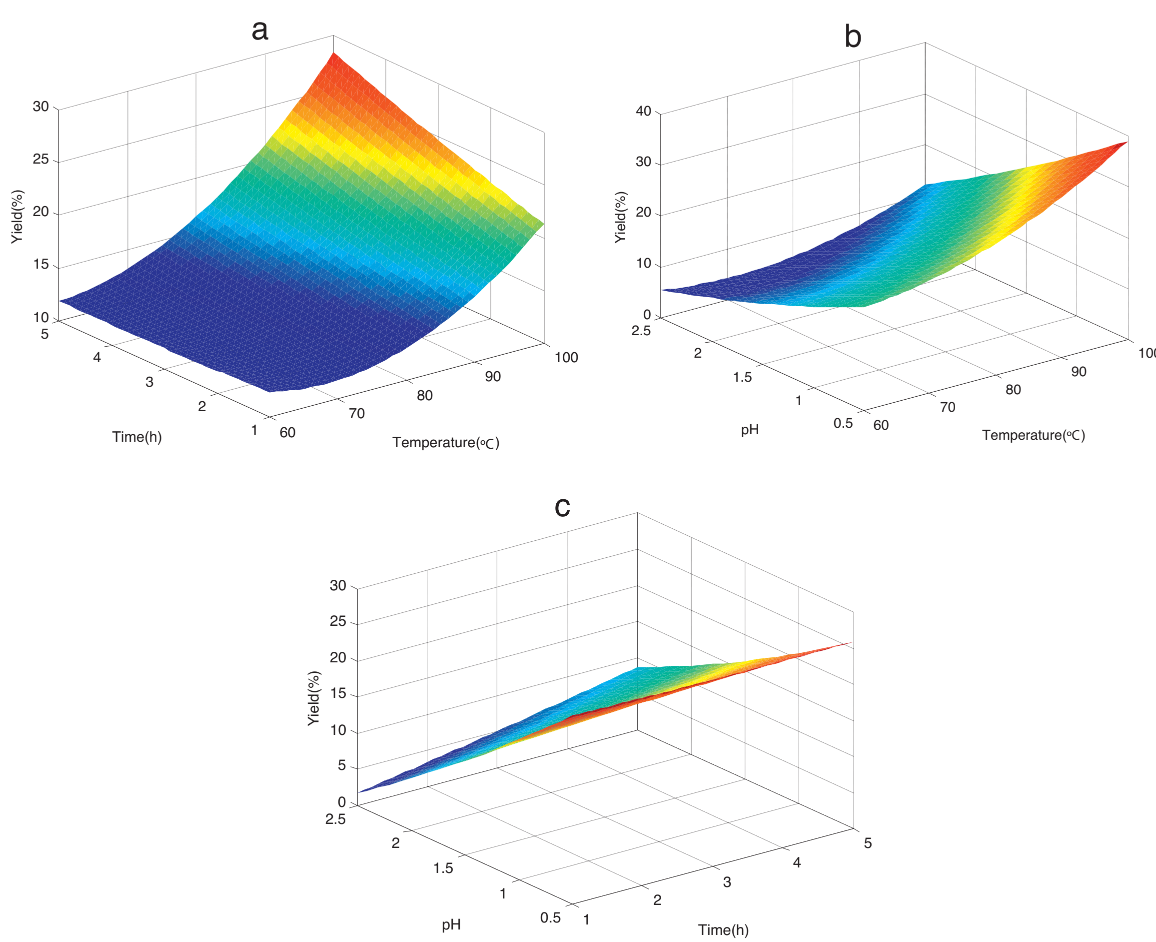

Fig. 1. Response surfaces for the yield of SBPP: (a) effect of extraction temperature and time on the yield at a constant extraction pH (1.5); (b) effect of extraction temperature and pH on the yield at a constant extraction time (3 h); and (c) effect of extraction time and pH on the yield at a constant extraction temperature (80 °C).

