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Black plum peel syrup as a potential health-promoting beverage for the elderly: optimization of physicochemical properties via Response Surface Methodology (RSM)

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ABSTRACT

Background: Plum peel, the major by-product of plum processing, is a valuable source of vitamins, minerals and antioxidant activity. Using this material, a nutrient-rich syrup as a potential healthy food for older adults can be produced. This study aimed to optimize the formulation of black plum peel syrup with black plum peel puree (40–60%) and pectin (0–0.5%) using response surface methodology (RSM).

Methods: Mixture of black plum peel was prepared from plum processing factories and different formulations of black plum peel syrup were produced. Optimization of the formulation was done using RSM based on chemical composition, antioxidant activity, phenolic compounds and rheological behavior of the products.

Results: Increasing the proportion of black plum peel puree increased antioxidant activity and total phenolic compounds. Higher pectin concentration was associated with reduced acidity and increased total phenolic compounds. Increasing black plum peel puree up to 50% decreased acidity, flow behavior index, and apparent viscosity, whereas further increases (50-60%) cause increasing values of these parameters. When the pectin increased up to 0.3%, antioxidant activity and viscosity increased and reduced when pectin was increased from 0.3% to 0.4%. Overall, black plum puree had the strongest effect on physicochemical properties of syrup. The optimal formulation consisted of 40.62% black plum peel puree and 0.2% pectin.

Conclusions: Using black plum peel to produce syrup as a food with high nutritional value can be a good way to reduce environmental pollution. This product can be suitable for the older adults due to its nutritional, antioxidant activity, and useful texture and flavor.

Introduction

Aging is one of the most significant demographic trends of current century. This means that the proportion of elderly people is the fastest-growing part of the world's population. By 2050, the number of individuals aged 60 years and older is expected to reach about 2 billion, while in many countries nearly 15% of their total population will be over 80 years old. Consequently, the number of 80-years individuals worldwide is expected to exceed 400 million by 2050. Therefore, these changes highlight the growing need for food and nutrition by the older adults- especially those are very old and weak requires urgent attention. Elderly people experience increasing difficulties in chewing and swallowing oral contents due to anatomical and physiological changes, and loss of teeth resulting in malnutrition. Therefore, it is recommended that foods for patients suffering from dysphagia should have appropriate textural characteristics, including soft texture and small particle size (e.g., less than 1.5 mm). Moreover, the bite should be moist, sticky, slippery, and then easy to swallow. Texture-modified food is a term that refers to foods with a softer texture and/or reduced particle size as well as concentrated liquids (beverages) designed for the geriatric market segment with eating disorders. Texture-modified foods include soft, crushed, pureed or liquid processed foods as well as liquids that have been thickened to varying degree (1). Among the texture-modified foods, syrup can be a good option for older adults due to its nutritional values and health benefits. Syrup is a popular drink that is prepared by mixing pureed fruits, flowers and herbs, sweeteners (usually sugar), acids (mainly citric acid) and other additives such as gums (usually pectin) and aromatics to achieve the desired concentration and is usually served cold (2). Plum (*Prunus subg. Prunus*) belongs to the family *Rosaceae* is a single seed fruit rich in variety of vitamins and minerals with different sizes, colors and flavors. Due to its phenolic compounds, anthocyanins, carotenoids and pectin, plum can be used as a useful food in the human diet. The presence of bioactive compounds such as antioxidants and anthocyanins has made plums to be considered as a wonderful food (3). The presence of polyphenols and antioxidant activity in plums and stone fruits causes anti-blood pressure, anti-obesity, anti-diabetes, reducing cardiovascular diseases, reducing the growth of cancer cells and relieving thirst and heatstroke (2, 4, 5).

During the drying process of plums, a significant amount of plum peel is remained as a by-product. Because of its high moisture content, large volume, and its improper disposal of this material, may lead to environmental pollution and waste management cost. Furthermore, when used as animal feed, it may contribute to the spread of diseases, especially fungal contamination in the environment. Despite its positive effects, no application has been reported this valuable food, while this material contains valuable compounds including anthocyanins and phenolic compounds. In addition, due to the large volume of this product and its nutritional and therapeutic properties, it can be used to prepare products with high added value. Since plum peel has the nutritional properties of plum and has an attractive color, it can also be used in syrup production (2, 6, 7). Although numerous studies have investigated fruit-based syrup (4, 8-17), not much research has been focused on the use of plum peel for syrup production (2, 4), particularly intended for the elderly. Therefore, the purpose of this research is to optimize the formulation of black plum peel syrup as a new and technological food suitable for the elderly, using RSM based on chemical composition, antioxidant activity, phenolic compounds and rheological behavior of the products.

Methods

Raw materials

The raw materials used in this study included frozen black plum peel, pectin, glucose syrup, sugar and citric acid. Plum peel was obtained from the plum processing factories, Neyshabur, Iran, Pectin (Green Ribbon, Citrus, 57-62% degree esterification) was purchased from NATUREX, Switzerland), citric acid was supplied from Jovein, Sabzevar, Iran, and glucose syrup and sugar from was obtained from Neyshabour supermarket, Khorasan Razavi, Iran.

Sample preparation

After washing and removing impurities, plum peel was crushed using an industrial grinder. Then crushed material then, was stored in the freezer until further experiments. for puree preparation, the crushed peel was mixed with water and filtered to obtain a homogeneous and smooth suspension. The resulting solution was referred to plum puree. Brix and pH of black plum puree were 24 and 3.79, respectively. To prepare the syrup, water was added to the black plum peel puree at a ratio of three times its weight and was heated together with glucose syrup and sugar to reach brix 60. Subsequently, pectin and citric acid were added and heating process continued until the final product reach 70% brix and pH below 3.2. After cooking, the samples were cooled to the room temperature (22˚C±2) for 24 hours and transferred into glass containers. The formulations consisted of the black plum peel and plum puree-sugar (40-60, 50-50 and 60-40%), 20% glucose syrup, 0.7 % citric acid and pectin at three levels (0, 0.25 and 0.5%).

Chemical composition of black plum peel syrup

Chemical compositions of the black plum peel syrup samples were analyzed as pH, brix, acidity TA and moisture content (by the oven method at 70℃). Total solids were obtained based on moisture content. Soluble solid (brix) was measured using a refractometer (RHBO_80, Link, Fuzhou, China). The hydrogen potential was controlled by a pH meter (Sartorius PB_11, Göttingen, Germany) and moisture content by an oven (Fan-Azma-Gostar, Iran). Acidity was determined according to the AOAC official method (18).

Determination of antioxidant activity and phenolic compounds

Preparation of plum peel syrup extract

Approximately 5 g of each syrup sample was weighed and mixed with 20 ml of methanol-water solution (50:50) and stirred for 1 h at room temperature a magnetic stirrer. Then, the mixtures were centrifuged at 3,500 rpm for 15 min, and the supernatant was collected. Subsequently, 20 ml of acetone-water solution (30:70) was added to the remaining residue and extracted for 60 min, followed by centrifugation. Both methanolic and acetone extracts were filtered through Whatman filter paper and mixed. The obtained extract was used to determine total phenolic compounds and antioxidant activity (2).

Total phenolic compounds of black plum peel syrup

Total phenolic compounds were determined using the Folin-Ciocalteu method. Briefly, 100 µL of each prepared extract were mixed with 5 ml of distilled water. Then, 500 µL of Folin-Ciocalteu reagent and 1.5 ml of 20% sodium carbonate were added and finally the volume was adjusted to 10 ml with distilled water. This mixture was incubated for 2 h at room temperature in the dark. Absorbance was measured at 765 nm using a UV/Vis spectrophotometer (Jenway, 7315). The results were expressed based on different concentrations (10-100 PPm) of Gallic acid using a calibration curve (2, 19).