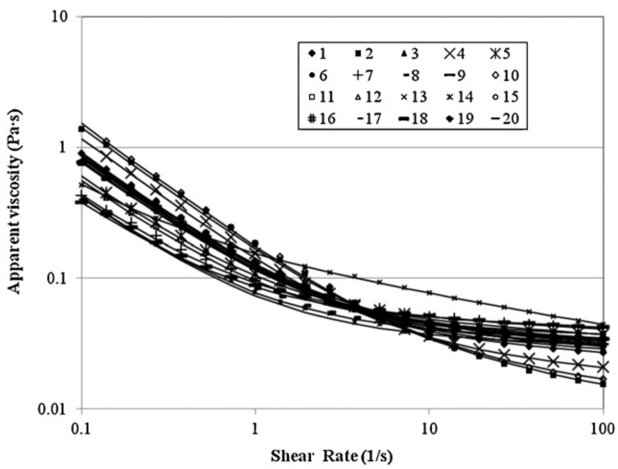

Fig. 2. Variation of apparent viscosity of SBPP at different extraction conditions. The symbols represent the experimental data and the solid lines represent the Herschel–Bulkley model. The numbers correspond to trials in the central composite design.

Fig. 3 suggests that the SBPP with high yield stress is obtained when extraction is carried out at higher temperature, pH value near 1.5 and for a longer time. According to Peinado et al. (2012) the presence of other hydrocolloids with pectin increases the yield stress of aqueous pectin solutions due to interaction among the various hydrocolloids molecules. Hence, the variation in yield stress of SBPP solutions extracted from different extraction regimes can be attributed to the simultaneous leaching of other hydrocolloids together with SBPP.

3.2.2. Emulsifying property

Table 3 shows that emulsifying property of SBPP was not significantly ($p > 0.05$) affected with the variation in the extraction conditions based on EAI values. The EAI values of the o/w emulsions stabilized by SBPP as shown in Table 3 are relatively high compared to the EAI of emulsions stabilized by other gums. For example, the EAI values of emulsions stabilized by 5% corn fiber gum (Yadav, Johnston, Hotchkiss, & Hicks, 2007) are lower than the EAI values obtained in this study.

The main reason affecting emulsifying property of SBPP is the amount of ferulic acid and protein present in it. One or both of these two functional groups can facilitate the adsorption of pectin onto the surface of the oil droplets to stabilize the emulsions. Furthermore, the molecular weight of SBPP can also significantly affect the emulsifying property by increasing the viscosity of the aqueous phase and slowing down the coalescence among the dispersed droplets (Funami et al., 2007; Leroux et al., 2003; Siew & Williams, 2008; Yapo, Wathelet, & Paquot, 2007b).

Based on the observation presented above, it can be concluded the acid-extracted pectin from sugar beet pulp (SBPP) can act as an effective emulsifier as well as a stabilizer of oil-in-water emulsions.

3.2.3. The color attributes

Fig. 4 presents the 3D response surface plots for tint value of SBPP products. Tint values of the treated samples extracted under different conditions are negative, which means that all the samples have a red hue. The interaction between extraction temperature