Antioxidant activity of black plum *peel syrup*

Antioxidant activity was evaluated using 2,2-diPhenyl-1-Picrylhydrazyl (DPPH) method. Six different concentrations of each syrup extract were made. One mL of DPPH ethanolic solution (0.3 mmol/L) was added to 2.5 mL of sample solution with different concentrations. In order to perform the reaction, the mixture was placed for 30 min at room temperature and darkness. Then, the absorbance of the samples against a suitable blank containing 1 ml of DPPH reagent and 2.5 ml of methanol was measured at a wavelength of 517 nm by a UV/Vis spectrophotometer. All analyses were performed in triplicate to ensure reliability (2, 19, 20).

Rheological properties of black plum peel syrup

The rheological properties of black plum peel syrup were measured using a rotational viscometer (Bohlin Instrument, Cirencester, UK) equipped with a bob-and-cup measuring system and a temperature-controlled unit (model F12-MC, Julabo Labortechnik, Seelbach, Germany). Measurements were performed at a constant temperature of 25 ± 0.5 °C, over a shear rate range of 20 to 300 s^{-1} with data collected for up to 400 s. The rheological behavior of the samples was interpreted using the power-law model [30]. All rheological tests were conducted in triplicate to ensure reliability (2, 21).

Statistical analysis

A Central Composite Rotatable Design (CCRD) was employed using Design-Expert Software (Version 11, Stat-Ease Inc., Minneapolis, Minnesota, USA) to evaluate the effects of pectin (X1: 0-0.5%) and black plum peel puree concentrations (X2: 40-60%) on the physicochemical properties of the black plum peel syrup. The experimental design consisted of five coded levels (−α, −1, 0, +1, +α), corresponding to low, medium, and high values of each factor, and generated a total of 13 experimental runs, including four factorial, four axial, and five center points to ensure rotatability and adequate estimation of experimental error. Coded levels for black plum peel puree was (40, 50 and 60%) and for pectin was (0, 0.25 and 0.5%). The experimental data were fitted to four polynomial models (linear, two-factor interaction [2FI], quadratic, and cubic). To assess model significance, Analysis of Variance (ANOVA) was performed, while model adequacy was assessed using the coefficient of determination (R^2), adjusted R^2, predicted R^2, p-values, and lack-of-fit tests. Models with significant regression ($p < 0.05$) and non-significant lack-of-fit ($p > 0.05$) were considered suitable for describing the system. The syrup formulations used in this study are presented in Table 1.

Results

The physicochemical properties of black plum peel syrup are presented in Table 2. The analytical results indicated that neither pectin and nor black plum peel purees significantly affected pH, °Brix and total solids of the black plum peel syrup ($p > 0.05$). However, both factors had a significant effect on acidity ($p < 0.01$). The measured values for pH, °Brix, total solids, and acidity ranged from 2.7 to 3.015, 70.50 – 74.50%, 45.84 – 48.94%, and 3.53–6.11%, respectively.

Table 1. black plum peel syrup formulations and experimental design based on the independent variables

No.	Pectin (%)	Black Plum Puree (%)
1	0	40
2	0.25	50
3	0.5	40
4	0.25	60
5	0	60
6	0.25	50
7	0.5	50
8	0.25	40
9	0.25	50
10	0	50
11	0.25	50
12	0.25	50
13	0.5	60

Table 2. The results of physicochemical properties of black plum peel syrup.

Properties	Mean	Std. Dev.
pH	2.88	0.07
Brix (%)	74.15	0.89
Total solids (%)	46.68	0.81
Acidity (%)	4.65	0.18
Antioxidant activity (%)	26.92	6.29
Total Phenolic compounds (mg/100 g)	0.50	0.09
K ($Pa.s^n$)	19.46	8.80
n	0.52	0.04
Apparent viscosity (Pa.s)	39.32	9.94

As presented in Table 3, the quadratic model described adequately the antioxidant capacity of the black plum peel syrup ($p < 0.05$). According to Table 4, the quadratic effects of both pectin and black plum peel were statistically significant ($p < 0.05$) and affected antioxidant capacity. As shown in Table 3, the 2FI model showed the variation in total phenolic compounds of the black plum peel syrup ($p < 0.01$). The results presented in Table 4 indicate that the linear effects of both black plum peel and pectin and their interaction, were statistically significant ($p < 0.01$).

The results of analysis showed that the addition of black plum puree and pectin did not significantly affect the consistency coefficients of the power-law model ($p > 0.05$).

Based on the optimization results, 0.2% pectin and 40.62% black plum peel puree yielded the most desirable physicochemical characteristics, with an excellent model fit ($R^2 = 0.999$).

Discussion

As shown in Table 3, the quadratic model provided the most appropriate description for the variation in acidity, with all terms showing significant statistical ($p < 0.001$). Table 4 further indicates that the linear parameter of pectin, the quadratic effects of black plum peel puree, and interaction between pectin and black plum peel puree significantly influenced on acidity ($p < 0.001$). Acidity decreased as the black plum puree concentration increased to 50% but increased at higher concentration (50 - 60%). In contrast, increasing pectin concentration resulted in a reduction in acidity of samples (Figure 1).

These results revealed a nonlinear trend in acidity in response to pectin and puree concentrations. Among the tested variables, the quadratic effect of black plum peel puree showed the strongest effect on acidity (Table 5). Egbekun et al. (22) reported moisture content, acidity, pH, and Brix of the syrup were reported 20%, 0.15%, 4.45, and 67%, respectively for syrup produced from black plum fruit. Similarly, Kunyanga et al. (23) reported pH, acidity and total solids of 3.2, 1% and 7.5%, respectively, in cactus fruit syrup. Wibowo et al. (11) observed that low-calorie sweetener in red dragon fruit had little effect on pH but significantly influenced total solids.

Table 3. Model selection for dependent variables

Variable/Model	Linear		2FI		Quadratic		Cubic		Residual	Total
	Sum of Squares	*p*-Value	Sum of Squares	*p*-Value	Sum of Squares	*p*-Value	Sum of Squares	*p*-Value	Sum of Squares	Sum of Squares
Acidity (%)	1.91	0.1370	1.36	0.0567	2.33	0.0002	0.16	0.0365	0.06	292.71
Antioxidant activity (%)	766.10	0.0457	141.92	0.2258	465.26	0.0351	108.76	0.087	181.21	10130.2
Total Phenolic compounds (mg/100 g)	0.6328	0.021	0.1481	0.0072	0.0306	0.3240	0.0785	0.001	0.0022	5.84
n	0.0092	0.6901	0.0160	0.267	0.0921	0.0004	0.0100	0.0019	0.0009	4.16
Apparent viscosity (Pa.s)	75.92	0.9031	177.29	0.5170	2817.27	0.0034	235.02	0.3540	456.34	17462.05

Table 4. Analysis of variance for determined parameters.

Variable		Model	X_1 *	X_2 **	X_1X_2	X_1^2	X_2^2	Residual	Pure Error	Cor Total
Acidity (%)	Sum of squares	5.60	1.89	0.0182	1.36	0.0012	1.95	0.2255	0.0600	5.83
	p-value	<0.0001	0.0001	0.4773	0.0003	0.8499	0.0001	-	-	-
Antioxidant activity (%)	Sum of squares	1373.28	118.80	647.31	141.92	378.57	1.43	289.97	0.0305	1663.25
	p-value	0.0138	0.1342	0.0055	0.1066	0.0193	0.8578	-	-	-
Total Phenolic compounds (mg/100 g)	Sum of squares	0.7809	0.3584	0.2744	0.1481	-	-	0.1113	0.0020	0.8922
	p-value	0.0002	0.0004	0.0011	0.0072	-	-	-	-	-
n	Sum of squares	0.1173	0.0065	0.0027	0.0160	0.0190	0.0915	0.0109	0.0006	0.1284
	p-value	0.0013	0.0811	0.2305	0.0151	0.0101	0.0001	-	-	-
Apparent viscosity (Pa.s)	Sum of squares	3070.48	71.69	4.23	177.29	2751.47	755.31	691.37	0.0479	3761.85
	p-value	0.0164	0.4224	0.8419	0.2222	0.0012	0.0279	-	-	-