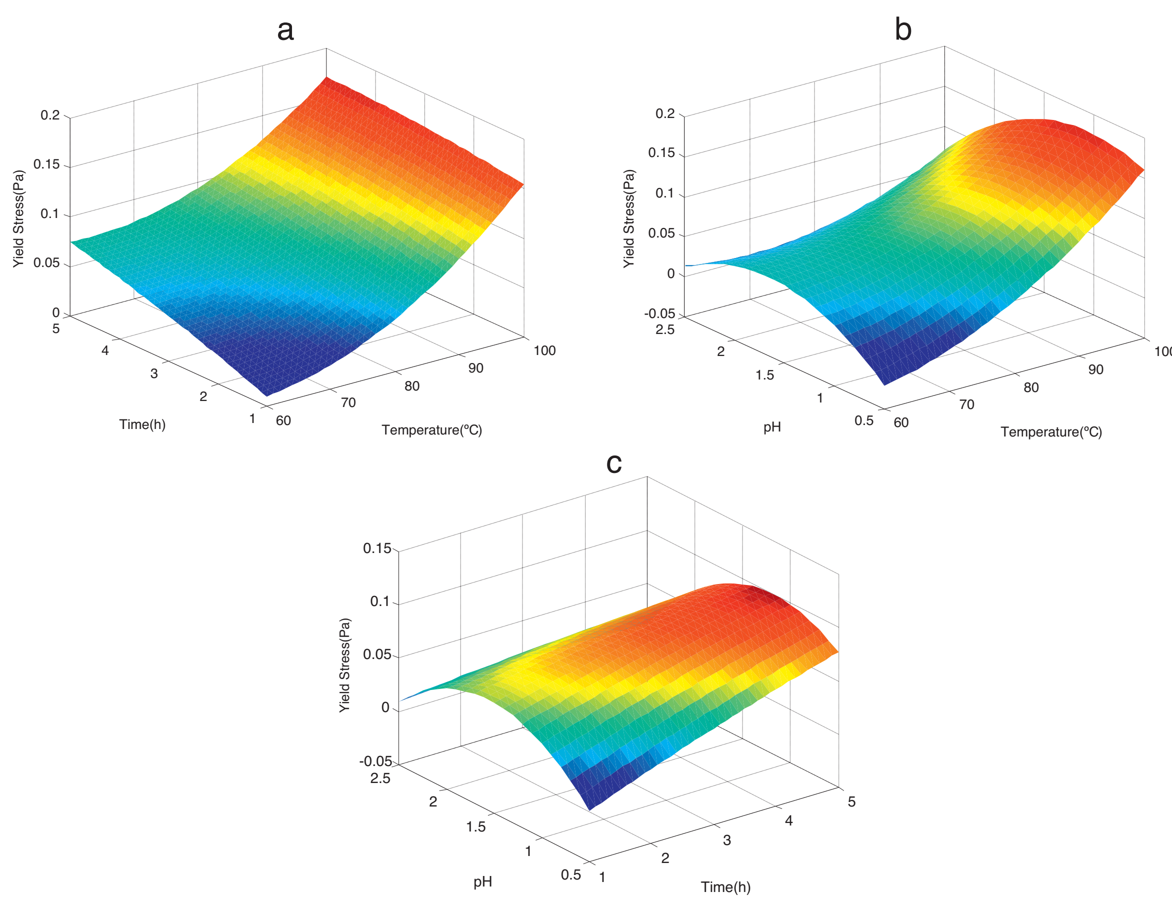

Fig. 3. Response surfaces for yield stress of SBPP: (a) effect of extraction temperature and time on yield stress at a constant extraction pH (1.5); (b) effect of extraction temperature and pH on yield stress at a constant extraction time (3 h); and (c) effect of extraction time and pH on yield stress at a constant extraction temperature (80 °C).