* X_1: Pectin; ** X_2: Black plum peel puree

Antioxidant capacity increased when increasing the black plum puree. However, increasing the pectin up to 0.3%, resulted in antioxidant activity, decreased when it was increased from 0.3 to 0.5% (Figure 2a). According to Table 5, the linear effect of black plum peel puree had the strongest effect on antioxidant capacity, likely due to its naturally high content of phenolic and antioxidant constituents. The antioxidant activity of the black plum peel syrup ranged from 3.84 to 33.63%. Wibowo et al. (11) reported that the low-calorie sweeteners had no significant effect on the betacyanin content (as an antioxidant) of red dragon syrup. Simarmata et al. (24) observed that increasing stevia increased the betacyanin level of beetroot syrup.

Total phenolic compounds increased with increasing the black plum peel and pectin (Figure 2b). The linear effects of plum puree and pectin exhibited the greatest influence on total phenolic compounds (Table 5). The total phenolic compounds of the samples ranged between 0.3 and 1.02 mg/100 g. Ouaabou et al. (14) studied the effect of storage duration and temperature on the bioactive and compounds of cherry syrup and reported no significant difference in total phenolic, total flavonoid, total anthocyanin, and antioxidant activity of samples up to 60 days of storage at − 20 °C and 4 °C. Although, significant reduction was observed after 30 days of storage at 28 °C and 40 °C from. As shown in Table 3, the quadratic model best described the variation in apparent flow behavior index of the black plum peel syrup ($p < 0.05$). As summarized in Table 4, the quadratic effects of both pectin and black plum peel puree and their interaction were statistically significant ($p < 0.05$). The flow behavior index increased as the pectin increased.5In contrast, increasing the black plum peel puree up to 50%, leads to a decrease in flow behavior index, while increased when it was increased from 50 to 60% (Figure 3a). The quadratic term of black plum peel puree had the most effect on flow behavior index (Table 5).

As shown in Table 3, the quadratic model best described the variation in apparent viscosity of the black plum peel syrup ($p < 0.01$). According to Table 4, the quadratic effects of both pectin and black plum peel puree were statistically significant ($p < 0.05$). Apparent viscosity decreased as the black plum peel puree increased up to 50% and rose when black plum peel puree increased from 50% to 60%. Also, it increased as the pectin increased up to 0.3% and reduced when pectin was increased from 0.3% to 0.4% (Figure 3b). Among the factors studied, the quadratic term of black plum peel puree had the greatest influence on apparent viscosity (Table 5).

Table 5. Designed equation models for dependent variables.

Dependent Variable	Equation	R^2	R^2-Adjusted	CV %
Acidity (%)	$y= +4.30 - 0.5617\ X_1{*} - 0.0550\ X_2{**} - 0.5825\ X_1X_2 + 0.0212\ X_1^2 + 0.8412\ X_2^2$	0.9613	0.9336	3.82
Antioxidant capacity (%)	$y= +31.26 + 4.45\ X_1 + 10.39\ X_2 + 5.96\ X_1X_2 - 11.71\ X_1^2 - 0.7197\ X_2^2$	0.8365	0.7194	31.89
Total Phenolic compounds (mg/100 g)	$y = +0.6166 + 0.2444\ X_1 + 0.2139\ X_2 + 0.1924\ X_1X_2$	0.9194	0.8618	15.51
n	$y = +0.5111 + 0.0328\ X_1 + 0.0212\ X_2 - 0.0632\ X_1X_2 - 0.0830\ X_1^2 + 0.182\ X_2^2$	0.9148	0.8539	7.09
Apparent viscosity (Pa.s)	$y = +39.40 + 3.46\ X_1 - 0.84\ X_2 - 6.66\ X_1X_2 - 31.56\ X_1^2 + 16.54\ X_2^2$	0.8162	0.6849	30.61

* X_1: Pectin; ** X_2: Black plum peel puree.

The consistency coefficients, flow behavior index, and apparent viscosity of black plum peel syrup ranged from 1.16 to 41.78 Pa·sn, 0.41 to 0.72, and 11.09 to 72.78 Pa·s, respectively. The formulated samples exhibited pseudoplastic (shear-thinning) behavior, characterized by a decrease in viscosity with increasing shear rate. Asiz et al., (25) studied the effect of low-methoxyl pectin (LMP, DE 31%) and high-methoxyl pectin (HMP, DE 85%) in date syrups at temperature of 25 - 90 °C. Their findings demonstrated that increasing the temperature resulted in a reduction in the viscosity, whereas pectin, particularly HMP, increased the viscosity, especially in 75 °C extracted syrup. González-Montemayor et al., (26) studied the effect of temperature on the rheological properties of concentrated aguamiel syrups of two Agave species, and reported that the syrups showed a non-Newtonian behavior, with the Power Law model, which provides the best description of the behavior of the products. To confirm the accuracy of thepredictive model, the optimized values were validated experimentally, showing strong agreement between the predicted and observed results for all measured parameters (Table 6).

Table 6. Validation of optimized formulation for black plum peel syrup.

Properties	Predicted	Observed
pH	2.95	2.98
Brix (%)	73.77	73.81
Total solids (%)	46.68	46.73
Acidity (%)	5.09	5.12
Antioxidant activity (%)	20.64	20.73
Total Phenolic compounds (mg/100 g)	0.40	0.47
K (Pa.s^n)	10.61	10.72
n	0.63	0.67
Apparent viscosity (Pa.s)	51.45	51.53

Kartini et al., (17) optimized the roselle syrup formulation, which contained cinnamon, cloves, and cardamom, xanthan gum and Tween 60 as stabilizing agent were used. Among six formulations prepared, the sample containing 1% xanthan gum and 0.1% Tween 60 showed the most desirable characteristicsincluding a pH of 2.73, viscosity of 403.3 cP, total anthocyanin content of 67.85 mg/L, and DPPH free radical inhibition of 50.43% and 64.05% at concentrations of 1% v/v and 2% v/v, respectively.

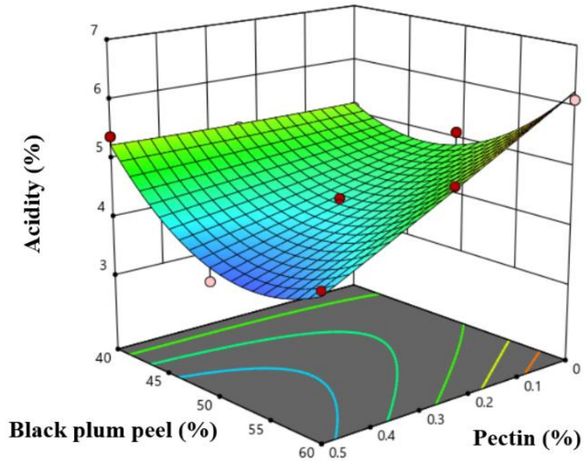

Figure 1. The effect of black plum peel puree and pectin concentration on the acidity of black plum peel syrup.