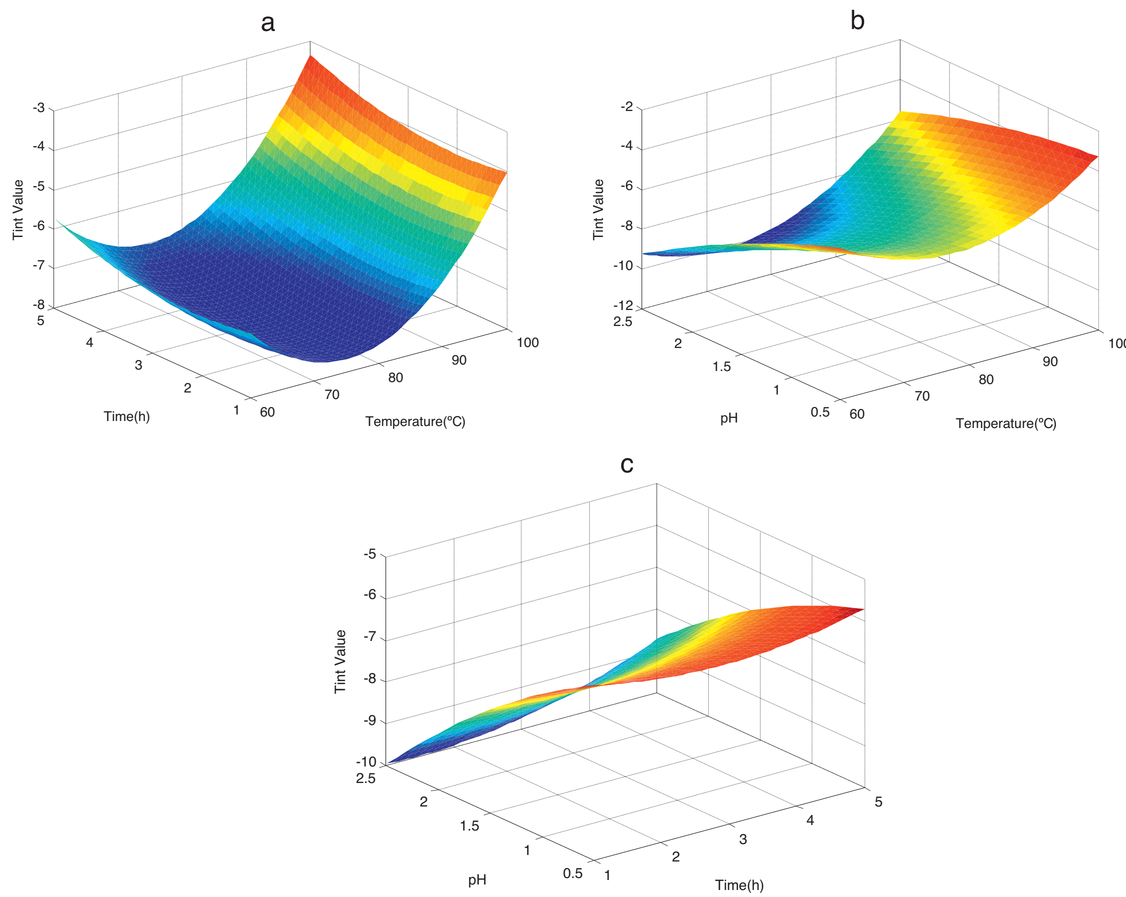

Fig. 4. Response surfaces for tint values of SBPP: (a) effect of extraction temperature and time on tint values at a constant extraction pH (1.5); (b) effect of extraction temperature and pH on tint values at a constant extraction time (3 h); and (c) effect of extraction time and pH on tint values at a constant extraction temperature (80 °C).

and time is reflected in Fig. 4a, in which the tint value decreases first and increases afterwards with the increase in extraction temperature. In other words, the lightest reddish tint is developed at the highest temperature and the red hue is also light at lower extraction temperature. Fig. 4b shows that the red hue has decreased when the pH value decreased suggesting that the redness became lighter at lower pH values. It appears that the color or hue substance present in SBPP undergo certain extent of degradation at low pH values. Fig. 4c shows an increasing trend of tint value with the decreasing pH. The effect of time in lower pH is not obvious, but the tint value increases with extending extraction time at higher pH. From the reasons argued above it appears that the product with best color attribute would be obtained at an extraction regime that combines highest extraction temperature, shortest extraction time and the lowest pH.

3.3. *Optimization of experiment*

According to the analysis of DPS software, the optimum production yield is obtained at the extraction regime that uses the extraction temperature of 96.8 °C, extraction time of 4.68 h and the extraction pH of 0.66. At this regime an extraction yield of 32.73% is achieved. It has to be noted here that the above optimum extraction regime does not take into account the functional properties of the SBPP.

Frequency analysis was used to obtain the optimum extraction condition. All the parameters except the EAI were used because the effect of extraction parameters on EAI was non-significant ($p > 0.05$). Frequency analysis indicated that the yield stress would be higher than 0.1 Pa at a probability of 95% in the extraction temperature, time and pH ranges of 93.5–95.7 °C, 2.97–3.76 h, and 1.20–1.57, respectively. Meanwhile the magnitude of the tint value might be above −6.0 at a probability of 95% in the extraction temperature, time and pH ranges of 86.3–93.8 °C, 2.59–3.41 h, and 0.91–1.23, respectively. Therefore, superimposing the response areas and balancing the influence of various factors, we found that the optimal temperature, time and pH of extraction to be 93.7 °C, 3 h, and 1.21, respectively. The yield of SBPP at this optimum extraction condition was 24.45%.

4. Conclusions

The extraction process of sugar beet pulp pectin (SBPP) was optimized using a central composite design, which consisted of 20 experimental points and 6 replicates. The second-order polynomial equation was used to predict the yield of SBPP in the design space. The optimization process aimed to obtain the SBPP with relatively higher yield, better color attribute as well as better rheological and emulsifying properties. The highest yield (32.73%) was obtained at the extraction temperature, time and pH values of 96.8 °C, 4.68 h and 0.66, respectively. The SBPP solutions exhibited Herschel–Bulkley behavior ($R^2 > 0.99$), and the highest yield stress was observed at extraction temperature, time and pH values of 96.8 °C, 4.68 h and 1.5, respectively. The emulsifying property of SBPP was not found to be significantly ($p > 0.05$) affected by the extraction conditions. And the best color was obtained at the temperature of 96.8 °C, time of 1.32 h and pH of 0.66. The optimal conditions considering the functional properties were found to be at the temperature, time and pH values of 93.7 °C, 3 h, and 1.21, respectively. At this optimum extraction condition the yield, yield stress, and tint value were found to be 24.45%, above 0.1 Pa and −6.0, respectively.

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