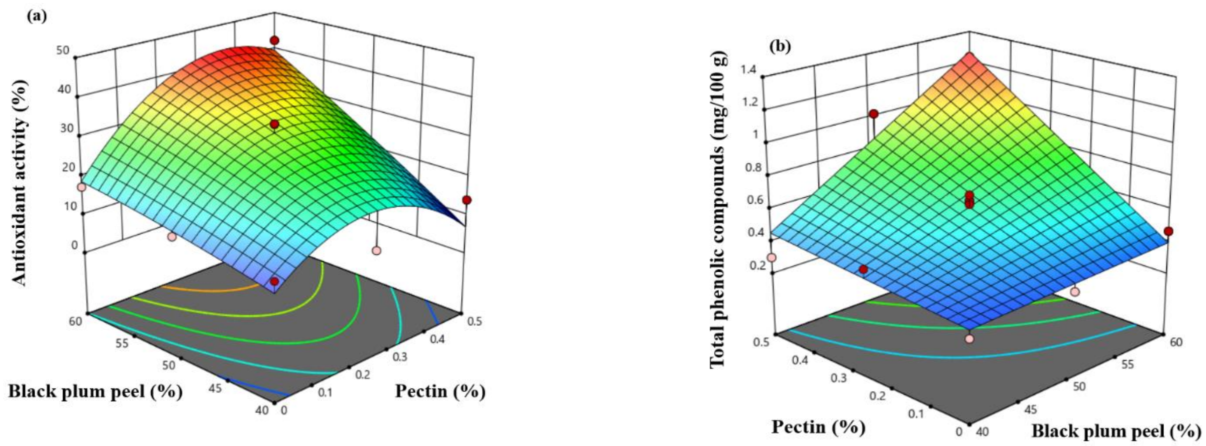

Figure 2. The effect of black plum peel puree and pectin concentration on the a) antioxidant activity and b) total phenolic compound of black plum peel syrup.

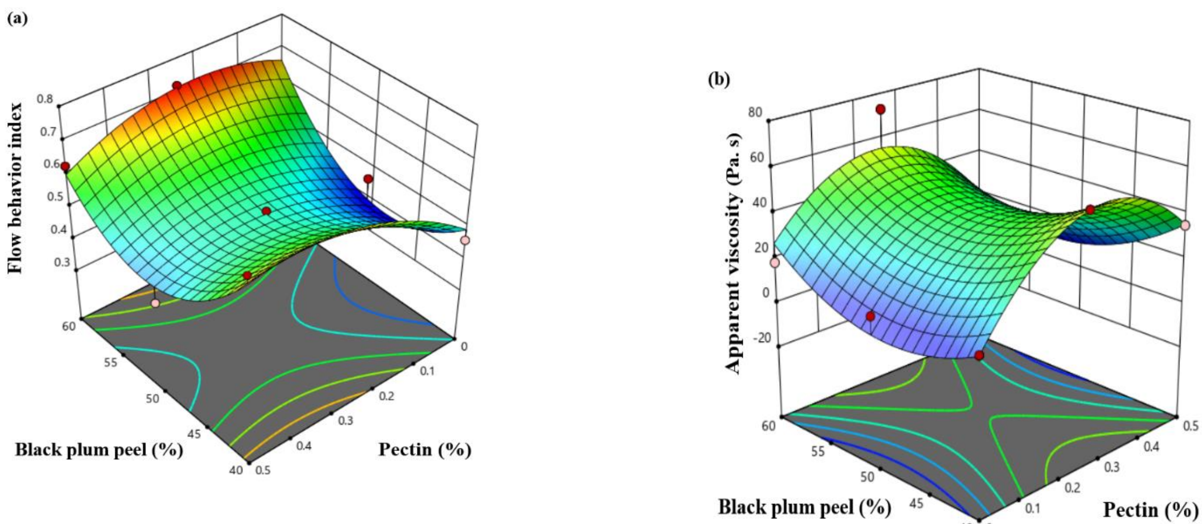

Figure 3. The effect of black plum peel puree and pectin concentration on the a) flow behavior index and b) viscosity of black plum peel syrup.

Conclusion

Black plum peel, as a major by-product of plum processing, representing a valuable source of antioxidant compounds and nutrients can be used to produce innovative food products. Its favorable nutritional and therapeutic effects, attractive color, desirable textural and flavor properties, make it a proper formulation of foods to address the older adults' needs. This study aimed to optimize the formulation of black plum peel syrup using response surface method (RSM). Increasing the black plum peel puree increased the antioxidant activity and phenolic compounds, which highlights its nutritional value for the elderly. RSM effectively identified the optimal formulation containing 40.62% black plum peel puree and 0.2% pectin. Beyond product optimization, this research demonstrates the potential of black plum peel as a functional food ingredient that contributes to reduce waste, improve resource efficiency. This food can be used as a food with modified texture for the elderly.

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Ethical Approval

The study was approved by the Neyshabur University of Medical Sciences Ethics Committee (Ethics Committee No.: IR.NUMS.REC.1398.020).

Conflict of Interests

The authors declare that they do not have any conflict of interest.

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